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*Research Article*

# Effects of Curing Mode on the Bond Strength of Resin-Modified Glass Ionomer Cements

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The purpose of this study was to evaluate the bond strength of resin-modified glass ionomer cements (RMGICs) with different curing mode. RelyX Luting 2(RL) and Nexus RMGI (NR) were bonded to enamel, dentin, cobalt-chromium alloy, and ceramic using light-curing and chemical-curing modes, separately. The bond strength of light curing was higher than that of chemical curing for all groups. The bond strength of RL and NR in the light-cured group was significantly higher than those in chemical curing for both enamel and cobalt-chromium alloy ($P < 0.05$). The most common mode of adhesive fracture was cohesive fracture. The difference of adhesive strength resulted from the difference of micromechanical interlocking of adhesive which came from the cohesive strength of RMGICs with light curing. FT-IR showed that light curing was followed by chemical curing, but the chemically cured samples could not be cured without an external light source. Thus, light curing is an essential procedure in improving bond strength when using dual-cured RMGICs in clinics.

## 1. Introduction

Resin-modified glass ionomer cements offer the advantages of higher bond strength, lower solubility, and fluoride release [1–4]. Therefore, resin-modified glass ionomer cement (RMGIC) is clearly a superior choice and is recommended for clinical use over conventional GIC, particularly for applications such as cementation of metal or ceramic prosthesis and orthodontic brackets [5, 6]. However, most RMGICs used in clinical settings are of the chemically cured type. When the prosthesis is fixed on the tooth, the patient is required to bite for a long time to ensure complete curing of the RMGIC. Furthermore, with chemical curing, removal of the cured RMGIC at the edges is inconvenient. Dual-cured RMGICs were developed to address these issues in clinical practice [7, 8]. In procedures using the RMGICs, the restorations are fixed on the tooth with the occlusion state, after which the dentist performs light-curing of the RMGIC in contact with the restoration and teeth edge and then removes the bonding material. After biting for 5 minutes, cementation of the restoration is complete. The process of light curing could shorten the occlusion time for patients and allow easy removal of the excess RMGIC, but there are few reports describing whether it can improve the bond strength [9–11]. Our previous studies have shown that the curing mode of dual-cured RMGIC can directly affect the flexural strength because the flexural strength in the light-curing mode is higher than that in the chemical-curing mode and also could improve the mechanical chimerism of RMGIC [12].

The present study hypothesise that the light-curing mode could further improve the bond strength of RMGICs. The bond strengths between dental tissues (enamel and dentin) and restorative materials (cobalt-chromium alloy and ceramic) are evaluated. The result of this research attempted to provide laboratory data supporting the choice of an appropriate curing method and adhesive system.

Table 1: RMGICs used in this study.

| Codes | Materials | Compositions | Manufacturers |
|---|---|---|---|
| RL | RelyX luting 2 | Paste A: fluoroaluminosilicate glass, proprietary reducing agent, 2-hydroxyethyl methacrylate, water, and opacifying agent<br>Paste B: methacrylated polycarboxylic acid, 2,2-bis [p- (2′-hydroxy-3′-methacryloxy propoxy) phenylene] propane, 2-hydroxyethyl methacrylate, water, potassium persulfate,<br>and zirconia silica fille. | 3M ESPE, USA |
| NR | Nexus RMGI | 2-Hydroxyethyl methacrylate, ytterbium trifluoride, 2-hydroxy-1,3-propanediyl bismethacrylate, (1-methylethylidene) bis [4,1-phenyleneoxy(2-hydroxy-3,1-propanediyl)] bismethacrylate, and water. | Kerr Corporation, USA |
| *M* | PD Casta H | Cobalt chromium alloy | Mountain Medico, USA |
| *C* | VITA mark II | Ceramics | VITA, Germany |

## 2. Materials and Methods

Two dual-cured RMGICs were used in this study, as listed in Table 1. These two RMGICs can be used for bonding the metal and ceramic restoration to dental tissue.

Specimens were divided into 16 groups (10 specimens per group): RL light-cured group, RL chemically cured group, NR light-cured group, and NR chemically cured group, and each group bonded with enamel (*E*), dentin (*D*), cobalt-chromium alloy, and ceramic. The specific groups and their respective codes are shown in Table 2.

A total of 120 caries-free bovine mandibular incisors of animals aged under 5 years were used for the measurement of bond strength. The roots were removed by a low-speed saw under cooling water, and residual soft tissue was removed clean with a scalpel. The crowns were embedded in a mold with a diameter of 20 mm and height of 14 mm with 'NISSIN's self-curing denture resin. When the resin was cured, the samples were polished with P240, P400, and P600 water sandpaper until a flat enamel or dentin surfaces larger than 3 mm diameter was exposed. Then, the enamel and dentin surfaces were etched for 20 and 15 s, respectively, with Bisco's 32% phosphoric acid etching agent; the tooth surface was rinsed with running water, and visible water on the surface was immediately removed with a brief stream of oil-free compressed air. The surface was kept moist in the preparation process.

We used casted 60 cobalt chrome alloy blocks of 10 mm length, 10 mm width, and 5 mm height, and cut 60 ceramic blocks of 12 mm length, 10 mm width, and 6.5 mm height. All the casting and cutting processes were strictly done in accordance with the instructions of the manufacturers. The casting cobalt chrome alloy blocks and cutting ceramic blocks were respectively embedded in a mold with a diameter of 20 mm, treated with a 14-mm-high layer of self-curing resin and exposed on one side to create a bonding surface. After the resin cured, the samples were polished under P240, P400, and P600 water sandpaper until the cobalt-chromium alloy and ceramic blocks showed a plane surface and then sandblasted for 3 s at a distance of 1 cm using 50 $\mu$m alumina particles at a pressure of 0.4 MPa. Then, the samples underwent ultrasonic cleaning for 60 s, followed by drying with a brief stream of oil-free compressed air.

A single-sided tape with a 3 mm diameter hole was applied to the bonding surface of enamel, dentin, cobalt-chromium alloy, or ceramic material, and the diameter of the circular hole was measured as the experimental adhesive diameter with a 3D digital display system (Smartscope MVP 200, Quality Vision International Inc., USA).

According to the manufacturer's instructions, a split mold (4 mm diameter and 2 mm height) was aligned vertically with the adhesive surface in the hole of tape with 3 mm diameter. For the chemical-curing mode, the cement was filled fully in the split mold and then chemically cured at room temperature for 10 minutes. For the light-curing and chemical-curing mode, the cement was filled in the split mold first with less than 0.5 mm following LED light (Coltolux, Switzerland) curing for 10 s and then the split mold was filled fully following light curing for 10 s. The sample was placed in a 37°C water bath (Memmert WNB10; Germany) for 24 h before the test.

The curing mechanism was characterized using a Fourier-transform infrared (FT-IR) spectrometer (Nicolet iN10 Spectrometer; MA, USA). Spectra in the range of 600–4000 $cm^{-1}$ were collected in 32 scans at a resolution of 4 $cm^{-1}$ and 900–3300 $cm^{-1}$ were evaluated. For the chemically cured sample, the spectra were collected at 1, 30, and 60 min. For the light-cured sample, the spectra were collected at 1 min, immediately after light curing, and then at 30 and 60 min.

The shear strength was measured by using the mechanical testing machine (INSTRON 3367; Illinois Tool Works Inc; USA) at a loading speed of 1.0 mm/min. The shear bond strength of each sample was automatically recorded, and the mean and standard deviation were calculated.

After the shear strength test, the morphology of the bonding interface was characterized by scanning electron microscopy (SEM; EVO 18; Zeiss, Oberkochen, Germany).

Statistical analysis comparing the mean values for the results of shear bond strength was performed with the software (IBM SPSS Statistics v19; IBM Corp). The paired-sample $T$ test was used to identify irregularities in the data distribution of different curing modes, and the least significant difference (LSD) test of one-way analysis of variance (ANOVA) was used to identify irregularities in the shear bond strength differences between the two kinds of RMGICs

Table 2: Table of groups and codes.

| Codes | RL | | NR | |
|---|---|---|---|---|
| | Light curing | Chemical curing | Light curing | Chemical curing |
| *E* | *E*1 | *E*2 | *E*3 | *E*4 |
| *D* | *D*1 | *D*2 | *D*3 | *D*4 |
| *M* | *M*1 | *M*2 | *M*3 | *M*4 |
| *C* | *C*1 | *C*2 | *C*3 | *C*4 |

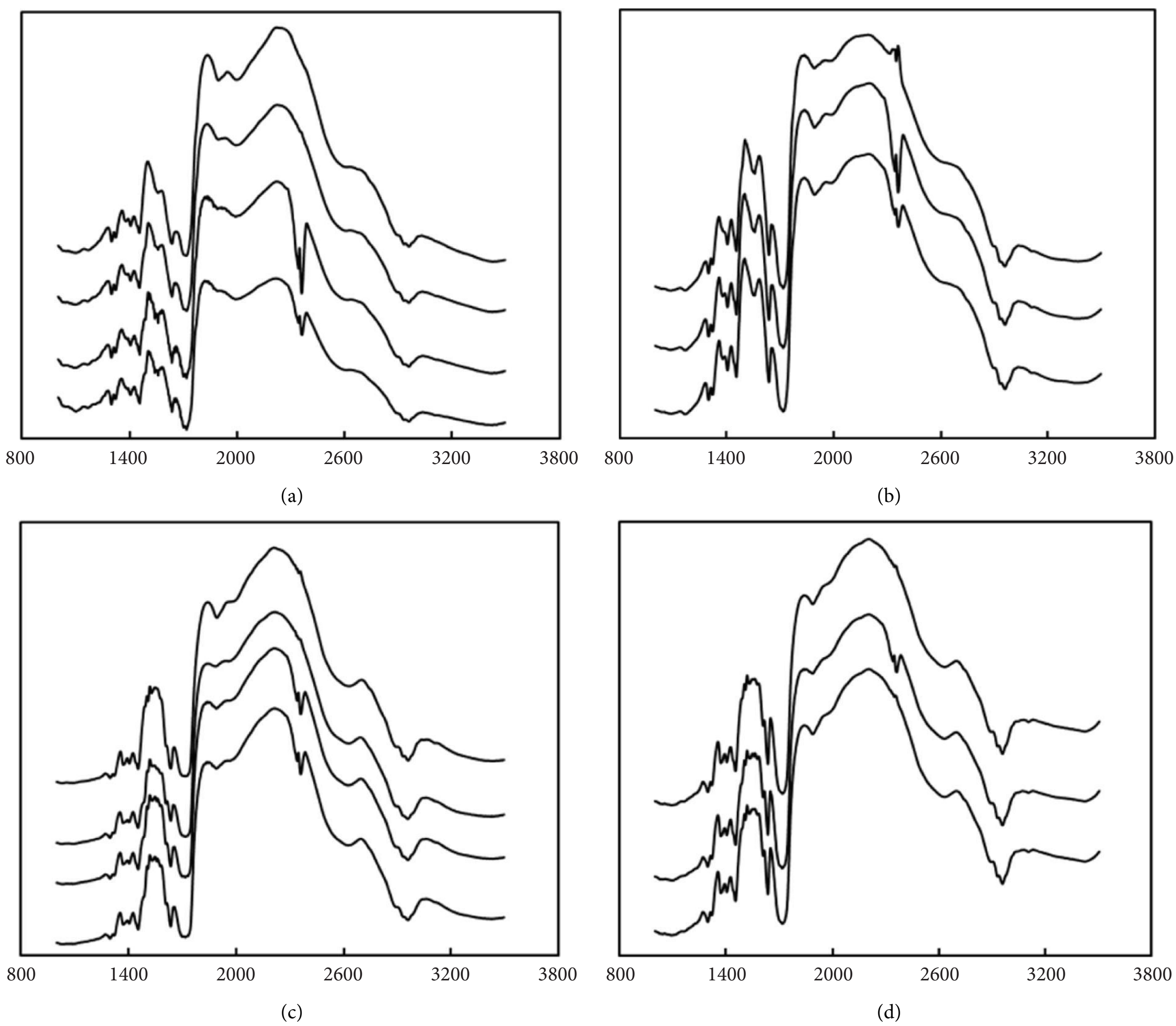


Figure 1: FT-IR spectra of cements. (a) Light-curing sample (RL): 1 min after mixing, light curing, 30 min after mixing, and 60 min after mixing (from top to bottom). (b) Chemical sample (RL): 1 min, 30 min, and 60 min after mixing (from top to bottom). (c) Light-curing sample (NR): 1 min after mixing, light curing, 30 min after mixing, and 60 min after mixing (from top to bottom). (d) Chemical sample (NR): 1 min, 30 min, and 60 min after mixing (from top to bottom).

and the same bonding material and the differences between the same RMGICs and different bonding materials ((*P*0.05) for all analyses).

## 3. Results

FT-IR spectra were used to qualitatively characterize the composition of the samples. As shown in Figure 1, the net peak absorbance height of methacrylate C=C bonds (C=C str, 1638 $cm^{-1}$) was used as the analytical frequency, while the peak absorbance height of ester bonds (C=O str, 1712 $cm^{-1}$) was used as a reference frequency. The C=C/C=O peak absorbance height ratios of groups B and C relative to group A were used to calculate the percentage of residual C=C bonds (RDB) on the top (group B) and bottom (group C) surfaces. The same peak height ratios were used to calculate the percentage changes in RDB before and after delayed exposure of group D specimens.

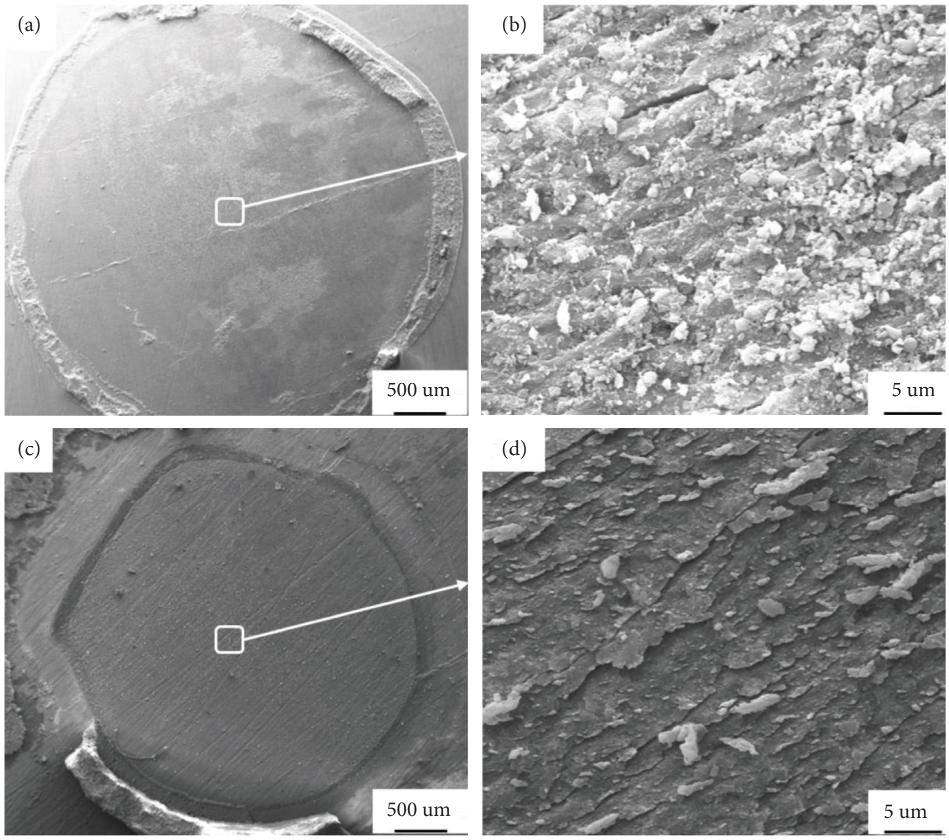


Figure 2: SEM images of the fracture interface for different bond strength on dentin: (a and b) 15 MPa, (c and d) 5 MPa.

Table 3: Shear bond strengths of various groups (MPa).

| Codes | RL | | NR | |
|---|---|---|---|---|
| | Light cured | Chemically cured | Light cured | Chemically cured |
| *E* | $13.26 \pm 3.62^{a}$ | $9.33 \pm 1.62^{a}$ | $15.81 \pm 3.60^{c}$ | $14.30 \pm 1.17^{c}$ |
| *D* | $10.81 \pm 2.76$ | $9.87 \pm 2.01$ | $9.42 \pm 1.17$ | $9.35 \pm 1.25$ |
| *M* | $7.87 \pm 2.60^{b}$ | $6.12 \pm 1.45^{b}$ | $12.00 \pm 2.76^{d}$ | $7.26 \pm 2.01^{d}$ |
| *C* | $9.62 \pm 2.27$ | $8.50 \pm 2.92$ | $7.09 \pm 1.41$ | $6.02 \pm 2.21$ |

a, b, c, d, $P < 0.05$.

The mode of failure was examined with SEM and typical samples are illustrated in Figure 2. The most common mode of fracture was cohesive fracture. For the sample with low bonding strength, the fracture surface was on the dentin.

As shown in Table 3, the bond strength of each light-cured group was higher than that of the corresponding chemically cured group for all the groups. The bond strengths of RL with enamel and alloy after light curing was $13.26 \pm 3.62$ MPa and $7.87 \pm 2.60$ MPa, which was significantly higher than that in the chemical curing (a and b: ($P$0.05)). The bond strengths of NR with enamel and alloy after light curing were $15.81 \pm 3.60$ MPa and $12.00 \pm 2.76$ MPa, which was significantly higher than the corresponding value in chemical curing (c and d: ($P$0.05)). The bond strength with enamel in the NR group was higher than that in the RL group. There was no significant difference between NR and RL when bonded with dentin.

The bond strength of NR with cobalt-chromium alloy with the light-curing group was higher than that of RL. When bonding with ceramics, RL was superior to NR, but there was no significant difference.

## 4. Discussion

The results of this study confirm the previous hypothesis that light curing could improve the bond strength, although short light curing or "tack curing" of RMGIC is used to create a semigel state in luting cements for an easier excess material cleanup and prevent the irritation of residues on tooth tissues [13]. RMGIC is indicated for the permanent cementation of metal-based and ceramic restorations of the dental tissue [14]. This specific laboratory study designed the bonding test between RMGICs and different corresponding materials, which is correlated to the clinical application.

Limited clinically relevant data exist examining the effect of light curing on the bonding properties of RMGICs, since many studies have evaluated RMGIC materials with or without light curing. The properties such as tensile strength, water uptake, wear rates, fluoride release, erosion, and compressive strength of RMGIC materials were affected significantly by light curing [15–17]. How the curing mode affect the bonding process and thus the bonding strength remains highly speculative [18]. Therefore, shear strength between RMGIC and restorations or the dental tissue was studied in the present study.

The increasing bonding strength trend with light curing related to the complex curing mechanism of RMGIC and its bonding mechanism with other materials [19, 20]. Analysis of FT-IR data in Figure 1 indicates clearly the curing process of different curing mode [21]. The acid-base reaction starts immediately between fluoroaluminosilicate glass and polycarboxylic acid after paste A and paste B are mixed [22]. The polymerization of methacrylated monomer in the mixer did not occur without an external light source [2]. Thus, the light-cured samples were actually dual-cured and so the cohesive strength of RMGIC was enhanced by the photopolymer cross-linked network. Our previous studies have shown that the light-curing mode of dual-cured RMGIC can directly heighten the flexural strength [12].

The bonding process between RMGIC and adherent materials included mainly micromechanical interlocking and chemical bonding [3]. The strength of micromechanical interlocking was related to surface morphology of adherent materials and cohesive strength of RMGIC. There is no comparability between different adherent materials, so it will not be discussed here. The present study mainly discusses the influence of different curing mode on the bonding performance of the same adherent materials with the same treatment. Theoretically, the chemical bonding property for the same chemical composition is not affected by the curing mode. Therefore, the difference of bonding strength is due to micromechanical interlocking strength which originate from cohesive strength of curing RMGIC [23]. As for the difference within the group, it mainly comes from the influence of technical sensitivity of the operation process. As the bond failure site morphology showed in SEM, curing mode alters the failure mode for RMGIC. Light curing specimens yielded a high incidence of mixed failure, which might be also related to an increase in cohesive strength of RMGIC. The percentage of adhesive failure along the dentin surface increases as the bond strength decreases. Furthermore, the differences between RL and NR maybe related to the differences in the chemical composition and content [24].

In order to clarify the influence of light curing, the laboratory study is slightly different from the clinical practice. Although a curing light for 2–3 second is recommended in manufactures' instruction, a longer time of 10 s is used to distinct the effect of light curing. Furthermore, other surface treatment such as hydrofluoric acid and silane coupling agent is not applied to minimize the impact of multiple factors. As a result, bonding strength cannot be directly related to the actual clinical data. However, the influence of light curing is not affected by these factors and the results have scientific and clinical significance. The results of this research show that light curing is effective to strengthen the bond strength of RMGICs. In this study, two commonly used RMGICs which could represent most resin-modified cements on the market were selected for research, but any new composition system may need further research.

## 5. Conclusions

The curing mode can directly affect the bond strength of dual-cured RMGICs because with the light-curing mode, the bond strength of each group with enamel, dentin, cobalt-chromium alloy, and ceramic, respectively, was higher than that with the chemical-curing mode. Therefore, light-curing is an essential procedure in improving bond strength when using dual-cured RMGIC in clinics.

## Data Availability

The data used in this study are available from the corresponding author on request.

## Disclosure

Yongxiang Xu and Yuan Li are the co-first authors.

## Conflicts of Interest

The authors declare that they have no conflicts of interest.

## Authors' Contributions

Yongxiang Xu and Yuan Li have equally contributed to the work reported. All authors have read and agreed to the published version of the manuscript.

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