Original Article

Influence of food-simulating liquids on bond strength of brackets bonded with a HEMA-free and HEMA-containing self-etching primer

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ABSTRACT

Objective: To evaluate the effect of food-simulating liquids on bond strengths of brackets bonded with a HEMA-free and a HEMA-containing self-etching primer.

Materials and Methods: Brackets were bonded to 280 bovine incisors that were divided into two groups: (1) Adper Prompt-L-Pop (Adper PLP)/Transbond-XT and (2) Transbond Plus self-etching primer (TSEP, HEMA-free)/Transbond-XT. Each group was evaluated under different storage conditions: 24 hours in water, thermocycling (T), T/12 weeks in water, T/12 weeks in 10% ethanol, T/12 weeks in 50% ethanol, T/12 weeks in 3% acetic acid, and T/12 weeks in olive oil. Shear bond strength was measured with a universal test machine.

Results: TSEP and Adper PLP showed a significantly higher bond strength at 24 hours than at T/12 weeks in 50% ethanol ($P = .000$). For Adper PLP, the bond strength at 24 hours was significantly higher than T/12 weeks in water ($P = .000$). Significant differences were not detected between the two bonding procedures for the different storage conditions ($P > .05$).

Conclusion: Owing to its hydrophilic nature, the bond strength produced by Adper PLP (a HEMA-containing self-etching primer) decreased significantly after T/12 weeks in water. Brackets bonded with both TSEP and Adper PLP showed significantly higher bond strengths at 24 hours than at T/12 weeks in 50% ethanol, probably due to the effect of ethanol at 50% on Transbond-XT. (*Angle Orthod.* 2012;82:346–350.)

KEY WORDS: Food-simulating liquids; HEMA-free self-etching primer; HEMA-containing self-etching primer; Shear bond

INTRODUCTION

Among the dental adhesives available today, self-etching adhesives are clinically easier to use and for this reason attractive for day-to-day use in a busy practice. They are based on polymerizable acidic monomers that condition and prime enamel and dentin simultaneously. It has been shown that these bond materials adhere to the tooth both micromechanically and chemically.[1] This twofold bonding mechanism is believed to be advantageous in terms of restoration durability. The micromechanical bonding component may provide resistance to abrupt debonding stress, and a chemical interaction may result in bonds that show greater resistance to hydrolytic breakdown and therefore greater durability.[2]

Most self-etching adhesives contain methacrylate monomers, which are highly hydrophilic. Among the hydrophilic monomers most used is 2-hydroxyethyl methacrylate (HEMA). It has been shown that the introduction of small quantities of HEMA (between 10% and 19%) improves bond strength to enamel significantly; however, the introduction of greater quantities (36%) decrease bond strength due to water attraction and osmosis through the polymerized adhesive layer.[3] HEMA-free adhesives have been developed with the aim of overcoming the disadvantages of HEMA-containing adhesives related to water retention.[4]

The chemical environment in the oral cavity can have a critical in vivo influence on the degradation of

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Table 1. Composition of the Adhesives According the Manufacturer's Material Safety Data Sheets

Adhesive	Composition	% by Weight
Transbond XT Paste	Silane-treated quartz	70–80
	Bisphenol A diglycidyl ether dimethacrylate	Oct 20
	Bisphenol A bis (2-hydroxyethil ether) dimethacrylate	May 10
	Dichlorodimethylsilane reaction product with silica	<2
TSEP	Methacrylate ester derivative	75–85
	Water	15–25
Adper PLP	Part A	
	Di-Hema phosphate	75–90
	Bisphenol A diglycidyl ether dimethacrylate	Oct 15
	Ethyl 4-dimethyl aminobenzoate	<2
	DL-camphorquinone	1–1.5
	Part B	
	Water	70–80
	2-hydroxyethyl methacrylate (HEMA)	17–28

dental materials. Materials in the mouth suffer an aging process provoked by diet, which can be simulated effectively by immersing the materials in food-simulating liquids (FSLs). Various authors have noted that the mechanical properties of restoration materials are affected by exposure to FSLs.[5–9] However, research into the effect of FSLs on bracket bonding is scarce[10,11] and to date, the influence of FSL on bond strength for HEMA-free self-etching primers has not been evaluated at all.

The aim of this study was to evaluate in vitro the effect of different FSLs on resistance to shear forces of brackets bonded with a HEMA-containing self-etching primer (Adper Prompt L-Pop [Adper PLP], 3M ESPE AG Dental Products, Seefeld, Germany) and a HEMA-free self-etching primer (Transbond Plus self-etching primer [TSEP], 3M Unitek Dental Products, Monrovia, Calif).

MATERIALS AND METHODS

Teeth

Two hundred eighty bovine lower central incisors were used. The teeth were washed in water to remove any traces of blood and then placed for 24 hours in a 0.1% thymol solution. Afterward, they were stored for 1 week in distilled water, which was changed periodically to avoid deterioration.

Brackets

Two hundred eighty upper central incisor brackets were used (Victory Series, 3M Unitek Dental Products). The base area of each bracket was calculated (mean = 10.25 mm^2) using image analysis equipment and MIP 4 software (Micro Image Processing Software, Digital Image Systems, Barcelona, Spain).

Bonding Procedure

Teeth were divided into two groups, and brackets were bonded to the buccal surfaces according to the instructions supplied by the manufacturer of each product. For all groups, buccal surfaces were polished with a rubber cup and polishing paste (Détartrine, Septodont, Saint-Maur, France).

Group 1 (n = 140): TSEP/Transbond XT. The enamel was treated with TSEP, which was gently rubbed onto the enamel for 3 seconds using the disposable applicator supplied with the system. A moisture-free air source was used to deliver a gentle burst of air to the primer. Transbond XT paste was applied to the base of the bracket, which was then placed on the tooth, pressing firmly. Excess adhesive was removed from around the base of the bracket. The adhesive was light cured, positioning the light guide of an Ortholux XT lamp (3M Unitek Dental Products) on each interproximal side for 10 seconds.

Group 2 (n = 140): Adper PLP/Transbond XT. Adper PLP was gently brushed onto the enamel for 15 seconds with the disposable applicator supplied with the system. A moisture-free air source was used to deliver a gentle burst of air to the primer. The self-etch primer was light cured for 10 seconds, and then the bracket was bonded with Transbond XT paste as in group 1 (Table 1).

Storage of Test Specimens

The specimens in each group (n = 140) were randomly divided into seven subgroups of 20 specimens. Each subgroup was subjected to one of the following storage conditions:

- Condition 1: Specimens were stored in distilled water at 37°C for 24 hours.[12]
- Condition 2: Specimens were subjected to thermocycling.[12] The thermocycling test comprised 500

cycles in distilled water at between 5°C and 55°C, with the specimens having been previously stored for 24 hours in water kept at 37°C. The exposure to each bath lasted 20 seconds, and the transfer time between baths was between 5 and 10 seconds.
- Condition 3: Specimens were thermocycled as for condition 2 and then stored in distilled water for 12 weeks.
- Condition 4: Specimens were thermocycled as for condition 2 and then stored in a 10% ethanol solution for 12 weeks.
- Condition 5: Specimens were thermocycled as for condition 2 and then submerged in a 50% ethanol solution for 12 weeks.
- Condition 6: Specimens were thermocycled as for condition 2 and then submerged in a 3% acetic acid solution for 12 weeks.
- Condition 7: Specimens were thermocycled as for condition 2 and then stored in olive oil for 12 weeks.

Substrates were changed weekly during the 12-week period and maintained at 37°C. The FSLs used for specimen conditioning were chosen according to Food and Drug Administration guidelines.[13] Distilled water simulates aqueous foods, whereas 3% acetic acid simulates acidic foods. The 10% ethanol aqueous solution simulates alcoholic beverages with up to 15% volume ethanol, while the 50% ethanol aqueous solution simulates drinks with high alcohol content. Olive oil simulates fatty foods.

Bond Strength Test

Shear bond strength was measured with a universal test machine (Autograph AGS-1KND, Shimadzu, Kyoto, Japan) with a 1-KN load cell connected to a metal rod with one end angled at 30°. The cross-head speed was 1 mm/min.[12]

Figure 1. The sharp end of the rod incised in the area between the base and the wings of the bracket exerting a force parallel to the tooth surface in an inciso-apical direction.

The teeth were set at the base of the machine so that the sharp end of the rod incised in the area between the base and the wings of the bracket, exerting a force parallel to the tooth surface in an inciso-apical direction (Figure 1).

The force required to debond each bracket was registered in Newtons (N) and converted into mega-Pascals as a ratio of Newton to surface area of the bracket (MPa = N/mm^2).

Statistical Analysis

Bond strengths for each bonding procedure were compared individually under the seven storage conditions. As the data were not distributed normally, they were analyzed using the Kruskal-Wallis test ($P < .05$), finding those groups that were significantly different with the Mann-Whitney test for two independent samples. To avoid an accumulation of errors due to multiple comparisons, the significance level ($P < .05$) was modified, dividing this between the number of comparisons made (Bonferroni correction), and $P < .002$ was considered significant.

The shear bond strengths of the two bonding procedures were compared across the seven different storage conditions. When the data fulfilled the criteria for normality, the existence of significant differences was analyzed using the *t*-test for two independent samples, and when data did not show a normal distribution, significant difference was evaluated using the Mann-Whitney test for two independent samples ($P < .05$).

RESULTS

Table 2 shows bond strength values for each of the groups evaluated. When bond strengths for each bonding procedure were compared under the seven storage conditions, TSEP showed a significantly

Table 2. Mean Shear Bond Strength (MPa) and Standard Deviation (SD) for Each Group (n = 20)[a]

Storage Condition	TSEP (Mean ± SD)	Adper PLP (Mean ± SD)
24 h	8.56 ± 3.40[a]	7.96 ± 2.46[a]
Thermocycled (T)	6.78 ± 2.11	6.87 ± 1.83
T/water	6.04 ± 2.54	4.98 ± 1.11[b]
T/ethanol 10%	6.95 ± 2.00	6.39 ± 1.74
T/ethanol 50%	5.14 ± 1.73[b]	4.68 ± 2.47[b]
T/acetic acid 3%	6.23 ± 2.75	6.65 ± 3.33
T/olive oil	6.86 ± 1.87	6.60 ± 1.50

Significant differences were not detected between TSEP and Adper PLP for the seven different storage conditions ($P > .05$). For each bonding procedure, different lowercase letters indicate significant differences ($P < .002$). Values unmarked by a letter did not show significant differences with any other ($P > .002$).

higher bond strength at 24 hours than when the specimens were thermocycled and immersed in ethanol 50% ($P = .000$). Like TSEP, Adper PLP showed a shear bond strength at 24 hours significantly higher than when thermocycled and immersed in ethanol 50% ($P = .000$). Adper PLP bond strength also decreased significantly after thermocycling and immersion in distilled water for 12 weeks ($P = .000$). Significant differences were not found for the rest of the storage conditions ($P > .002$).

Significant differences were not detected between the shear bond strengths of the two bonding procedures for the seven different storage conditions ($P > .05$).

DISCUSSION

In the oral environment, a number of diverse factors (temperature fluctuations, mechanical stress, enzymes, food and drink, etc.) interact simultaneously to accelerate the degradation of bond materials. When the aging factor is introduced into study design, bond strength tests can provide clinical information of greater relevance.[2] There are a number of different methods for the accelerated simulation of aging, one of them being the exposure of bond materials to FSLs.

Thermocycling is another method used for simulating material aging. Intraoral temperature fluctuation can cause repetitive contraction/expansion stress at the tooth-adhesive interface due to the bond material's greater thermal contraction/expansion coefficient in comparison with the tooth. Such stress may lead to cracks that propagate over bonded interfaces, and once a gap is created, shifting gap dimensions can cause inflows and outflows of oral fluids, a process known as percolation.[14] Hot water can accelerate hydrolysis of interface components, leading to uptake of water and extraction of breakdown products or poorly polymerized oligomers.[15,16]

When an accelerated aging test is carried out, it is important to include one or more control groups.[2] In the present study, two control groups were set up: a 24-hour group and a thermocycled group. As it is crucial to first determine the short-term bonding effectiveness of adhesives, the 24-hour group was established as a data baseline for assessing long-term effectiveness.[2] The thermocycled control group was introduced with the aim of distinguishing the effect of thermocycling on bond strength from the effects of FSLs, given that the test groups were kept in FSLs for 12 weeks after thermocycling. It has been observed that over a time span of this duration, all types of adhesive will undergo a mechanical and morphological degradation that resembles in vivo aging.[2]

For both TSEP and Adper PLP, no significant differences in bond strength were detected between the specimens that were immersed in distilled water for 24 hours and those that were thermocycled. The thermocycling was carried out following recommendations described in ISO TR11405 guidelines. However, some studies state that 500 cycles are insufficient for simulating or evaluating the effectiveness of bracket bonding in the long term.[14]

After subjecting specimens to thermocycling and immersing them in water for 12 weeks, Adper PLP (HEMA-containing self-etching primer) bond strength was significantly less than after 24 hours, while TSEP (HEMA-free self-etching primer) showed no effects. Despite the greater degree of Adper PLP's bond strength affectation, significant differences in bond strength were not found for either of the self-etching primers under this storage condition. Our results coincide with those of Torkabadi et al.,[3] who found that there was a significant reduction in bond strength of a HEMA-containing adhesive after storage in water, whereas bond strength produced by a HEMA-free adhesive decreased over time, but no significant differences were observed. The former is thought to be caused by a degradation of interface components through hydrolysis. But water can also infiltrate and decrease the mechanical properties of the polymer matrix by swelling and reducing the frictional forces between polymer chains, a process known as plasticization. Hydrophilic materials act as semipermeable membranes; by attracting water, they degrade faster than hydrophobic adhesives.[2] Water absorption is directly correlated with the hydrophilicity of the resin.[17,18]

Brackets bonded with both TSEP (HEMA-free) and Adper PLP (HEMA-containing) showed significantly higher bond strengths at 24 hours than when the specimens had been thermocycled and immersed in 50% ethanol. However, after immersion in 10% ethanol, bond strength was not affected. This decrease shared by both groups may be due to the effect of ethanol on the composite resin used for bracket bonding (Transbond XT). Hobson et al.[10] found that Transbond XT bond strength was significantly less after 12 weeks of storage in 50% ethanol, while 8% ethanol had no effect on bond strength. Other authors have observed that ethanol decreases composite bond strength,[7–11] surface hardness, resistance to wear,[5] and fracture toughness.[8] The diffusion of ethanol into the composite can result in structure microcracking that may subsequently weaken the bond. Ethanol has similar solubility characteristics to Bis-GMA, and this may further promote the infusion of ethanol into the composite, leading to further damage.[7] It has also been suggested that this effect of ethanol could be caused by the ethanol softening or dissolving and/or attacking the silane bond.[8]

As in other studies, the effect of immersion in acetic acid and olive oil did not have any effect on bond strength, and this has been attributed to the absence of a plasticizing effect on polymers.[19]

Despite Adper PLP's a significant decrease in bond strength after thermocycling and storage in distilled water for 12 weeks, no significant differences were found between the two self-etching primers in this storage condition or in any other. We have to take into account that this is an in vitro study, and we must be cautious when extrapolating results to a clinical situation.

CONCLUSIONS

- After subjecting specimens to thermocycling and immersion in distilled water for 12 weeks, bond strength for the HEMA-containing self-etching primer (Adper PLP) was significantly less than after 24 hours, whereas HEMA-free self-etching primer (TSEP) was not affected.
- Both brackets bonded with TSEP, and those bonded with Adper PLP showed significantly higher bond strengths at 24 hours than when thermocycled and immersed in ethanol 50%.
- Thermocycling and immersion in 10% ethanol, 3% acetic acid, and olive oil did not significantly affect the self-etching primers' bond strength.

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