

Adhesive Thickness Effects on the Bond Strength of a Light-Cured Resin-Modified Glass Ionomer Cement

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Abstract: These in vitro studies investigated the effect of adhesive thickness on the tensile and shear bond strength of a light-cured, resin-modified glass-ionomer cement (FO). A light-cured conventional composite resin (CO) was used as the control material. Mesh-based metal brackets were bonded to extracted human premolars using FO and CO. The adhesive thickness was controlled by a special device and 0, 0.25, and 0.5 mm thicknesses were tested for both bonding agents. All bonded specimens were stored in distilled water at 37°C for 48 hours and thermocycled between 5°C and 55°C for 200 cycles before testing. Analysis of variance showed that bond strength was significantly affected by the adhesive thickness ($P < .001$) and type of adhesive ($P = .001$). There were statistically significant differences between the mean bond strengths of the groups at the $P < .05$ level of significance. For all adhesive thicknesses, CO had higher bond strength values than those of FO in both test modes. The bond strength values were also analyzed using a Weibull analysis, which showed the most favorable adhesive thickness, and the 5% and 90% probabilities of failures was 0.25 mm in the FO groups. Bracket-adhesive interface failure was predominant for all groups in tensile testing, but enamel-adhesive interface failures increased with increased adhesive thickness in shear testing for the FO. This study suggests that adhesive thickness under a bracket could be particularly important when using a FO in direct bonding. (*Angle Orthod* 2005;75:254-259.)

Key Words: Adhesive thickness; Shear bond strength; Tensile bond strength; Bonding adhesives

INTRODUCTION

Imperfect adaptation of a bracket base to the tooth surface results in a variable thickness of adhesive. A minimal adhesive thickness has been reported necessary to achieve optimal bond strength,¹ but increased thickness also has been reported to weaken the joint, because of the introduction of imperfections and increased polymerization shrinkage.²

An earlier study comparing the bond strengths of two chemically cured composite resins showed tensile bond

strength unaffected by increasing adhesive thickness, whereas shear bond strength decreased as thickness increased.³ However, in another study,⁴ tensile bond strength was decreased with increased adhesive thickness with both chemically cured and no-mix composite resins. Jost-Brinkmann et al⁵ reported similar results for no mix-composite resins. They found the chemically cured composites produced the highest tensile bond strengths, with the adhesive thickness having no apparent influence. Interestingly, the no-mix resin cured adequately only in a layer 0.2 mm or less and did not cure at all in greater thicknesses. The results also indicated that light-cured adhesives achieve maximum bond strength at 0.2 mm but are considerably weaker at 0 mm.

Mackay⁶ reported that increasing the thickness of two chemically-cured and two light-cured composite resins from 0 to 0.26 mm had no statistically significant effect on their mean shear bond strength, although the trend was for decreased strength.

All these studies investigated the effect of adhesive thickness on the bond strength of conventional chemically cured, no-mix, or light-cured composite resins. However, to date, there is no data available concerning the effect of adhesive thickness on the bond strength of light-cured, resin-modified glass-ionomer bonding cements. Therefore, the aims of

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this investigation were (1) to investigate the effects of the adhesive thickness on the in vitro tensile and shear bond strengths of a light-cured, resin modified glass-ionomer cement (FO) and to compare the results with those of a conventional light-cured composite resin (CO) and (2) to compare the bond failure sites under both tensile and shear type of forces.

MATERIALS AND METHODS

Substrate

Two groups of 120 human premolars were used in the study. The teeth were extracted from 14- to 17-year-old patients undergoing orthodontic treatment. The inclusion criteria required perfect buccal enamel, with no caries and no extraction damage.

Adhesives and bracket

A FO (Fuji Ortho LC, GC Corporation, Tokyo, Japan) was chosen to test the effect of adhesive thickness on bond strength of glass ionomer cements. A light-cured orthodontic adhesive (Transbond XT, 3M Unitek, Monrovia, Calif) was used as the control group (CO). The brackets used were mesh-based (Midi Diagonali, Leone Sesto, Fiorentino, Italy) standard 0.018- × 0.030-inches slot stainless steel premolar brackets.

Sample preparation

All teeth were cleaned with a flour of pumice slurry. The labial surfaces of the CO crowns were etched with a 37% phosphoric acid liquid for 30 seconds, rinsed with water for 20 seconds, and dried with compressed air. In the FO group, a 10% polyacrylic acid solution was applied to the labial enamel surface for 20 seconds, rinsed with water, and slightly dried with a light flow of air.

The control and test groups (CO and FO), were further divided into three subgroups of 40 samples each according to the thickness of the adhesive. Half of the samples in each subgroup were tested under tensile forces, and the other half were tested under shear forces. The adhesive thicknesses were: in subgroups 0, adapted to 0 mm (bracket base and the tooth surface in contact); in subgroups 25, adapted to 0.25 mm; and in subgroups 50, adapted to 0.5 mm.

A device designed for indirect bonding in lingual orthodontics (TARG + TR System, Ortonorm Ltd, Istanbul, Turkey) was used to control the thickness of the adhesive between the bracket base and the enamel surface (Figure 1). The roots of the teeth were embedded in dental stone, and the bracket was moved forward until it contacted the labial surface of the tooth. The best fit of the bracket base and the enamel surface was found, and the distance between the two tips of the machine (the distance between the lingual surface of the tooth and the bracket slot) was recorded. This recording was accepted as zero.

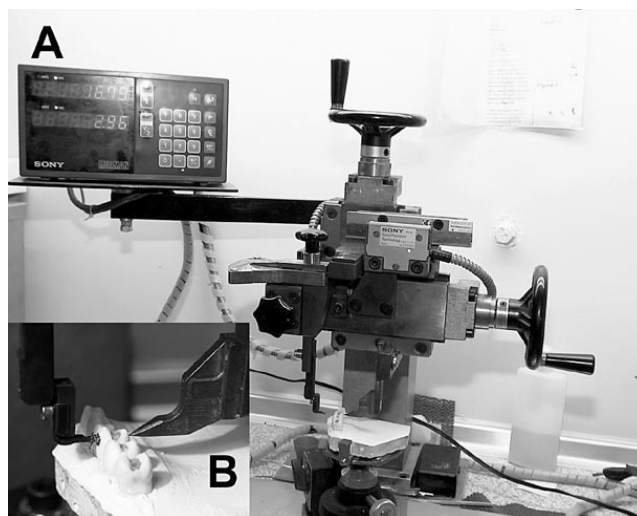


FIGURE 1. (A) Device used to control adhesive thickness. (B) Bracket at the removing tip of the machine.

After withdrawal of the bracket from the enamel surface, the adhesive was applied to the bracket base. Subsequently, the bracket holding tip was slowly moved forward again until the desired adhesive thickness was read from the digital screen of the machine. The excessive adhesive was carefully removed with a scaler. The tooth-bracket combination was then exposed to a visible light (Ortholux XT, 3M Unitek) for 10 seconds each from the cervical, occlusal, mesial, and distal directions.

Finally, the bracketed tooth was stored in distilled water at 37°C for 24 hours and thermocycled in water between 5 ± 2°C and 55 ± 2°C for 200 cycles before mounting. A jig was specially constructed to mount the tooth-bracket combination in a position where the bracket base was parallel to the cylinder surface and the bracket was at the center of the plastic molding cup filled with dental stone. A period of five minutes was allowed for initial setting before the mounted specimens were again placed in distilled water at 37°C for 24 hours before testing.

Bond strength testing

To simulate tensile and shear type forces, special testing jigs (Figures 2 and 3) were constructed and attached to the jaws of a Lloyd LRX testing machine (Lloyd Instruments Plc, Fareham, Hampshire, England). The peak force levels, automatically recorded on the testing machine, were converted to stress per unit area. A crosshead speed of one mm per minute was used in both test modes.

Variables evaluated

The bond strengths for tensile and shear testing, bond failure sites, and the presence of visible enamel damage were evaluated. Adhesive remnant index (ARI)⁷ scores were used for the classification of the failure site.



FIGURE 2. (A) Tensile test equipment. (B) Close-up view of the cast nickel-chromium bracket holder.

Statistical analysis was performed using analysis of variance (ANOVA) and any significant differences revealed by this procedure were further investigated using the Tukey honest significant difference multiple-range test with a 95% confidence limit ($P < .05$). The bond strengths were also evaluated as a function relating the probability of failure to applied stress by means of Weibull analysis.^{8,9} To analyze the failure sites, contingency tables were designed and subjected to the chi-square (χ^2) test.

RESULTS

Tensile and shear bond strengths

The descriptive statistics for each group tested is summarized in Table 1. The results revealed that the bond strength was significantly affected by the adhesive thickness ($P < .001$), the type of adhesive ($P = .001$), and the mode of testing ($P < .001$).

In the CO group, the mean tensile bond strength decreased as the adhesive thickness increased. However, in the FO group, the highest mean tensile bond strength was achieved at 0.25 mm ($FO_{25} = 7.6$ MPa). The ANOVA showed significant differences among the six subgroups tested under tensile type of loading ($F = 8.13$, $P = .000$) at the 95% confidence level (Table 1). Using the tensile bond strength as the dependent variable in a factorial AN-

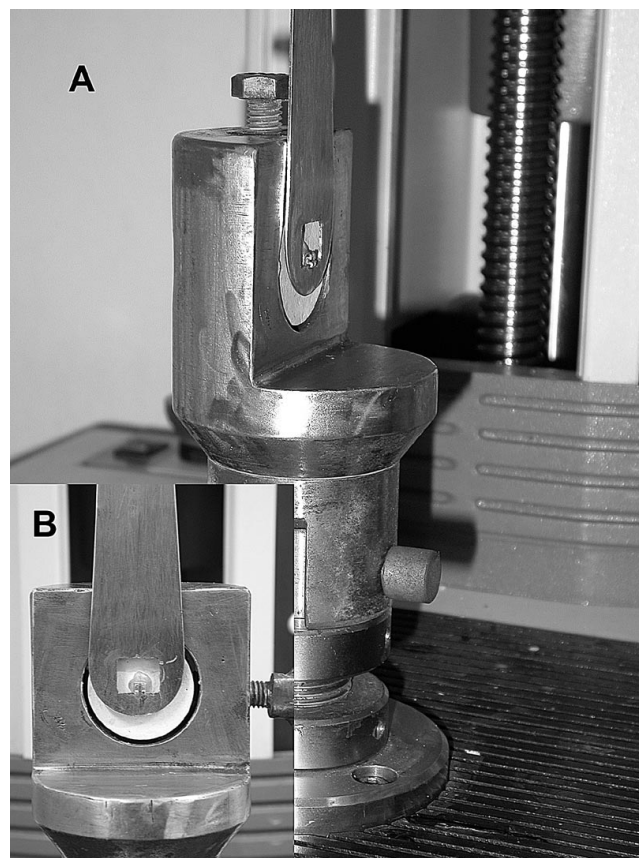


FIGURE 3. (A) Shear test equipment. (B) Close-up view of the stainless steel plate hooked under bracket tie-wings.

OVA, a significant interaction was found between the adhesive thickness (particularly at the 0 mm) and the type of the adhesive ($P = .000$).

Contrary to their mean tensile bond strengths, in the control group, the mean shear bond strength increased as adhesive thickness increased. FO_{25} , as in tensile testing, showed the highest shear bond strength (16.5 MPa) between the FO groups. The ANOVA revealed a statistically significant difference in the shear bond strength levels among the six groups tested ($F = 22.18$, $P = .000$) at the 95% confidence level (Table 1). Factorial ANOVA also showed a significant interaction between the adhesive thickness (particularly at the 0.5 mm) and the type of the adhesive ($P = .000$) in shear testing.

The results of the Weibull analysis of bond strengths are presented in Table 1. The predictability of a group is given in the Weibull modulus (m value). Higher m values indicate a more predictable system and, possibly, a more clinically reliable system. In both test modes, the FO groups produced lower m values than the CO groups at the three adhesive thicknesses.

The characteristic strength (σ_0) in the Weibull analysis refers to the bond strength at which 63.2% of the samples fail and is similar to the mean derived from the ANOVA

TABLE 1. Descriptive Statistics, ANOVA, and Parameters of the Weibull Analysis of Tensile and Shear Bond Strengths for Each Group^a

Test	Group ^b	n	Mean (MPa)	SD (MPa)	Range (MPa)	Tukey HSD ^c	Weibull modulus <i>m</i>	Correlation coefficient	Characteristic strength σ_0 (MPa)	Bond strength at 5% probability of failure $\sigma_{.05}$ (MPa)	Bond strength at 90% probability of failure $\sigma_{.90}$ (MPa)
Tensile	FO ₀	20	5.5	2.1	2.8–9.2	A	2.84	0.964	6.2	2.1	8.6
	FO ₂₅	20	7.6	2.2	4.8–12.0	B	3.76	0.969	8.4	4.0	10.5
	FO ₅₀	20	6.4	1.9	2.5–9.9	AB	3.95	0.979	7.3	3.6	9.7
	CO ₀	20	9.5	2.1	6.4–13.4	BC	5.13	0.984	10.3	6.1	12.2
	CO ₂₅	20	8.4	1.8	5.8–11.7	B	5.24	0.981	9.1	5.7	11.3
	CO ₅₀	20	7.7	2.1	4.0–11.6	B	4.10	0.987	8.5	4.6	11.1
Shear	FO ₀	20	12.3	2.9	8.2–18.2	E	4.58	0.979	13.4	7.5	16.0
	FO ₂₅	20	16.5	3.3	10.3–22.3	D	5.81	0.984	17.8	11.0	20.4
	FO ₅₀	20	13.2	3.4	7.9–19.5	DE	4.47	0.980	14.6	8.6	19.2
	CO ₀	20	16.1	2.4	11.2–21.8	D	6.71	0.943	17.2	12.3	20.0
	CO ₂₅	20	18.3	2.9	13.9–24.2	F	6.74	0.969	19.5	13.5	22.0
	CO ₅₀	20	21.2	3.5	14.6–28.6	DE	6.49	0.967	22.8	15.6	26.3

^a ANOVA indicates analysis of variance; HSD, honest significant difference; FO, light-cured, resin-modified glass ionomer cement groups; CO, light-cured composite resin groups.

^b 0, adhesive thickness was 0 mm; 25, adhesive thickness was 0.25 mm; 50, adhesive thickness was 0.5 mm.

^c Groups showed with different letters were significantly different at $P = .05$ level according to Tukey HSD test.

TABLE 2. Frequency and Percentage Occurrence (%) of the Adhesive Remnant Index (ARI) for Each Group Tested^a

Test Mode	Group ^b	n	ARI = 0	ARI = 1	ARI = 2	ARI = 3
Tensile	FO ₀	20	0 (0)	1 (5)	9 (45)	10 (50)
	FO ₂₅	20	2 (10)	4 (20)	6 (30)	8 (40)
	FO ₅₀	20	2 (10)	5 (25)	6 (30)	7 (35)
	Total (FO)	60	4 (6.7)	10 (16.7)	21 (35)	25 (41.7)
	CO ₀	20	1 (5)	1 (5)	9 (45)	9 (45)
	CO ₂₅	20	1 (5)	2 (10)	9 (45)	2 (40)
	CO ₅₀	20	2 (10)	1 (5)	6 (30)	11 (55)
	Total (CO)	60	4 (6.7)	4 (6.7)	24 (40)	28 (46.7)
Shear	FO ₀	20	3 (15)	5 (25)	6 (30)	6 (30)
	FO ₂₅	20	4 (20)	8 (40)	5 (25)	3 (15)
	FO ₅₀	20	7 (35)	6 (30)	4 (20)	3 (15)
	Total (FO)	60	14 (23.3)	19 (31.7)	15 (25)	12 (20)
	CO ₀	20	1 (5)	4 (20)	6 (30)	9 (45)
	CO ₂₅	20	1 (5)	5 (25)	7 (35)	7 (35)
	CO ₅₀	20	4 (20)	7 (35)	6 (30)	3 (15)
	Total (CO)	60	6 (10)	16 (26.7)	19 (31.7)	19 (31.7)

^a ARI scores: 0 = no adhesive left on the tooth, 1 = less than half of the adhesive left on the tooth, 2 = more than half of the adhesive left on the tooth, and 3 = all adhesive left on the tooth.

^b FO indicates light-cured, resin-modified glass ionomer cement groups; CO, light-cured composite resin groups; 0, adhesive thickness was 0 mm; 25, adhesive thickness was 0.25 mm; 50, adhesive thickness was 0.5 mm.

which assumes a normal distribution. The ranking of the characteristic strengths of all groups was the same as those of their mean bond strengths. Values of tensile and shear forces required for 5% and 90% probabilities of failures ($\sigma_{.05}$, $\sigma_{.90}$) revealed that, at lower force levels, the FO groups were more likely to fail than the CO groups. FO₀ showed the lowest values for the 5% and 90% probabilities of failures in both tensile ($\sigma_{.05} = 2.1$ and $\sigma_{.90} = 8.6$ MPa) and shear ($\sigma_{.05} = 7.5$ and $\sigma_{.90} = 16.0$ MPa) testing. Although CO₀ reached the highest values ($\sigma_{.05} = 6.1$ and $\sigma_{.90}$

= 12.2 MPa) in tensile testing, CO₅₀ had the highest values ($\sigma_{.05} = 15.6$ and $\sigma_{.90} = 26.3$ MPa) in shear testing (Table 1).

Failure sites

The distribution of failure sites (ARI scores) is given in Table 2. The χ^2 analysis of test mode (tensile, shear) vs failure site (total frequencies were used) revealed a statistically significant difference for the CO ($P = .019$) and FO

groups ($P = .003$). However, there was no significant difference between the distribution of ARI scores between the CO and FO groups when the failure sites were separately analyzed in tensile ($P = .40$) and shear test ($P = .13$) modes.

Under tensile forces, both bonding adhesives predominantly underwent bracket-adhesive interface failures (ARI scores 2 and 3). However, in shear testing, the FO₂₅ and FO₅₀ groups predominantly showed bond failures at the enamel-adhesive interface (ARI scores 0 and 1). None of the samples showed any grossly visible enamel fracture.

DISCUSSION

Tensile and shear bond strengths

In the present study, technique inconsistencies were minimized by using the same type of bracket for both control and test groups and by developing easily reproducible test methods. However, some unavoidable factors might still affect the outcome of specific tests.

Tensile testing requires specimen and substrate alignment, and a number of complex jigs have been designed for in vitro bond strength studies so that the forces act at right angles to the surface of the specimen.¹⁰⁻¹² However, peel and shear forces can still occur, despite these alignment jigs because of the complex geometry of orthodontic brackets.^{13,14}

According to beam theory, the further the applied force is from the bonding interface, the higher the applied moment.¹⁵ However, contrary to this intuitive conclusion, as force was applied farther from the tooth surface by increasing adhesive thickness from 0 to 0.25 mm, the shear bond strength increased with both adhesives. This increase in the shear bond strength continued when the adhesive thickness increased from 0.25 to 0.5 mm for the control group. This conflict in shear testing was explained by Katona,¹⁵ who used a finite element model to show that it is impossible to apply a pure shear load to a bracket because of an unavoidable inherent bending moment. Therefore, simpler uniform cross-section beam concepts are not necessarily applicable to tooth-bracket combination because of the geometric complexity.¹⁵

Although the mean tensile and shear bond strengths of the test group increased with increasing adhesive thickness from 0 to 0.25 mm, they decreased when the adhesive thickness was further increased from 0.25 to 0.5 mm. A possible explanation for this could be the polymerization reactions, namely, a slow acid-base reaction (between glass powder and organic acid) and an immediate photochemically induced polymerization (within the resin), taking place between the two components of the light-cured, resin-modified glass-ionomer cement. The cross-linking between these two polymerization reactions might need a greater thickness of the cement (more than 0 mm) to achieve high bond strength. However, complete blending, polymeriza-

tion, and cross-linking of the two phases might not occur when the adhesive thickness increased to 0.5 mm.

In this study, the mean tensile bond strength of the control group decreased when the thickness of the adhesive increased. These results are in conflict with those presented by Jost-Brinkmann et al⁵ who reported an increase when the thickness of the light-cured adhesives increased from 0 to 0.2 mm. However, it should be noted that they used bovine incisors and different light-cured composite resins.

As stated earlier, the mean shear bond strength of the control group increased when the adhesive thickness increased. Although these findings are consistent with those predicted by finite element modelling,^{15,16} they contradict those reported for light-cured and chemically cured composite resins in previous in vitro bonding studies.^{3,6}

Mean shear bond strengths similar to those obtained in the present study were recorded by Rix et al,¹⁷ who tested the micromesh-based metal brackets bonded with Transbond XT and Fuji Ortho LC resins on human premolar teeth. Another study,¹⁸ using the same bonding adhesives and bovine incisors, yielded higher mean shear bond strength values than those of the present study. However, in both studies, the adhesive thickness was not controlled.

Although the Weibull analysis is not routinely used in orthodontic bond strength studies, it is used to relate the results of in vitro studies to clinical performance.^{19,20} In the present study, a wide range of m values (Weibull modulus) were obtained (Table 1). FO₀ showed the lowest values for the 5% and 90% probabilities of failures and low m values in both test modes. Therefore, a high number of bond failures have to be expected when the light-cured, resin-modified glass-ionomer cement is applied at 0 mm thickness.

Failure sites

The present study indicates that all FO groups failed in tensile testing and FO₀ groups in shear testing predominantly at the bracket-adhesive interface (ARI scores 2 and 3) corroborating the findings of many authors.²¹⁻²³

The remaining FO groups, FO₂₅ and FO₅₀, predominantly failed at the enamel-adhesive interface as has been reported by other studies using resin-modified glass ionomer cements.^{18,24,25} The failure sites for the CO group were predominantly at the bracket-adhesive interface in both test modes. This is a common finding for in vitro bond strength studies using mesh-based metal brackets.^{4,5,22}

Clinical implications

All groups tested under tensile and shear forces in this study yielded mean bond strengths equivalent to those given to predict clinical success.^{26,27} The thickness of the adhesive is under the control of variable factors, such as the amount and viscosity of the adhesive resin, pressure applied during the bonding, and changes in the temperature and humidity in the oral environment.

Instead of controlling all these variable factors, a simple modification at the mesh-based bracket structure could create a homogeneous thickness of the adhesive resin. For example, small stops could be manufactured at the corners of the brackets to provide the necessary space to gain optimum adhesive thickness. Application of controlled bonding pressure through a bracket holder that has a pressure gauge could be another way to gain a homogenous adhesive thickness between the bracket and enamel. Of course, the other variables affecting the viscosity of bonding agent also should be strictly controlled during bonding.

The effect of the adhesive thickness on the bond strength of resin-modified glass ionomer cement under torque loading was not explored. Further investigations are needed to examine this subject.

CONCLUSIONS

- The light-cured, resin-modified glass-ionomer cement had its highest mean bond strength at the 0.25 mm thickness in both tensile and shear test modes.
- Although mean tensile bond strengths decreased, mean shear bond strengths of the light-cured composite resin control groups progressively increased when the adhesive thickness increased from 0 to 0.5 mm.
- The results of the Weibull analysis indicate that the adhesive layer thickness of the light-cured, resin-modified glass-ionomer cement could be more than 0 mm to gain clinically more reliable bond strength during orthodontic treatment.

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