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Effect of Light-Cure Initiation Time on Polymerization Efficiency and Orthodontic Bond Strength with A Resin-Modified Glass-Ionomer

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Structured Abstract

Objectives – The polymerization and acid–base reactions in resin-modified glass-ionomers (RMGI) are thought to compete with and inhibit one another. To examine the effect of visible light-cure (VLC) delay on the polymerization efficiency and orthodontic bond strength of a dual-cured RMGI.

Setting and Sample Population – The Orthodontics Graduate Program at Marquette University. An *in vitro* study utilizing 72 freshly extracted human bicuspid teeth.

Materials and Methods – A RMGI light-cured immediately, 2.5, 5, or 10min after mixing comprised the experimental groups. Isothermal and dynamic temperature scan differential scanning calorimetry (DSC) analysis of the RMGI was performed to determine extents of VLC polymerization and acid–base reaction exotherms. Human premolars ($n = 18/\text{group}$) were bonded with the RMGI. Shear bond strength and adhesive remnant index (ARI) scores were determined.

Results – Differential scanning calorimetry results showed the 10-min-delay RMGI group experienced significantly ($p < 0.05$) lower VLC polymerization compared with the other groups. Acid–base reaction exotherms were undetected in all groups except the 10-min delay group. No significant differences ($p > 0.05$) were noted among the groups for mean shear bond strength. A chi-square test showed no significant difference ($p = 0.428$) in ARI scores between groups.

Conclusions – Delay in light-curing may reduce polymerization efficiency and alter the structure of the RMGI, but orthodontic shear bond strength does not appear to be compromised.

Introduction

Glass-ionomers (GI) were invented in the late 1960s and first introduced into dentistry in 1972 (1). Glass-ionomers set via an acid–base reaction between polymers of polyacrylic acid and fluoroaluminosilicate bases (2). The setting reaction begins when hydrogen ions from the polyacrylic acid attack and decompose the silica glass particles. A rapid reaction, in which the released Ca^{2+} ions interact with the polyacrylic acid chains, occurs, followed by a slower reaction between the chains and Al^{3+} , with the end result being a structure of cross-linked polyacrylic acid units. Glass-ionomers are advantageous in that they are capable of chemically bonding to tooth structure and can release fluoride over a period of time. Disadvantages of GIs include moisture sensitivity and low initial strength. Resin-modified glass-ionomers (RMGI) were developed to overcome the disadvantages of conventional GIs by adding polymerizable components similar to those found in composite resins. Dental composite resins consist of an organic matrix usually composed of dimethacrylates as well as inorganic fillers. When exposed to visible light, a photoinitiator reacts with an amine-reducing agent to generate free radicals, causing the dimethacrylate monomers to form a chain reaction, creating polymers of the resin.

Although, simplistically, RMGIs may be viewed as a combination of traditional GIs and composite resins, they are complex materials, as the acid–base and polymerizable components must coexist within one formulation. To achieve this, RMGIs typically contain 2-hydroxyethyl methacrylate (HEMA) to act as a solvent and photopolymerizable monomer, allowing the aqueous and organic phases to become miscible. Resin-modified glass-ionomers also contain calcium fluoroaluminosilicate glasses, just as the typical GIs do, which are the source for cross-linking ions for the acid–base process and to act as filler for the resin phase. Additional components include photoinitiators, polyacrylic acid (which may or may not have the HEMA grafted onto it), and water. The setting reaction of a RMGI is complicated because of the interacting process of chemical cure through acid–base reactions with the cross-linking, polymerization reaction attributed to visible light-curing (VLC). The photopolymerization reaction will be affected by the polarity of the acid–base nature, and the acid–base process will be inhibited by the presence of organic matter, as well as through the reduced diffusion of reactants through the cross-linked network (3-5). In this system, the resin photopolymerization reaction occurs at a much

faster pace than the acid–base reaction, but it relies entirely on the availability of monomer and its mobility/diffusion, which is affected by the amount of material already cross-linked in the matrix network by the acid–base reaction. It is, thus, acceptable to assume that, as one reaction affects the extent and speed of the other, if the initiation time of the photopolymerization reaction was modified, it would alter the balance of acid–base vs. photopolymerization of the material. This would result in a product that might physically perform differently based on the extents of reactions that took place; for example, if light-curing was delayed, the RMGI would set more because of the acid–base reaction, less photopolymerization would take place, and the material may possess physical properties closer to that of a GI. On the other hand, if photopolymerization occurred early on, the acid–base reaction would be diminished, resulting in a material with physical properties more similar to a composite resin.

Some studies have evaluated the properties of RMGIs with or without light-curing. Light-curing has been shown to affect diametral tensile strength (6), water uptake (7), wear rates (8), fluoride release (9), erosion, and compressive strength (10). Two studies have examined whether delayed light exposure has an effect on orthodontic bond strength using a RMGI (11, 12). The data of the one study (11) appear to be also presented in the other (12). Nevertheless, using bovine mandibular incisors, they examined tensile and shear bond strength using a RMGI (Fuji Ortho LC; GC America Inc., Alsip, IL, USA) that was light-cured 5, 10, 20, and 40min after mixing the powder/liquid. Although the mean bond strength decreased approximately 20% with time from the 5- to 40-min delay groups, there was no statistically significant difference between any time interval groups. However, light-cure (LC) delays of 20 and 40min, and perhaps 10min for a quadrant, are impractical clinically. Additionally, a recent report using thermal analysis to examine a restorative RMGI (Fuji II LC; GC America Inc.) showed a decrease in LC reaction exotherm, which would be expected to occur even within the working time of the material (13). Therefore, utilizing more clinically relevant LC delay times, the objective of this study was to examine the effect of LC delay on the polymerization efficiency and orthodontic bond strength of a capsulized RMGI. The hypotheses of the research were that delay in an orthodontic RMGI light-activation 1) allows for greater acid–base reaction, 2) reduces resin polymerization extent, 3) results in a RMGI of a different structure, and 4) this different structure will affect the physical properties of the material and decrease the bond strength when used to bond brackets to enamel.

Materials and methods

Fuji Ortho LC (universal shade capsules; GC America Inc.) was the RMGI investigated in this two-component research consisting of (1) differential scanning calorimetry (DSC) analysis to investigate the extent of acid–base and/or photopolymerization reaction and (2) bond strength determination. Four experimental groups were established via the RMGI being light-cured immediately (0), 2.5, 5, and 10min after mixing. The material was prepared following the manufacturer's instructions using a mechanical mixer for 10s (ProMix; Dentsply International, York, PA, USA) under dim light conditions to reduce ambient light-curing of the material. When prescribed, visible light polymerization was performed for 40s using a light-curing unit (Optilux 501; Kerr, Danbury, CT, USA) with an irradiance of 600mW/cm² as measured with a commercial radiometer (Model 100 Optilux Radiometer; Kerr).

DSC analysis

Immediately after mixing, the RMGI was placed in a pre-weighed 40- μ l aluminum crucible and transferred to a DSC (822e; Mettler-Toledo, Columbus, OH, USA) synchronized in time with mixing. Five experimental protocols were initially tested to determine the effect of temperature on the acid–base reaction of the RMGI in the absence of light-curing ($n = 5$ /group). As the manufacturer recommends refrigerating the material to extend working time, three of the five RMGI groups were chilled in a refrigerator set at 5°C, with one group remaining at the chilled temperature for the DSC analysis, another group heated to 23°C (room temperature; RT), while the

other group was heated to 35°C (oral temperature). This is analogous to leaving the chilled material on a cooled glass slab (prior to bonding), leaving it exposed to room temperature, or placing it in the mouth (as in bonding), respectively. In this study, 35°C has been designated as an average oral temperature, noting differences that arise owing to location within the mouth and the mouth being open/closed (**14**). The other two RMGI groups were kept at room temperature (23°C), with one group remaining at room temperature for the DSC analysis, while the remaining group was heated to 35°C for the analysis. This represents a clinician using non-refrigerated material and similarly leaving it at room temperature or placing it in the mouth, respectively. Thus, these five groups may be designated by the initial temperature of the RMGI and the DSC analysis as: chilled-5°C, chilled-23°C, chilled-35°C, RT-23°C, and RT-35°C. For this DSC analysis, heat flow was monitored for 40min at the indicated isothermal analysis temperature (5, 23, or 35°C). An exothermic peak, ascribed to the acid–base exothermic reaction (**13**), was evaluated with the time at its summit noted. The peaks were not integrated to yield overall enthalpy, because the varying profile of the peaks could lead to bias even with a standardized integration approach.

Differential scanning calorimetry analysis was also conducted on the RMGI to determine the effect of delay of light-curing on polymerization efficiency. Experimental groups were established via the RMGI being light-cured immediately (0), 2.5, 5, and 10min after mixing ($n = 10$ /group). The RMGI capsules were refrigerated (5°C) prior to mixing. Immediately after mixing, the RMGI was placed in a pre-weighed crucible and transferred to the DSC synchronized in time with mixing. Differential scanning calorimetry measurements initially consisted of isothermal (35°C) heat flow evaluation for 25min, to allow for measurement of polymerization and/or acid–base reaction exotherms. At the prescribed times, the RMGI was light-cured for 40s. This produced an exotherm arising from the heat input of the light-curing unit and the polymerization reaction. Next, forward (at 10°C/min) and reverse (at 20°C/min) dynamic temperature scans were conducted between 35 and 300°C. This dynamic scan degrades the material, producing an endotherm that serves as an indicator of glass-ionomer/resin material character (**13, 15**) and its original structure (**16**). The final thermal segment was a 15min, 35°C isothermal period with VLC initiated four times for the determination of mean enthalpy contributed from the VLC process. This energy was then subtracted from the initial VLC exotherm, resulting in an exotherm solely from the polymerization reactions (**17**). The polymerization exotherm is directly related to degree of conversion (**18**). Differential scanning calorimetry measurements were completed in a closed air environment, except during VLC, in which measurements were conducted with the light guide tip approximately 2mm from the test material surface. One trained operator (first author) conducted all of the VLC DSC experiments. Additionally, as will be discussed later, an immediate group was also examined utilizing the same protocol, except that the RMGI capsule was warmed to 35°C prior to mixing ($n = 5$).

Bond strength determination

Bonding

With approval from the Institutional Review Board at Marquette University, freshly extracted human premolars were collected and stored in deionized water. The water was replaced frequently to limit bacterial proliferation, and the chosen teeth were free of fractures, caries, and restorations. All teeth were prepared by sectioning off the roots 2mm below the cemento-enamel junction, utilizing a high-speed handpiece and tapered diamond bur, pumicing for 10s with a rubber prophylactic cup and fluoride-free pumice (Whip-Mix Corp., Louisville, KY, USA), rinsing with water, and storing in deionized water at 35°C prior to bonding. The 72 teeth were randomly divided into four groups of 18 to constitute the aforementioned immediate (0), 2.5, 5, and 10min RMGI LC delay groups.

Every bracket was bonded one at a time by one author (first author) using a direct bond technique and stainless steel brackets with a 0.022-inch slot, 0° tip, and 0° torque (Victory Series™ Universal Bicuspid Twin; 3M Unitek, Monrovia, CA, USA). Before bonding, each tooth was rinsed with fresh deionized water, dried thoroughly with oil-free compressed air, and the buccal surface was scrubbed with 10% polyacrylic acid conditioner (GC Ortho

Conditioner; GC America Inc.) for 20s with a cotton tip applicator. The teeth were rinsed again and excess moisture was removed with a light flow of air for 1–2s. Under dimmed light, the prepared RMGI was dispensed onto the bracket base, which was immediately positioned onto the buccal aspect of the tooth, aligned to the center of the tooth, and seated with firm pressure. Visible light polymerization was initiated at the prescribed time after mixing [immediate (0), 2.5, 5, or 10min] for 10s at all sides of the bracket, angled at 45° from the enamel–bracket interface, for a total of 40s. Prior to light-curing, the teeth were kept under a box to prevent ambient light penetration.

Mounting and shear bond strength testing

The bonded teeth were mounted in resin (Great Lakes Orthodontics, Tonawanda, NY, USA) up to the central groove of the tooth and stored in fresh distilled water at 37°C for 24h. A universal testing machine (Instron Corp., Canton, MA, USA) equipped with a 50-kgf-capacity load cell (Instron) was used to debond the brackets from the teeth using a shear load applied to the bracket at a crosshead speed of 0.1mm/min. Specimens were positioned such that the loading blade was directed parallel to the long axis of the tooth with contact made as close to the bracket/tooth interface as possible. Shear load forces to debond the bracket from the tooth were recorded and converted to MPa using a bracket base area of 10mm².

Adhesive remnant index classification

After debonding, the bracket base and tooth were analyzed under optical microscopy at 10× magnification using external illumination and given a score according to the adhesive remnant index (ARI). One of the four possible outcomes was recorded as follows: 0 = no adhesive left on tooth/all adhesive left on bracket, 1 = less than half of the adhesive left on tooth, 2 = more than half of the adhesive left on the tooth, and 3 = all of the adhesive left on the tooth.

Statistical analysis

Differences in DSC parameters and shear bond strength between the four groups were analyzed using a one-way analysis of variance (anova) followed by a *post hoc* Tukey test when indicated. A Weibull analysis was performed to determine bond strength reliability, Weibull modulus, characteristic strength, and probability of failure at 6.0MPa. The latter value was selected following a recommendation that shear/peel bond strength should be approximately 6kg at 24h (6kg/10mm²=6MPa) (19). In addition, a chi-square test was used to compare the ARI scores between the groups. Significance for all statistical tests was set at $p < 0.05$, and the analysis was performed using SPSS Statistics 17.0 (SPSS Inc., Chicago, IL, USA).

Results

The times of the maximum acid–base exotherm for the chilled-5°C, chilled-23°C, chilled-35°C, RT-23°C, and RT-35°C groups were 26.8 ± 0.4 , 24.4 ± 1.8 , 5.9 ± 0.8 , 23.0 ± 1.7 , and 5.4 ± 0.4 min, respectively. **Figure 1** displays a comparative thermogram of these groups. For the light-cured RMGI, DSC exothermic and endothermic values are listed in **Table 1**. Comparing the VLC polymerization exothermic values, the 10-min delay group had significantly ($p < 0.05$) lower polymerization efficiency compared with the other groups. The immediate, 2.5-, and 5-min delay groups were not significantly different from each other ($p > 0.05$). No acid–base exotherms were recorded in the immediate, 2.5, and 5min delay groups, while a slight, but noticeable exothermic peak was observed around 5–7min for the 10-min delay group (**Fig. 2**). The dynamic scan endotherm enthalpy values were not significantly ($p > 0.05$) different among the groups, but the temperature for the 10-min VLC delay endotherm peak was significantly ($p < 0.05$) lower compared with the immediate, 2.5, and 5min delay groups, which were not significantly different from each other ($p > 0.05$). Typical endotherms are presented in **Fig. 3**.

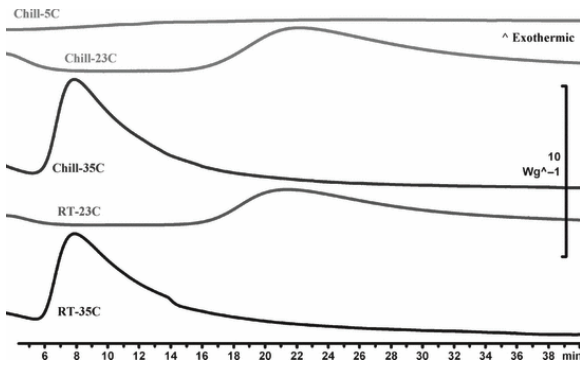


Figure 1 Isothermal differential scanning calorimetry thermograms for the resin-modified glass-ionomers stored and analyzed at various temperatures.

Table 1. Differential scanning calorimetry analysis parameters

| Group | Light-activated polymerization exotherm (J/g) | Acid–base reaction exotherm (J/g) | Dynamic scan endotherm (J/g) | Temperature of endotherm peak (°C) |
|----------------------|---|-----------------------------------|------------------------------|------------------------------------|
| Immediate LC | 22.7 ± 5.4 AB | Not detected | 29.5 ± 5.2 A | 184 ± 16 A |
| 2.5-min delay LC | 23.4 ± 4.5 AB | Not detected | 25.9 ± 6.7 A | 186 ± 15 A |
| 5-min delay LC | 21.0 ± 5.9 B | Not detected | 21.9 ± 3.8 A | 170 ± 4 AB |
| 10-min delay LC | 8.4 ± 6.4 C | 5.5 ± 1.5 | 22.5 ± 9.3 A | 158 ± 14 B |
| Immediate LC at 35°C | 30.3 ± 7.6 A | Not detected | 28.5 ± 2.8 A | 181 ± 9 A |

LC, Light-cure.

Within each parameter, different letters denote significant ($p < 0.05$) differences that exist.

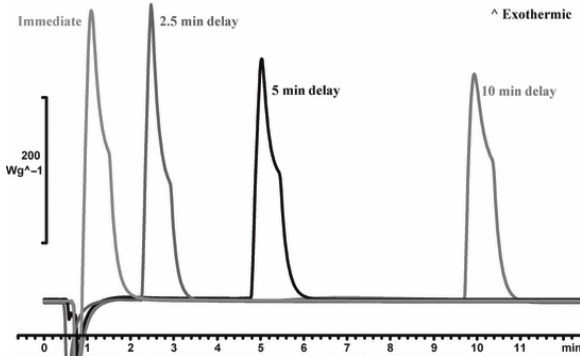


Figure 2 Comparison of light-cure polymerization exotherms.

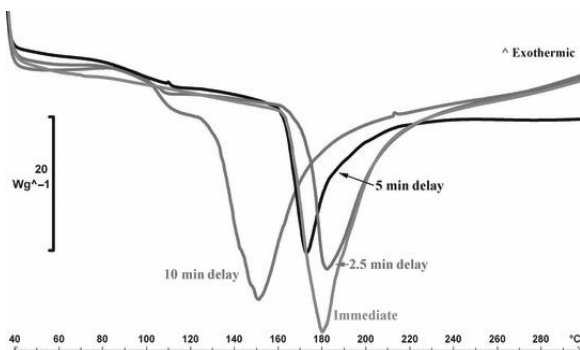


Figure 3 Comparison of dynamic scan differential scanning calorimetry endotherms.

Table 2 displays the mean shear bond strength and Weibull analysis results. Anova indicated no significant ($p > 0.05$) difference in bond strength existed among the four VLC delay groups. The Weibull modulus was fairly similar between groups, although the immediate group showed a greater value indicative of less variability in bond strength. Similarly, the immediate group presented with the lowest probability of failure at a bond strength of 6.0 MPa. This is further observed in **Fig. 4**, which shows the probability of failure vs. shear bond strength. Adhesive remnant index scores are listed in **Table 3**. A chi-square test found no significant difference ($p = 0.428$) between groups. An ARI score of 1 was found in at least 83% of the teeth for all groups, indicating a majority, but not all, of the adhesive remained on the bracket following debonding.

Table 2. Shear bond strength and Weibull analysis results

| Group | Mean \pm standard deviation (MPa) | Weibull modulus (β) | Characteristic strength (α ; MPa) | Probability of failure at 6.0 MPa (%) |
|------------------|-------------------------------------|-----------------------------|---|---------------------------------------|
| Immediate LC | 14.3 \pm 2.3 | 6.8 | 16.1 | 0.1 |
| 2.5 min delay LC | 14.8 \pm 3.2 | 4.9 | 16.0 | 0.7 |
| 5 min delay LC | 12.8 \pm 3.1 | 4.4 | 14.1 | 2.2 |
| 10 min delay LC | 14.5 \pm 2.7 | 5.5 | 15.6 | 0.4 |

LC, Light-cure.

One-way anova indicated no significant ($p > 0.05$) differences exist for mean shear bond strength.

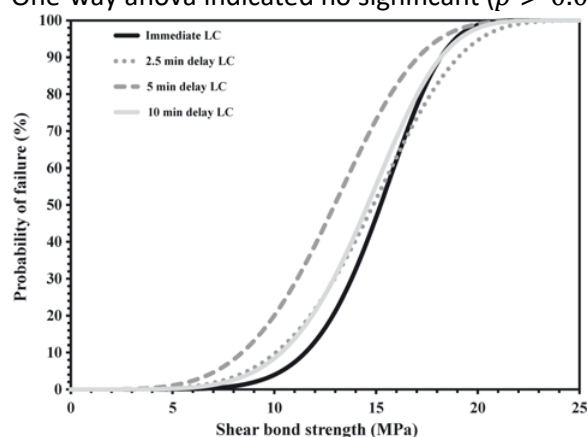


Figure 4 Probability of failure vs. shear bond strength.

Table 3. Adhesive Remnant Index (ARI) scores by group

| Group | ARI scores* | | | |
|------------------|-------------|----|---|---|
| | 0 | 1 | 2 | 3 |
| Immediate LC | 1 | 16 | 1 | 0 |
| 2.5-min delay LC | 0 | 16 | 2 | 0 |
| 5-min delay LC | 0 | 18 | 0 | 0 |
| 10-min delay LC | 2 | 15 | 1 | 0 |

LC, Light-cure.

*A chi-square test showed no significant difference ($p = 0.428$) between groups.

Discussion

Nicholson and Anstice (4, 5) hypothesized early on that the acid–base and VLC polymerization reactions in RMGIs would compete with and inhibit each other. Experimentally, Young (3) showed that after the VLC-induced photopolymer cross-linked network has formed, diffusion of acid–base reactants is reduced. Eliades and Palaghias (20) also showed that the acid–base reaction rate is slower in VLC vs. dark-cured RMGIs. Berzins et al.

(13) tested the theory that there was a competition of reactions in RMGIs. They discovered that as time was allowed for the acid–base reaction to occur, the VLC polymerization exotherm significantly decreased. This is partially supported in the present study. There were no significant differences in VLC polymerization exotherm between the immediate, 2.5, and 5min VLC delay groups, but the 10min VLC delay group had significantly lower polymerization conversion. The decrease in VLC polymerization exotherm and the presence of acid–base exotherm in the 10-min VLC delay group would indicate that the acid–base reaction inhibits the VLC polymerization. With regard to the lack of difference noted between the immediate, 2.5, and 5min VLC delay groups, it must be noted that refrigerated capsules (5°C) were used in these experiments. Although the DSC sensor was set at 35°C, the immediate LC RMGI was still more likely near its refrigerated temperature at the time of light-curing and, this would greatly impair diffusion of monomer reactants, and thus polymerization efficiency. The other delay groups had more time on the sensor, allowing it to equilibrate to the increased temperature of the DSC sensor, thus allowing its monomer reactants greater diffusion. To address this consideration, an immediate group was prepared with the capsule heated to 35°C prior to mixing. As would be expected, its mean VLC polymerization exotherm was increased and was significantly greater than the 5min VLC delay group (**Table 1**). Thus, in general, it appears delay in light-curing of this orthodontic RMGI allows more acid–base reaction to occur, limiting diffusion of polymerizable components, and decreasing polymerization efficiency.

With regard to the rate of the acid–base reaction, the orthodontic RMGI reacts more slowly than the restorative RMGI tested in a previous study (13). The acid–base exotherm peak was near the 3–4min mark for a restorative RMGI (13), while it was near 5–7min for the orthodontic RMGI, as viewed in the 10-min delay group in **Fig. 2**. The manufacturer must alter the components/composition of the RMGI to extend the working/setting time to allow for multiple brackets to be placed as well as allow for bracket position adjustment after placement but prior to light-curing. Of course, refrigerating the material also contributes to this, but on a fairly limited level as discussed later. The dark cure DSC data further confirms the slower reaction in the orthodontic RMGI (**Fig. 1**). At a DSC analysis temperature of 35°C, the average acid–base exotherm peak time was 5.4 and 5.9min for when the capsule was stored in the refrigerator or room temperature, respectively. The data presented earlier and in **Fig. 1** indicate the greatest determinant of the peak acid–base reaction time is not at what temperature the capsule is stored, but rather what temperature the RMGI is exposed to after mixing, i.e. a cooled glass slab, room temperature, or oral temperature. This is not surprising, as reactive components will have decreased diffusion rates at lower temperatures, or alternatively worded, increasing temperature will increase chemical reaction rates. Berzins et al. (13) found the exothermic peak of the acid–base reaction to be decreased in time by 50% with an increase in temperature of 10°C (37–47°C). An increase from 23 to 35°C in this study resulted in decreased times of approximately 75%. The curve profiles in **Fig. 1** provide further illustrative evidence of the effect of temperature on reaction rate. The RMGI exposed to 35°C presented with sharper peaks, whereas exposure to 23°C resulted in broadening of the peak, and finally very little exothermic activity was observed when the RMGI was exposed to 5°C. Once again, the diffusibility of the reactants at a given temperature will determine the reaction's rate and extent. It should be acknowledged that a myriad of possibilities exist as to the temperature profile, i.e. temperature over time, of the RMGI material during the bonding process. Using a refrigerated capsule as the manufacturer recommends, the RMGI used for the very first bracket to be situated on a tooth would quickly go from exposure to the refrigeration temperature to oral temperature, whereas the last bracket to be bonded with the RMGI would have more time exposed to room temperature or that of a cooled glass slab if used.

Differential scanning calorimetry analysis showed no differences ($p > 0.05$) with regard to the dynamic scan endotherm, which did not correlate with the study on a restorative RMGI (13) that showed a significant increase ($p < 0.05$) in values as VLC delay increased. However, endothermic peak temperatures did follow a similar trend observed previously (13) with the 10-min delay group being significantly less than the other delay groups ($p <$

0.05). As the dynamic scan endotherm is thought to be an indicator of GI/resin material character, with lower values more consistent with GI, it appears allowing the acid–base reaction to occur resulted in a material more similar to a GI in structure.

The setting reaction competition in RMGIs is curious scientifically, but what effect does it have clinically in orthodontics? The bond strength determination component of this research was designed to discern this. As mentioned previously, two studies similar in design explored the effect of delayed light-curing on orthodontic bond strength (**11**, **12**). However, at least two of their four delay groups are not practical clinically (20 and 40 min delay groups). This study utilized more clinically relevant LC delay times to examine its effect on orthodontic bond strength. For instance, allowing 30–60s per tooth for adhesive application, bracket seating, bracket positioning, and excess adhesive removal, bonding a quadrant before light-curing would take between 3 and 6 min, noting that the manufacturer’s instructions mention placing all brackets in a quadrant or full arch prior to the light-curing step, although the latter is perhaps not a common practice. Thus, the first bracket to be bonded would be delayed in light-curing by this amount. Of course, working time in addition to the amount of material available in a single capsule may limit the use to one capsule for every three teeth. Factoring in 40s of light-curing per tooth, the third bracket bonded with the first capsule would experience a delay of 4.3–7.3min before light-curing. Light-curing after dispensing only one capsule would reduce these delay times by 35–50%; alternatively, consideration of a full arch would double the times. The experimental groups in the current study are among these clinically expected LC delay times. Results showed no significant difference in shear bond strength whether the RMGI was light-cured earlier or later within 10min. The immediate LC group did show slightly favorable Weibull analysis results, but not noteworthy enough to provide a recommendation adopting this practice. Furthermore, this would result in an increase in the number of capsules used to bond a case, although if it were deemed more reliable that would have to be weighed against the cost of rebonding a debonded bracket. Tavas and Watts (**19**) suggested that 6kg (or 6MPa in this study) of shear/peel bond strength is needed at 24h, and the current data showed a consistent value greater than that for all groups, which confirms previous studies that RMGIs are reliable orthodontic bonding agents.

The fact that the 10-min delay group showed significantly lower polymerization efficiency and a different structure via the DSC testing, but presented with similar bond strength to the other groups, appears contradictory. However, several possibilities exist to explain this. First, as RMGIs bond to tooth structure via both micromechanical interlocking and chemical bonding (**21**), one might guess that a greater GI character in the RMGI may increase the chemical bond nature of the adhesive, increasing bond strength (**22**). However, if this comes at the expense of a greater percentage of VLC polymerization, the resulting properties of the material may be more GI-like and lack bond strength (**23**). It is possible that these opposing effects combined to result in no difference in bond strength compared with the other groups.

Alternatively, it is possible the enamel preparation was not sufficient to discriminate between the RMGI groups with presumed, varied physical properties. In this study, a 10% polyacrylic acid conditioner was applied for 20s, following the manufacturer’s instructions. Komori and Ishikawa (**24**) observed that conditioning enamel with 10% polyacrylic acid produced a smooth surface without evidence of enamel-prism-etching patterns, while preparation with 37% phosphoric acid did. They suggested use of a 10% polyacrylic acid conditioner is insufficient for providing a mechanical bond of the adhesive. Bishara et al. (**25**) found that using a 20% polyacrylic acid conditioner significantly ($p < 0.05$) increased the shear bond strength of a RMGI compared with a 10% polyacrylic acid conditioner. Cacciafesta et al. (**26**) observed a greater RMGI bond strength after using a 37% phosphoric acid etch than with 10% polyacrylic acid. Also, Godoy-Bezerra et al. (**27**) determined that enamel conditioning with 10% polyacrylic acid did not increase the shear bond strength of a RMGI. Based on these studies, one can hypothesize that the 10% polyacrylic acid conditioner provided little preparation of the enamel surface, resulting in an enamel-adhesive bond insufficient to differentiate between the bonding groups.

Further investigation of the bonding properties of the RMGI groups could be conducted with increased preparation of the enamel surface to determine whether it was a factor.

Adhesive remnant index scores (**Table 3**) showed a majority of resin remained on the bracket and not on the tooth for all groups. This further suggests the amount of enamel etching was minimal in accordance with Bishara et al. (**28**). In their RMGI LC delay studies, Komori and Ishikawa (**11**) showed similar results that most of the resin remained on the bracket with no significant differences between groups ($p > 0.05$), whereas Ando et al. (**12**) found the resin remain mostly on the tooth following tensile bond strength testing.

Conclusions

Within the confines of this *in vitro* study, it may be concluded that delay in light-curing an orthodontic RMGI does allow for greater acid–base reaction, thereby reducing the degree of conversion of the polymerizable components, and altering the structure of the material. However, the orthodontic bond strength of the material remains unaffected within clinically relevant delays in light-curing.

Clinical relevance

When bonding brackets with a resin-modified glass-ionomer, the orthodontist typically will mix the RMGI and apply several brackets to the teeth before light-curing the material. During this time delay between mixing and light-curing, which may be variable depending upon number of brackets bonded, the glass-ionomer components are able to react. Does this affect the material and its bonding capacity? This project was intended to determine how clinically relevant LC delay times influence the structure of a RMGI and whether bond strength is influenced by light-curing earlier or later after mixing.

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