Original Article

A Comparison of Shear Bond Strength and Debonding Characteristics of Conventional, Moisture-Insensitive, and Self-etching Primers In Vitro

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Abstract: Bond failure is often attributed to moisture contamination. To overcome this commonly encountered problem, materials have been developed that are hydrophilic and that are believed to offer better bond strength in moisture-contaminated environment. Shear bond strength was compared among three materials: conventional Transbond XT primer (3M Unitek), moisture-insensitive primer (MIP, 3M Unitek), and self-etch primer (Transbond plus, 3M Unitek). Bond strength was tested under laboratory conditions with brackets bonded on both dry enamel and enamel contaminated with natural saliva. Self-etch primer showed maximum bond strength under both dry and wet conditions. Conventional primer was comparable with the former under dry conditions but did not offer clinically adequate bond strength in cases of moisture contamination. Both MIP and self-etch primer showed adequate bond strength superior to that of conventional primer in case of moisture contamination. All primers showed typical debonding characteristics of separation at the bracket-adhesive interface or within the adhesive itself, with the exception of the conventional primer used with moisture-contaminated enamel. (*Angle Orthod* 2004;74:264–268.)

Key Words: Contamination; Hydrophilic; Hydrophobic; Adhesive

INTRODUCTION

Genesis of the acid-etch technique and the subsequent adaptation of direct bonding in orthodontics has revolutionized the placement of orthodontic appliances. Rapid strides in material science over the years have produced progressively advanced materials making the direct bonding procedure more precise, comfortable, and time-effective.

However, the stress is still on clinically adequate bond strength, which is affected by bracket base design, adhesive used, bonding protocol, etc.[1] Bonding is a technique-sensitive procedure, and moisture is cited as the most common cause for bond failure.[2–5] Contamination causes plugging of porosities caused by acid etching and a reduction in surface energy. Thus, the penetration of the resin is impaired, and the micromechanical retention is compromised.

Despite their hydroxyl groups, conventional bis-phenol A glycidal methacrylate (Bisgma) resins are hydrophobic and are efficient only in a dry environment.[2,6] A possible solution to this problem has been offered by the development of the moisture-insensitive primer (MIP). These are developed based on dentin-bonding agents, which have hydrophilic components, such as hydroxyethyl methacrylate (HEMA) and maleic acid dissolved in acetone, that are efficient even in the presence of moisture.[7,8]

Another novel concept is the sixth-generation bonding systems, where etching and priming agents are combined into a single acidic primer solution. These self-etch primers help the clinician save time, reduce cross-contamination, and reduce wastage. Because they are hydrophilic, it is logical to presume that they may be effective in situations with minimal moisture contamination.[9]

Although literature exists in which the bond strengths of MIP and self-etching primers have been independently compared with conventional primers, no reported study has compared the bond strength of all three.

In addition to offering good bond strength, bonding agents should enable easy debonding and cleanup procedure without causing enamel damage.

Therefore, this study was undertaken for two reasons:

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Accepted: May 2003. Submitted: February 2003.

- To evaluate and compare the shear bond strength of self-etch primer (Transbond Plus), MIP, and conventional (Transbond XT) primer under both dry and contaminated conditions;
- To study the debonding characteristics and site of bond failure of specimens bonded with the above primers.

MATERIALS AND METHODS

Bonding system

Transbond XT was the adhesive used. Three types of primers were tested under both dry and contaminated conditions:

- Conventional primer: Transbond XT (3M Unitek);
- MIP: Transbond MIP (3M Unitek);
- Self-etching primer: Transbond plus (3M Unitek).

Teeth

One hundred twenty human upper premolars (extracted for orthodontic purpose) were collected and stored in a solution of 0.1% (wt/vol) thymol to prevent dehydration and bacterial growth. The criterion for selection included intact buccal enamel.

The teeth were fixed in a self-cure acrylic block (35 × 9 × 9 mm) such that the roots were completely embedded in the acrylic up to the cementoenamel junction. The blocks were color coded, and the samples were then segregated into six groups of 20 samples each:

- Groups bonded without salivary contamination (1, 2, and 3);
- Groups bonded with salivary contamination (4, 5, and 6).

Brackets

Upper premolar brackets (Gemini series, 80-gauge mesh, Unitek) were used.

Bonding procedure

Groups bonded without contamination underwent the following:

- Oil-free prophylaxis;
- Etching with 37% H_3PO_4 for 15 seconds;[3,5,10]
- Primer application.

For group 3, self-etch primer was rubbed onto the enamel surface for three seconds according to the manufacturer's instructions. (Etching and priming were combined into a single step.) On air thinning for 15 seconds, the surface appeared glossy.

Groups bonded with contamination underwent the following:

- Oil-free prophylaxis;
- Etching with 37% H_3PO_4 for 15 seconds.[3,5] For group 6, self-etch primer was rubbed onto the enamel surface for three seconds.
- Saliva contamination: Natural saliva was collected from the operator within an hour after brushing, without any food consumed in-between. Two coats of saliva were applied to the etched enamel surface, and the excess was blotted, leaving the surface moist.
- Primer application: Except for group 6 because etching and priming were done in one step before moisture contamination.

Brackets were then bonded with Transbond XT and cured for 40 seconds (10 seconds each on the mesial, distal, occlusal, and gingival margins of the bracket) using a Q-lux visible light–curing unit (intensity 480 nm). Bonded specimens were stored in distilled water at room temperature for 24 hours before testing.

Bond strength testing

A Lloyd Universal testing machine (Model No. LR 100K) was used to test shear bond strength. The specimen mounted in its acrylic block was secured to the lower grip of the machine (fixed head). To maintain a consistent debonding force, a custom-made blade was fixed in the upper grip (movable head) connected to the load cell. The blade was positioned in such a way that it touched the bracket.

A cross-head speed of one mm/minute was used. The computer recorded the force to debond the bracket in Newtons. The bond strength was calculated in megapascals using the formula

$$\text{bond strength mPa} = \frac{\text{force in Newtons}}{\text{surface area of brackets in mm}^2}.$$

The surface area of the bracket was 10.61 mm^2, as given by the manufacturer. The debonded specimens were screened using a stereomicroscope under 40× magnification, and scoring was done using the modified adhesive remnant index (ARI).[11] Photographs were taken using a Leica optical microscope.

Statistical analysis

The mean and standard deviations of the shear bond strengths were subjected to a one-way analysis of variance (ANOVA). Multiple-range test by Tukey–honestly significant difference procedure was used to identify the significant groups at the 5% level.

The Kaplan-Meier survival analysis for shear bond strength was done. A log-rank test was used to calculate the overall *P* value for the test of equality of survival distributions among different groups and to identify the significant groups at the 5% level after adjusting for multiple comparisons using the Bonferroni-correction method.

The ARI scores were subjected to Kruskal-Wallis one-way ANOVA to calculate the *P* value. Mann-Whitney *U*-

TABLE 1. Students 't' Test For Shear Bond Strength

	Shear Strength		Comparison of Groups	*t* Value	*P* Value
	Mean	SD			
1. Conventional primer (dry)	9.54	3.86	1 vs 2	0.29	.78 (ns)
			1 vs 3	1.51	.14 (ns)
			1 vs 4	4.38	$<$.001
			1 vs 5	0.49	.63 (ns)
			1 vs 6	1.23	.23 (ns)
2. MIP (dry)	9.27	1.71	2 vs 3	2.65	$<$.01
			2 vs 4	5.79	$<$.01
			2 vs 5	0.35	.73 (ns)
			2 vs 6	2.29	$<$.05
3. Self-etch primer (dry)	11.104	2.56	3 vs 4	7.13	$<$.001
			3 vs 5	2.8	$<$.01
			3 vs 6	0.39	.70 (ns)
4. Conventional primer (contaminated)	4.69	3.10	4 vs 5	5.31	$<$.001
			4 vs 6	6.94	$<$.001
5. MIP (contaminated)	9.07	1.99	5 vs 6	2.46	$<$.05
6. Self-etch primer (contaminated)	10.79	2.43		$F = 14.51$, $P < .001$	

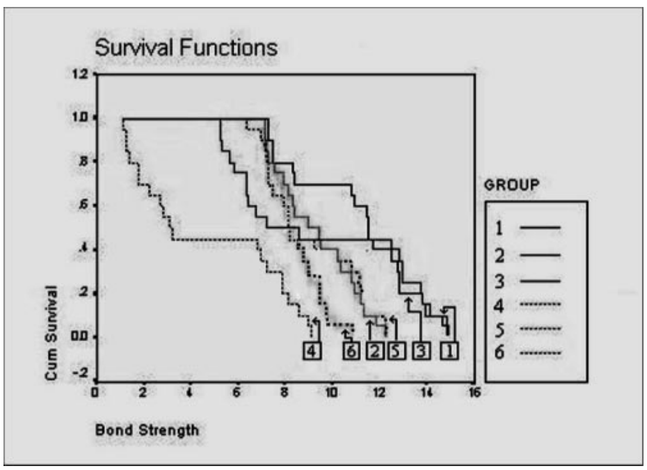

FIGURE 1. Kaplan-Meier Survival Plots

test was used to identify the significant groups after adjusting for multiple comparison tests by Bonferroni-correction method.

RESULTS

Under dry conditions, self-etch primer showed the highest shear bond strength, followed by conventional primer and MIP (Table 1). Under contaminated conditions, MIP showed the highest bond strength, followed by self-etch primer and conventional primer. All groups showed bond strengths significantly higher than that of group 4. Group 3 was significantly superior to group 6, but no other contrasts were statistically significant.

The Kaplan-Meier survival plots showed that the survival curve of group 4 was significantly different from those of all the other groups (Figure 1). Median bond strengths of all the groups were significantly greater than that of group 4. In addition, median bond strength of group 3 was significantly greater than those of groups 2, 5, and 6.

TABLE 2. Mean SD and Test of Significance of Mean ARI Scores Between Different Study Groups

Group	Mean	SD	*P* Value	Significant Groups
1	2.40	1.47	$<$.0001	1 vs 4 ($P < .0001$)
2	2.90	1.07		2 vs 4 ($P < .0001$)
3	2.85	0.93		3 vs 4 ($P < .0001$)
4	4.85	0.37		4 vs 5 ($P < .0001$)
5	3.30	0.92		4 vs 6 ($P < .0001$)
6	2.05	1.36		

When the ARI scores were evaluated, there was a significant association between ARI scores of different groups (Table 2). Group 4 showed significantly higher scores than all other groups; however, no other contrasts were significant.

DISCUSSION

Although the direct bonding of orthodontic brackets has dramatically improved the clinical practice of orthodontics, moisture contamination still poses a problem, especially while bonding posterior teeth and in cases of surgically exposed teeth.

Although traditional Bisgma resins are hydrophobic and are not efficient in a wet environment, MIPs have been used and found to offer comparable strength under both dry and wet conditions.[4,6]

These primers are adaptations of dentin-bonding agents, which have hydrophilic components such as HEMA, which allows a lower contact angle and an extension of the molecule, which readily bonds to the resin composite. It is even more effective when dissolved in acetone solvent.[7] Although enamel has a lesser organic content than dentin, the same principle is successful.[4,6,12]

Self-etch primers, which are relatively new entrants into the orthodontic bonding scene, offer a distinct clinical advantage because they combine etching and priming in a single stage. The reactive components are esters from bivalent alcohols with methacrylic and phosphoric acid or its derivatives. The phosphate residue is to etch the enamel, whereas the methacrylate component of the molecule is available for copolymerization with the bonding agent and composite resin. There is no need to rinse off reaction products or residual phosphoric acid esters because both are subsequently polymerized into the bonding layer. However, there are conflicting reports with reference to their bond strength.[13–15]

Although these primers are purported to be hydrophilic, they have not been specifically recommended for use in a moisture-contaminated environment. Therefore, this study compared the bond strengths of conventional primer, MIP, and self-etch primer under both dry and contaminated conditions.

The study was designed according to the recommendations of Fox et al.[16] Each group consisted of 20 samples (premolars were used). After bonding, the samples were stored in distilled water for 24 hours and then tested in a Lloyd machine at a cross-head speed of one mm/minute. In addition to the ANOVA, which considers only the mean bond strengths, the Kaplan-Meier survival analysis was done because it considers the entire spread of data and thus helps evaluate clinical performance better.[17]

The results of the ANOVA showed that under dry conditions, the bond strengths of all the primers were comparable. Under contaminated conditions, both self-etch primer and MIP showed acceptable and comparable bond strengths; however, the bond strength displayed by conventional primer was not adequate.[1] There was a significant difference in the bond strength displayed by self-etch primer under wet and dry conditions. The Kaplan-Meier survival distribution curves showed that group 4 was significantly different from all the other groups.

Under dry conditions, self-etch primer showed a performance significantly superior to that of MIP. Self-etch primer displayed adequate bond strength under both dry and contaminated conditions; however, there was a significant difference while comparing the median bond strengths under dry and wet conditions. Prati et al,[18] who studied bond strength of self-etch primers in dentin, reported that they are superior to conventional primers despite their limited resin-infiltrated dentin layer thickness (RIDL). Other studies using self-etch primers on enamel showed that they offered clinically adequate bond strength but are inferior to conventional primers.[13,14]

However, these studies compared these materials only under dry conditions and not on wet or contaminated enamel. The results of this study proved that self-etch primers offered adequate bond strength under both dry and contaminated conditions. However, the bond strength was significantly reduced in the presence of contamination.

MIP showed promising results when used under moist conditions while considering the median values of the Kaplan-Meier analysis. This is in contrast to the observations of Littlewood et al,[7] who found the results disappointing on applying the Weibull analysis. Newman et al[19] reported that adhesion promoters containing pyromellitic glycerol dimethacrylate and HEMA and other acrylates can enhance bond strength and promote bonding under slightly moist conditions.

Evaluation of ARI scores carried out half an hour to one hour after debonding showed that there was a statistically significant difference between conventional primer under contaminated conditions and all other groups. All primers displayed bond failure within the adhesive or at the adhesive-bracket interface, the only exception being conventional primer under contaminated conditions, which showed bond failure at the enamel-adhesive interface with no composite left behind on the tooth surface. In view of the low bond strength, this reflects an inability of the resin to flow into the saliva-contaminated enamel surface and does not reflect debonding at the enamel-adhesive interface and loss of enamel.

CONCLUSIONS

Conventional primer does not offer adequate bond strength under conditions of moisture contamination. Conventional primer does offer adequate and comparable bond strength to self-etch primer and MIP under dry conditions and, therefore, may be more cost-effective. MIP and self-etching primer offered adequate bond strength under contaminated conditions also. Self-etch primer displayed considerably superior performance under dry compared with contaminated conditions.

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