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ORIGINAL ARTICLE

Effect of an acidic primer on shear bond strength of orthodontic brackets

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A unique characteristic of some new etching systems is that they combine the conditioning and priming agents into a single acidic primer solution. The purpose of this study was to determine the effects on the shear bond strength and the bracket/adhesive failure mode when an acidic primer and other enamel etchants were used to condition the enamel surface before bonding. The brackets were bonded to extracted human teeth according to one of four protocols following the manufacturers instructions. Group I, teeth were etched with 37% phosphoric acid, the brackets were then bonded with System 1+ adhesive (Ormco Corporation. Orange, Calif.); group II, teeth were etched with 10% maleic acid, the brackets were also bonded with System 1+ adhesive; group III, an acidic primer that contains both the acid (phenyl-P) and the primer (hema and dimethacrylate) were placed on the enamel for 30 seconds. The adhesive used on this group was a lightly filled resin that contains Bis-GMA and HEMA. (Clearfil Liner Bond 2. J.C. Moritta, Kuraway, Japan); Group IV, the same acidic primer was used as in group III, the adhesive used was highly filled (Panavia 21. J.C. Moritta) and contains Bis-GMA. The present in vitro findings indicated that the use of acidic primers to bond orthodontic brackets to the enamel surface could provide clinically acceptable shear bond forces ($\bar{x} = 10.4 \pm 4.4$ MPa) when used with a highly (77%) filled adhesive (Panavia 21). These debonding forces were comparable to those obtained when the enamel was conditioned with either Phosphoric ($\bar{x} = 11.8 \pm 4.1$ MPa) or Maleic ($\bar{x} = 10.9 \pm 4.4$ MPa) acids. With the use of a lightly (10%) filled adhesive (Clearfil Liner Bond 2), the shear bond strength was significantly lower ($\bar{x} = 5.9 \pm 5.6$ MPa). It is of interest to note that there was a tendency to have less residual adhesive remaining on the tooth when an acid primer was used than when phosphoric and maleic acids were used. This might be of advantage to the clinician because it will require less time to clean the teeth after debonding. (Am J Orthod Dentofacial Orthop 1998; 114:243-7.)

Conventional adhesive systems use three different agents, an enamel conditioner, a primer solution, and an adhesive resin, in the process of bonding orthodontic brackets to enamel. A unique characteristic of some new etching systems is that they combine the conditioning and priming agents into a single acidic primer solution, for simultaneous use on both enamel and dentin.[1,2] Combining conditioning and priming into a single treatment step results in improvement in both time and cost-effectiveness to the clinician and indirectly to the patient.

These new systems were used originally on dentin.[1,3] Theoretically, the acidic part of the primer (phenyl P) dissolves the smear layer and incorporates it into the mixture. Acidic primer solutions also demineralize the dentin and encapsulate the collagen fibers and hydroxyapatite crys-

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tals.[2] This simultaneous conditioning and priming allows penetration of the monomer into the dentin. The adhesive resin component will then diffuse into the primed dentin, producing a "hybrid layer."[3]

In a 1997 in vitro study, Gordan[4] investigated whether acidic primer solutions allowed for the elimination of the etching step when bonding to dentin and enamel. It was found that the mean bond strength for Clearfil Liner Bond 2 (J.C. Moritta, Kuraway, Japan) with the use of an acidic primer was 20.4 ± 3.4 MPa and with the Denthesive II (Heraeus Kulzer, South Bend, Ind.) was 5.4 ± 1.6 MPa. The results suggested that for restorative purposes, Clearfil Liner Bond 2 could be used with an acidic primer, but when using Denthesive II bonding system, traditional enamel etching with phosphoric acid was needed to provide adequate bond strength.

In the early 90s, maleic acid was introduced as an alternative etching material in an attempt to control the depth of the enamel etch. Barkmeier and Erickson[5] compared the use of 10% maleic acid to 37% phosphoric acid and reported that the resulting bond strengths were essentially similar, 38.0 ± 6.5 and 38.3 ± 8.0 MPa, respectively. Triolo et al.[6] concluded that bond strengths when using maleic acid were time dependent, in other words, the longer the etching time, the greater the bond strengths. Scanning electron microscopy of the enamel surface treated with 10% maleic acid and 37% phosphoric acid revealed a similar morphologic pattern but the depth of the etched surface was significantly less with maleic acid.[6]

Orthodontists use the acid-etch bonding technique as a primary means of attaching brackets to the enamel surface. Maintaining a sound unblemished enamel surface after debonding orthodontic brackets is a primary concern to the clinician. As a result, bond failure at the bracket-adhesive interface or within the adhesive is more desirable (safer) than at the adhesive-enamel interface, because enamel fracture and crazing have been reported at the time of bracket debonding.[7] It is possible that the depth of the etched enamel surface created by phosphoric acid may be a factor contributing to the incidence of enamel fracture.[8-10] Therefore, alternative enamel conditioners, such as maleic acid, and the newly introduced acidic primers that contain phenyl P may be beneficial, if they can maintain a clinically useful orthodontic bracket bond strength while decreasing the depth of enamel dissolution.

The purpose of this study was to determine the effects on the shear bond strength and the bracket/adhesive failure mode when using acidic primer and other enamel etchants to condition the enamel surface before bonding.

MATERIAL AND METHODS

Teeth

Forty-eight freshly extracted human molars were collected and stored in a solution of 0.1% (weight/volume) thymol. The criteria for tooth selection included: intact buccal enamel not subjected to any pretreatment chemical agents, e.g., hydrogen peroxide, with no cracks from the presence of the extraction forceps, and no caries. The teeth were cleansed and then polished with pumice and rubber prophylactic cups for 10 seconds.

Brackets

Orthodontic metal brackets (Victory Series. 3M Unitek, Monrovia, Calif.) were used in this study. The average bracket base surface area was determined to be 12.2 mm.[2]

Bonding procedure

The brackets were bonded to the teeth according to one of four protocols following the manufacturers instructions.

- Group I. Teeth were etched with 37% phosphoric acid. The brackets were then bonded with System 1+ adhesive (Ormco Corporation. Orange, Calif.). System 1+ has two components: an activator primer liquid placed on the tooth and the adhesive paste placed on the bracket.
- Group II. Teeth etched with 10% maleic acid. The brackets were also bonded with System 1+ adhesive.
- Group III. An acidic primer that contains both the acid (phenyl-P) and the primer (hema and dimethacrylate) is placed on the enamel for 30 seconds. The adhesive used on this group was a lightly filled resin that contains Bis-GMA and HEMA. (Clearfil Liner Bond 2. J.C. Moritta, Kuraway, Japan).
- Group IV. The same acidic primer was used as in group III. The adhesive used was highly filled (Panavia 21. J.C. Moritta) and contains Bis-GMA.

Each bracket was subjected to a 300 gm compressive force after which excess bonding resin was removed with a small scaler.

Debonding procedure

The teeth were embedded in acrylic in phenolic rings (Buehler, Ltd., Lake Bluff, Ill.). A mounting jig was used to align the facial surface of the tooth to be perpendicular with the bottom of the mold. All samples were stored in deionized water at 37° C for 48 hours. Each tooth was oriented with the testing device as a guide, so its labial surface was parallel to the force during the shear strength test. A steel rod with one flattened end was attached to the crosshead of a Zwick test machine (Zwick Gm bH & Co., Ulm, Germany). An occlusogingival load was applied to the bracket producing a shear force at the bracket-tooth interface. A computer electronically connected with the Zwick

Table I. Descriptive statistics and results of the analysis of variance comparing the shear bond strength in mega pascals of the four groups evaluated

Groups tested	$\bar{x}$	SD	Range	p
System I				
Phosphoric	11.8	4.1	6.4-19.1	A
Maleic	10.9	4.4	3.3-17.1	A
Acid Primer				
Clearfil	5.9	5.6	1.1-16.4	B
Panavia	10.4	4.4	3.0-16.1	A

F-value = 4.32, p = 0.0091.
$\bar{x}$ = mean, SD = standard deviation.
p = Probability; groups with the same letter are not significantly different from each other.

Table II. Frequency distribution and the results of the chi-squared analysis of the Adhesive Remnant Index of the four groups evaluated

Groups tested	ARI Scores*				
	1	2	3	4	5
System I					
Phosphoric	—	5	6	1	—
Maleic	—	4	8	—	—
Acid Primer					
Clearfil	—	—	4	10	1
Panavia	—	—	4	6	1

χ^2 = 26.82, p = 0.001
*1, all composite on tooth; 2, >90% of composite on tooth; 3, <10% but >90% of composite remains on tooth; 4, <10% composite on tooth; 5, no composite remains on tooth.

test machine recorded the results of each test. Shear bond strengths were measured at a crosshead speed of 5 mm/min.

Residual adhesive

After debonding, the teeth and brackets were examined under ×10 magnification. Any adhesive remaining after bracket removal was assessed according to the Adhesive Remnant Index (ARI) and scored with respect to the amount of resin material adhering to the enamel surface.[11] The ARI scale has a range between 5 and 1, with 5 indicating that no composite remained on the enamel; 4, less than 10% of composite remained on the tooth surface; 3, more than 10% but less than 90% of the composite remained on the tooth; 2, more than 90% of the composite remained; and 1, all of the composite remained on the tooth, as well as the impression of the bracket base. The teeth were scored by two investigators to determine as well as agree on the extent of the adhesive remaining on the bracket and/or the tooth. The ARI scores were also used as a more complex means of defining the site of bond failures between the enamel, the adhesive, and the bracket base.

Statistical analysis

Descriptive statistics including the mean, standard deviation, minimum and maximum values were calculated, for each of four groups of teeth tested.

The analysis of variance was used to determine if significant differences were present in bond strength, followed by a Duncan's Multiple Range Test to identify which of the groups were different. The chi-squared test was also used to determine significant differences in the ARI scores between the different groups. For the purpose of the statistical analysis groups 1 and 2 were combined, as were groups 4 and 5 when appropriate. Significance for all statistical tests was predetermined at $p \leq 0.05$.

RESULTS

Shear bond strength comparisons

The descriptive statistics for the shear bond strengths of the various groups tested are presented in Table I.

The results of the analysis of variance indicated that the shear bond strength when using Clearfil Liner Bond with an acidic primer was significantly lower ($\bar{x}$ = 5.9 ± 5.6 MPa) than when using Panavia 21 with the same acidic primer ($\bar{x}$ = 10.4 ± 4.4 MPa) or when using System 1+ with either phosphoric acid ($\bar{x}$ = 11.8 ± 4.1 MPa) or maleic acid ($\bar{x}$ = 10.9 ± 4.4 MPa) enamel etchants.

Adhesive remnant index (ARI) comparisons (Table II)

The results of the chi-squared comparisons indicated that there was a significant difference (p = 0.001) between the bonding systems when using the acidic primers as compared with the scores obtained when the teeth were etched with either phosphoric or maleic acids. With the use of the acidic primers there is a higher frequency of ARI scores of 3 and 4; this indicates less composite is remaining on the tooth than when phosphoric and maleic acids are used as enamel etchants with a resultant ARI scores of 2 and 3. With any of the systems tested, there were no ARI score of 1 and very few of 5 (Table II).

DISCUSSION

The direct bonding of orthodontic brackets has revolutionized and improved the clinical practice of orthodontics. However, there is a need to improve our ability to maintain a clinically useful bond strength while minimizing the damage to the tooth during debonding. Traditionally, the use of acid

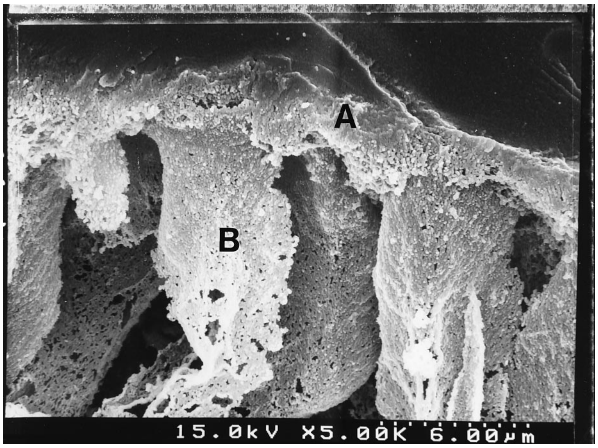

Fig. 1. Photomicrograph (SEM) of adhesive penetration into enamel surface etched with maleic or phosphoric acids. Resin tags are thick and uniform penetrating into the etched enamel. **A,** Adhesive resin; **B,** adhesive resin tag. (Original magnification ×5000.)

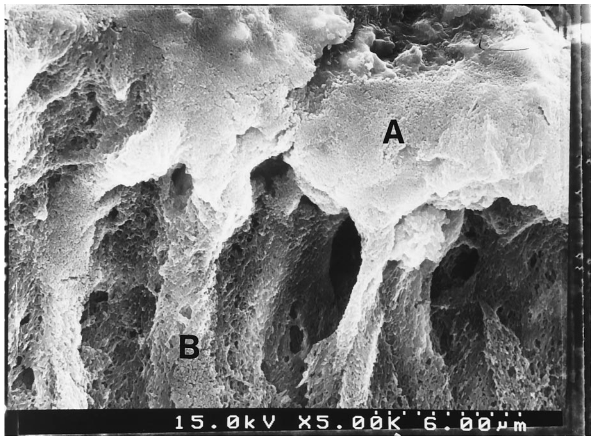

Fig. 2. Photomicrograph (SEM) of adhesive penetration into enamel surface etched with the acidic primer. Resin tags penetrating into etched surface are thinner and less uniform than in Fig. 1. **A,** Adhesive resin; **B,** Adhesive resin tag. (Original magnification ×5000.)

etchants followed by primer materials was an essential part of the bonding procedure in order to allow good wetting and penetration of the adhesive that will bond the bracket to the enamel surface. Recently, 10% maleic acid was used as a conditioner to etch both enamel and dentin in an attempt to control the depth of the etch.[5,6,12]

The use of the new acidic or self-etching primers for orthodontic purposes has not been evaluated. These new primers are thought to simplify the clinical handling of adhesive systems by combining the etching step with primer application in one mix.[1,3] The present study compared two bonding systems, Clearfil Liner Bond 2 and Panavia 21 that use an acidic primer with a conventional orthodontic adhesive, System 1+ used with either phosphoric acid or maleic acid as enamel etchants.

The present findings indicated that the use of acidic primers to bond orthodontic brackets to the enamel surface can provide clinically acceptable shear bond forces ($\bar{x} = 10.4 \pm 4.4$ MPa) when used with a highly (77%) filled adhesive (Panavia 21). These debonding forces were comparable to those obtained when the enamel was conditioned with either phosphoric ($\bar{x} = 11.8 \pm 4.1$ MPa) or maleic ($\bar{x} = 10.9 \pm 4.4$ MPa) acids. With the use of a lightly (10%) filled adhesive (Clearfil Liner Bond 2), the shear bond strength was significantly lower ($\bar{x} = 5.9 \pm 5.6$ MPa). It needs to be remembered that this is an in vitro study and care should be taken when interpreting the results without simulating the oral environment.

It is of interest to note that there was a tendency to have less residual adhesive remaining on the tooth when an acidic primer was used (ARI scores of 4 and 5) than when using phosphoric and maleic acids (ARI scores of 2 and 3). This might be of advantage to the clinician because it will require less time to clean the teeth after debonding, in addition, to the fact that the enamel etchant and the primer are placed in one step.

The change in the bracket-adhesive-enamel failure modes when an acidic primer is used might be explained by examining scanning electron micrographs of the enamel surfaces. In Fig. 1, one can observe that the resin tags with a phosphonic acid etch are thick and uniform. On the other hand, when using an acidic primer as illustrated in Fig. 2, the resin tags are thin and less uniform, which is conducive to a weaker bond, hence less adhesive will remain on the tooth at the time of debonding.

More research is needed to determine which adhesive-acidic primer combinations are most suitable for orthodontic purposes.

CONCLUSION

Acidic primers containing both the enamel etchant and primer, can be successfully used in bonding orthodontic brackets. The type of adhesive used (highly filled versus lightly filled) may have a significant effect on the shear bond strength, with the highly filled adhesives providing more clinically acceptable bond strength. In addition, the use of acidic primers decreased the amount of residual adhesive left on the enamel surface after debonding.

REFERENCES

1. Chigira H, Koike T, Hasegawa T, Itoh K, Wakumoto S, Hyakawa T. Effect of the self etching dentin primers on the bonding efficacy of dentine adhesive. Dent Mater J 1989;8:86-92.
2. Nishida K, Yamauchi J, Wada T, Hosoda H. Development of a new bonding system [Abstract 267]. J Dent Res 1993;72:137.
3. Nakabayashi, N. Dentinal bonding mechanisms. Quint Int 1991;22:73-4.
4. Gordan VV. Acidic primers in dentin and enamel: shear bond strength and microleakage [Master Thesis]. University of Iowa, 1997.
5. Barkmeier WW, Erickson RL. Shear bond strength of composite to enamel and dentin using Scotchbond multi-purpose. Am J Dent 1994;7:175-9.
6. Triolo PT Jr, Swift EJ Jr, Mudgil A, Levine A. Effects of etching time on enamel bond strengths. Am J Dent 1993;6:302-4.
7. Britton JC, McInnes P, Weinberg R, Ledoux WR, Retief DH. Shear bond strength of ceramic orthodontic brackets to enamel. Am J Orthod Dentofacial Orthop 1990;98:348-53.
8. Harris AM, Joseph VP, Rossouw PE. Shear peel bond strengths of esthetic orthodontic brackets. Am J Orthod Dentofacial Orthop 1992;102:215-9.
9. Chaconas SJ, Caputo AA, Niu SL. Bond strength of ceramic brackets with various bonding agents. J Dent Res 1989;68:945.
10. Guess MV, Watanabe LG, Beck FM, Crall MG. The effect of silane coupling agents on the bond strength of a polycrystalline ceramic bracket. J Clin Orthod 1988;22:788-92.
11. Oliver RG. The effect of different methods of bracket removal on the amount of residual adhesive. Am J Orthod Dentofacial Orthop 1988;93:196-200.
12. Olsen ME, Bishara SE, Damon P, Jakobsen JR. Evaluation of Scotchbond multi-purpose and maleic acid as alternative methods of bonding orthodontic brackets. Am J Orthod Dentofacial Orthop 1997;111:498-501.