Original Article

Assessing the Performance of a Methyl Methacrylate-Based Resin Cement with Self-Etching Primer for Bonding Orthodontic Brackets

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Abstract: Questions over the usefulness of a self-etching primer with resin adhesive in the bonding of orthodontic brackets remain unsolved. The purpose of this study was to determine the effects of using Multibond, a new methyl methacrylate (MMA)-based resin cement with self-etching primer, on the shear bond strength of orthodontic brackets compared with Superbond C&B, which is a well-known MMA-based resin cement containing phosphoric acid etching. Metal or plastic brackets were bonded to etched or self-etching primed bovine teeth using Superbond C&B or Multibond. The shear bond strengths were measured after immersion in water at 37°C for 24 hours. Data were analyzed by two-way analysis of variance and Scheffe's test. The surface appearances of the teeth after phosphoric acid etching or self-etching priming were observed by field-emission scanning electron microscopy (FE-SEM). Metal brackets bonded with Multibond had a significantly lower shear bond strength than metal brackets bonded with Superbond C&B. No significant differences in shear bond strength were observed between Multibond and Superbond C&B when plastic brackets were bonded to the enamel. The shear bond strength of metal brackets bonded with Multibond was comparable with that of plastic brackets bonded with Superbond C&B. Adhesive remnant index score showed a tendency of more residual resin cement remaining on the teeth when metal brackets were bonded with Multibond. FE-SEM observation revealed less dissolution of the enamel surface resulting from treatment with Multibond self-etching primer as compared with phosphoric acid. Thus, the Multibond system may be a candidate for bonding orthodontic brackets with the advantage of minimizing enamel loss. (*Angle Orthod* 2003;73:702–709.)

Key Words: Self-etching primer; Shear bond strength; Adhesive remnant index; Phosphoric acid etching; Resin cement

INTRODUCTION

Direct bonding of orthodontic brackets is now routinely performed for esthetic reasons. The direct bonding adhesives provide clinically acceptable bond strengths. Most of the manufactures recommend phosphoric acid etching for resin adhesives, and orthodontists commonly use the acid-etch bonding technique when attaching brackets to the enamel surface.[1–3]

However, the phosphoric acid etching technique requires rinsing and drying the tooth after application of the etching agents. The etching procedure is sometimes troublesome, and there is a risk of contamination during the etching process in orthodontic clinics. Moreover, phosphoric acid etching has been blamed for decalcification and the development of white spot lesions around bonded orthodontic appliances.[4,5] Mechanical damage to the enamel during debonding and removal of the remaining resin after acid etching has been reported.[6–8]

In conservative dentistry, self-etching primers are being used more frequently to replace phosphoric acid etching in composite resin restorations, and their efficacy regarding adhesion to dentin and enamel has been reported.[9–12] Self-etching primers function as both an etching agent and a primer. Rinsing of the enamel after application of the self-

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Accepted: January 2003. Submitted: November 2002.

TABLE 1. Materials Used in This Study

Material	Component	Composition[a]	Batch No.	Manufacturer
Superbond C&B	Etching agent	65 wt% Phosphoric acid	2005-02	Sun Medical Co Ltd, Shiga, Japan
	Polymer powder	Polymethyl methacrylate	2005-03	
	Monomer liquid	4-META, MMA	2004-07	
	Catalyst	Partly oxidized TBB	2004-01	
Multibond	Primer A	Phosphate monomer, acetone	105	Tokuyama Dental Corp, Tokyo, Japan
	Primer B	Water, acetone, borate catalyst	295	
	Polymer powder	Polymethyl methacrylate, benzoyl peroxide	504	
	Monomer liquid	MAC-10, MMA, amine, acrylate monomer	507	

[a] 4-META indicates 4-methacryloloxyethyl trimellitate anhydride; MMA, methyl methacrylate; TBB, tri-*n*-butyl borane; MAC-10, 11-methacryloxy-1,1-undecanedicarboxylic acid.

etching primer is not required. Thus, the use of a self-etching primer reduces the number of clinical steps and saves clinical operation time because separate acid-etching and water-rinsing steps are eliminated and the application requires simply drying with air.

Superbond C&B (Sunmedical Co Ltd, Shiga, Japan) is a unique methyl methacrylate (MMA)-based adhesive resin cement that has been widely used for bonding orthodontic brackets and has earned an exceptional reputation for strong bonding.[13–15] This resin cement is also known as C&B-Metabond (Parkell Inc, Farmingdale, NY, USA) in North America. Superbond C&B consists of polymer powder, monomer liquid, polymerization catalyst, and a phosphoric acid etchant. Tight bonding of orthodontic brackets to the enamel is achieved by 65 wt% phosphoric acid etching. On the other hand, a new type of MMA-based resin cement with a self-etching primer named Multibond (Tokuyama Dental Corp, Tokyo, Japan) has been developed recently. Multibond is also known by the brand name of M-Bond (J. Morita, USA Inc, Irvine, Calif) in North America. This resin cement consists of polymer powder, monomer liquid, and a self-etching primer instead of the phosphoric acid etchant.

The purpose of this study was to determine the effects of the new MMA-based resin cement with a self-etching primer Multibond on the shear bond strength of orthodontic brackets as compared with the shear bond strength of orthodontic brackets bonded with Superbond C&B. The surface appearances of teeth after phosphoric acid etching or self-etching priming were also observed by field-emission scanning electron microscopy (FE-SEM).

MATERIALS AND METHODS

Experimental protocols

Seventy-two freshly extracted bovine incisors were used in this study. They were randomly allocated to four groups of 18 teeth in each group. The roots of the teeth were cut off, leaving the crowns to be embedded. The teeth were embedded in acrylic resin with the buccal surfaces available for bonding. After curing the acrylic resin, the teeth surfaces to be bonded were cleansed and polished with pumice and rubber prophylactic cups for 10 seconds.

Metal orthodontic brackets (stainless steel, Standard Edgewise 100-1100, Dentsply-Sankin K.K., Tokyo, Japan) and plastic brackets (polycarbonate with glass fiber, Clear bracket, Twin Standard 150-1100, Dentsply-Sankin K.K., Tokyo, Japan) were used in this study. The average bracket surface area was determined to be 9.64 mm^2 for metal brackets and 11.29 mm^2 for plastic brackets. The metal or plastic brackets were bonded to teeth with Superbond C&B or Multibond, according to the procedures described below. The materials used in this study are listed in Table 1.

Superbond C&B For Metal or Plastic Bracket Bonding

The teeth were etched with 65 wt% phosphoric acid for 30 seconds, washed for 20 seconds, and air dried. The catalyst of partly oxidized tri-*n*-butyl borane (TBB) was added to the monomer liquid to prepare an activated monomer liquid. The polymer powder and activated monomer liquid were mixed and used to bond the metal or plastic brackets to the enamel using the brush-dip technique. The monomer mixture of 4-methacryloloxyethyl trimellitate anhydride and MMA was polymerized by partly oxidized TBB initiator.

Multibond for metal or plastic bracket bonding

Primer A and primer B were mixed to form a self-etching primer containing phosphoric ester methacrylate, acetone, water, and a borate catalyst. The primer was placed on the enamel for 30 seconds. Excessive primer solution was evaporated using compressed air. Then the metal or plastic brackets were bonded to the enamel by a mixture of the polymer powder and monomer liquid.

Each bracket was subjected to a 300-g force, according to reports by Bishara et al,[16,17] 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

TABLE 2. Shear Bond Strengths Between Brackets and Enamel

	Resin cement					
	Superbond C&B			Multibond		
Bracket	Mean[a]	SD[b]	Range	Mean	SD	Range
Metal bracket	18.9 A, a	4.5	11.8–28.3	14.6 B, a	3.6	9.2–19.3
Plastic bracket	13.1 A, b	4.3	7.3–21.5	10.6 B, c	3.2	6.6–16.5

[a] Mean values with the same superscripts are significantly different ($P > .05$). Uppercase letters indicate the comparison of brackets within the same resin cement, and lowercase letters indicate the comparison of cements within the same bracket. Significant differences were found between metal brackets and plastic brackets in the Superbond C&B group (A vs A, $P < .05$) and in the Multibond group (B vs B, $P < .05$) and between Superbond C&B and Multibond in the metal bracket group (a vs a, $P < .05$). No significant difference was found between Superbond C&B and Multibond in the plastic bracket group (b vs c, $P > .05$).

[b] SD indicates standard deviation.

method,[18,19] using a testing machine (TCM-500CR, Shinkoh, Tokyo, Japan) at a cross-head speed of two mm/min.

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).[20] The amount of residual material adhering to the enamel surface was scored according to the method reported by Oesterle et al.[21] The ARI score takes values from 0 to 3—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.

A complementary test of bonding to the human enamel was also performed. Due to the limited number of human enamel samples, the shear bond strength of metal brackets bonded with Superbond C&B was examined by the same method described above. Ten human enamel samples were embedded in acrylic resin. The surface of the human enamel was polished with pumice and rubber prophylaxis for 10 seconds. After 30-second etching with phosphoric acid, the etched surface was washed and dried. Metal brackets were bonded with Superbond C&B, and the shear bond strengths were measured after immersion in water at 37°C for 24 hours.

Statistical analysis

Eighteen specimens were tested for each procedure. The bond strengths were analyzed using two-way analysis of variance (ANOVA) and Scheffe's test for multiple comparisons of the means. The chi-square (χ^2) test was used to determine significant differences in the ARI scores among the four procedures. Significance for all statistical tests was predetermined at $P < .05$. In case of a significant difference in χ^2 test, complementary tests were preformed to ascertain differences between groups.

FE-SEM observation

The bovine enamel surfaces were cleansed and then polished with pumice and rubber prophylactic cups as described above. In one specimen, the bovine 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.[15]

In another specimen, the tooth surface was treated with a mixture of primer A and primer B (self-etching primer solution) of Multibond for 30 seconds. Excess solution was then evaporated using compressed air. The specimen was also dehydrated, dried, and ion coated using the same method described above.

The surface appearances of the phosphoric acid–etched and self-etch–primed tooth specimens were observed using a FE-SEM (JSM-6340F, JEOL, Tokyo, Japan). The appearance of the enamel surface polished with pumice and rubber prophylactic cups was also observed.

RESULTS

Comparison of shear bond strengths

The results of the shear bond strength measurements are listed in Table 2. Two-way ANOVA showed significant differences in bond strengths between the two types of resin cement ($F = 11.512$, $P < .05$), and between the two types of brackets ($F = 23.894$, $P < .05$). No two-way interactions were found for the types of resin cements and the types of brackets ($P > .05$).

The mean shear bond strength of metal orthodontic brackets was significantly greater than that of plastic brackets when bonded with Superbond C&B or with Multibond resin cement ($P < .05$). Metal brackets bonded with Superbond C&B and those bonded with Multibond differed significantly in shear bond strength ($P < .05$), but there was no significant difference in bond strength between plastic brackets bonded with Superbond C&B and those bonded with Multibond ($P > .05$).

The complementary bonding test to the human enamel showed a mean shear bond strength of 20.8 ± 4.9 MPa when metal bracket was bonded to phosphoric acid–etched enamel with Superbond C&B. This value was comparable with that of the bovine enamel.

Comparison of ARI

The results of ARI scores are shown in Table 3. Chi-square test showed significant difference in ARI score among the four procedures ($\chi^2 = 36.147$, $P < .0001$). Complementary tests showed a significant difference between metal brackets bonded with Multibond and the other three groups ($P < .001$). A significant difference was also found between metal brackets bonded with Superbond C&B and plastic brackets bonded with Multibond ($P = .0339$).

FE-SEM observation

Figure 1 shows the FE-SEM micrographs of enamel surfaces that have been (1) polished, (2) etched with phosphoric acid, or (3) treated with the self-etching primer of Multibond.

After cleansing and polishing, smooth and roughened areas were present on the enamel surface. The smooth area was covered with organic materials derived from saliva, and minute focal holes[22] were observed on the roughened surface (Figure 1a, arrow). Focal holes are distinctly demarcated holes with a depth varying from fractions of a micrometer to 10 μm or occasionally greater.

Phosphoric acid etching produced a roughened enamel surface, but the dissolution pattern was different from results described previously.[19] There was no distinct dissolution of enamel prisms or enamel peripheries. The enamel surface was a finely roughened surface with a random arrangement of enamel crystals.

In the FE-SEM micrograph of an enamel surface after treatment with Multibond self-etching primer (Figure 1c), 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. The surface was covered with some organic materials, and no enamel crystals were observed.

DISCUSSION

In the orthodontic field, the efficacy of using self-etching primers for the bonding of orthodontic brackets has been reported. Bishara et al[23,24] reported that an acidic self-etching primer containing phenyl-P provided clinically acceptable shear bond forces when used with a highly filled composite adhesive (Panavia 21, Kuraray Medical Inc, Tokyo, Japan) but did not give sufficient bond strength when used with a lightly filled composite adhesive (Clearfil Liner Bond 2, Kuraray Medical Inc) or the traditional composite resin adhesive Transbond XT (3M Unitek, Monrovia, Calif). The use of a newly developed self-etching primer Prompt L-Pop (ESPE Dental AG, Seefeld, Germany) resulted in clinically acceptable bond strengths when used with Transbond XT.[16] The effectiveness of fluoride-releasing self-etch acidic primers on the shear bond strength of orthodontic brackets was also evaluated.[17] Yamada et al[19] reported that a self-etching primer containing MDP (10-methacryloyloxy-decamethylene phosphoric acid) gave a significantly lower bond strength than phosphoric acid etching when used with composite resin adhesive, but the same self-etching primer showed bond strength comparable with that of poly(acrylic acid) etching when used with a resin-modified glass ionomer cement. More research is needed to identify an effective orthodontic self-etching primer bonding system.

The newly introduced Multibond resin cement uses a self-etching primer instead of phosphoric acid etching. Moreover, the addition of the polymerization initiator into the monomer liquid is not required. The clinical process and handling procedures of Multibond are improved when compared with Superbond C&B.

Phosphoric acid etching produces a roughened enamel surface by dissolving the hydroxyl apatite of the enamel and forming enamel resin tags. Although the enamel-etching technique is a useful and accepted orthodontic procedure for bonding orthodontic brackets, there is a need to improve this method, ie, to maintain clinically useful bond strength while minimizing the amount of enamel loss. In this study, FE-SEM observations revealed that the Multibond self-etching primer produced less enamel dissolution when compared with phosphoric acid etching, as was also reported for other self-etching primers,[19] and that the residual resin of the self-etching primer was present on the enamel surface. Pashley and Tay[25] have recently developed a new method for the preparation of etched enamel samples for SEM where the remaining resin components are dissolved by sonicating the samples in absolute ethanol after dehydrating in an ascending series of ethanol. The present samples for FE-SEM observation were prepared without the sonication step, which accounted for the observation of residual resin component. A comparison of two methods, with and without sonication, should be further investigated. But according to this study, enamel loss may be reduced using Multibond self-etching primer.

The shear bond strength of metal orthodontic brackets bonded with Multibond was significantly lower than that of metal brackets bonded with Superbond C&B. Mechanical interlocking of cured resin formed on the roughened enamel surface is the main contribution to shear bond strength of orthodontic brackets bonded with composite resin adhesive.[26] Recent studies of conservative dentistry have suggested that self-etching primers with lower decalcifying ability are less effective than phosphoric acid etching when used to bond ground enamel with a thick smear layer or an intact unground enamel.[25,27] Yamada et al[19] also found that the shear bond strengths of orthodontic brackets after self-

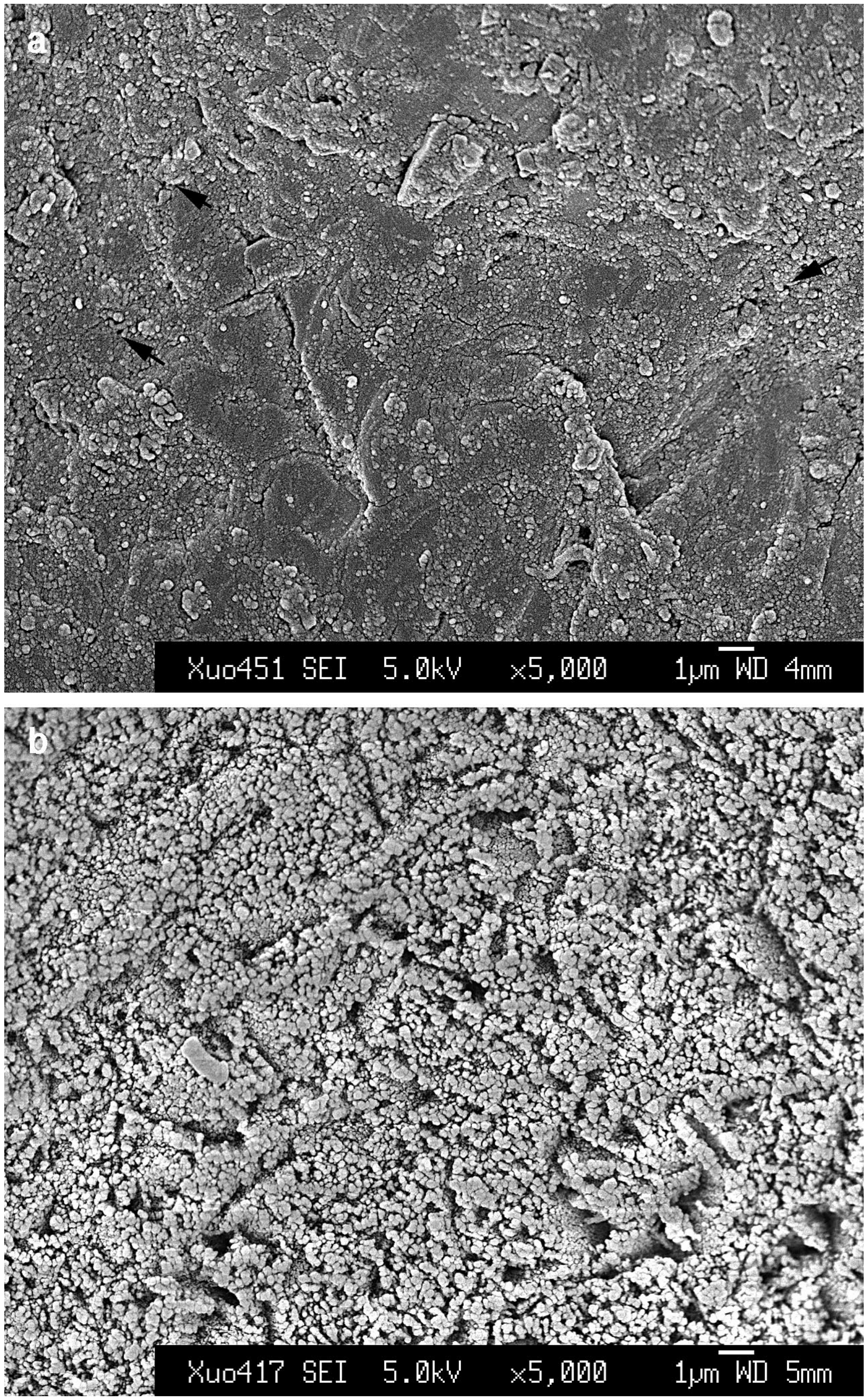

FIGURE 1. Field-emission scanning electron microscopy micrographs of enamel surfaces. (a) Cleansed surface: minute focal holes (arrow) are observed. (b) Phosphoric acid–etched surface: roughened enamel surfaces are evident. (c) Self-etching primed surface: the enamel surface appears almost flat and the presence of minute focal holes (arrow) can also be identified.

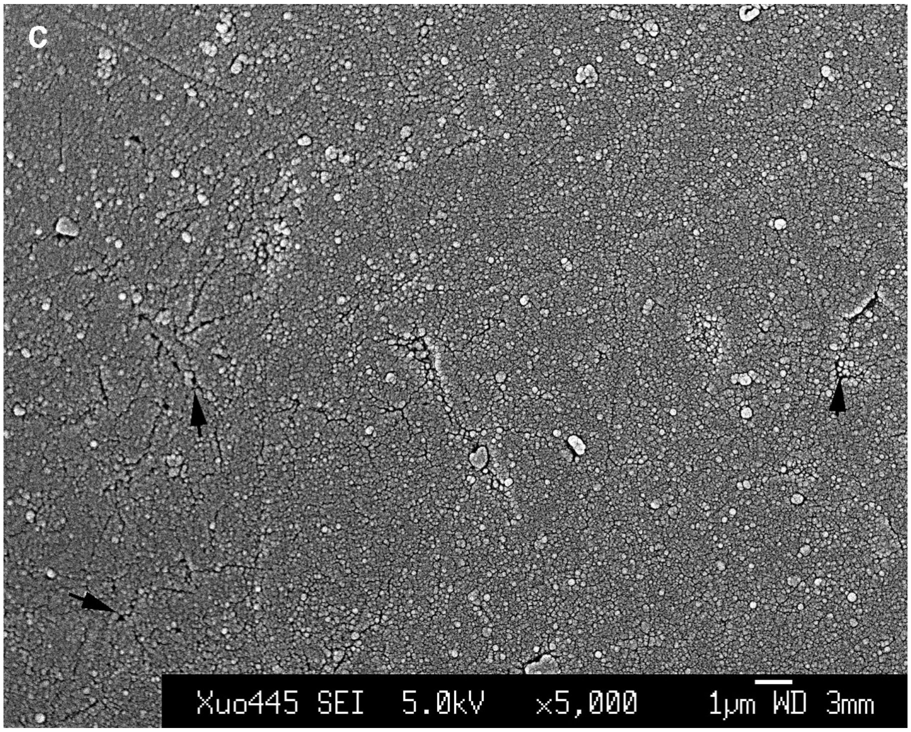

FIGURE 1. Continued.

TABLE 3. Frequency Distribution of the Adhesive Remnant Index (ARI)[a]

	ARI Scores				
	0	1	2	3	*n*
Metal bracket bonded with Superbond C&B	14	4	—	—	18
Plastic bracket bonded with Superbond C&B	17	1	—	—	18
Metal bracket bonded with Multibond	4	12	2	—	18
Plastic bracket bonded with Multibond	18	—	—	—	18

[a] χ^2 value = 36.147, $P < .0001$. A significant difference was detected between metal brackets bonded with Multibond and the other three groups ($P < .001$). A significant difference was also found between metal brackets bonded with Superbond C&B and plastic brackets bonded with Multibond ($P = .0339$).

etching primer treatment were significantly lower than the shear bond strengths of those after phosphoric acid etching. The results of this study support the results of Yamada et al.[19]

However, there were no significant differences in shear bond strength between Multibond and Superbond C&B when they were used to bond plastic brackets to the enamel, and the shear bond strength for metal brackets bonded with Multibond was comparable with that of plastic brackets bonded with Superbond C&B. Moreover, shear bond strengths of about 10 MPa were obtained for plastic brackets bonded with Multibond. This value is higher than the shear bond strength of metal brackets bonded with resin-modified glass ionomer cement that has the clinical advantage of fluoride release, radio-opacity, and low thermal conductivity.[19] Bishara et al[24] reported that a shear bond strength of 7 MPa to the enamel was clinically acceptable for bonding to the enamel surface. These data suggest that the use of Multibond may be clinically acceptable for bonding metal or plastic orthodontic brackets.

In this study, bovine teeth were used as a substitute for human teeth because of the morphological similarity between bovine and human enamel and the difficulties in obtaining human teeth. Shinha et al[28] and Komori and Ishikawa[29] evaluated the bond strength of light-cured glass ionomer cements or light-cured composite resin adhesive using bovine enamel. However, the results obtained using bovine teeth sometimes cannot be extrapolated to human teeth. To verify the applicability of the present findings using bovine teeth to human teeth, we performed a complementary test on the human enamel. Our results revealed comparable bond strengths in human and bovine samples when metal brackets were bonded to phosphoric acid–etched enamel using Superbond C&B. The final evaluation of the efficacy of Multibond self-etching primer for clinical usefulness should be conducted using human teeth.

The appearances of the enamel surface after treatment with the phosphoric acid etchant of Superbond C&B are different from those of results of Yamada et al.[19] This was due to a difference in the concentration of the phosphoric acid, which was 40% in previous etching agents and 65% in the etchant of Superbond C&B. Higher concentrations of phosphoric acid remove less superficial enamel during the etching procedures.[30]

Both Multibond and Superbond C&B gave significantly lower bond strength with plastic brackets than with metal brackets. Liu et al[31] reported significantly lower shear bond strengths of plastic brackets compared with metal brackets when bonded to human premolars. Gang et al[32] found that sandblasting and silane coupling treatment of plastic brackets improved the bond strengths to the human enamel. Some surface treatment may be needed to improve the bond strength of plastic brackets to the enamel.

The findings in the ARI scores is noteworthy. Significant differences in ARI scores were observed among the four procedures. There was a tendency of more residual resin cement remaining on the tooth when Multibond was used to bond metal brackets. Therefore, use of Multibond in bonding metal orthodontic brackets to the enamel may have a lower risk of enamel fracture at the time of debonding, but perhaps more clinical time is required to remove the remaining adhesive from the enamel after debonding. Further studies should be performed to determine the effectiveness of Multibond under simulated clinical conditions. Furthermore, not all self-etching primers perform equally, and comparative studies with other products are necessary.

CONCLUSIONS

The present findings indicate that a newly introduced MMA-based resin cement with self-etching primer Multibond has a potential for clinical use in bonding metal or plastic orthodontic brackets to teeth, with the advantage of minimizing the amount of enamel loss and reducing the number of clinical steps during bonding.

ACKNOWLEDGMENTS

A part of this study was supported by a grant from the Ministry of Education, Culture, Sports, Science and Technology to promote 2001—Multidisciplinary Research Project (in 2001–2005). We express our gratitude to Sun Medical and Tokuyama Dental for supplying the adhesive resins for this study.

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