Tensile Bond Strength of 4-META/MMA-TBB Resin to Saliva Contaminated Teeth Using Self-Etching Primer

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Abstract
The aim of this study was to evaluate the tensile bond strength of 4-META/MMA-TBB resin to enamel using a self-etching primer with saliva contamination. The polished human molar surface was etched with phosphoric acid or Megabond self-etching primer with or without saliva contamination, then bonded with Superbond C&B resin cement. The tensile bond strengths were measured after immersion in water at 37 °C for 24 hours. Without saliva contamination, there was not significantly different between phosphoric acid etching and Megabond (P > 0.05). When the surfaces were saliva contaminated, using phosphoric acid etching showed decreasing in bond strength significantly (P < 0.05). Comparing to using Megabond, the bond strength decreased nonsignificantly (P > 0.05). The SEM showed the difference of enamel surface after phosphoric acid etching and Megabond. In conclusion, comparing the tensile bond strength, Megabond is a better candidate for etching enamel than phosphoric acid when clinical used.

Keywords:
Tensile bond strength, 4-META/MMA-TBB resin, Megabond self-etching primer, phosphoric acid etching, saliva contamination

Introduction
Superbond C&B (Sunmedical Co. Ltd., Shiga, Japan) is a unique adhesive resin cements and has been widely used for bonding orthodontic brackets to enamel in direct bonding technique. It is methyl methacrylate (MMA) initiated by partly oxidized tri-n-butyl borane (TBB) in the presence of polymethyl methacrylate (PMMA) and contains the adhesive monomer, 4-methacryloxyethyl trimellitate anhydride (4 META), dissolved in MMA (1-3). This resin is known as 4-META/MMA-TBB resin, and is also known as C&B Metabond (Parkell Inc, Farmingdale, NY) in North America. The bonding to enamel with Superbond is achieved by 65% by weight phosphoric acid etching.

Instead of phosphoric acid, self-etching primer have become widely used in composite resin restorations (4, 5). Self-etching primers function as both an etching agent and a primer. Water rinsing is not required. Thus, the use of self-etching primer reduces of clinical steps and operation time compared to phosphoric acid etching. In case of orthodontic treatment, the use of phosphoric acid etchings show the association with enamel loss and a risk of enamel cracks and scratches after debonding brackets (6, 7). In addition, phosphoric acid etching has also been blamed for decalcification and the development of white spot lesions around bonded orthodontic appliances (8). Thus, some self-etching primer systems are now introduced for bonding orthodontic bracket to enamel (9, 10).

During clinical procedures, it is difficult to complete the prevention of saliva and blood contamination. Reports show that using phosphoric acid etchings, contaminations can decrease the bond strength when bonding with resins (11-13). Compare to using self-etching primers, contaminations did not affect the shear bond strength (9, 10).

Previously, we demonstrated that the use of self-etching primer with Superbond C&B produced com-
patible bond strength more than with phosphoric acid etching, with less enamel fracture after debonding the orthodontic bracket from enamel. Saliva contamination significantly decreased the shear bond strength with phosphoric acid etching, but did not cause any decrease of bond strength with self-etching primer treatment (9).

The shear or tensile bond strength evaluation was always considered in traditional bond strength studies. Each bond strength is different in some circumstances, such as the adhesive thickness of the bonding cement (14), bonding durability (15). We already evaluated the effectiveness of the use of self-etching primer with Superbond C&B by measuring the shear bond strength as described above (9). Kitayama et al (15). compared the tensile and shear bond strength of resin-reinforce glass ionomer cement to glazed porcelain, and they reported that shear bond strength was higher approximately three to four times more than tensile bond strengths. Smith and Reynolds suggested that tensile bond strength of approximately 5-7 MPa was adequate for clinical success (16). The tensile bond strength of Superbond C&B resin to bovine enamel using self-etching primer were already studied (17, 18), but not to human enamel. Because it is important to evaluate the efficacy of the use of self-etching primer with Superbond C&B in orthodontic treatments, the evaluation using tensile bond strength will be useful.

The present study aimed at comparing the tensile bond strength to enamel between self-etching primer and phosphoric acid with and without saliva contamination when bonding with Superbond C&B. The obtained tensile bond strengths were evaluated by comparing with previous shear bond strength.

Materials and Methods

Tensile Bond Strength Measurement

Fig. 1 shows the schematic drawing of the specimen for tensile bond strength measurement. A total of 80 human molar teeth were used in this study. They were randomly allocated into 4 groups of 20 teeth each. The buccal surface were cleansed and then polished with pumice and rubber prophylactic cups for 10 seconds. In all protocols, Superbond C&B resin cement was used for bonding. As self-etching primer, Megabond (Kuraray Medical Inc., Tokyo, Japan) was used. Megabond is composed of 10-methacryloxydecyl dihydrogen phosphate (MDP), 2-hydroxyethyl methacrylate, a polyfunctional dimethacrylates. This self-etching primer is a component of the Clearfil Megabond System (Kuraray Medical Inc.), as known as Clearfil SE Bond outside Japan.

Group 1: Phosphoric acid etching. The teeth were etched with 65% phosphoric acid gel for 30 seconds, washed for 20 seconds, and air-dried.

Group 2: Phosphoric acid before contamination. The teeth were etched with 65% 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 30 seconds to simulate extremely severe clinical conditions. Then blowing off the saliva for 5 seconds.

Group 3: Self-etching primer. The teeth were etched with Megabond Self-etching primer for 30 seconds. The excessive solution was evaporated using compressed air.

Group 4: Self-etching primer before contamina-
tion. The teeth were etched with Megabond self-etching primer for 30 seconds. The excessive 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.

The area of adhesion was standardized by using a piece of 300 μm-thick masking tape with a hole measuring 4.0 mm in diameter.

The Superbond C&B resin cement was prepared by adding the catalyst (partially oxidized TBB) to the monomer liquid to become active monomer liquid. The polymer powder and active monomer liquid were mixed and applied to bond a sandblasted stainless steel rod to the enamel using brush dip technique. After curing the resin, all specimens were stored in water at 37 °C for 24 hours. Then, the bond strength was measured using computer-controlled Instron testing machine (TG-5kN), Minebea, Tokyo, Japan.

The comparison in mean tensile bond strength between 2 groups was analyzed by two-way analysis of variance (ANOVA) and Fisher’s test for multiple analysis. The level of significance was predetermined at P<0.05.

FE-SEM observation

The human enamel surfaces were cleansed and then polished with pumice and rubber prophylactic cups as described above. Two specimens were prepared. One was etched with phosphoric acid for 30 seconds and washed for 20 seconds. Another specimen was treated with Megabond self-etching primer for 30 seconds, and excess solution was evaporated using compressed air. The primed enamel surface was rinsed with acetone for 30 seconds to remove the organic components of the self-etching primer. Both etched and self-etching primed specimens were dehydrated through a graded series of ethanol, dried in a critical apparatus, and ion-coated with platinum.

The surface appearances of etched and primed tooth specimens were observed using a field-emission scanning electron microscope (FE-SEM; JSM-6340F, Tokyo, Japan).

Results

The results of tensile bond strength of phosphoric acid etching and self-etching primer with and without saliva contamination are presented in Fig. 2. Two-way ANOVA showed significant differences in bond strength between phosphoric acid etching and self-etching primer treatment (P<0.05), and also showed significant differences in bond strength between with and without saliva contamination (P<0.05). Two-way interaction was found for the types of pretreatment; etching and self-etching priming and with and without saliva contamination (P<0.05).

Without saliva contamination, there was no significantly difference in tensile bond strength between phosphoric acid etching and self-etching primer (P>0.05). In contrast, when teeth were contaminated with saliva, self-etching primer produced showed significantly higher tensile bond strength than phosphoric acid etching (P<0.05). When used with self-etching primer, there was no significant difference between with and without saliva contamination (P>0.05). Phosphoric acid etching showed significant decrease in tensile bond strength after saliva contamination (P<0.05). Macroscopic observation of debonded specimens showed an adhesive-resin enamel failure in all groups. No enamel fracture was observed after debonding.

Fig. 2. Tensile bond strength between stainless steel rod and human enamel when used with 4-META/MMA-TBB resin. *, **: significant difference
Fig. 3 showed the FE-SEM micrographs of human enamel surfaces that have been etched with phosphoric acid (Fig. 3a) and treated with Megabond primer (Fig. 3b). Phosphoric acid etching produced a finely roughened enamel surface with random arrangement of enamel crystals. Dissolution of both enamel prisms and peripheries was observed. The appearances of enamel surface after Megabond primer treatment was different from that observed after phosphoric acid etching (Fig. 3b). There was no distinct dissolution pattern and the enamel surface appeared almost flat. No enamel crystals were observed.

Discussion
In the present study, we demonstrated the efficacy of self-etching primer with the use of Superbond C&B when teeth were contaminated with saliva by measuring the tensile bond strength.

When tooth was not contaminated with saliva, no significant difference existed in the tensile bond between phosphoric acid and self-etching primer. It is generally recognized that mechanical retention on roughened enamel contributes to the adhesion of dental resins to enamel (19). However, the appearance of self-etching primed enamel surface was completely different from that etched by phosphoric acid: there was no distinct dissolution and no roughening of self-etching primed enamel surface. These finding suggest that the main adhesion contributor comes not from the mechanical retention obtained by macro-resin tags but from the formation of micro-resin tags (18).

The present study found that saliva contamination did not decrease the tensile bond strength to self-etching primed enamel. It is hypothesized as following although the detailed mechanism is unknown. Because the self-etching primer treatment did not produce distinct dissolution pattern on enamel, the saliva cannot penetrate into the primed enamel and the contaminated saliva on self-etching primed enamel is too thin. Thus, adhesive resin can bond to saliva contaminated enamel.

In the comparison between tensile and shear bond strength, the tensile bond strength obtained in the present study was approximately one third of previous shear bond strength (15–21 MPa) (9). Thomas et al. evaluated the tensile and shear stressed in orthodontic attachment adhesive layer with 3D finite element analysis (20). They concluded that a typical tensile load induces predominantly tensile stresses in the cement interfaces and that the test is relatively insensitive to minor misalignments errors. In contrast, the shear load produces both tensile stressed and compressive stressed in the cement interface that are of comparable magnitude or greater as compared to the shear stress. This is the one of the reason for the decrease of tensile bond strength compared with
shear bond strength, although the detailed reason is not clear.

At any event, present tensile bond strength is clinical acceptable value for bonding orthodontic bracket even if teeth is contaminated with saliva, according to the suggestion by Smith and Reynolds (16). In addition, self-etching primer treatment has a benefit for less damage for enamel dissolution. The use of Megabond primer with Superbond C&B for bonding orthodontic bracket to enamel may have a lower risk of enamel fracture at the time of debonding.

The lower value of tensile bond strength compared with shear bond strength also suggested that removal of bonded orthodontic bracket by tensile force is more desirable than shearing force in orthodontic clinic. In the clinical situation, the purpose is not to obtain the highest possible bond strength, but to obtain adequate bond strength for orthodontic treatment, and safe debonding without any damage in enamel is very important.

In conclusion, the present study revealed the usefulness of Megabond self-etching primer with Superbond C&B resin cement by measuring the tensile bond strength besides the measurement of shear bond strength.

References
20. Thomas RL, de Rijk, WG, Evans CA: Tensile and