

Effect of Self-Etching Primers on Bond Strength— Are They Reliable?

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Abstract: Currently introduced self-etching primers combine conditioning and priming agents into a single product. The purpose of this study was to determine the effects of using three self-etching primers on the shear bond strength (SBS) of orthodontic brackets and on the bracket/adhesive failure mode. Brackets were bonded to extracted human teeth according to one of four protocols. In the control group, teeth were etched with 37% phosphoric acid. In the experimental groups, the enamel was conditioned with three different self-etching primers, Clearfil SE Bond (CSE), Etch & Prime 3.0 (EP3), or Transbond Plus (TBP), as suggested by the manufacturer. The brackets were then bonded with Transbond XT in all groups. The present in vitro findings indicate that conditioning with TBP before bonding orthodontic brackets to the enamel surface resulted in a significantly ($P \leq .001$) higher SBS (mean, 16.0 ± 4.5 MPa) than that found in CSE, EP3, and the control (acid-etched [AE]) groups. CSE produced bond strength values (mean 11.5 ± 3.3 MPa) that are statistically comparable to those produced by acid etching (mean 13.1 ± 3.1 MPa). The use of EP3 for enamel conditioning resulted in the lowest mean SBS value (mean 9.9 ± 4.0 MPa). A comparison of the adhesive remnant index scores indicated that there was more residual adhesive remaining on the teeth that were treated with conventional acid etching than in the CSE and EP3 groups. In the TBP group, the failure sites were similar to those of the AE group but different from those of the CSE group. (*Angle Orthod* 2003;73:64–70.)

Key Words: Self-etching primers; Bond strength; SEM examination

INTRODUCTION

Many researchers have studied adhesion to enamel.¹ Although different modalities have been tested,^{2–4} at present, phosphoric acid etching seems to be the most frequently used method of enamel surface preparation. One of the potential disadvantages of etching with phosphoric acid is that the acid causes demineralization of the most superficial layer.^{5–8} To control excessive enamel loss, maleic and polyacrylic acids have been used as alternatives for phosphoric acid. The use of polyacrylic acid has been found to result in a reduction in bond strength.^{9–11}

A unique characteristic of some new bonding systems in operative dentistry is that they combine conditioning and priming agents into a single acidic primer solution for simultaneous use on both enamel and dentin.^{12,13} Combining con-

ditioning and priming into a single treatment step results in improvement in both time and cost-effectiveness for clinicians as well as for patients. In a self-etching primer, the active ingredient is a methacrylated phosphoric acid ester. The phosphoric acid and the methacrylate group are combined into a molecule that etches and primes at the same time. The phosphate group on the methacrylated phosphoric acid ester dissolves the calcium and removes it from the hydroxyapatite. But rather than being rinsed away, the calcium forms a complex with the phosphate group and gets incorporated into the network when the primer polymerizes. Agitating the primer on the tooth surface serves to ensure that fresh primer is transported to the enamel surface. Etching and monomer penetration to the exposed enamel rods are simultaneous. In this manner, the depth of the etch is identical to that of the primer penetration. Three mechanisms act to stop the etching process. First, the acid groups attached to the etching monomer are neutralized in a similar way, as is phosphoric acid, by forming a complex with the calcium from the hydroxyapatite. Second, as the solvent is driven from the primer during the airburst step, the viscosity rises, slowing the transport of acid groups to the enamel interface. Finally, as the primer is light cured and the primer monomers are polymerized, transport of acid groups to the interface is stopped.¹⁴

These new systems were also found to be effective when bonding the brackets to the enamel. In recent laboratory

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TABLE 1. Principle Ingredients of the Three Self-Etching Primers as Provided by the Manufacturers

Groups Tested	Primer ^a /Catalyst♣	Bond ^a /Universal♣
Transbond Plus	<ul style="list-style-type: none">• Water• Methacrylated phosphoric acid esters• Phosphine oxide• Stabilizer• Fluoride complex• Parabenes	
Clearfil SE Bond ^a	<ul style="list-style-type: none">• 10-Methacryloyloxydecyl dihydrogen phosphate (MDP)• 2-Hydroxyethyl methacrylate (HEMA)• Hydrophylic dimethacrylate• Camphorquinone• N,N-Diethanol-<i>p</i>-toluidine• Water	<ul style="list-style-type: none">• MDP• Bis-phenol A diglycidylmethacrylate (Bis-GMA)• HEMA• Hydrophobic dimethacrylate• Camphorquinone• N,N-Diethanol-<i>p</i>-toluidine• Silanated colloidal silica
Etch & Prime 3.0♣	<ul style="list-style-type: none">• Pyro-phosphate• HEMA• Initiators• Stabilizers	<ul style="list-style-type: none">• HEMA• Ethanol• Distilled water• Stabilizer

^a Also known as Clearfil Mega Bond in Japan.

studies on acidic primers by Bishara et al,¹⁵⁻¹⁷ two different acidic primers for restorative dentistry and/or adhesives for restorative dentistry and orthodontics were tested. The results indicated that both self-etching primer systems resulted in significantly reduced bond strength values. It was suggested that more research was needed to determine whether currently available orthodontic adhesive systems could be used with an acidic primer.¹⁶

Recently, a new acidic primer (Transbond Plus, 3M Unitek, Monrovia, Calif) to be used for orthodontic purposes was introduced. The purpose of this study was to determine the efficacy of three different self-etching primers on the shear bond strength (SBS) of orthodontic brackets and the bracket/adhesive failure mode in comparison with conventional 37% phosphoric acid etching.

MATERIALS AND METHODS

Eighty noncarious human maxillary and mandibular premolars, extracted for orthodontic purposes, were used in this study. Teeth with hypoplastic areas, cracks, or gross irregularities of the enamel structure were excluded from the study. The teeth were stored in distilled water after extraction. The water was changed weekly to avoid bacterial growth. The sample was randomly divided into four groups of 20 each. Each tooth was mounted vertically in a self-cure acrylic so that the crown was exposed. The buccal enamel surfaces of the teeth were pumiced, washed, and dried before surface preparation.

Enamel surfaces were prepared, and the brackets were bonded to the teeth according to one of the following protocols:

Acid-etched group as the control

In the acid-etched (AE) group, 20 teeth were etched with 37% phosphoric acid gel (Email Preparator, Vivadent,

Liechtenstein) for 30 seconds. The teeth were thoroughly washed and dried. Liquid primer of the Transbond XT was applied with a brush tip before bracket bonding.

Clearfil SE Bond group

Clearfil SE Bond (CSE) is a light-cured bonding system that consists of a self-etching primer and a bonding agent. The primer offers simultaneous treatment of both dentine and enamel. In the CSE group, CSE group primer was applied for 20 seconds and dried with mild airflow. Then the bonding agent was applied, subjected to gentle airflow, and light cured for 10 seconds.

Etch & Prime 3.0 group

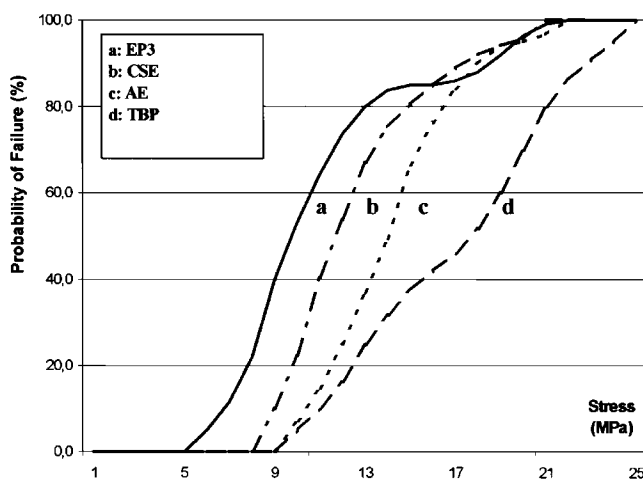
Etch & Prime 3.0 (EP3) is a light-cured, self-etching, acetone-free, two-component enamel and dentine adhesive material and is available in 6 mL of each of EP3 *Universal* and EP3 *Catalyst*. In the EP3 group, the enamel surface was air-dried. One drop each of *Universal* and *Catalyst* were mixed thoroughly in a dish. A generous quantity of primer was applied onto the enamel with a disposable brush and was allowed to act for 30 seconds. Excessive solvent was removed with air for five seconds, and the primer was cured for 10 seconds.

Transbond Plus group

The orthodontic self-etch primer Transbond Plus (TBP) is predosed so that it is used for single applications. For activation, the two components, ie, acid and the primer, were squeezed together, and the resulting mix was applied directly onto the tooth surface. The area to be etched was rubbed with the solvent for three seconds and was gently air-dried for two seconds. Table 1 describes the principle

TABLE 2. Descriptive Statistics and the Results of the Duncan's Multiple Range Test Comparing the Shear Bond Strengths (in MPa) of the Four Groups Tested

Groups Tested	N ^a	Mean	SD ^b	Range	Duncan Test ^c
Transbond Plus	20	16.0	4.5	8.8–23.5	A
Acid	20	13.1	3.1	8.2–20.4	B
SE Bond	20	11.5	3.3	8.0–19.6	BC
Etch & Prime 3.0	20	9.9	4.0	4.5–19.2	C

^a N, Sample size.^b SD, Standard deviation.^c Groups with different letters are significantly different from each other.**FIGURE 1.** Probability of failure of different conditioning methods at particular shear stress values.

ingredients of the self-etching primers as supplied by the manufacturers.

Bracket bonding

Eighty stainless steel premolar brackets (GAC International Inc., Central Islip, NY) with a base surface area of 12.6 mm² were used for this study. After surface preparation, the brackets were bonded on premolars with Transbond XT (3M Unitek), and any excess resin was removed with an explorer before the resin was polymerized. A conventional halogen light source (Ortholux XT, 3M Dental Products, St Paul, Minn) was used for curing for 20 seconds from each of mesial and distal sides. The total time required for preparing and bonding a single tooth in the groups AE, CSE, EP3, and TBP was 100, 70, 85, and 45 seconds, respectively. After complete sample preparation, all samples were kept in distilled water at 37°C for 24 hours for short-term storage.¹⁸

Before debonding, the embedded specimens were secured in a jig attached to the base plate of a universal testing machine (Model 500, Testometric, Lancashire, UK). A chisel-edge plunger was mounted in the movable crosshead

TABLE 3. Frequency Distribution of the ARI Scores of the Four Groups Tested^a

Groups Tested ^b	ARI Scores ^c					N ^d	Multiple Comparison		
	1	2	3	4	5		CSE	AE	TBP
EP3	—	1	4	5	10	20	**	***	
CSE	—	13	2	3	2	20		***	***
AE	17	2	—	1	—	20			***
TBP	15	3	2	—	—	20			ns

^a $\chi^2 = 84.999$; $P < .05$.^b AE indicates acid etched; ARI, adhesive remnant index; CSE, Clearfil SE Bond; EP3, Etch & Prime 3.0; ns, not significant; TBP, Transbond Plus.^c ARI Scores: 1 indicates all of the composite, with an impression of the bracket base, remained on the tooth; 2, more than 90% of the composite remained; 3, more than 10% but less than 90% of the composite remained on the tooth; 4, less than 10% of composite remained on the tooth surface; 5, no composite remained on the enamel.^d N: Sample size.** $P < .01$.*** $P < .001$.

of the testing machine and was positioned so that the leading edge aimed at the enamel-adhesive interface before being brought into contact at a crosshead speed of 0.5 mm/min. The force required to dislodge the brackets was measured in Newtons (N), and the SBS (1 MPa = 1 N/mm²) was then calculated by dividing the force values by the bracket base area (12.6 mm²).

After being debonded, the teeth and brackets were examined under 10× magnification. Any adhesive that remained after bracket removal was assessed and scored according to the modified adhesive remnant index (ARI).¹⁰

Scanning electron microscopic examination

For scanning electron microscopic (SEM) evaluation, premolars with brackets bonded after acid etching and TBP application were used. Two premolars from each group were sectioned longitudinally between the bracket wings to examine the differences in cured resin-etched enamel and cured resin-hybridized enamel in cross section. To evaluate the resin tag formation, half of these bracketed specimens were kept in 37% phosphoric acid solution until the tooth structure was totally dissolved to reveal the resin tags. For further investigation of the TBP effect on enamel, additional samples treated with TBP only were prepared by cracking the premolars longitudinally. The samples were dehydrated in ascending grades of alcohol, sputter-coated with 200-nm gold-palladium, and examined under a Jeol JSM 5200 (Tokyo, Japan) scanning electron microscope operated at 20–25 kV.

Statistical analysis

Descriptive statistics that included the mean, standard deviation, and minimum and maximum values were calculated.

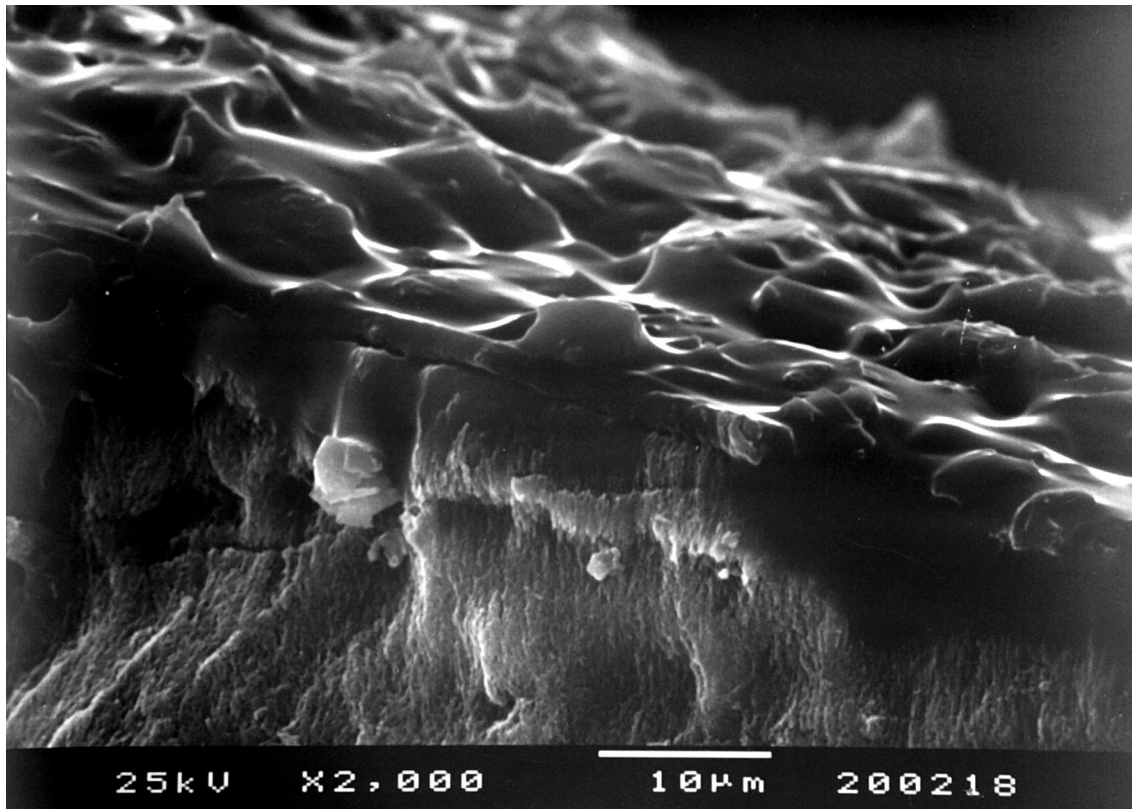


FIGURE 2. TBP-treated enamel in cross section and the outer surface of TBP layer on enamel. Magnification, 2000 \times .

ed for each of the four test groups. A one-way analysis of variance (ANOVA) was used to determine whether significant differences were present in the bond strengths among the four groups. When a statistically significant difference was present, a Duncan's multiple range test was used to identify which of the groups were different. The chi-square test was also used to determine significant differences in the ARI scores among the groups. Multiple comparisons of ARI scores were also performed. Significance for all statistical tests was predetermined at a probability value of .05 or less.

RESULTS

SBS comparisons

The descriptive statistics for the SBSs of the four groups tested are presented in Table 2. As can be seen from the table, the highest bond strength values were produced by the TBP group, followed by the AE, CSE, and EP3 groups. The ANOVA and Duncan's multiple range test revealed that TBP (16.0 ± 4.5 MPa), when compared with the other conditioning methods, yielded significantly higher bond strength values. CSE (11.5 ± 3.3 MPa) produced bond strength values that are statistically comparable to those of acid etching (13.1 ± 3.1 MPa). The use of EP3 for enamel conditioning resulted in the lowest mean SBS value (9.9 ± 4.0 MPa). The survival graphs (Figure 1) indicate that half

of the brackets remain bonded at 18 MPa in the TBP group, at 14 MPa in the control group, at 11 MPa in the SEB group, and at 8 MPa in the EP3 group. The survival curves of the last three groups are almost parallel to each other, which indicates a uniform decrease in the survival of brackets with increasing stresses. On the other hand, in the TBP group, the curve is less steep, and this indicates a lower correlation of bond failure with the increasing shear stress values.

ARI comparisons

The results of the chi-square comparisons indicated that there were significant differences between the four groups tested (Table 3) ($P \leq .001$). There was a greater frequency of ARI scores of 1 with the TBP and AE groups, which indicated that failures were mainly in the adhesive-bracket interface. Differences in ARI scores were not statistically different between the TBP and AE groups. In the CSE group, the failures were mostly cohesive within the resin (ARI score = 2, 3, and 4). Scores of the CSE group were statistically different from that of the AE and TBP groups. In the EP3 group, there was a higher frequency of ARI scores of 4 and 5, which indicated that less residual adhesive remained on the enamel than on the bracket base after debonding. The ARI scores of the EP3 group were statistically different from those of the other three groups.

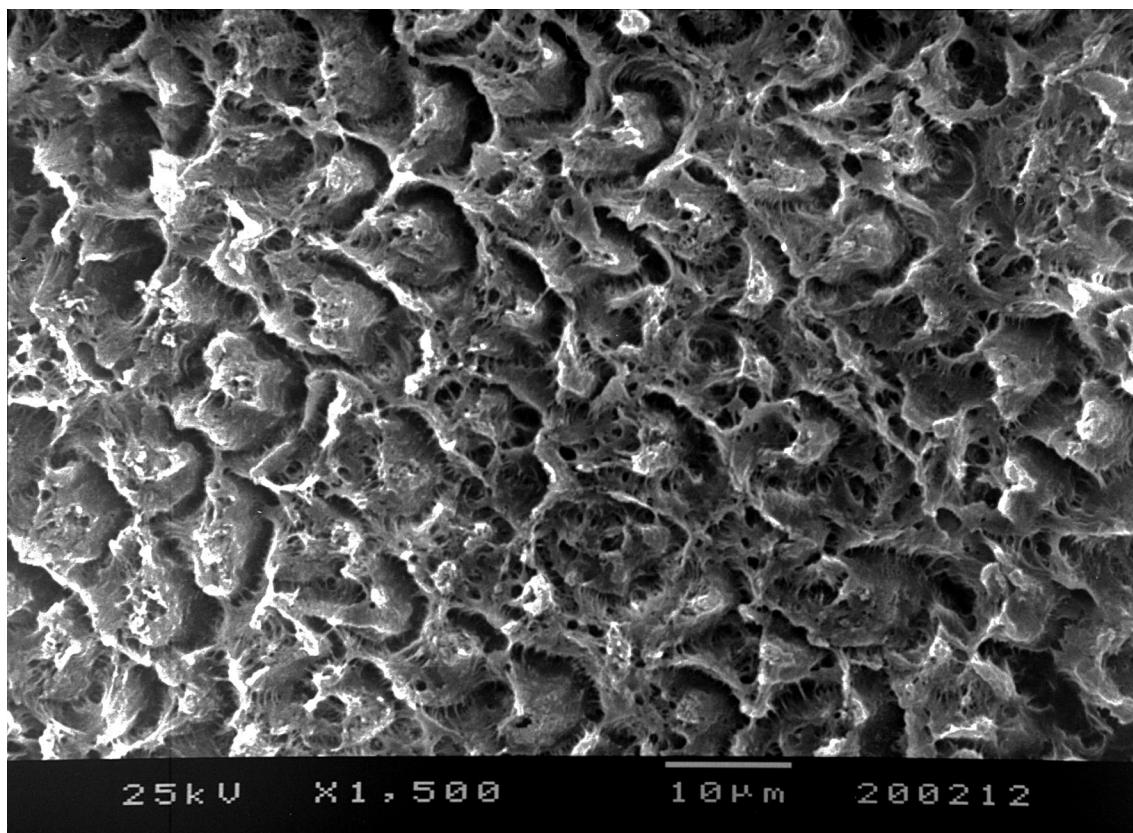


FIGURE 3. SEM view of the adhesive under the bracket base after complete removal of the AE enamel. Note the exact replica of the honeycomb appearance of phosphoric acid-etched enamel. Magnification, 1500 \times .

SEM examination

There were no distinct features in the enamel-resin interface to differentiate between the SEM micrographs of the cross-sections of the AE and TBP-treated enamels. Figure 2 shows a cross section from a TBP-treated enamel surface in which the outer enamel surface and TBP layer was not sectioned with the saw but was cracked. An irregular but smooth hybrid layer, 3–4 μm in thickness, covers the enamel prisms. Figures 3 and 4 show the adhesive under the bracket base after dissolution of the tooth structure in acid to reveal and compare the resin tag formation in the AE and TBP-treated enamels, respectively.

In the AE samples (Figure 3), a distinct honeycombed structure with micro- and macrotag formations is present. In the TBP-treated samples (Figure 4), an irregular tag formation with no apparent indentations of enamel prism or core material is present.

DISCUSSION

The present study evaluated the SBS values produced by three self-etching primers and compared them with those produced by the conventional acid-etching technique. The lowest SBS values were recorded in the two self-etching primer groups (CSE and EP3) designed to be used for re-

storative purposes. The CSE bonding system has a pH value of 2.0 and is comparatively less acidic than EP3 and TBP, which have pH values of 0.75 and 1.0, respectively. The efficacy of self-etching primers on unground enamel, however, does not depend upon their etching aggressiveness.²⁰ Even with the existing differences in the composition of the three self-etching primers, it is very difficult to find a correlation between the monomer structure and the bond strength.¹⁹

The low bond strength values produced by the aggressive self-etchant EP3 in our study were in accordance with the findings of Toledano et al.¹⁹ Contradictory to our results, on the other hand, Pashley and Tay,²⁰ comparing the acid-etching, CSE, and Prompt-L-Pop (PLP; restorative version of TBP) groups, found significantly lower microtensile bond strength values with the self-etching primers than with acid etching. But when the self-etching primers were rinsed and replaced with the control resin, the bond strengths in the PLP group were no longer significantly different from those produced by 32% phosphoric acid etching. In the same study, similar to our results, an aggressive etching effect and a thick hybrid layer formation after PLP application were reported.

Bishara et al.¹⁵ discussed the selective compatibility of early acidic primers with different adhesives and mentioned

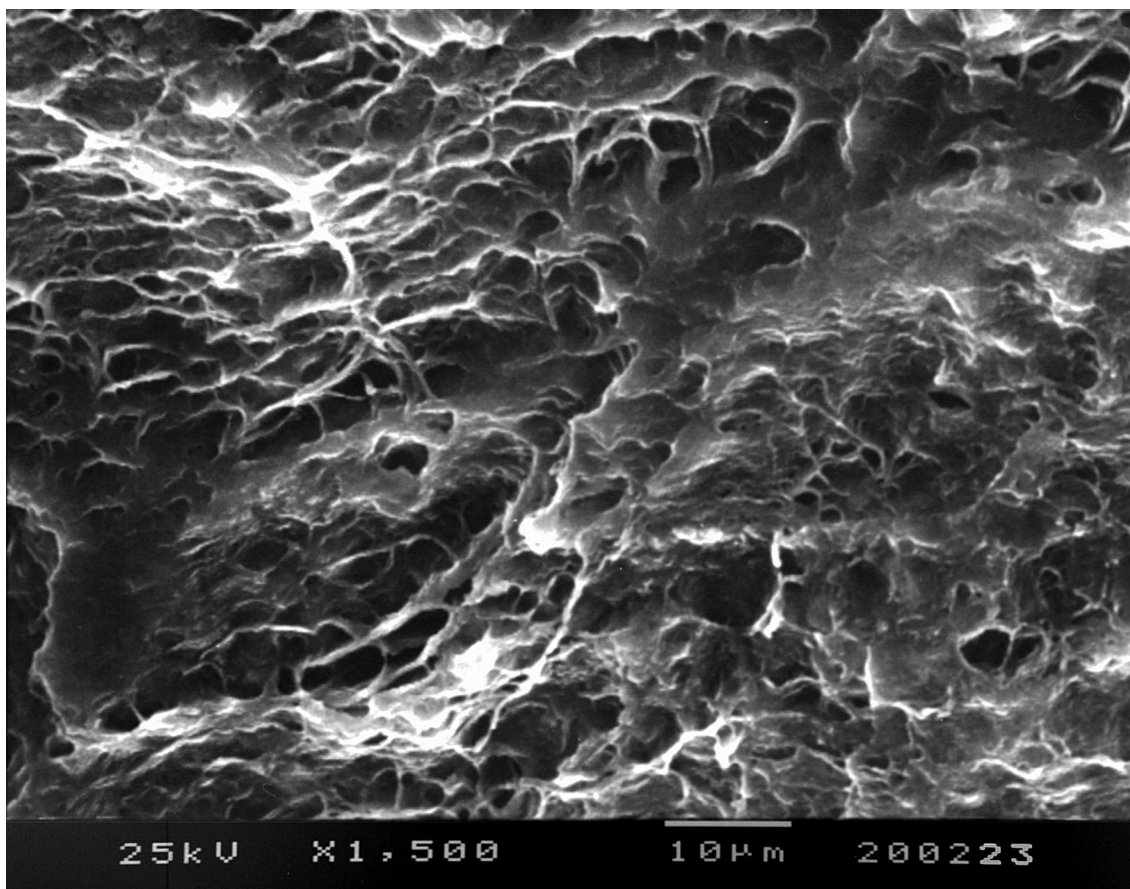


FIGURE 4. SEM view of the adhesive under the bracket base after complete removal of the TBP-treated enamel. Magnification, 1500 \times .

the need for acidic primers manufactured for orthodontic purposes. TBP, as the only orthodontic acidic primer in the market today, was superior to the other three conditioning methods both in terms of SBS and the time required for application.

It was interesting to observe that the average bond strength in the TBP group was significantly higher than that of the conventional AE technique. Self-etching primer produces a highly porous surface on aprismatic enamel. The etching effect approaches that of phosphoric acid despite the absence of additional gross retentive structures that are produced by differential dissolution of the underlying enamel prisms. One important advantage to simultaneous etching and priming is that the primer penetrates the entire depth of the etch, ensuring an excellent mechanical interlock.

SEM examination of the impressions of the AE (Figure 3) and TBP-treated (Figure 4) enamels showed different surface characteristics. Despite the lack of tag formation, high bond strengths obtained with TBP and the fact that more adhesive remained on enamel after debonding (ARI scores = 1 and 2) implicate the presence of different mechanisms that constitute adhesion. Conversely, several other investigators have concluded that there is no correlation be-

tween the bond strength and the tag length and that the adhesive strength of the resin to enamel is mainly attributable to the resin's ability to penetrate between the enamel crystallites and rods.^{21,22}

Bishara et al¹⁵ reported on tag formation but their results differed from the current study's observations. Using CSE, they found weaker bonds and lesser residual adhesive on enamel after debonding in the acidic primer group than in the phosphoric acid group. According to the authors,¹⁵ the thin and less uniform resin tags observed in the SEM photographs of acidic primer-treated enamel were conducive to poor adhesion. Because the acidic primer and the adhesives investigated are different, comparison of their observations with the current one is difficult.

In a recent study, Bishara et al¹⁷ evaluated the use of a newly developed restorative acidic primer (PLP) for bracket bonding. The findings indicated that the use of a self-etch primer produced lower but clinically acceptable SBS values. The low bond-strengths may partly be explained with the different application time of PLP. In the restorative version, the manufacturer recommends 15-seconds application times to remove the smear layer on dentin, whereas three seconds are sufficient for enamel to bond orthodontic attachments. Like the present study, this was an in vitro

study, and one must exercise care in applying the findings in clinical situations.

CLINICAL CONSIDERATIONS AND CONCLUSIONS

After our encouraging laboratory findings and having in mind all the shortcomings of an in vitro setting, we have bonded 218 attachments (brackets and tubes) on 12 patients since June 2001. To date only four attachments have failed (three mandibular second molars and one lower incisor).

The intermediate steps such as acid etch for 30–60 seconds, wash thoroughly, dry, and then prime are no longer necessary with self-etching primers. TBP is packaged for single-patient use to avoid contamination, is fast to apply, and is cost effective when used for full bonding.

For optimal bonding, we recommend the following procedure:

- Dry the tooth surface.
- Apply TBP. Rub it thoroughly for at least three seconds, and always wet the surface with new solution to ensure monomer penetration. The presence of water in the chemical composition of TBP may necessitate air-drying, but as the operator moves from one side to the other, the solvent evaporates and drying is no longer necessary.
- Bond the bracket with Transbond XT.

At the moment, we do not have enough data on the compatibility of TBP with orthodontic adhesives other than light-cured Transbond. But within the limitations of our laboratory and clinical findings, it is clear that the hybrid layer produced after TBP application on enamel is capable of bonding strongly to enamel surfaces and by no means reduces the bond strength of orthodontic brackets.

Self-etching primers are effective, but only future long-term clinical studies will prove their clinical reliability.

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REFERENCES

1. Driessens FC. Chemical adhesion in dentistry. *Int J Dent*. 1977; 27:317–323.
2. Zachrisson BU, Büyükyılmaz T. Recent advances in bonding to gold, amalgam and porcelain. *J Clin Orthod*. 1993;27:661–675.
3. Usumez S, Orhan M, Usumez A. Laser etching of enamel for direct bonding with an Er,Cr:YSGG hydrokinetic laser system. *Am J Orthod Dentofacial Orthop*. In press.
4. Canay Ş, Kocadereli İ, Akça E. The effect of enamel air abrasion on the retention of bonded metallic orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 2000;117:15–19.
5. Shannon IL. Caries risk in teeth with orthodontic bands: a review. *J Acad Gen Dent*. 1972;20:24–28.
6. Stratemann MW, Shannon IL. Control of decalcification in orthodontic patients by daily self-administered application of a water free 0.4 percent stannous fluoride gel. *Am J Orthod*. 1974;66:273–279.
7. Zachrisson BU. Cause and prevention of injuries to teeth and supporting structures during orthodontic treatment. *Am J Orthod*. 1976;69:285–300.
8. Boyd RL. Comparison of three self-applied topical fluoride preparations for control of decalcification. *Angle Orthod*. 1993;63:25–30.
9. Triolo PT Jr, Swift EJ Jr, Mudgil A, Levine A. Effects of etching time on enamel bond strengths. *Am J Dent*. 1993;6:302–304.
10. Olsen ME, Bishara SE, Damon P, Jakobsen JR. Evaluation of Scotch bond multi-purpose and maleic acid as alternative methods of bonding orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 1997;111:498–501.
11. Smith DC. A new dental cement. *Br Dent J*. 1968;125:381–94. Taken from *Quint Int*. 1990;21:41–45.
12. 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*. 1989;8:86–92.
13. Nishida K, Yamauchi J, Wada T, Hosoda H. Development of a new bonding system [abstract]. *J Dent Res*. 1993;72:137.
14. Cinader D. Chemical processes and performance comparisons of Transbond Plus self etching primer. *Orthod Perspect*. 2001;8:5–6.
15. Bishara SE, Gordan VV, VonWald L, Olson ME. Effect of an acidic primer on shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 1998;114:243–247.
16. Bishara SE, Gordan VV, VonWald L, Jakobsen JR. Shear bond strength of composite, glass ionomer, and acidic primer adhesive systems. *Am J Orthod Dentofacial Orthop*. 1999;115:24–28.
17. Bishara SE, VonWald L, Lafoon JF, Warren JJ. Effect of a self-etch primer/adhesive on the shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 2001;119:621–624.
18. International Organization for Standardization. ISO TR 11405 dental materials—guidance on testing of adhesion to tooth structure. Geneva, Switzerland: WHO; 1993.
19. Toledano M, Osorio R, de Leonardi G, Rosales-Leal JJ, Ceballos L, Cabrerizo-Vilchez MA. Influence of self-etching primer on the resin adhesion to enamel and dentin. *Am J Dent*. 2001;14:205–210.
20. Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives. Part II: etching effects on unground enamel. *Dent Mater*. 2001;17:430–444.
21. Shintchi MJ, Soma K, Nakabayashi N. The effect of phosphoric acid concentration on resin tag length and bond strength of a photo-cured resin to acid-etched enamel. *Dent Mater*. 2000;16:324–329.
22. Galan D, Williams PT, Kasloff Z. Effects of warm air-drying and spreading on resin bonding. *Am J Dent*. 1991;4(6):277–280.