

Comparison of Shear Bond Strength, Water Sorption and Solubility of 3 Glass Ionomer Cements for Direct Bonding of Orthodontic Brackets *in vitro*

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Abstract

The purpose of this study was to evaluate shear bond strength, water sorption and solubility of three brands of glass ionomer cement adhesives; RU-HBM1 (a resin-modified glass ionomer cement prototype), Hy-bond Glasionomer CX (GIC) and Fuji Ortho LC (RMGIC) as orthodontic adhesives. For shear bond strength test, thirty extracted human premolar teeth were divided into 3 groups (n=10) and bonded to stainless steel brackets. Shear bond strength was measured by a universal testing machine with a 1.0 mm/min crosshead speed. For the water sorption and solubility tests, six disc specimens were prepared for each group. Water sorption and solubility of the different adhesives were calculated by the weight of samples before and after immersion in artificial saliva and after desiccation. Data were analyzed by one-way ANOVA. The RU-HBM1 had significantly higher shear bond strength than Hy-bond Glasionomer CX but lower strength than Fuji Ortho LC ($p<0.05$). In addition, the RU-HBM1 had the lowest mean water sorption value and was the only orthodontic adhesive that had a positive mean solubility value. In conclusion, RU-HBM1 provided adequate shear bond strength for clinical orthodontic purposes and showed a lower water sorption parameter than commercial glass ionomer cements.

Keywords: Glass Ionomer Cement, Resin-modified Glass Ionomer Cement, Orthodontic Bracket, Shear Bond Strength, Water Sorption, Solubility

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Introduction

Direct bonding of orthodontic brackets with resin composite has been commonly used in contemporary orthodontics ever since the acid etch technique was introduced by Buonocore in 1955.¹ This technique improves esthetics, greatly reduces chairside time, and decreases gingival irritation from band placement. However, direct bonding with resin composite has some undesirable consequences such as a risk of decalcification of the enamel surface around orthodontic brackets, localized enamel cracks during debonding and scratching or enamel abrasion from excessive adhesive removal.²

Glass ionomer cement (GIC) has been developed as an alternative adhesive in direct orthodontic bracket bonding. GIC has many advantages such as biocompatibility, chemical bonding to enamel and dentin and resistance to erosion.^{3,4} The major benefit of GIC is fluoride release and uptake.^{5,6} Nevertheless, the bond strength of GIC is lower than resin cement. As a result, the failure rate of bracket bonding to teeth is higher.⁷⁻⁹

Resin-modified glass ionomer cement (RMGIC) has a higher adhesive strength than GIC owing to the addition of polymerized resins such as 2-hydroxyethyl methacrylate (HEMA), while its fluoride-releasing ability is maintained.¹⁰ Interestingly, RMGIC can be successfully used to bond orthodontic brackets because of its relatively high bond strength.¹¹⁻¹⁶ Bishara *et al.*¹⁷ reported that, with etched enamel and wet conditions, the shear bond strength of RMGIC was comparable with that of traditional light-cured resin composite. Moreover, during bracket debonding, RMGIC can be easily removed from the enamel surface without damaging the tooth structure.^{18,19}

Nowadays, commercial RMGICs used in Thailand are imported. To reduce the cost of dental treatment, A Research Unit-Herbal Medicine, Biomaterial and Material 1 (RU-HBM1), a RMGIC prototype has been developed. A previous study reported that RU-HBM1 has an adequate working time, depth of curing and flexural strength as

well as a biocompatibility with pulp cells.²⁰ However, shear bond strength, water sorption and solubility of this cement have not been investigated yet. Therefore, the purpose of this study was to examine shear bond strength, water sorption and solubility of RU-HBM1 *in vitro*. The null hypothesis is that there are no difference in shear bond strength, water sorption and solubility between RU-HBM1 and the other adhesives.

Materials and Methods

All study protocols were approved by the Human Research Ethics Committee of the Faculty of Dentistry, Chulalongkorn University (HREC-DCU 2016-005).

Shear bond strength test

Tooth sample preparation

Thirty upper first premolar teeth extracted from healthy young orthodontic patients were collected, cleaned of debris and then stored in 0.1 % (weight/volume) thymol solution to inhibit bacterial growth.^{21,22} Teeth with caries, enamel defects and restorations were excluded.

Brackets (Maestro bracket, Ortho Organizers, Carlsbad, California, USA) were bonded to each tooth at the middle third of the buccal surface using double-sided adhesive tape. In order to have the buccal surface of the tooth parallel to the applied force during the shear bond strength test, a guiding index was made by attaching two brackets to the opposite rims of a PVC pipe (21 mm in diameter and 5 mm in length) and inserting guiding wire (0.017" x 0.025" rectangular stainless steel wire) into slots of the guiding brackets (Fig. 1A). A prepared crown was ligated to the guiding wire at the center with an elastomeric o-ring. Then, a crown attached to the guiding index was placed on a PVC pipe (21 mm in diameter and 25 mm in length), containing self-cured acrylic (Fig. 1B). The

palatal half of the tooth was embedded in self-cured acrylic at the settled position. When the acrylic was set, the position of the bracket was marked on the enamel surface. Afterwards, the bracket and double-sided

adhesive tape were removed. The embedded teeth were cleaned with a rubber cup using fluoride-free pumice for 10 s and rinsed with water.

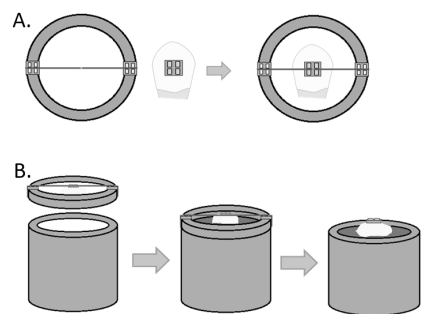


Figure 1 Sample preparation. (A) A prepared crown was ligated to the guiding index with an elastomeric o-ring at the center of the guiding wire, (B) A prepared crown attached to the guiding index was placed on a PVC pipe which contained self-cured acrylic. The palatal half of the tooth was embedded in self-cured acrylic at the settled position.

The samples were randomly divided into 3 groups as follows:

- Group 1: GI CX, Hy-bond Glasionomer CX, Shofu, Japan (powder/liquid ratio of 2:1 w/w, 40 s mixing time) (n=10)
- Group 2: Fuji Ortho LC, GC, Japan (3:1 w/w, 30 s mixing time) (n=10)
- Group 3: RU-HBM1, Research Unit of Herbal Medicine, Biomaterial and Material for Dental Treatment, Chulalongkorn University, Thailand (1:1 w/w, 30 s mixing time) (n=10)

The powder and liquid components of Hy-bond Glasionomer CX, Fuji Ortho LC and RU-HBM1 are shown in Table 1.

Table 1 Powder and liquid components of materials

Material	Composition	
	Powder	Liquid
Hy-bond Glasionomer CX	Fluoroalumino silicate glass, HY agent, Pigments	Acrylic acid-tricarboxylic acid co-polymer solution, Tartaric acid
Fuji Ortho LC	Alumino-silicate glass	Polyacrylic acid (20-22 %) 2-Hydroxyethyl methacrylate (35-40 %) 2,2,4,Trimethyl hexamethylene dicarbonate (5-7 %) Triethyleneglycol dimethacrylate (4-6 %) Polyacrylic acid (29 %)
RU-HBM1	Fluoroalumino silicate glass	2-Hydroxyethyl methacrylate (33 %) Triethyleneglycol dimethacrylate (7 %) Photoinitiator

Acid conditioning of enamel surface was required before the bonding procedure. The buccal surface of the teeth was conditioned with 37 % phosphoric acid for 30 s, rinsed with water for 10 s and air-dried. The adhesive was applied to the bracket base and the brackets were placed at the settled position. The excessive adhesive was removed with an amalgam carver without any disturbance to bracket position.

All brackets were bonded to the teeth by the same operator. Group 1 was chemically cured. Groups 2 and 3 were polymerized by a LED light curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) with a power density of 1200 mW/cm² for 40 s (10 s for each side of the bracket: mesial, distal, occlusal, gingival).

Shear bond strength measurement

After the bonding procedure, all samples were kept in distilled water at 37°C for 24 h. Exception for group 1 which would be waited for 10 min after cementation (according to manufacturer’s instruction). The shear bond strength test was performed using a universal testing machine (EZTest EZ-S Series, Shimadzu, Japan) with a cross-head speed of 1.0 mm/min. The shear force was recorded in newton (N). The shear bond strength (MPa) was then calculated as the ratio of shear force to bracket base area (14.42 mm²).

Water sorption and solubility test

Sample preparation

In accordance with ISO 4049 standardization²³, six disc-shaped specimens were prepared for each group

using a stainless steel mold with 15.0±0.1 mm diameter and 1.0±0.1 mm thickness. Glass slides were put on the top and bottom of the mold.

The samples of each group were prepared using the same powder and liquid proportion as in the shear bond strength test. The GI CX group was cured chemically and left for 10 min following the manufacturer’s instruction. To ensure the complete polymerization in the Fuji Ortho LC and RU-HBM1 groups, the cement was light-cured on both sides with overlapping sections. After curing, the samples were removed from the mold. The peripheries of all samples were polished by 1000-grit abrasive paper to remove irregularities. The diameter of the finished samples was at least 14.8 mm.

Water sorption and solubility measurement

The samples were stored in desiccators at 37±1°C. After 22 h, the samples were transferred to another desiccator at 23±1°C for 2 h. Each specimen was weighed using a digital balance (40SM-200A, Precisa, Zurich, Switzerland). This cycle was repeated until the weight loss between consecutive cycles was not more than 0.1 mg, at which point the mass was recorded as m₁. After the final drying cycle, the circular surface of each specimen was separated into 4 quarters by 2 perpendicular lines (Fig. 2). The mean thickness of each sample was the average of the thickness at the center of the specimen and at 4 points on the circumference. The mean diameter was the average of the length of 2 perpendicular lines. The volume of each specimen (v) was then calculated.

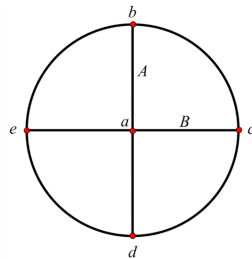


Figure 2 Points and lines for measurement of mean diameter and mean thickness of the specimens. The surface of the discs was divided into 4 equal quarters by line A and B. The mean diameter was the average length of line A and B. The mean thickness was the average thickness of the disc at point a, b, c, d and e.

The specimens were stored separately in 10 ml artificial saliva and maintained at 37 ± 1 °C for 7 days. After removal of the samples, their surfaces were patted dry until they were free from visible moisture, waved in air for 15 s, and weighed 1 min after removal from artificial saliva. This mass was recorded as m_2 .

Finally, the specimens were reconditioned to a constant mass in the desiccators following the aforementioned protocol, until a constant weight (m_3) was achieved.

Water sorption and solubility were calculated according to the Oysaed and Ruyter formulas²⁴.

$$\begin{aligned} \text{Sorption (A)} &= m_2 - m_3 / v \\ \text{Solubility (S)} &= m_1 - m_3 / v \end{aligned}$$

Statistical Analysis

The data were collected and expressed in mean \pm standard deviation (SD). Shear bond strength, water sorption and solubility were analyzed by one-way analysis of variance (ANOVA). Depending on the homogeneity of variance, multiple comparisons of shear bond strength and solubility were evaluated by Tamhane’s T2 test, while those of water sorption were calculated by Scheffé multiple comparison. A *p*-value less than 0.05 was considered to be statistically significant.

Statistical Analysis

Shear bond strength test

Fuji Ortho LC had the highest shear bond strength, followed by RU-HBM1. GI CX had the lowest mean shear bond strength (Fig. 3). There were significant differences among the tested groups (*p*<0.001).

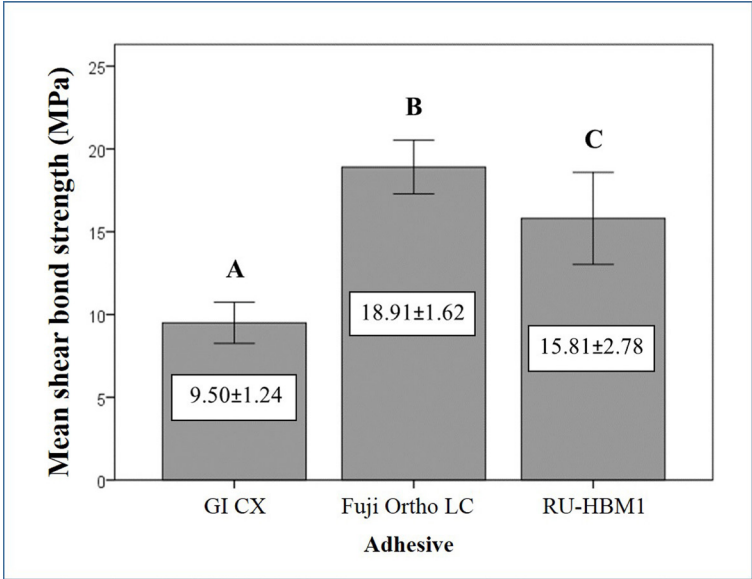


Figure 3 Average shear bond strength of GI CX, Fuji Ortho LC and RU-HBM1 (n=10). Bars labeled with different letters indicate a significant difference between the groups

Water sorption and solubility test

Mean water sorption values are presented in Fig. 4. RU-HBM1 had the lowest water sorption, followed

by GI CX and Fuji Ortho LC. One-way ANOVA showed significant differences among the tested groups ($p<0.001$).

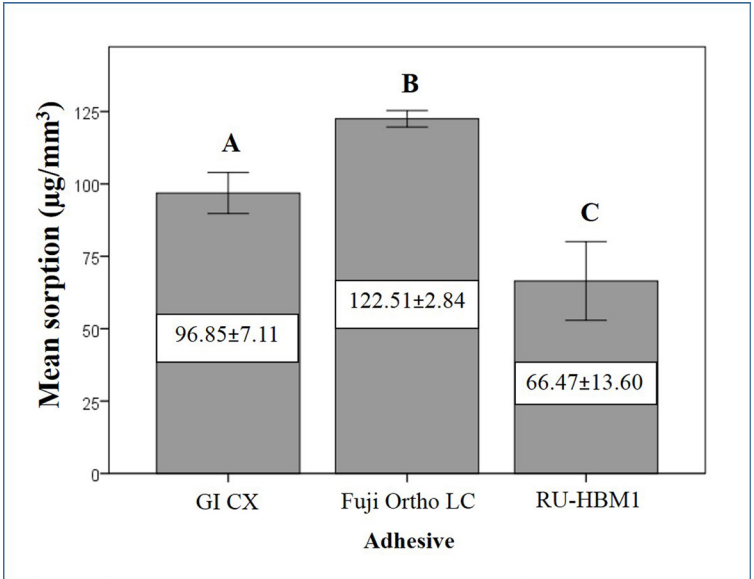


Figure 4 Average water sorption of GI CX, Fuji Ortho LC and RU-HBM1 (n=6). Bars labeled with different letters indicate a significant difference between the groups.

For solubility, RU-HBM1 was the only adhesive that had a positive mean solubility value, while GI CX and Fuji Ortho LC had a negative mean solubility value (Fig. 5). One-way ANOVA showed that solubility was significantly different among the tested groups ($p<0.001$).

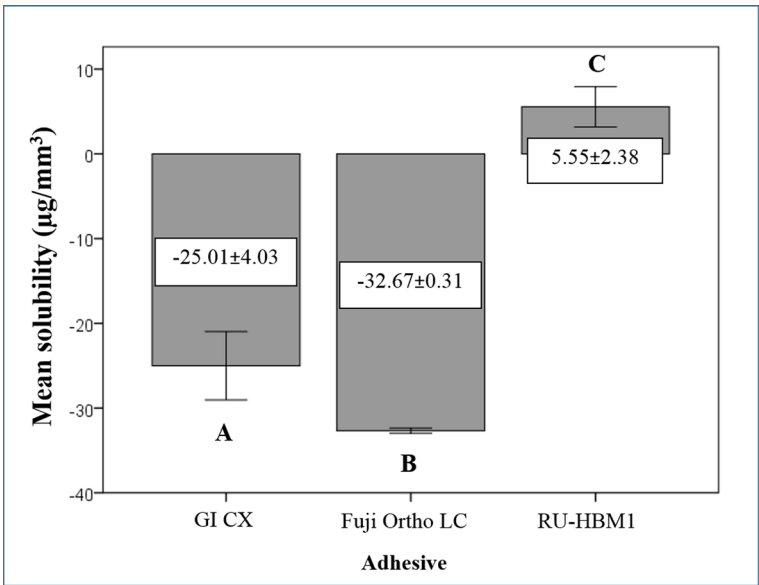


Figure 5 Average solubility of GI CX, Fuji Ortho LC and RU-HBM1 (n=6). Bars labeled with different letters indicate a significant difference between groups.

Discussion

Resin composite is the most common material for direct bonding of orthodontic brackets.^{25,26} Due to the disadvantages of resin composite such as enamel loss or fracture during debonding and high risk of white spot lesions around the brackets.^{27,28} RMGIC has been proposed as an alternative bonding material. Due to its enamel-friendly characteristics and its biological as well as chemical properties, RMGIC seems to be a potential orthodontic bonding material.^{29,30}

In this study, the experimental enamel surface was etched with 37 % phosphoric acid in dry conditions ahead of RMGIC cementation. There are many techniques to improve the shear bond strength of RMGIC. Conditioning the enamel with an appropriate acid is one of the favorite approaches. Many research groups reported that conditioning enamel with acid etching prior to bonding with RMGIC results in higher bond strengths.^{17,31-36} Using 37 % phosphoric acid as etching agent provides more shear bond strength than 10 % or 20 % polyacrylic acid.^{34,37} Unlike that of resin cement, the bond strength of RMGIC is not significantly affected by moisture.^{31,32} Moreover, the bond strength of Fuji Ortho LC on etched enamel was significantly higher when the enamel was dry than when contaminated with water.³⁸

GIC materials are known to be highly sensitive to moisture. The freshly set cement is susceptible to lose and take up water across its surface. As a result, micro-cracks in the cement can be found in both dry and moist conditions.^{39,40} However, micro-cracks caused by setting in dry condition can be closed after rehydration.⁴¹ In this study, the brackets were bonded to dry enamel surfaces according to the manufacturer's instruction. The micro-crack closure could occur because the samples were kept in distilled water at 37°C for 24 h after bonding the bracket to the tooth surface.

One of the limitations of this study is that we couldn't use the same duration of mixing time in all 3

materials. The mixing time of Hy-bond Glasionomer CX was longer than the others because it was more difficult to blend its powder and liquid into homogenous mixture. However, the mixing time of each material was followed its manufacturer's instruction. Continued mixing of the material accelerates the setting reaction. Working time and initial setting time can be decreased considerably by longer mixing time. Moreover, increasing mixing time was able to improve mechanical properties such as compressive strength and modulus of elasticity.⁴²

Particle size has considerable effects on the microstructure of GICs and their mechanical properties such as wear resistance, surface hardness and compressive strength. An increased proportion of smaller particles, resulting in a larger surface area, corresponded to higher strength.^{43,44} When macrogranular glass particles of GIC were replaced by nanogranular glass particles, the compressive strength and Young's modulus increased. However, the relative water uptake was not significantly different.⁴⁵ The effect of altering mixing time and powder particle size on shear bond strength, sorption and solubility would be interested for further study.

In this study, Hy-bond Glasionomer CX and Fuji Ortho LC were used to represent commercial conventional GIC and RMGIC, respectively. The null hypothesis was rejected. Our data revealed that there were significant differences in shear bond strength among the three experimental groups. The mean shear bond strength of the RU-HBM1 and Fuji Ortho LC still met the minimum requirements of shear bond strength for clinical orthodontic treatment, i.e. 5.9 to 7.8 MPa.⁴⁶ In addition, we observed that the bond failure type of the RU-HBM1 was mostly a bracket-adhesive interface failure, while failures for GI CX and Fuji Ortho LC mostly occurred at the enamel-adhesive interface. This data suggests that the RU-HBM1 has a stronger bond between the enamel and adhesive rather than the other groups. The possibility of enamel

fracture during debonding could be lower with the RU-HBM1, resulting in a more intact, sound enamel surface. However, considerable chair time is needed to remove the residual adhesive with increasing possibility of damaging the enamel surface during the cleaning process.⁴⁷ Anyway, enamel abrasion can be prevented by carefully polishing technique with less abrasive burs. In contrast, enamel fracture caused by debonding procedure with the failure at the enamel-adhesive interface is unpredictable and difficult to prevent. Therefore, the RU-HBM1 is a promising direct orthodontic bonding material

Water sorption and solubility are critical parameters for evaluation of orthodontic bonding cements. Water uptake could cause deterioration of filler-resin matrix bonding and degradation, resulting in worse structural and mechanical properties.^{48,49} The water sorption characteristic of RMGIC depends on both composition and degree of polymerization. However, a major effect on the water sorption of RMGIC is from the resin compositions.^{49,51} Hydrophilic constituents such as HEMA or resin molecules that contain hydrophilic moieties clearly increased water sorption values.^{50,52}

From our data, the RU-HBM1 had a lower mean water sorption than GI CX and Fuji Ortho LC. In the progression of the acid-base reaction, both GIC and RMGIC take up water as an integral part of their structures. A possible explanation is that the acid-base reaction of the RU-HBM1 completed more rapidly than that of the two other groups. As a result, GI CX and Fuji Ortho LC continuously take up water into their structures over a longer period than the RU-HBM1. Moreover, comparing between Fuji Ortho LC and RU-HBM1, RU-HBM1 has lower hydrophilic components as shown in Table 1. As a result, it took up water less than Fuji Ortho LC.⁵⁰ A high water sorption value of Fuji Ortho LC has been previously reported.⁵³

In this study, the RU-HBM1 was the only adhesive that showed positive mean solubility values, while the others displayed a negative mean solubility. A negative water solubility of GI CX and Fuji Ortho LC has been

previously reported.^{53,54} This may be because the acid-base reaction of GI CX and Fuji Ortho LC was prolonged and water molecules were continuously bonded into their structures. Therefore, the materials gained weight and expanded.⁵⁰ In contrast, a possible explanation of the positive solubility value for RU-HBM1 is that it had a more rapid acid-base reaction and polymerization than the other two materials. The weight loss from m_1 to m_3 may be because water molecules did not bond to the structure after the acid-base reaction ended. Therefore, the water was only trapped in the space of the matrix, filler or matrix-filler interface. Then, the water was vaporized out of the sample after drying in the desiccator. Moreover, the dissolution of unpolymerized monomers and fillers into the artificial saliva may be another reason of weight loss.^{48,55}

In this study, the RU-HBM1 was prepared in a powder-liquid form. Accordingly, it had to be mixed before bonding. The mixing process of RMGIC makes this bonding technique more inconvenient compared with resin composite. For future studies, a ready to use RU-HBM1 in paste form should be developed for more clinical convenience in direct orthodontic bonding. Moreover, the ability of this RU-HBM1 to act as a fluoride reservoir would be interesting for further investigation.

Conclusion

In conclusion, under the conditions of this *in vitro* study, the RU-HBM1 had adequate shear bond strength for use as an orthodontic bonding material. Moreover, its water absorption was lower than that of commercial GIC, while its solubility was higher.

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