Original Article

Effects of Thermocycling on the Bond Strength of a Resin-Modified Glass Ionomer Cement: An In Vitro Comparative Study

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Abstract: This study investigated the effects of thermally induced stresses (thermocycling) on the shear bond strength of resin-modified, chemically cured, glass ionomer cement for use as an orthodontic bonding agent. A conventional no-mix composite resin was also used as a control. Mesh-based metal orthodontic brackets were bonded to extracted human premolars using either the resin-modified glass ionomer cement or the no-mix composite resin. Specimens were stored either in water at 37°C for 24 hours for baseline data or thermocycled between 5°C and 55°C for 200 and 20,000 cycles before testing the in vitro shear bond strengths. Thermocycling reduced shear bond strengths for all specimens. The resin-modified glass ionomer cement showed a 11.1% decrease after 200 thermocycles and 26.5% decrease after 20,000 thermocycles, whereas the no-mix adhesive resin showed only 5.7% and 17.9% reductions, respectively. Analysis of variance showed statistically significant differences between the mean shear bond strengths of the groups at the $P < .001$ level of significance. For the resin-modified glass ionomer cement groups, the predominant bond failure site was at the bracket-adhesive interface. The results of this study suggest strongly that resin-modified glass ionomer cements offer a viable alternative to conventional no-mix composite resins, with satisfactory in vitro shear bond strength even after 20,000 thermocycles. (*Angle Orthod* 2003;73:692–696.)

Key Words: Shear bond strength; Thermally induced stress; Bonding adhesives

INTRODUCTION

Glass ionomer cements were invented by Wilson and Kent and have the ability to bond unetched enamel physicochemically, thereby eliminating the need for acid etching of the enamel surface.[1,2] They also release fluoride ions over long periods into adjacent enamel and have the capacity to absorb fluoride from fluoride gels, increasing their release of fluoride ions.[3,4] However, several in vitro studies have reported that the bonding strength of glass ionomer cements to enamel and the bracket is significantly lower than that of conventional composite resin.[5–7]

Recently, the development of resin-modified glass ionomer cements has provided a new adhesive resin, which combines the advantageous characteristics of composite resins and glass ionomer cements. Thus, the physical and mechanical properties of glass ionomer cements became similar to those of composite resins while the fluoride release was maintained.[8] Several studies reported that in vitro testing of these new products provided enough bond strength to withstand orthodontic forces during actual in vivo application.[9,10] However, many of these studies did not show how the bond strength of these cements were affected by the humidity and the body temperature changes in the long term. As Buonocore[11] stated, the in vitro bond strength tests could include the thermal cycling of the specimens to assess the durability of the bond. Otherwise, in vitro results might not be indicative of the effect of oral moisture conditions on bond strength.

This in vitro study was therefore designed to investigate the effects of thermocycling on the shear bond strengths of a resin-modified, chemically cured, glass ionomer cement used for bonding of orthodontic brackets and to compare this bonding agent with a no-mix conventional composite resin. The bond failure sites were also investigated.

MATERIALS AND METHODS

Teeth used

Ninety human premolars extracted for orthodontic reasons were cleared of debris and stored in distilled water.

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The criterion for tooth selection was perfect labial enamel, with neither cracks nor damages caused by extraction forceps.

Adhesives and bracket

The resin-modified glass ionomer cement used in this study was a chemically cured system specifically formulated for orthodontic bonding (Fuji Ortho, GC Corporation, Tokyo, Japan). A no-mix composite resin (Leone, Leone Sesto, Fiorentino, Italy) made for the bonding of orthodontic brackets was also used for the purposes of comparison. All brackets used for the test and control groups were foil-mesh–based stainless steel upper premolar brackets (Midi Diogonal, Leone Sesto, Fiorentino, Italy).

The selected teeth were prepared for accurate placement into plastic mounting cups by removing two-thirds of their roots. They were then mounted in plastic cups filled with a low-temperature–setting resin of the polyester type (Metset Mounting Plastics, Buehler UK Ltd, Coventry, UK), leaving the labial surface exposed. The mounted teeth were stored at room temperature in distilled water and randomly assigned to one of the test groups.

Bonding

Before bonding, the labial surfaces of the mounted teeth in all groups were polished using a nonfluoride pumice paste in a prophy cup, rinsed with water, and dried with an air spray. For the control groups, the etching procedure was accomplished with 37% phosphoric acid gel for 30 seconds, rinsed with water for 20 seconds, and dried with compressed air. Then 45 brackets were bonded using the no-mix composite resin according to manufacturer's recommendations.

The labial surfaces of the other 45 samples selected for bonding with resin-modified glass ionomer cement were pretreated with an application of 10% polyacrylic acid for 20 seconds, rinsed with water for 20 seconds, and slightly dried with a light flow of air before bonding. Fuji Ortho is a powder and liquid system, in which the powder and liquid were mixed in recommended proportions for 30 seconds before this mixture was applied to the bracket base. After seating the bracket on the tooth surface with firm pressure, excess adhesive was removed with a scaler. Three brackets were bonded with each mix. The tooth-bracket combinations were then left undisturbed for 10 minutes at room temperature before being stored in water at 37°C for 24 hours.

Storage conditions

Two groups of samples (resin-modified glass ionomer = F and no-mix adhesive resin = N) were further divided into three subgroups. Each subgroup consisted of 15 samples. The storage conditions of subgroups were

- In subgroups 24, tooth-bracket combinations were stored in distilled water at 37°C for 24 hours.
- In subgroups T1, samples were stored in distilled water at 37°C for 24 hours and then thermocycled in water between 5 ± 2°C and 55 ± 2°C for 200 cycles.
- In subgroups T2, samples were stored in distilled water at 37°C for 24 hours and then thermocycled in water between 5 ± 2°C and 55 ± 2°C for 20,000 cycles.

During thermocycling, the dwelling time for the specimens in each well was 30 seconds, and the transfer time between the wells was four seconds. The specimens were stored at 37°C in distilled water for 24 hours to provide baseline data for comparative purposes. Two cycle times for thermocycling were used. One was to measure the effects on bond strength of short and long time exposure to moisture at oral temperature and the other was to simulate accelerated aging by thermally induced stresses.

Bond strength testing

In this study, shear bond strength tests were carried out using a Lloyd LRX testing machine (Lloyd Instruments Plc., Fareham, Hampshire, UK). To create accurate shear-type forces, a special jig was constructed and attached to the jaws of the testing machine. Each test specimen was placed into a holding ring positioned in the lower jaw of the testing machine so that the bracket base of the sample was parallel to and centered on the direction of the force applied. A stainless steel plate was fixed to the upper jaw of the testing machine, with the lower part of the plate hooked under the gingival tie-wings of the brackets (Figure 1). The brackets always should be loaded from beneath the tie-wings to reduce the peeling moment for in vitro shear testing.[12,13] The machine was activated with a crosshead speed of one mm/minute until failure was noticed. The peak force levels, automatically recorded on the testing machine, were converted to stress per unit area (MPa) by dividing the force (N) by the mean unit area of the base of the bracket (11.9 mm^2).

After testing, the separated assemblies were recovered and examined under an optical microscope at 20× magnification to determine the site of failure.

Statistical analysis

A comparison between the groups was made using analysis of variance (ANOVA). When ANOVA showed statistically significant differences between the groups tested, Tukey's honest significant difference (HSD) multiple-range test was performed to find significant differences in bond strengths between any two groups at the 0.95 level of confidence.

The failure sites were classified using the adhesive remnant index (ARI)[14] and compared using the chi-square (χ^2) test.

FIGURE 1. Shear test equipment used in this study.

TABLE 1. Descriptive Statistics of Shear Bond Strengths for Each Group[a]

Group	N	Mean (MPa)	SD	Minimum	Maximum	Tukey's HSD*
N_{24}	15	22.9	4.9	17.3	31.0	A
N_{T1}	15	21.6	4.6	16.1	28.3	A
N_{T2}	15	18.8	4.4	13.8	27.2	AB
F_{24}	15	16.2	4.2	11.1	24.0	BC
F_{T1}	15	14.4	3.7	8.0	19.3	C
F_{T2}	15	11.9	3.4	8.2	19.0	C

[a] F indicates resin-modified glass ionomer cement groups; N, no-mix composite resin groups; 24, stored in distilled water at 37°C for 24 hours; T1, thermocycled in water between 5 ± 2°C and 55 ± 2°C for 200 cycles; T2, thermocycled in water between 5 ± 2°C and 55 ± 2°C for 20,000 cycles.

* Groups shown with different letters were significantly different at $P = .05$ level according to Tukey's HSD test.

TABLE 2. Frequency and Percentage Occurrence of the adhesive remnant index (ARI) for Each Group Tested[a]

Group[b]	N	ARI = 0	ARI = 1	ARI = 2	ARI = 3
N_{24}	15	6 (40%)	4 (26.7%)	3 (20%)	2 (13.3%)
N_{T1}	15	5 (33.3%)	5 (33.3%)	2 (13.3%)	3 (13.3%)
N_{T2}	15	5 (33.3%)	4 (26.7%)	3 (20%)	3 (20%)
Total (N)	45	16 (35.5%)	13 (28.8%)	8 (17.7%)	8 (17.7%)
F_{24}	15	3 (20%)	3 (20%)	3 (20%)	6 (40%)
F_{T1}	15	3 (20%)	2 (13.3%)	3 (20%)	7 (46.7%)
F_{T2}	15	2 (13.3%)	1 (6.7%)	2 (13.3%)	10 (66.7%)
Total (F)	45	8 (17.7%)	6 (13.3%)	8 (17.7%)	23 (51.5%)

[a] Adhesive remnant index (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; 3, all adhesive left on the tooth.

[b] F indicates resin-modified glass ionomer groups; N, no-mix composite resin groups; 24, stored in distilled water at 37°C for 24 hours; T1, thermocycled in water between 5 ± 2°C and 55 ± 2°C for 200 cycles; T2, thermocycled in water between 5 ± 2°C and 55 ± 2°C for 20,000 cycles.

RESULTS

After either 200 or 20,000 cycles, there was a reduction of the mean shear bond strengths of both the resin-modified glass ionomer cement and no-mix adhesive resin groups (Table 1). The conventional no-mix composite resin had the highest mean shear bond strength (22.9 MPa) when specimens were stored in distilled water at 37°C for 24 hours (N_{24}). The lowest value (11.9 MPa) was given with the resin-modified glass ionomer cement after 20,000 thermocycles (F_{T2}). Using the bond strength (MPa) as the dependent variable, ANOVA showed a significant difference between the groups tested ($F = 14.73$, $P = .000$).

The grouping of these differences by Tukey's HSD multiple-range test indicated that the groups N_{24} (22.9 MPa) and N_{T1} (21.6 MPa) demonstrated higher mean shear bond strength than the groups F_{24} (16.2 MPa), F_{T1} (14.5 MPa), and F_{T2} (11.9 MPa). The mean shear bond strength of N_{T2} (18.8 MPa) was also significantly higher than F_{T1} and F_{T2} (Table 1).

Table 2 shows the distribution of ARI scores (failure sites) expressed as frequency of occurrence. The resin-modified glass ionomer groups, namely, F_{24}, F_{T1}, and F_{T2}, showed a predominant ARI score of 3, whereas the no-mix composite resin groups, N_{24}, N_{T1}, and N_{T2}, predominantly had ARI scores of 0 and 1. In other words, the resin-modified glass ionomer cement groups predominantly failed at the bracket-adhesive interface (ARI scores of 2 and 3), whereas the failure site was frequently at the enamel-adhesive interface (ARI scores of 0 and 1) for the no-mix composite resin. After thermocycling, there was an increase in ARI score of 3 in the resin-modified glass ionomer groups. The χ^2 test showed a statistically significant difference ($P = .001$) between the adhesive types. When performing the χ^2 test, for each adhesive group, total frequencies ($F = F_{24} + F_{T1} + F_{T2}$ and $N = N_{24} + N_{T1} + N_{T2}$) were used because of the low expected frequencies of the cells. There was no visible enamel fracture in any of the groups.

DISCUSSION

A balance in bond strength must be achieved when choosing a bracket-adhesive combination for fixed orthodontic treatment. Bond strength should not only be high enough to resist the forces during the course of orthodontic treatment but also low enough to allow the removal of the bracket without any complications at the end of orthodontic treatment. Therefore, high mean bond strength does not necessarily mean better clinical performance.

Guidelines for adequate in vitro shear bond strength have not been reported. However, some reports have suggested that previous bonding studies might be used as a guide to analyze the shear bond strength.[15] Shear bond strength studies using metal brackets have reported bond strengths in the 12.1–20.7 MPa range. The groups in this study generated shear bond strengths ranging from 12 to 23 MPa that is almost within the range stated in the literature.

After thermocycling, there was a reduction in the mean shear bond strengths for both the no-mix adhesive and resin-modified glass ionomer cement groups. This effect of thermocycling on bond strength for different adhesives was also found in several other studies.[5,9,15–18] However, there is no standardization for thermocycling times between the different studies. Cycling times range from 100 to 2500 from one study to another.

In this study, the reduction of the mean shear bond strengths was around 11.1% for the 200 times thermocycled group (F_{T1}) and 26.5% for the 20,000 times thermocycled group (F_{T2}) with the resin-modified glass ionomer cement, whereas only around 5.7% (N_{T1}) and 17.9% (N_{T2}) reductions were found with the no-mix adhesive resin groups, respectively.

The decrease in the bond strengths of thermally cycled specimens relative to those that were not cycled may possibly be explained by the absorption of water and the alternating stressing of the system resulting from the large mismatch of the thermal expansion coefficient of the adhesives (for resin composites $\alpha = 20–55$ ppm/°C; for glass ionomer cements $\alpha = 8$ ppm/°C) with those of the stainless steel bracket (for 316 L stainless steel $\alpha = 16$ ppm/°C) and enamel ($\alpha = 12$ ppm/°C).[19,20] These differences between the thermal coefficients of three components of the system are likely to affect adversely the adhesion of the resin to other parts of the system. The cyclical stress may cause any debonded regions at the interfaces to grow progressively in size. Because the resin-modified glass ionomer cement consists of a mixture of two components, namely, glass ionomer and resin adhesive, this extra interface between the two might make this cement more prone to this adverse effect.

Although technique inconsistencies were minimized by using a standardized sample preparation and testing method (performed by one investigator), the ranges and standard deviations of bond strength were high in all groups. This could be due to the variations in the buccal surface morphology of the premolar teeth, the amount of the adhesive resin applied to the bracket base, and the application force during bonding. Another reason for the wide ranges of bond strength might also be the inability of the operator to place all the bonded specimens into the sample jig in such a manner that their openings underneath the tie-wings were exactly at the middle of the jig. In addition, the outlier values could not be excluded from the data pool because of the limited number of the samples.

A direct comparison between the results of the present study and those of others is somewhat difficult because there has been no standardization of testing techniques in the dental literature and because of the variety of materials and methods used.[21] However, despite these variations, the present results may at least in part be compared with those of previous studies in which similar test methods and materials were used.

In this study, the mean shear bond strength values for metal brackets bonded with the resin-modified glass ionomer cement (Fuji Ortho) were lower than those reported by Komori and Ishikawa,[9] who reported an average value of 20.1 MPa for the same cement without thermocycling. They also reported a mean shear bond strength value of 17.9 MPa (10.9% decrease) with the same resin-modified glass ionomer cement after thermocycling. However, they did not report the number of cycles applied during thermocycling and used flattened bovine enamel in their study.

Mean shear bond strengths similar to those obtained in the present study were recorded by Lippitz et al,[10] who tested the mesh-based metal brackets bonded with resin-modified glass ionomer cements (Advance, Fuji Duet, Fuji Ortho LC) on human premolars. Another study, using a broadly comparable method and using a light-cured, resin-modified glass ionomer cement (Fuji Ortho LC), yielded lower mean shear bond strength values than those of the present study.[4] Lower mean shear bond strength values were also recorded by Jou et al[22] and Cacciafesta et al,[23] although in both studies, light-cured resin-modified glass ionomer cements and ceramic brackets were used.

The ARI scores indicate that bond failure predominantly occurs at the bracket-adhesive interface (ARI scores of 2 and 3) with the resin-modified glass ionomer cement. Similar findings were also reported for metal and ceramic brackets bonded with resin-modified glass ionomer cements.[7,9,10,24] The occurrence of bracket-adhesive interface failures was observed more in the thermocycled groups of resin-modified glass ionomer cement. Although the application of shear-type forces is not the intended technique for debonding of the metal brackets, the occurrence of this type of failure site for the brackets bonded with this adhesive may offer a clinical advantage in protecting the adhesive-enamel interface from damage. It could also act as a ‘‘safety valve’’ in protecting the enamel if excessive tensile or shear type of forces were accidentally applied.

CONCLUSIONS

The results of the present in vitro study indicate that although there was a reduction in the mean shear bond strength after thermocycling, the resin-modified glass ionomer cement (Fuji Ortho) showed bond strengths within the guidelines given in the literature and a preferable bond failure site.

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