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# Efficacy of Using Self-etching Primer with a 4-META/MMA-TBB Resin Cement in Bonding Orthodontic Brackets to Human Enamel and Effect of Saliva Contamination on Shear Bond Strength

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## ABSTRACT

The objective of this study was (1) to evaluate the effectiveness of Megabond when used with Superbond C&B, a 4-methacryloxyethyl trimellitate anhydride (4-META)/methyl methacrylate (MMA)-tri-*n*-butyl borane (TBB) resin, to bond orthodontic metal brackets to human enamel and (2) to examine the influence of saliva contamination on shear bond strength. Metal brackets were bonded to phosphoric acid-etched or Megabond-treated human premolars using Superbond C&B resin cement. The effects of saliva contamination after acid etching or self-etch priming, and the effect of re-etching or self-etch priming after saliva contamination on shear bond strength were also assessed. The shear bond strengths were measured after immersion in water at 37°C for 24 hours. Data were analyzed using two-way analysis of variance and Fisher's protected least significant difference test for multiple comparisons. There were no significant differences in shear bond strength between phosphoric acid etching and self-etch priming for no contamination, saliva contamination, and repeat treatment (etching or priming) after saliva contamination. With phosphoric acid etching, saliva contamination significantly decreased the shear bond strength. Repeat phosphoric acid etching after saliva contamination did not significantly improve the bond strengths. With self-etching primer treatment, however, saliva contamination did not cause any decrease of bond strength. Phosphoric acid etching produced more enamel fracture than self-etching primer treatment. Field-emission scanning microscopy revealed less dissolution of enamel surface resulted from self-etching primer compared with phosphoric acid. These results suggest that Megabond when used with Superbond C&B resin cement may be a good candidate for bonding orthodontic brackets to human enamel.

**KEY WORDS:** Self-etching primer, Phosphoric acid etching, Saliva contamination, Shear bond strength, 4-META/MMA-TBB resin.

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**INTRODUCTION** [Return to TOC](#)

Adhesive resin cements are presently 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, is a unique MMA-based adhesive resin cement that has been used widely for bonding orthodontic brackets and has earned a reputation for strong

bonding.<sup>1-4</sup> This resin cement is also known as C&B Metabond (Parkell Inc, Farmingdale, NY) in North America. Tight bonding of orthodontic brackets to enamel with Superbond C&B is achieved by 65% by weight phosphoric acid etching. It is reported that variation of the concentration of phosphoric acid from 20 to 65% by weight did not produce 4-META/MMA-TBB resin-etched enamel bonds of different strengths, although demineralization decreased with the increasing concentration of phosphoric acid.<sup>5</sup> Thus, manufacturers recommended the application of 65% by weight phosphoric acid etchant for the adhesion of 4-META/MMA-TBB resin to enamel to minimize the enamel loss.

In conservative dentistry, self-etching primers have been used widely instead of phosphoric acid in composite resin restorations, and their efficacy regarding adhesion to dentin and enamel has been reported.<sup>6-8</sup> Self-etching primers function both as an etchant and a primer. Rinsing of the enamel after application of the self-etching primer is not required. Thus, substituting the 65% by weight phosphoric acid etching agent with a self-etching primer in the Superbond C&B system for bonding orthodontic brackets would reduce the number of clinical steps and save clinical operation time because separate acid-etching and water-rinsing steps are eliminated, and the application simply requires drying with air. The time saved by using a self-etching primer is more than that spent in the preparation of the adhesive before bonding. Moreover, phosphoric acid techniques are associated with enamel loss and a risk of enamel cracks and scratches after debonding.<sup>9-11</sup> Phosphoric acid etching has also been blamed for decalcification and the development of white spot lesions around bonded orthodontic appliances.<sup>12,13</sup> Therefore, use of a self-etching primer with Superbond C&B resin cement in bonding orthodontic brackets to enamel would also avoid the aforementioned risk caused by phosphoric acid etching.

Most commercially available direct-bonding adhesives require a contamination-free enamel surface to obtain successful clinical results.<sup>14,15</sup> However, it is very difficult to completely prevent saliva and blood contamination of cleansed and dried enamel surfaces when bonding attachments to impacted or lingual tooth surfaces. Various studies have reported reduction of shear bond strength when resin is directly bonded to saliva-contaminated etched enamel compared with the uncontaminated surfaces.<sup>16-18</sup>

Itoh et al<sup>19</sup> reported the influence of contamination by water, human saliva, and blood on the bonding of metal brackets with Superbond C&B to phosphoric acid-etched enamel surface. They found that saliva and blood contamination decreased the bond strength in orthodontic brackets bonded to the polished anterior surface of bovine teeth.

The aims of the present study were (1) to evaluate the effectiveness of using a self-etching primer with Superbond C&B resin cement to bond orthodontic brackets to human enamel and (2) to investigate the influence of saliva contamination on shear bond strength of orthodontic brackets bonded with Superbond C&B cement to self-etch primed and acid-etched human enamel surfaces.

## MATERIALS AND METHODS [Return to TOC](#)

A total of 120 premolar teeth were used in this study. They were randomly allocated to six protocols of 20 teeth each. 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 polished with pumice and rubber prophylactic cups for 10 seconds. The teeth were not ground before etching/self-etch priming and bonding.

Orthodontic metal brackets (Standard Edgewise 100-1100, Dentsply-Sankin K.K., Tokyo, Japan) for premolars were used in this study. The average bracket surface area was determined to be 9.64 mm<sup>2</sup>. The brackets were bonded to the teeth according to one of six protocols (n = 20).

Megabond (Kuraray Medical Inc., Tokyo, Japan) was used as a self-etching primer. Megabond is composed of 10-methacryloxydecyl dihydrogen phosphate (MDP), 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

The teeth were randomly divided into six groups and bonded according to one of the six protocols shown below. In all protocols, the Superbond C&B resin cement was used for bonding. First 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 activated monomer liquid were mixed and used to bond metal brackets to the enamel surface using the brush-dip technique.

*Protocol 1: Phosphoric acid etching.* The teeth were etched with 65% by weight phosphoric acid gel for 30 seconds, washed for 20 seconds, and air-dried. Then, the metal orthodontic brackets were bonded to the etched enamel surface using Superbond C&B resin cement according to the above procedures.

*Protocol 2: Phosphoric acid etching before contamination.* The teeth were etched with 65% by weight phosphoric acid gel for 30 seconds. After rinsing and drying, the etched surface was contaminated with 20 µl of human fresh whole saliva. The contaminant fluids were left on the surface for 30 seconds to simulate extremely severe clinical conditions. After blowing off the saliva with air for 5 seconds, metal orthodontic metal brackets were bonded to the enamel according to the above procedures.

*Protocol 3: Phosphoric acid etching before contamination and repeat phosphoric acid etching.* The teeth were etched with 65% by weight phosphoric acid gel for 30 seconds. After rinsing and drying, the etched surface was contaminated with 20 µl of human fresh whole saliva, which was left on the surface for 30 seconds. After blowing off the saliva with air for 5 seconds, the contaminated teeth were re-etched with 65% by weight phosphoric acid gel for 30 seconds. Orthodontic metal brackets were bonded to enamel according to the above procedures.

*Protocol 4: Self-etching primer.* An acidic self-etching primer, Megabond, was placed on the enamel for 30 seconds. Excessive primer solution was evaporated using compressed air. Then orthodontic metal brackets were bonded to the enamel using Superbond C&B resin cement according to the above procedures.

*Protocol 5: Self-etching primer before contamination.* Megabond was placed on the enamel for 30 seconds. Excessive primer solution was evaporated using compressed air. Then, the self-etching primed enamel was contaminated with 20 µl of human fresh whole saliva. The contaminant fluids were left on the surface for 30 seconds to simulate extremely severe clinical conditions. After blowing off the saliva with air for 5 seconds, orthodontic metal brackets were bonded to the enamel according to the above procedures.

*Protocol 6: Self-etching primer before contamination and re-self-etching primer.* Megabond was placed on the enamel for 30 seconds. Excessive primer solution was evaporated using compressed air. Then, the self-etching primed enamel was contaminated with 20 µl of human fresh whole saliva, which was left on the surface for 30 seconds. After blowing off the saliva with air for 5 seconds, the contaminated teeth were again treated with Megabond for 30 seconds. The excess solution was evaporated using compressed air. Then, orthodontic metal brackets were bonded to the enamel according to the above procedures.

### **Bonding assessments**

Each bracket was subjected to a 300-g force, according to Bishara et al,<sup>20</sup> and excess bonding resin was removed with a small scaler. After curing the resin, all samples were stored in deionized water at 37°C for 24 hours. Shear bond strength was measured according to Noguchi's methods<sup>21–23</sup> using a testing machine (TCM-500CR, Shinkoh, Tokyo, Japan) at a cross-head speed of 2 mm/min. The shear bond strengths were expressed in megapascals.

After debonding, the teeth and brackets were examined under 10× magnification. The debonding characteristics for each specimen were determined using the adhesive remnant index (ARI).<sup>24</sup> ARI scores were scored as 0 to 3: 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; and 3, all the adhesive remained on the tooth with a distinct impression of the bracket base. The enamel fracture was also scored according to the method of Schaneveldt and Timothy.<sup>25</sup>

### **Statistical analysis**

Twenty specimens were tested for each procedure. Differences in mean measurements among the six protocols were analyzed by two-way analysis of variance (ANOVA) and Fisher's test for multiple comparisons. The chi-squared ( $\chi^2$ ) test was used to analyze differences in ARI scores among the six protocols. The level of significance for all statistical tests was predetermined at  $P < .05$ . When a significant difference was found by the chi-squared test, complementary tests were performed to ascertain differences among groups.

### **Field emission–scanning electron microscope observation**

The human enamel surfaces were cleansed and then polished with pumice and rubber prophylactic cups as described above. The tooth surface was etched with the phosphoric acid etching agent included in Superbond C&B for 30 seconds and washed for 20 seconds. After washing, the specimen was dehydrated through a graded series of ethanol, dried in a critical drying apparatus, and ion-coated with platinum, according to the method of Itoh et al<sup>19</sup>.

In another specimen, the tooth surface was treated with Megabond for 30 seconds, and the excess solution was evaporated using compressed air. The treated enamel surface was rinsed with acetone for 30 seconds to remove the organic components of the self-etching primer. The specimen was also dehydrated, dried, and ion-coated by the method described previously.

The surface appearances of the acid-etched– and self-etching primer–treated tooth specimens were observed using a field-emission scanning electron microscope (FE-SEM; JSM-6340F, JEOL, Tokyo, Japan). The appearance of the human enamel surface polished with pumice and rubber prophylactic cups was also observed.

## **RESULTS [Return to TOC](#)**

### **Comparison of shear bond strengths**

The results of shear bond strength (MPa) measurements are listed in [Table 1](#). Two-way ANOVA showed significant differences in

bond strength between with and without contamination ( $F = 4.190$ ,  $P < .05$ ), and no significant differences between phosphoric acid etching and self-etching primer treatment ( $F = 0.786$ ,  $P > .05$ ). Two-way interactions were found for the types of pretreatment; etching and self-etching priming and with and without contamination ( $P < .05$ ).

No significant difference in shear bond strength was observed between phosphoric acid and Megabond when the teeth were not contaminated with saliva (protocol 1 vs protocol 4,  $P > .05$ ). When the teeth were contaminated with saliva, there was also no significant difference in shear bond strength between phosphoric acid etching and Megabond (protocol 2 vs protocol 5, protocol 3 vs protocol 6; both  $P > .05$ ).

In phosphoric acid-etched teeth, saliva contamination significantly decreased the shear bond strength of Superbond C&B resin cement to etched enamel compared with no contamination (protocol 2 vs protocol 1,  $P < .05$ ), and repeat etching with phosphoric acid after saliva contamination also gave significantly lower bond strength compared with no contamination (protocol 3 vs protocol 1,  $P < .05$ ).

In contrast, when the teeth were treated with self-etching primer, there were no significant differences among no contamination, saliva contamination, and repeat priming after saliva contamination (protocol 4 vs protocol 5 vs protocol 6; all  $P > .05$ ).

### Comparison of ARI

The results of frequency distribution of ARI scores and frequencies of enamel fracture after debonding are shown in [Table 2](#). The chi-squared test showed significant differences in ARI score among the six procedures ( $\chi^2 = 24.710$ ,  $P = .0059$ ). A significant difference in ARI scores was found among the self-etching primer groups (protocols 4, 5, and 6;  $\chi^2 = 13.636$ ,  $P = .0086$ ) but no significant difference among the phosphoric acid etching groups (protocols 1, 2, and 3;  $\chi^2 = 13.636$ ,  $P = .2698$ ). Comparing the phosphoric acid etching groups with self-etching primer groups, a significant difference existed between the two groups ( $\chi^2 = 9.527$ ,  $P = .0085$ ). Phosphoric acid etching produced more enamel fracture than self-etching priming.

### FE-SEM observation

[Figures 1–3](#) show the FE-SEM micrographs of human enamel surfaces that have been (1) polished, (2) etched with phosphoric acid, and (3) treated with Megabond.

After cleaning and polishing, smooth and roughened areas were present on the enamel surface. The smooth areas were covered with organic materials derived from saliva, and minute focal holes<sup>26</sup> were observed on the roughened surface ([Figure 1](#), arrow). Scratches produced by polishing with pumice and rubber prophylactic cups were observed.

Phosphoric acid etching produced a roughened enamel surface. There was no distinct dissolution of enamel prisms or enamel peripheries, and the enamel surface was finely roughened with random arrangement of enamel crystals ([Figure 2](#)).

In the FE-SEM micrograph of the enamel surface after treatment with Megabond ([Figure 3](#)), the pattern was different from that observed after phosphoric acid etching. There was no distinct dissolution pattern, and the enamel surface appeared almost flat. Minute focal holes (arrow) were also identified. No enamel crystals were observed.

### DISCUSSION [Return to TOC](#)

Few reports have examined the efficacy of self-etching primer in the adhesion of Superbond C&B resin cement to enamel surface. Hayakawa and Nemoto<sup>27</sup> examined the efficacy of self-etching primer containing methacryloxyethyl phosphate in the adhesion of Superbond C&B resin cement to ground bovine enamel. They reported that their self-etching primer, which contains methacryloxyethyl phosphate, HEMA, and ferric chloride was useful in obtaining strong adhesion of Superbond C&B to bovine enamel. However, they provided no data on bonding to human enamel.

In the present study, we examined the effectiveness of Megabond when used with Superbond C&B resin cement for the bonding of orthodontic brackets to human enamel. Our analysis showed no significant difference in shear bond strength between phosphoric acid and Megabond, regardless of the presence or absence of saliva contamination. Megabond was therefore useful for the bonding of orthodontic brackets to enamel when used with Superbond C&B resin cement.

On the other hand, a previous report indicated that Megabond gave significantly lower shear bond strength than phosphoric acid etching agent when used with composite resin adhesive.<sup>23</sup> Moreover, recent studies in conservative dentistry have suggested that self-etching primers that have milder actions are less effective than phosphoric acid when used to bond ground enamel with a thick smear layer or intact unground enamel.<sup>28,29</sup> These previous findings contradict the present results. This discrepancy was because of the difference in adhesive resins. Superbond C&B resin cement contains 4-META, a well-known adhesive monomer. Hotta et al<sup>30</sup> reported that 4-methacryloxy trimellitic acid conformity (4-MET), which is a hydrolysis product of 4-META, promotes effective diffusion of monomer into enamel. The difference in monomer penetration efficiency probably influenced the shear bond strength.

Newman et al<sup>31,32</sup> investigated the effectiveness of Megabond as an adhesion promoter by applying Megabond to an enamel surface after etching with phosphoric acid or polyacrylic acid. In the present study, we applied Megabond directly on the enamel surface without any etching procedure, and we found that use of Megabond treatment instead of phosphoric acid etching in the Superbond C&B system was effective for the bonding of orthodontic brackets to human enamel.

Phosphoric acid etching produces a roughened enamel surface by dissolving the hydroxyl apatite of enamel and forming enamel resin tags.<sup>33</sup> Although the enamel etching technique is a useful and accepted orthodontic procedure for bonding orthodontic brackets, there is a need to improve this method, that is to maintain clinically useful bond strength while minimizing the amount of enamel loss. In the present study, FE-SEM observation reveals a smaller extent of enamel dissolution on treatment with Megabond compared with phosphoric acid etching. The findings in the present study indicate that enamel loss may be reduced by using Megabond.

Contamination by oral fluids such as saliva and plasma has been reported to reduce the bond strength of direct bonding of adhesive to enamel.<sup>16–18</sup> Itoh et al<sup>19</sup> investigated the influence of contamination of water, human saliva, and blood on the bonding of metal brackets with Superbond C&B to phosphoric acid-etched bovine enamel. They reported that saliva and blood contamination decreased the bond strength in orthodontic brackets bonded to the polished anterior surface of bovine teeth. The data obtained in the present study of human teeth confirms their results. When human enamel was etched with phosphoric acid, saliva contamination significantly decreased the shear bond strength of orthodontic brackets, and repeat etching after saliva contamination did not significantly improve the bond strength. Silverstone et al<sup>34</sup> have reported that a biofilm forms quickly over etched enamel after exposure to saliva. It is well known that this biofilm significantly lowers the bond strength between resin and etched enamel. Therefore, a major reason for reduced bond strength seems to be the presence of this biofilm on the enamel surface. However, even on saliva-contaminated surfaces, Superbond still provides a clinically acceptable bond strength of 12–20 MPa for bonding orthodontic brackets.<sup>19</sup>

When human enamel was treated with self-etching primer, saliva contamination did not significantly decrease the shear bond strength of orthodontic brackets. There were no significant differences in shear bond strength among the three protocols of no contamination (protocol 2), saliva contamination (protocol 4), and repriming after saliva contamination (protocol 6).

Bishara et al<sup>35</sup> used a new self-etching primer, Angel I (3M/ESPE, Minneapolis, Minn), and assessed the effect of saliva contamination on the shear bond strengths of orthodontic metal brackets precoated with adhesive resin. They reported that human saliva contamination either before or after the application of the self-etching primer did not affect the shear bond strength significantly, although there was on average a 25% reduction in the mean shear bond strength. In the present study, there was no reduction in the mean shear bond strength after saliva contamination when Megabond was used with Superbond C&B resin cement.

The findings of ARI scores combined with enamel fracture are noteworthy. There was a tendency of less enamel fracture when human enamel was treated with Megabond both in the absence and presence of saliva contamination. Therefore, use of Megabond with Superbond C&B resin cement for bonding orthodontic brackets to enamel may have a lower risk of enamel fracture at the time of debonding.

In the clinical situation, the purpose is not to obtain the highest possible bond strength with the adhesive, but to obtain adequate bond strength for orthodontic treatment purposes and, equally important, safe debonding after treatment. The usefulness of Megabond with Superbond C&B resin cement for bonding orthodontic brackets to enamel should be further investigated by considering the clinical situation, for example long-term bond strength.

## CONCLUSIONS [Return to TOC](#)

The present findings provide evidence in human teeth that when using Superbond C&B as an orthodontic direct-bonding adhesive, Megabond is a better candidate than phosphoric acid etchant for preparing the enamel surface. Use of Megabond saves water-rinsing time and leads to less enamel surface loss when debonding. In addition, saliva contamination does not affect the bond strength, and repeat treatment with the self-etching primer is not necessary after saliva contamination.

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**TABLE 1.** Comparison of Bond Strengths (MPa) among Six Procedures

	No Contamination (Protocols 1 and 4)			Saliva Contamination (Protocols 2 and 5)			Repeat Etching/Priming After Saliva Contamination (Protocols 3 and 6)		
	Mean <sup>a</sup>	SD <sup>b</sup>	Range	Mean	SD	Range	Mean	SD	Range
Phosphoric acid etchant	20.4 <sup>a,b</sup>	6.4	11.4–33.3	14.8 <sup>a</sup>	4.1	7.7–21.6	16.7 <sup>b</sup>	5.9	5.3–27.6
Megabond self-etching primer	17.8	4.5	12.4–28.8	17.3	3.4	11.2–23.3	19.2	4.0	11.2–26.7

<sup>a</sup> Mean values with the same superscripts are significantly different. No significant difference in shear bond strength was found between phosphoric acid etchant and Megabond self-etching primer under conditions of no contamination (protocol 1 vs protocol 4,  $P > 0.05$ ) and saliva contamination (protocol 2 vs protocol 5, protocol 3 vs protocol 6, both  $P > 0.05$ ). Significant differences were found among the three protocols of phosphoric acid etchant ( $P < 0.05$ ), but no significant differences were detected among the three protocols of self-etching primer.

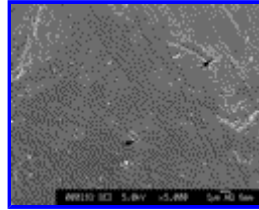
<sup>b</sup> SD indicates standard deviation.

**TABLE 2.** Frequency Distribution of the Adhesive Remnant Index (ARI) and Enamel Fracture (EF) During Debonding<sup>a</sup>

Procedure	ARI				EF
	0	1	2	3	
Protocol 1 (phosphoric acid etching)	10	2	0	0	8
Protocol 2 (phosphoric acid etching followed by saliva contamination)	14	3	0	0	3
Protocol 3 (phosphoric acid etching followed by saliva contamination and then re-etching)	14	0	0	0	6
Protocol 4 (self-etch priming)	14	1	0	0	5
Protocol 5 (self-etch priming followed by saliva contamination)	16	4	0	0	0
Protocol 6 (self-etch priming followed by saliva contamination and then repriming)	14	6	0	0	0

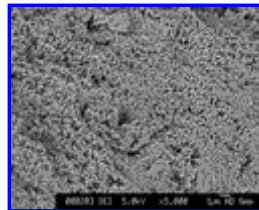
<sup>a</sup>  $\chi^2 = 24.710$ ,  $P = .0059$ ; a significant difference was detected between phosphoric acid and Megabond self-etching primer ( $\chi^2 = 9.527$ ,  $P = .0085$ ). There was a significant difference in ARI score among the self-etching primer groups ( $\chi^2 = 13.636$ ,  $P = .0086$ ) but no significant difference among phosphoric etching groups ( $\chi^2 = 13.636$ ,  $P = .2698$ ).

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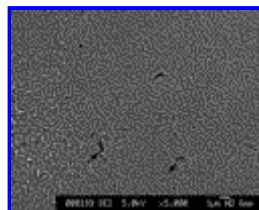
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**FIGURE 1.** Field-emission scanning electron micrographs of enamel surface. Polished surface: minute focal holes (arrow) are observed.



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**FIGURE 2.** Field-emission scanning electron micrographs of enamel surface. Phosphoric acid-etched surface: finely roughened enamel surface is evident



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**FIGURE 3.** Field-emission scanning electron micrographs of enamel surface. Self-etching primed surface: the enamel surface appears almost flat and the presence of minute focal holes (arrow) can also be identified. SEM conditions are shown in the bars

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