Bonding of Light-Cured Glass Ionomer Cement to Polycarbonate Resin Treated With Experimental Primers

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Abstract: The effect of experimental primers on the shear bond strength of polycarbonate composite resin with light-cured glass ionomer cements was investigated. Mixtures of methylmethacrylate (MMA) with the comonomers 2-hydroxyethyl methacrylate (HEMA), triethyleneglycol methacrylate (TEGDMA), and bisphenol-A-glycidymethacrylate (bisGMA) were used as primers. Polycarbonate composite resin rods of circular cross section and plates were bonded, with and without precured and nonprecured primers, using 2 light-cured glass ionomer cements (commercially available [LC] and experimental [EX]). In addition, commercial polycarbonate composite resin brackets with precured 50% TEGDMA/MMA primer were bonded to etched human enamel with both cements. Shear bond strengths were measured. Results were compared by ANOVA and Scheffe's tests at $P = .05$. The 30% HEMA/MMA, 50% TEGDMA/ MMA, 10% bisGMA/MMA, and 30% bisGMA/MMA primers produced the higher shear bond strengths (9.5 to 20.8 MPa) with LC and EX to polycarbonate composite resin. The 50% TEGDMA/MMA primer was most effective in improving the shear bond strengths of both LC and EX. Precured 50% TEGDMA/ MMA primer on a commercial resin bracket was effective in providing good shear bond strength to enamel. (*Angle Orthod* 2000;70:357–365.)

Key Words: Polycarbonate resin; Light-cured glass ionomer; Experimental primers; Brackets; Shear bond strength

INTRODUCTION

Glass ionomer cements are able to chemically bond to enamel and to release fluoride ions.1,2 These properties would provide some benefits to orthodontics, and some researchers have evaluated their use.^{3–6} However, they found that glass ionomer bond strengths to enamel were significantly less than those of resin composites to etched enamel.^{4,6}

Recently, new light-cured glass ionomer cements for orthodontic bonding have become commercially available. Light-cured glass ionomer cements can hasten the setting reaction because of dual or triple reactions.⁷⁻¹⁰ Fricker¹¹ reported that there were no significant differences in the failure rates of direct-bonded orthodontic metal brackets cemented with Fuji Ortho LC light-cured reinforced glass ionomer (GC, Tokyo, Japan) compared with a bonding resin composite in a 12-month clinical trial. However, the bond strength of the light-cured glass ionomer cement to polycarbonate composite resin brackets is very low. If the bond strength to resin brackets and polycarbonate composite resin lingual buttons can be improved, more extensive application of the light-cured glass ionomer cement in orthodontics will result.

We hypothesized that treatment of the resin bracket base surface with a primer coupling agent could improve the bonding of light-cured glass ionomer cement to the resin bracket. To test this hypothesis, experimental primers consisting of methylmethacrylate (MMA) with 2-hydroxyethyl methacrylate (HEMA), triethyleneglycol methacrylate (TEGDMA), and bisphenol-A-glycidymethacrylate (bis-GMA) were prepared. The purpose of this study was to determine the effect of applying new experimental primers to polycarbonate composite resin on the shear bond strength to light-cured glass ionomer cement.

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TABLE 1. Materials

^a HEMA indicates hydroxyethyl methacrylate.

MATERIALS AND METHODS

A commercial light-cured glass ionomer cement (LC; Fuji Ortho LC, GC Co, Tokyo, Japan), and an experimental light-cured glass ionomer cement (EX; 3M Unitek Dental Products, Monrovia, Calif), were used as direct-bonding adhesives (Table 1). A polycarbonate composite resin material (Sankin Kogou K. K., Tokyo, Japan), which has a similar composition (85% polycarbonate and 15% inorganic filler by weight) to a commercial polycarbonate composite resin bracket, was used as an adherend to evaluate various experimental primer compositions.

Primer mixtures were prepared composed of MMA with 10, 30, 50, 70, and 90% of each of the comonomers HEMA, TEGDMA, and bisGMA. Photoinitiators consisting of 1% camphorquinone and 1% methacrylic acid 2 dimethylaminoethyl ester were added to the mixtures. Primers consisting of photoinitiators and 100% HEMA, 100% TEGDMA, and 100% MMA were also prepared. A primer containing 100% bisGMA was not used because of its inherently high viscosity.

Shear bond strength of the cement to polycarbonate composite resin

Polycarbonate composite resin plates ($10 \times 10 \times 2$ mm) and rods of circular cross section (5 mm diameter \times 2 mm long) were used as adherends. The resin plates were embedded with acrylic resin in acrylic tubes. The surfaces of the resin plates and rods were polished with 600-grit SiC paper under continuous running water and dried with oilfree compressed air. There were 2 experimental groups for investigation of the effect of different surface treatments.

Group 1: Primers precured. After applying the experimental primers to both polycarbonate resin surfaces by using a brush, the primers were air-thinned with oil-free compressed air for 10 seconds. After thinning, both resin surfaces with primers were illuminated for 40 seconds with a visible light-curing unit (Luxor, Model 4000, ICI, Macclesfield, England) to precure the primers. The LC and EX cements were mixed according to the manufacturer's directions and applied to the bottom surfaces of the rods. The rods with the cements were pressed on the resin plates under a load of 200 g for 10 seconds, and the excess cement was removed. Application of this constant load was accomplished by means of a frame supporting a rod upon which a 200-g weight was placed. The specimens with the cements were illuminated for 40 seconds with a visible light around the perimeter of the rod and immersed in 37° C water for 24 hours.

Group 2: Primers not precured. After completion of the group 1 testing, certain primer compositions were selected on the basis of high shear bond strength production for both cements. The primers selected for both cements were 30% HEMA/MMA and 50% TEGDMA/MMA solutions. For the bisGMA/MMA primers, 30% bisGMA/MMA was chosen for LC, and 10% bisGMA/MMA was chosen for EX. These percentages were selected because they produced the highest bond strengths when the primers were precured. For this group, the primers were air thinned after application with oil-free compressed air for 10 seconds. The mixed cements were applied to the bottom surfaces of the resin rods without precuring the primers. The remaining steps were completed as for group 1.

The shear bond tests were performed with a universal testing machine at a crosshead speed of 1 mm/min (Figure 1). The mean shear bond strengths were analyzed using 1 way and 2-way analyses of variance and Scheffe's multiple comparison test at a significance level of .05.

Shear bond strength of polycarbonate composite resin bracket to human enamel

Eighty human premolars freshly extracted for orthodontic reasons were used. The roots of the teeth were removed, leaving the crowns to be embedded in acrylic tubes with plaster. Enamel surfaces were polished with polishing cream (Polishing Paste 1, Bee Brand Medico Dental Co Ltd, Tokyo, Japan) in a brush, washed with water for 10 seconds, and dried with oil-free compressed air for 10 seconds. One-half of the teeth were conditioned for 20 seconds by use of 10% polyacrylic acid. The remaining teeth were conditioned for 20 seconds by use of 37% phosphoric acid. All teeth were washed with water for 10 seconds and dried with oil-free compressed air for 10 seconds.

The base surfaces of commercial polycarbonate resin brackets (Sankin Kogou K. K., Tokyo, Japan) for premolars

FIGURE 1. Schematic representation of shear bond test apparatus.

were treated with 50% TEGDMA/MMA according to the results from group 1. This primer was selected on the basis of the shear bond strength results from group 1. Resin brackets without primer treatment were also used. The mixed cements (LC and EX) were applied to the base surfaces of the resin brackets. The brackets with the cements were pressed on the etched enamel under a load of 200 g

for 10 seconds. Constant loading of the brackets was accomplished by means of a frame supporting a rod upon which a 200 g weight was placed. While under load, excess cement was removed, and the polymerization process was initiated utilizing visible light illumination (Luxor). The bonding cements were illuminated for 40 seconds from the top surface and from each side of the brackets. The specimens were then immersed in 37^oC water for 24 hours.

The shear bond tests were performed with a universal testing machine at a crosshead speed of 1 mm/min (Figure 1). The mean shear bond strengths were analyzed using 1 way and 2-way analyses of variance and Scheffe's multiple comparison test at a significance level of .05.

Scanning electron microscopy observation

Scanning electron microscopic (SEM) examination was performed on the following specimens: resin surfaces treated with various primers prepared as described above for group 1, polished resin surfaces, and fractured resin surfaces after shear bond testing. The specimens were dried in a desiccator under vacuum for 24 hours. These surfaces and the base surfaces of the resin brackets with and without primer were coated with a thin layer of gold-palladium alloy and observed with SEM (JSM-330, JEOL Ltd, Tokyo, Japan).

RESULTS

Shear bond strength of the cement to polycarbonate composite resin

The shear bond strengths of LC and EX as a function of comonomer concentration in the primers is summarized in Tables 2 and 3 (together with statistical differences) and

TABLE 2. Relationship Between Shear Bond Strength of Commercially Available Cement and Comonomer Concentration in Primersa

	Shear Bond Strength, MPa						
Primer	0%	10%	30%	50%	70%	90%	100%
HEMA/MMA ^b	3.8 ± 1.2 A	$7.2 + 3.1$ A	$9.5 + 1.2$ A	$6.9 + 2.4$ A	7.3 ± 1.3 A	6.1 \pm 1.5 A	6.0 \pm 1.7 A
TEGDMA/MMA	$3.8 \pm 1.2 B$	4.3 ± 1.6 B	$8.0 \pm 2.9 B$	$15.0 + 2.8 \text{ C}$	$14.2 + 2.1$ C	$15.0 \pm 3.7 C$	$14.9 + 4.4$ C
BisGMA/MMA	3.8 ± 1.2 D	$11.8 + 2.1$ F	$13.5 + 2.5$ F	$7.2 + 1.9$ DF	$6.8 + 2.4$ DF	3.7 ± 0.9 D	\cdots

^a Values with the same letters indicate no significant difference ($P > .05$).

^b HEMA indicates 2-hydroxyethyl methacrylate; MMA, methylmethacrylate; TEGDMA, triethyleneglycol methacrylate; and bisGMA, bisphenol-A-Glycidymethacrylate.

TABLE 3. Relationship Between Shear Bond Strength of Experimental Cement and Comonomer Concentration in Primersa

	Shear Bond Strength, MPa						
Primer	0%	10%	30%	50%	70%	90%	100%
HEMA/MMAb	2.3 ± 0.8 A	3.9 ± 1.1 AB	$17.6 + 3.0$	11.1 \pm 1.1 C	9.7 ± 1.8 BC	8.1 ± 2.4 BC	$3.7 + 1.2$ AB
TEGDMA/MMA	2.3 ± 0.8 D	4.9 ± 2.2 D	18.2 ± 3.4 EF	$20.8 + 4.3$ F	19.3 ± 2.3 EF	20.2 ± 3.8 EF	$14.2 + 3.0 F$
BisGMA/MMA	2.3 ± 0.8 G	$9.5 + 2.9$ H	$6.3 \pm 1.0 \text{ GHz}$	$3.2 + 0.6$ G	3.3 ± 0.9 G	$1.1 + 0.3$ G	\cdots

^a Values with the same letters indicate no significant difference ($P > .05$).

^b HEMA indicates 2-hydroxyethyl methacrylate; MMA, methylmethacrylate; TEGDMA, triethyleneglycol methacrylate; and bisGMA, bisphenol-A-Glycidymethacrylate.

FIGURE 2. Effect of precured primer type and concentration on shear bond strength of commercially available cement to polycarbonate composite resin.

FIGURE 3. Effect of precured primer type and concentration on shear bond strength of experimental cement to polycarbonate composite resin.

FIGURE 4. Effect of primer precuring on shear bond strength of commercially available and experimental cement to polycarbonate composite resin.

a Values with the same letters indicate no significant difference $(P > .05)$.

b HEMA indicates 2-hydroxyethyl methacrylate; MMA, methylmethacrylate; TEGDMA, triethyleneglycol methacrylate; and bisGMA, bisphenol-A-Glycidymethacrylate.

TABLE 5. The Effect of Primer Precuring on Shear Bond Strength of Experimental Cement to Polycarbonate Resin^a

		Shear Bond Strength, MPa
Primer	Precured	Not Precured
30% HEMA/MMA ^b 50% TEGDMA/MMA 30% BisGMA/MMA	17.6 ± 3.0 A 20.8 ± 4.4 9.5 ± 2.9	19.2 ± 4.1 A 6.2 ± 0.4 3.2 ± 0.1

^a Values with the same letters indicate no significant difference $(P > .05)$.

b HEMA indicates 2-hydroxyethyl methacrylate; MMA, methylmethacrylate; TEGDMA, triethyleneglycol methacrylate; and bisGMA, bisphenol-A-Glycidymethacrylate.

FIGURE 5. Effect of polycarbonate bracket base surface pretreatment on shear bond strength of commercially available and experimental cement to etched enamel. Bracket base surface pretreatment consisted of precured 50% TEGDMA/MMA.

Figures 2 and 3. Neither light-cured glass ionomer cement adhered to the polycarbonate resin without primer treatment.

For LC and EX, 2-way analysis of variance showed that there were significant differences in shear bond strength among the types of primers and among the concentration of comonomers in the primers ($P < .05$). The TEGDMA/ MMA primer group was most effective in improving the shear bond strength of the cements, the bisGMA/MMA

	Shear Bond Strength, MPa						
		Untreated Bracket	Treated Bracket				
Adhesive	10% Polyacrylic	37% Phosphoric	10% Polyacrylic	37% Phosphoric			
LC	4.1 \pm 1.9 A	4.4 ± 1.5 A	6.5 ± 2.0	9.9 ± 1.7			
EX	$5.6 \pm 2.3 B$	$5.5 \pm 1.5 B$	7.9 ± 1.8 C	$9.5 \pm 2.0 C$			

TABLE 6. Shear Bond Strength of LC and EX Between Brackets Treated and Untreated With 50% TEGDMA/MMA Primer to Enamel Etched With 10% Polyacrylic or 37% Phosphoric Acidsa

^a Values with the same letters indicated no significant difference ($P > .05$).

b LC indicates commercially available cement; EX, experimental cement; TEGDMA, triethyleneglycol methacrylate; and MMA, methylmethacrylate.

primer group was least effective, and the HEMA/MMA primer group was intermediate.

For LC, shear bond strength values ranged from 3.8 MPa to 9.5 MPa for the HEMA/MMA primer group. Scheffe's test showed no significant differences in this group. The shear bond strength of specimens with TEGDMA/MMA primer significantly ($P < .05$) increased up to 50% concentration of TEGDMA in the primer but did not change with a further increase in concentration. The shear bond strength of specimens with 50–100% TEGDMA/MMA primers ranged from 14.2 MPa to 15.0 MPa. The shear bond strength of specimens with the bisGMA/MMA primer group increased significantly ($P < .05$) up to 30% concentration of bisGMA and decreased significantly ($P < .05$) with further increasing concentration. The specimens with 10% bisGMA/MMA and 30% bisGMA/MMA primers showed the higher shear bond strengths (11.8 MPa to 13.5 MPa).

For EX, Scheffe's test showed that the shear bond strength of specimens with HEMA/MMA primer group increased significantly ($P < .05$) up to 30% concentration of HEMA and decreased significantly ($P < .05$) with further increases in concentration. The specimens with 30% HEMA/MMA primer showed the highest shear bond strength (17.6 MPa). The shear bond strength of the specimens with the TEGDMA/MMA primer group increased significantly ($P < .05$) up to 50% concentration of TEGD-MA and decreased significantly ($P < .05$) with further increases in concentration. The shear bond strength of specimens with 50–100% TEGDMA/MMA primers ranged from 14.2 MPa to 20.8 MPa. The shear bond strength of specimens with bisGMA/MMA primer group increased significantly ($P < .05$) up to 10% concentration of bisGMA in the primer and decreased significantly $(P < .05)$ with further increasing concentration. The specimens with 10% bisGMA/MMA primer showed higher shear bond strength (9.5 MPa) than the others, except for that with 30% bis-GMA/MMA primer.

The effect of primer precuring on the shear bond strength of the cements to polycarbonate composite resin is summarized in Tables 4 and 5 and in Figure 4. For LC and EX,

no significant difference in shear bond strength between precuring and nonprecuring treatment was observed with 30% HEMA/MMA primer, whereas the shear bond strengths of specimens with the other primers were significantly $(P < .05)$ enhanced by precuring treatment.

Shear bond strength of polycarbonate composite resin brackets to human enamel

The effects of 50% TEGDMA/MMA treatment of a commercial polycarbonate resin bracket on the shear bond strength to etched human enamel are summarized in Table 6 and Figure 5. The specimens treated with 50% TEGD-MA/MMA primer had significantly ($P < .05$) higher shear bond strengths than those without primer for both LC and

FIGURE 6. Scanning electron micrographs of typical polished (below) and MMA-treated (above) polycarbonate composite resin surfaces.

FIGURE 7. Scanning electron micrographs of typical polycarbonate composite resin surfaces treated with various primers (left: HEMA/MMA primers; middle: TEGDMA/ MMA primers; and right: bisGMA/MMA primers). The top micrographs show primers with 10% comonomer. The first middle micrographs show primers with 30% comonomer. The second middle micrographs show 50% comonomer. The bottom micrographs show primers with 70% comonomer.

EX to enamel etched with both polyacrylic and phosphoric acids.

Scanning electron microscopy observation

Figure 6 shows SEM micrographs of typical polished and MMA-treated polycarbonate composite resin surfaces. The SEM micrograph revealed many scratches on the polished polycarbonate composite resin surface. After treatment with

MMA primer, the scratches were removed from the surface, and the resin surface exhibited a wavy appearance.

Figure 7 shows SEM micrographs of typical polycarbonate composite resin surfaces treated with various primers. The resin surfaces treated with MMA/HEMA and HEMA/ TEGDMA primers that gave higher shear bond strengths were rougher. In the specimens treated by MMA/bisGMA primer, the resin surfaces became smoother with increasing concentration of bisGMA.

FIGURE 8. Scanning electron micrograph of typical fracture surfaces of the polycarbonate composite resin after shear bond test (above: resin cohesive fracture; below: resin-cement interface failure).

Figure 8 shows typical fracture surfaces of the polycarbonate composite resin after shear bond testing. The specimens showing higher shear bond strength exhibited cohesive fracture for both LC and EX, whereas the specimens showing lower shear bond strength were accompanied by interfacial failure.

Figure 9 shows SEM micrographs of typical polycarbonate composite resin bracket surfaces with and without 50% TEGDMA/MMA primer. The surface of the polycarbonate composite resin bracket was almost flat, and some lines on the surface were visible. The surface of polycarbonate composite resin bracket with 50% TEGDMA/MMA primer was rough, and many porosities were visible.

Figure 10 shows typical fracture surfaces on the enamel side and the bracket side after shear bond testing. For specimens without primer, all areas on the surface of etched enamel were covered with the fractured cement, and the ditches on the cement surface were clearly visible. Fractured cement was not visible on the bracket surface. However, when primer was used, all areas of the bracket surface were covered with the cement, and no fractured cement was visible on the surface of etched enamel.

DISCUSSION

Glass ionomer cements do not inherently bond to polycarbonate composite resin. Therefore, some type of coupling agent is required to develop clinically meaningful

FIGURE 9. Scanning electron micrographs of typical polycarbonate composite resin bracket surfaces with (above) and without (below) 50% TEGDMA/MMA primer.

bond strengths. Further, it would be advantageous for such a coupling agent to cause a surface roughening of the polycarbonate resin material in order to simultaneously achieve a measure of micromechanical attachment. The approach taken in this investigation is based on the potential for methacrylate monomers to partially dissolve the surface of the polycarbonate resin because of its linear polymer structure. Selection of comonomers was based on the HEMA content of the glass ionomer cements and the bisGMA and TEGDMA content of composite resin restoratives.¹²⁻¹⁴ Composite restoratives containing these ingredients have been shown to bond to glass ionomer cements.

The shear bond strength of both the LC and EX cements to polycarbonate composite resin was greatly improved, to varying degrees, by treatment with the experimental primers tested. This increase in bond strength appears to have been the result of a combination of the formation of covalent bonds to the precured primers and the roughening of the polycarbonate surface by the primers. For the HEMA/ MMA and TEGDMA/MMA primer groups, the surface roughness of the resin was dependent on the concentration of comonomer in the primers. The 30% HEMA/MMA and 50% TEGDMA/MMA primers produced the roughest surfaces and the highest shear bond strength values for both LC and EX. The specimens showing higher shear bond strength demonstrated cohesive fracture during bond testing for both cements.

FIGURE 10. Scanning electron micrographs of typical fracture surfaces on the enamel side (left) and the bracket side (right) after shear bond test (top micrographs show cement-bracket interface failure, and bottom ones show enamel-cement interface failure).

Results with the bisGMA/MMA primer group were much more variable. With LC, bond strength peaked at 30% concentration of bisGMA and then decreased at higher concentrations. For EX, bond strength peaked at 10% concentration and decreased at higher concentrations. All concentrations of the bisGMA/MMA primer group higher than 10% produced bond strengths substantially lower than the other primer groups tested. The cause for the diminished bond strength with the bisGMA/MMA primer may be related to its much higher viscosity, which renders it difficult to wet the surface of the polycarbonate resin. The poorer wettability leads to less attacking of the surface and, consequently, smoother surfaces. However, even though the surfaces were very smooth, 10% and 30% bisGMA/MMA primers provided shear bond strengths of 13.5 MPa and 9.5 MPa for LC and EX, respectively. Higher shear bond strengths of LC to the resin treated with bisGMA/MMA primer group were achieved than with EX. We hypothesized that the mixture of LC may penetrate more easily into the air-inhibited layer on the precured primer because of differences in the concentration of HEMA in the mixture of cement.

Although elimination of precuring of the primer can reduce chair time somewhat, this procedure may have potentially detrimental effects on the shear bond strength. The results of this study indicated that shear bond strengths without precuring of the primer tended to be lower than when a precuring procedure was employed. An exception was noted with 30% HEMA/MMA primer for both cements. A possible explanation for the lower shear bond strengths with the TEGDMA/MMA and bisGMA/MMA primers may be related to the hydrophobic nature of the residual TEGDMA or bisGMA monomers limiting their capacity to mix with the cements.

The combination of 50% TEGDMA/MMA primer and precuring technique was most effective in increasing the shear bond strength of the cements to the polycarbonate resin. Therefore, the effects of this bonding regimen on shear bond strength of a commercial polycarbonate composite resin bracket was examined and compared to brackets without primer application. Further, interactions with phosphoric and polyacrylic acids were considered. Higher shear bond strengths with both cements were obtained with primer-treated brackets to enamel etched with 37% phosphoric acid. It is important to note that the primer treatment changed the location of bond failure from the cement-resin interface to the enamel-cement interface failure. The resulting shear bond strengths of primer treated brackets to etched enamel (6.5 MPa to 9.9 MP) are within the range of about 5.9 to 7.8 MPa, which Reynolds suggested is necessary for successful orthodontic treatment.15

The results of this study suggest that acceptable clinical shear bond strengths to polycarbonate composite resin bracket can be obtained with light-cured glass ionomer cements if an appropriate experimental primer, particularly precured 50% TEGDMA/MMA primer, is used.

CONCLUSIONS

This study evaluated the effect of new experimental primers on the shear bond strength of polycarbonate composite resin with 1 commercial and 1 experimental lightcured glass ionomer cements. The results lead to the following conclusions:

- 1. The shear bond strengths of both glass ionomer cements to polycarbonate composite resin were dependent on the types and the concentration of comonomer.
- 2. The treatment of polycarbonate composite resin specimens with HEMA/MMA and TEGDMA/MMA primer groups greatly improved their shear bond strengths to light-cured glass ionomer cements.
- 3. The 50% TEGDMA/MMA primer was most effective in improving the shear bond strengths of both glass ionomer cements tested.
- 4. Precuring provided better shear bond strength results than not precuring for TEGDMA/MMA and bisGMA/ MMA primer groups.
- 5. Treatment of the base surface of a commercial polycarbonate composite resin bracket with a combination of 50% TEGDMA/MMA primer and precuring technique provided good shear bond strengths.

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