Original Article

Bonding Durability of Using Self-Etching Primer with 4-META/ MMA-TBB Resin Cement to Bond Orthodontic Brackets

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Abstract: This study determines the bonding durability when a self-etching primer is used with Superbond C&B (a 4-methacryloxyethyl trimellitate anhydride/methyl methacrylate–tri-*n*-butyl borane resin) to bond orthodontic brackets to enamel. Thermocycling test was used to assess bonding durability. Metal brackets were bonded to the phosphoric acid–etched or Megabond self-etching primer–treated enamel surface of human premolars using Superbond C&B. The shear bond strengths were measured after immersion in water at 37°C for 24 hours or after 2000 or 5000 cycles of thermocycling between 5°C and 55°C. Data were analyzed using 2-way analysis of variance and Fisher's protected least significant difference test for multiple comparisons. There was no significant difference in shear bond strength between phosphoric acid and Megabond self-etching primer treatment before the thermocycling test. After 2000 and 5000 thermal cycles, significant decreases in shear bond strength were observed with phosphoric acid etching. On the contrary, no significant differences in shear bond strength were observed after 2000 and 5000 thermal cycles with Megabond self-etching primer. The adhesive remnant indices were not significantly different between phosphoric acid etching and Megabond self-etching primer treatment either before or after thermal cycles. This study suggested that when used with Superbond C&B in bonding orthodontic brackets, Megabond self-etching primer is superior to phosphoric acid as an enamel preparation agent in providing durable bond strength. (*Angle Orthod* 2005;75:260–265.)

Key Words: Self-etching primer, Phosphoric acid etching, Shear bond strength, 4-META/MMA-TBB resin, Thermocycling test

INTRODUCTION

Currently, adhesive resin cements are widely used for bonding orthodontic brackets to enamel. Superbond C&B (Sunmedical Co Ltd, Shiga, Japan), a 4-methacryloxyethyl trimellitate anhydride (4-META)/methyl methacrylate (MMA)–tri-*n*-butyl borane (TBB) resin cement, is a unique MMA-based adhesive resin cement used widely for bonding orthodontic brackets and has earned a reputation for strong bonding.[1–4] This resin cement is also known as C&B Megabond (Parkell Inc, Framingdale, NY). Manufacturers recommended the application of 65% weight phosphoric acid etchant for tight adhesion of the 4-META/MMA-TBB resin to the enamel.[5]

Self-etching primers that function both as an etchant and a primer have been used widely to substitute phosphoric acid etching in composite resin restoration, and their efficacy regarding the adhesion to dentin and enamel has been reported.[6–8] Rinsing of the enamel after application of the self-etching primer is not required. Using a self-etching primer in lieu of phosphoric acid and an unfilled resin would reduce the number of steps as well as the time required for bonding orthodontic brackets to teeth. The time saved by the use of a self-etching primer is more than that spent in the preparation of the adhesive before bonding. Moreover, phosphoric acid etching techniques have been claimed to cause the iatrogenic damage to the enamel.

Van Waes et al[9] used a computerized 3-dimensional scan-

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ner to measure enamel loss caused by orthodontic bonding and debonding after phosphoric acid etching and reported an average loss of enamel of 7.4 μm. Using field emission scanning electron microscopy, Kawasaki et al[4] observed more dissolution of the enamel surface resulting from phosphoric acid etching than from self-etching primer treatment. Some in vitro studies reported enamel fracture after debonding the orthodontic bracket bonded to etched enamel and that the amount of enamel fracture was proportional to the length of etching time.[10–13] In some clinical studies, a composite resin adhesive with phosphoric acid etching produced a greater number of white spots on the enamel at debonding and more enamel damage than glass-ionomer cement.[14,15] Pascotto et al[16] concluded that the use of a glass-ionomer cement for bonding may be encouraged because it decreases the development of caries around orthodontic brackets compared with composite resin/phosphoric acid etching.

Recently, Sirirungrojying et al[17] evaluated the effectiveness of a self-etching primer in the bonding of orthodontic brackets to enamel using 4-META/MMA-TBB resin and found that the application of a commercially available self-etching primer, Megabond primer (Kuraray Medical Inc, Tokyo, Japan), produced no significantly different bond strength compared with phosphoric acid etching. They concluded that Megabond primer when used with Superbond C&B resin cement may be a good candidate for bonding orthodontic brackets to human enamel.

In the clinical situation, long-term bracket to enamel bond strength may be important for successful and effective patient treatment. Some reports evaluated the long-term bonding durability using the thermocycling test. Previous thermocycling studies varied widely in the number of cycles used, ranging from 100 to 20,000. Ishikawa et al[18] reported that when orthodontic brackets were bonded to enamel with resin-reinforced glass-ionomer cement, there was no statistical difference in bond strength between teeth that had undergone 2000 thermal cycles and those that had not. On the other hand, Bishara et al[19] reported that cyanoacrylate adhesive had a clinically adequate bond strength at 24 hours after initial bonding but lost about 80% of its strength after 500 cycles of thermocycling between 5°C and 55°C. Kitayama et al[20] evaluated the bonding durability of resin-reinforced glass-ionomer cement to glazed porcelain using 2000 thermal cycles and found that the tensile bond strength decreased significantly, whereas shear bond strengths showed no decrease after the thermal cycling test. Arici and Arici[21] reported a reduction in mean shear bond strength for both resin-modified glass-ionomer cement and composite adhesive used to bond orthodontic metal brackets to enamel after both 200 and 20,000 thermal cycles.

The bonding durability of Superbond C&B to enamel has also been assessed. Mogi[1] reported that the tensile bond strength of Superbond C&B resin cement used on bovine enamel etched with phosphoric acid decreased to almost 60% of the initial bond strength after 1 year of immersion in water at 37°C. Miwa et al[22] evaluated the tensile bond strength between enamel and orthodontic bracket bonded with Superbond C&B after phosphoric acid etching after thermocycling and found a decrease in tensile bond strength after 3000 and 10,000 thermal cycles. Hayakawa and Nemoto[23] also reported a significant decrease in tensile bond strength after 5000 thermal cycles when Superbond C&B was used on bovine enamel etched with phosphoric acid. However, no reports correlate the number of thermal cycles with clinical time under oral conditions.

This study assesses the effectiveness of a self-etching primer when used with 4-META/MMA-TBB resin to bond orthodontic brackets to enamel by determining the bonding durability using the thermocycling test. Two thousand and 5000 thermal cycles were used in this study, as reported in previous studies.[18,20,22]

MATERIALS AND METHODS

A total of 120 extracted human premolars were used in this study. Initially, they were randomly divided into a phosphoric acid group and self-etching primer group. The teeth were embedded in acrylic resin with the buccal surfaces available for bonding. After curing the acrylic resin, the tooth surfaces to be bonded were cleansed and polished with pumice and rubber prophylactic cups for 10 seconds to simulate a routine clinical procedure.

Orthodontic metal brackets (Super mesh STD Edgewise 131-45B, Tomy International Inc, Tokyo, Japan) were used in this study. The average bracket surface area was 11.188 mm^2. Megabond self-etching primer (Kuraray Medical Inc) was tested. Megabond primer is composed of 10-methacryloxydecyl dihydrogen phosphate, 2-hydroxyethyl methacrylate (HEMA), and polyfunctional dimethacrylates. This primer is a component of the Clearfil Megabond System (Kuraray Medical Inc), also known as Clearfil SE Bond outside Japan.

Bonding procedures

Protocol 1—phosphoric acid etching. A tooth was etched with 65% phosphoric acid gel, which was in the Superbond C&B kit, for 30 seconds, washed for 20 seconds, and air-dried. Then, a metal orthodontic bracket was bonded to the etched enamel surface using Superbond C&B resin cement. The catalyst, a partly oxidized TBB initiator, was added to the monomer mixture of 4-META and MMA to prepare an activated polymerized monomer liquid. Then, the polymer powder and the activated monomer liquid were mixed and used to bond metal brackets to the treated enamel surface using the brush-dip technique.

Protocol 2—self-etching primer treatment. An acidic self-etching primer, Megabond self-etching primer, was directly placed on the polished enamel for 30 seconds. Excessive primer solution was evaporated using compressed

TABLE 1. Conditions Used in This Study

Condition No.	Pretreatment Reagent	Number of Thermal Cycles	Number of Extracted Human Premolars
1	Phosphoric acid etching	0	20
2	Phosphoric acid etching	200	20
3	Phosphoric acid etching	5000	20
4	Megabond self-etching primer	0	20
5	Megabond self-etching primer	200	20
6	Megabond self-etching primer	5000	20

TABLE 2. Shear Bond Strengths (MPa) Before and After Thermocycling[a]

	Number of Thermal Cycles								
	0			2000			5000		
Pretreatment Agent	Mean	SD	Range	Mean	SD	Range	Mean	SD	Range
Phosphoric acid etching	20.4[Aa]	6.3	11.4–33.3	10.4[Bb]	3.7	5.3–20.0	10.5[Bd]	5.1	4.4–19.6
Megabond self-etching primer treatment	17.8[Ca]	4.4	12.4–28.8	17.5[Cb]	3.4	12.7–24.5	15.3[Ce]	4.3	9.7–24.4

[a] Mean values with different letters are significantly different ($P < .05$). Uppercase letters indicate the comparison of shear bond strength within the same pretreatment, and lowercase letters indicate the comparison of shear bond strength at the same number of thermal cycles. Phosphoric acid etching produced significant differences in shear bond strength after thermocycling compared with no thermocycling ($P < .05$). No significant differences in shear bond strength were found between no thermocycling and after 2000 or 5000 thermal cycles in the case of Megabond self-etching primer treatment ($P > .05$). In the absence of thermocycling, there was no significant difference in shear bond strength between phosphoric acid etching and self-etching primer treatments ($P > .05$). Significant differences in shear bond strength existed between phosphoric acid etching and self-etching priming at 2000 or 5000 thermal cycles ($P < .05$).

air. Then, an orthodontic metal bracket was bonded to the primed enamel surface using Superbond C&B resin cement according to the procedures described above.

Each bracket was subjected to a 300-g force, according to the report of Bishara et al,[24] and excess bonding resin was removed with a small scaler.

Bonding assessments

After bonding, teeth in the phosphoric acid group and those in the self-etching primer group were randomly divided into three groups of 20 each. Twenty teeth in the phosphoric acid group and 20 in the self-etching primer group were debonded after immersion in deionized water at 37°C for 24 hours. Twenty teeth in the phosphoric acid group and 20 in the self-etching primer group were kept in deionized water at 37°C for 24 hours, subjected to thermocycling test for 2000 cycles, and then debonded. Thermocycling was performed between 5°C and 55°C according to the methods recommended by the International Organization for Standardization's recommendation.[25] The specimens were stored in distilled water for 24 hours to provide baseline data for comparative purpose. The exposure in each bath was 60 seconds, and the transfer time between baths was less than 5 seconds. The remaining 20 teeth in the phosphoric acid group and 20 in the self-etching primer group were kept in deionized water at 37°C for 24 hours, subjected to thermocycling test for 5000 cycles, and then debonded. These conditions are summarized in Table 1.

Shear bond strength was measured according to the methods recommended by the International Organization for Standardization[17,25] using a testing machine (TCM-500CR, Shinkoh, Tokyo, Japan) at a crosshead speed of 2 mm/min. After debonding, the teeth and brackets were examined with 10× magnifications. The debonding condition of each specimen was scored using the adhesive remnant index (ARI).[26] The ARI scores ranged from 0 to 3, ie, score 0 = no adhesive remained on the enamel; 1 = less than half of the adhesive remained on the tooth surface; 2 = more than half of the adhesive remained on the tooth; 3 = all the adhesive remained on the tooth with a distinct impression of the bracket base. Enamel fracture was also scored according to the method of Schaneveldt and Foley.[27]

Statistical analysis

Twenty specimens were tested for each procedure. Two-way analysis of variance (ANOVA) and Fisher's test for multiple comparisons were used to detect statistical differences in mean measurements among the 6 procedures. The chi-square (χ^2) test was used to analyze statistical differences in ARI scores and enamel fracture scores among the 6 protocols. Significance for all statistical tests was predetermined at $P < .05$.

RESULTS

Comparison of shear bond strengths

The results of shear bond strength measurements (MPa) are listed in Table 2. Two-way ANOVA showed significant differences in bond strength between phosphoric acid etch-

TABLE 3. Results of Analysis of Variance for Shear Bond Strength

Source of Variation	df	Sum of Squares	Mean Squares	F-Value	*P*-Value
Pretreatment reagent	1	289.945	289.945	12.990	0.0005
Number of thermal cycles	2	888.846	444.423	19.911	<0.0001
Pretreatment reagent/Number of thermal cycles	2	519.580	259.790	11.639	<0.0001
Residual	114	2544.553	22.321		

TABLE 4. Frequency Distribution of the Adhesive Remnant Index (ARI)[a,b]

No. of thermal cycles	Pretreatment	ARI = 0	ARI = 1	ARI = 2	ARI = 3	EF
0	Phosphoric acid etching	10	2	0	0	8
	Self-etching primer treatment	14	1	0	0	5
2000	Phosphoric acid etching	16	1	0	0	3
	Self-etching primer treatment	15	4	0	0	1
5000	Phosphoric acid etching	12	4	0	0	4
	Self-etching primer treatment	14	2	0	0	4

[a] EF, enamel fracture.

[b] There were no significant differences in ARI scores among six procedures ($\chi^2 = 12.101$, $P = .2784$). Enamel fracture was included in the chi-square test.

ing and self-etching primer treatment (F = 12.990, $P = .0005$) and between the number of thermal cycles (F = 19.911, $P < .0001$). Two-way interactions were found for the type of pretreatment agent (phosphoric acid or self-etching primer) and the number of thermal cycles (F = 11.639, $P < .0001$). The results of ANOVA for shear bond strength are summarized in Table 3.

In the absence of thermocycling, no significant difference in shear bond strength was observed between phosphoric acid etching and Megabond self-etching primer treatment ($P > .05$). However, the bond strength after 2000 or 5000 thermal cycles obtained was significantly higher with Megabond self-etching primer treatment than with phosphoric acid etching ($P < .005$).

In premolars treated with phosphoric acid, 2000 and 5000 thermal cycles significantly decreased the bond strength compared with no thermocycling ($P < .05$). There was no significant difference in the shear bond strength between 2000 and 5000 thermal cycles ($P > .05$). When the teeth were treated with self-etching primer, there were no significant differences between 0, 2000, and 5000 thermal cycles ($P > .05$).

Comparison of ARI

The results of frequency distribution of ARI scores and frequencies of enamel fracture after debonding are shown in Table 4. Chi-square test that included the ARI scores and enamel fracture showed no significant difference in ARI score among the 6 conditions ($\chi^2 = 12.101$, $P = .2784$). Enamel fractures were observed after debonding in all 6 conditions.

DISCUSSION

This study showed that the durability of the bonding of orthodontic brackets to enamel using Superbond C&B was influenced by the method of enamel pretreatment, ie, phosphoric acid etching or self-etching primer treatment. Phosphoric acid etching significantly decreased the bond strength after thermocycling. The results from this study are consistent with those of Mogi,[1] Miwa et al,[22] and Hayakawa and Nemoto.[23]

However, the shear bond strength of orthodontic brackets bonded to enamel etched with phosphoric acid was about 10 MPa after 5000 thermal cycles. Bishara et al[28] reported that a shear bond strength of 7 MPa was clinically acceptable for bonding to the enamel surface. According to them, the shear bond strength of orthodontic bracket bonded to enamel etched with phosphoric acid after 5000 thermal cycles may be a clinically acceptable value.

In the clinical situation, it is more important to obtain adequate bond strength during orthodontic treatment for safe debonding rather than to attain the greatest possible bond strength. With in vitro debonding, the enamel surface is examined under a dissecting microscope and the amount of adhesive remaining on the tooth is recorded using the ARI.[26] ARI scores are used to define the site of bond failure between the enamel, the adhesive, and the bracket base. Schaneveldt and Foley[27] reported the ARI scores including the enamel fracture.

In this study, some enamel fractures were also observed in all 6 conditions tested, even after 5000 thermal cycles. Therefore, the failure patterns in this study were scored according to the method of Schaneveldt and Foley.[27] The results of this study indicated that both phosphoric acid etching and self-etching primer treatment may produce enamel fracture at the debonding of orthodontic brackets. Debonding of orthodontic brackets should be performed very carefully to avoid enamel fracture, even when the tooth has been treated with Megabond primer. More clinical research is necessary to assess the effectiveness of self-etching primer treatment.

Treatment with Megabond self-etching primer maintained the initial bond strength after thermocycling. Newman et al[29] reported that Megabond promoted the bonding under slightly monistic conditions because it contains HEMA and is hydrophilic. This may be one reason for the durable bond strength of the Megabond self-etching primer treatment. Hayakawa and Nemoto[23] suggested that the reason for the decrease of the bond strength after thermal cycles was due to the deterioration of the physical properties of Superbond C&B. It is speculated that Megabond self-etching primer treatment produced less deterioration of the physical properties of Superbond C&B than phosphoric acid etching, although the details are not clear.

Sirirungrojying et al[17] reported that Megabond self-etching primer treatment produced less roughening of the enamel surface than phosphoric acid etching and claimed that enamel loss was reduced by self-etching primer treatment as compared with phosphoric acid etching. Although it remains unclear how many thermal cycles are needed to predict long-term stability of the bonding of orthodontic bracket to enamel using Superbond C&B resin, data obtained in this study show less degradation of the bracket-adhesive-enamel construct for Megabond primer compared with phosphoric acid etching. This study indicated that the Megabond self-etching primer provides stable and sustained bond strength when used with Superbond C&B resin cement to bond orthodontic bracket to enamel.

CONCLUSIONS

This study provides evidence in human teeth that when using Superbond C&B as an orthodontic direct-bonding adhesive, Megabond self-etching primer is superior to phosphoric acid as an enamel preparation agent in providing durable bond strength. Another merit of using Megabond self-etching primer is the reduction in the number of clinical steps during bonding.

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