The use of easily debondable orthodontic adhesives with ceramic brackets

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We experimentally produced an easily debondable orthodontic adhesive (EDA) containing heat-expandable microcapsules. The purpose of this *in vitro* study was to evaluate the best debondable condition when EDA was used for ceramic brackets. Shear bond strengths were measured before and after heating and were compared statistically. Temperatures of the bracket base and pulp wall were also examined during heating. Bond strengths of EDA containing 30 wt% and 40 wt% heat-expandable microcapsules were 13.4 and 12.9 MPa, respectively and decreased significantly to 3.8 and 3.7 MPa, respectively, after heating. The temperature of the pulp wall increased 1.8–3.6°C after heating, less than that required to induce pulp damage. Based on the results, we conclude that heating for 8 s during debonding of ceramic brackets bonded using EDA containing 40 wt% heat-expandable microcapsules is the most effective and safest method for the enamel and pulp.

Keywords: Bond strength, Debonding, Microcapsule, Orthodontic adhesive

INTRODUCTION

Adhesives used with orthodontic brackets require not only sufficient bond strength to withstand mastication and orthodontic forces, but also sufficient moderation to allow debonding, because excessive bond strength usually results in intense pain and/or enamel fractures. Retief¹⁾ stated that enamel fractures can occur with bond strengths as low as 13.5 MPa. Although wide ranges of bond strengths using ceramic brackets have been reported, some investigators^{2–4)} have reported forces in excess of 29.4 MPa. Redd and Shivapuja⁵⁾ also reported that enamel damage was more likely to result from debonding ceramic brackets than from debonding stainless-steel brackets, because most bond failures associated with enamel damage were generated at the enamel/adhesive interface. Additionally, ceramic brackets are more difficult to remove from the teeth because the wings of the bracket base may break and remain in place.

Several attempts to solve problems such as enamel surface damage during debonding have been reported. Boyer *et al.*⁶⁾ tested ultrasonic debonding, Brouns *et al.*⁷⁾ examined thermal debonding, and other researchers^{8–11)} have explored the use of laser devices. Each of these methods has associated problems, such as lengthy treatment times, excessive heating, and expensive devices. Tsuruoka *et al.*¹²⁾ developed an easily debondable adhesive (EDA) containing heat-expandable microcapsules and demonstrated that bond strengths decreased by about one-third after 8–10 s of heating when the EDA was used with stainless-steel brackets. However, the heat conductivity of a ceramic bracket, which is generally thicker and larger than a stainless-steel bracket to avoid fracture, would differ from that of

a stainless-steel bracket. The size and design of the bracket influence heat conductivity. Ceramic brackets are also more difficult to remove from the teeth, as described above. Thus, the application of EDA to ceramic brackets would be more reasonable than application to stainless-steel brackets. The purpose of this *in vitro* study was to evaluate the best debondable conditions when EDA and ceramic brackets were used.

MATERIALS AND METHODS

Easily debondable materials

The base bonding material used in this experiment was 4-META/MMA-TBB resin adhesive (Orthomite SuperBond, Sun Medical, Moriyama, Japan), which has polymer and monomer components. To prepare the EDA powder, we mixed thermo-expandable microcapsules (Matsumoto Microsphere F-36D, Matsumoto Yushi-Seiyaku, Osaka, Japan) into the polymer powder, according to the method of Tsuruoka *et al.*¹²⁾

Measurement of temperature at the adherent surface of the bracket base

The brackets were heated with a heater (Ultra Five Heater, Hakko, Nagano, Japan) set at 300°C. Bracket base temperatures of the ceramic (Crystalline IV, Tomy International, Tokyo, Japan) and stainless-steel (New DynaLock, 3M Unitek, Monrovia, CA, USA) brackets were measured with a K-type thermocouple (Okazaki Manufacturing, Kobe, Japan) (Fig. 1). Bracket thicknesses were 2.15 mm (ceramic) and 1.80 mm (stainless steel).

Tooth specimens and bracket bonding

Freshly extracted bovine permanent mandibular incisors

Fig. 1 Temperature measurement at the adherent surface of the bracket base during heating. a: tip of heater, b: bracket, c: sensor head of the K-type thermocouple.

($n=112$) were collected from a slaughterhouse. The criteria for tooth selection were intact labial enamel (lacking fractures caused during extraction) and absence of caries. The teeth were divided randomly into eight groups of eight specimens each, corresponding to the number of variables tested.

Soft tissues were removed from each tooth. After separating the crown from the root, the pulp was extirpated and the crown was stored in distilled water until further use. The crown was then embedded in self-curing acrylic resin (Tray Resin, Shofu, Kyoto, Japan) to facilitate its placement in the testing machine. The labial surface of each crown was flattened to facilitate the application of shearing force and polished with waterproof silicon-carbide papers (#400, #600), as described previously^{13–15}. The enamel surfaces were rinsed with water and dried with an oil-free airstream.

One operator bonded 112 ceramic maxillary central incisor brackets with a 0.018-inch slot as follows. The enamel surface was etched for 30 s, rinsed, and dried according to the manufacturer's instructions. Adhesive was applied and the bracket was firmly seated. Excess adhesive was then removed with a dental probe without disturbing the bracket, and the specimens were immersed in distilled water at 37°C for 24 h. The average bracket base surface area was 12.58 mm².

Brackets in the heating groups were heated from above with the heater using the optimal heating time (8 s) recommended by Tsuruoka *et al.*¹² for stainless-steel brackets. Brackets in the heating groups were also bonded using optimal concentrations of EDA (30 wt%, 40 wt%) suggested by Tsuruoka *et al.*¹² and heated for 10 s and 12 s to explore the appropriate heating times for ceramic brackets. After heating, each bracket was cooled immediately with water (Fig. 2).

Fig. 2 Heating of a ceramic bracket. After bonding with easily debondable adhesive, the bracket was heated from above for 8, 10, and 12 s. a: tip of heater, b: ceramic bracket.

Shear bond strength measurement

All samples were tested in shear mode on a universal testing machine (5567, Instron, Norwood, MA, USA). Specimens ($n=8$) were secured in the lower jaw of the machine with the bracket base parallel to the shear force, then stressed in an occlusogingival direction at a crosshead speed of 1 mm/min.

Assessment of residual adhesive

After the measurement of shear bond strength, each specimen was examined under an optical microscope (SZ-3003; As one, Osaka, Japan) at 15× magnification to identify the fracture pattern of the bonded surface. The residual adhesive on each tooth was assessed using the adhesive remnant index (ARI)¹⁶, as follows: 0, no adhesive remaining; 1, <50% adhesive remaining; 2, ≥50% adhesive remaining; 3, all adhesive remaining with a distinct impression of the bracket base.

Measurement of temperature increase in the pulp chamber

To evaluate the temperature increase in the pulp chamber during heating, we used four fresh human permanent first premolars that had been extracted during orthodontic treatment. The protocol for this experiment was reviewed and approved by the Ethics Committee of the Department of Dentistry, Nihon University. The extracted teeth were stored in distilled water at 4°C until use. Brackets were bonded to the teeth with bonding material containing 40 wt% microcapsules. Each tooth was drilled with an air turbine from the lingual cemento-enamel junction toward the labial bonded bracket. The sensor head (0.5 mm diameter) of a K-type thermocouple was then placed in contact with the inner surface of the pulp wall, facing the labial surface where the bracket was bonded. The average distance from the labial surface to the pulp wall was 3.62 mm. After heating for 8, 10, and 12 s, the heated bracket was cooled immediately with water. The

temperature of the inner surface of the pulp wall was measured at a room temperature of $24\pm1^{\circ}\text{C}$; this measurement was performed four times for each of the three heating times (Fig. 3).

Statistical analyses

To calculate shear bond strength, statistical analyses were performed on all data, and descriptive statistics, including the mean, standard deviation (SD), minimum (min), median, and maximum (max), were calculated for each group using the software (SPSS, SPSS Inc., Chicago, IL, USA). Differences among groups were sought using the Kruskal-Wallis H-test, followed by the Mann-Whitney U-test with Bonferroni correction. Differences in temperature at the bases of the ceramic and stainless-steel brackets were compared using the unpaired *t*-test. Differences in the distribution of ARI scores were analyzed using the chi-squared test with Yates' correction. *P* values less than 0.05 were considered to indicate statistical significance for all tests.

Fig. 3 Measurement of pulp temperature increase. The tip of the thermocouple was placed in contact with the inner surface of the pulp wall, which faced the heated bracket. After heating, the bracket and tooth were cooled immediately with water.
a: tip of heater, b: ceramic bracket, c: sensor head of the K-type thermocouple.

RESULTS

Figure 4 shows the time course of temperatures at the adherent surfaces of the bracket bases after heating (300°C). The temperature at which microcapsules began to expand was from 8 s in both brackets. No significant difference in temperature was detected between ceramic and stainless-steel brackets at any time point.

Tables 1 and 2 show the descriptive statistics for the shear bond strength of each group before and after 8 s of heating, and Table 3 shows ARI scoring results. No significant difference in bond strength before heating was observed among 0 wt% and other concentrations of EDA. ARI scores did not differ significantly among concentrations. Bond strengths of EDA containing 20, 30, and 40 wt% microcapsules were significantly less after heating for 8 s than before heating. The bond strength of EDA containing 20 wt% microcapsules decreased to 70% of the bond strength before heating, and those of EDA containing 30 and 40 wt% microcapsules decreased to 30–40% of bond strength before heating.

The median bond strength of 30 wt% EDA was 13.4 MPa in specimens that were not heated. This value

Fig. 4 Time course of temperature at the adherent surface of the bracket base during heating. No significant difference was observed between brackets at any time point.

Table 1 Shear bond strengths at each concentration without heating

Microcapsule concentration (wt%)	Shear bond strength (MPa)					
	Median	Min	Max	Mean	(SD)	Sig
0	18.2	11.7	21.8	18.1	(3.4)	—
10	15.9	12.2	23.0	16.4	(3.2)	n.s.
20	15.3	10.9	16.4	14.9	(1.8)	n.s.
30	13.4	11.4	17.2	13.8	(1.9)	n.s.
40	12.9	10.3	14.6	12.7	(1.3)	n.s.

SD: standard deviation; Min: minimum value; Max: maximum value.

Sig: n.s. indicates no significant difference in bond strength between each concentration and 0% EDA.

Table 2 Shear bond strengths in each concentration after 8 seconds of heating

Microcapsule concentration (wt%)	Shear bond strength (MPa)				
	Median	Min	Max	Mean (SD)	Sig
0	16.7	12.4	19.7	16.4 (2.7)	n.s.
10	14.6	12.0	16.8	14.4 (1.5)	n.s.
20	11.2	7.7	13.9	10.8 (2.2)	*
30	6.5	4.7	7.9	6.3 (1.1)	*
40	4.6	2.4	6.3	4.5 (1.6)	*

SD: standard deviation; Min: minimum value; Max: maximum value.

Sig: * indicates that the bond strength of each concentration was significantly ($p<0.05$) less than that of EDA in the same concentration before heating. n.s. indicates no significant difference in bond strength of the same concentration before and after heating.

Table 3 Adhesive remnant index (ARI) of each adhesive

Heating time (seconds)	Microcapsules concentration (wt%)																
		0			10			20			30			40			
		Score	0	1	2	3	0	1	2	3	0	1	2	3	0	1	2
0		3	1	2	2	4	0	2	2	3	1	3	0	7	1	0	0
8		1	2	1	4	4	1	2	1	5	0	0	3	8	0	0	0

ARI scores: 0, no adhesive left on tooth surface; 1, less than 50% of adhesive left on tooth surface; 2, more than 50% of adhesive left on tooth surface; 3, all adhesive left on the tooth surface.

No significant difference was detected among concentrations.

Table 4 Shear bond strengths of each concentration

Microcapsule concentration (wt%)	Heating time (seconds)	Shear bond strength (MPa)				
		Median	Min	Max	Mean (SD)	Sig
30	0	13.4	11.4	17.2	13.8 (1.9)	—
	8	6.5	4.7	7.9	6.3 (1.1)	*
	10	6.2	2.2	10.6	6.1 (2.9)	*
	12	3.8	2.4	8.4	4.6 (2.3)	*
40	0	12.9	10.3	14.6	12.7 (1.3)	—
	8	4.6	2.4	6.3	4.5 (1.6)	*
	10	5.4	3.9	6.1	5.2 (0.8)	*
	12	3.7	2.1	5.7	3.8 (1.5)	*

SD: standard deviation; Min: minimum value; Max: maximum value.

Sig: * indicates that the bond strength in each concentration was significantly ($p<0.05$) less than that of unheated specimens.

Table 5 Adhesive remnant index (ARI) of each adhesive

Microcapsule concentration (wt%)	Heating time (seconds) Score	0				8				10				12			
		0	1	2	3	0	1	2	3	0	1	2	3	0	1	2	3
30*		7	1	0	0	8	0	0	0	0	0	3	5	0	0	3	5
40*		7	1	0	0	5	3	0	0	0	1	2	5	0	0	1	7

ARI scores: 0, no adhesive left on tooth surface; 1, less than 50% of adhesive left on tooth surface; 2, more than 50% of adhesive left on tooth surface; 3, all adhesive left on the tooth surface.

* indicates significant differences ($p<0.05$) among heating times.

Fig. 5 Time course of temperature increase at each heating time. After each heating time, the bracket and tooth were cooled immediately with water (15°C). All curves peaked approximately 7 s after heating, and temperatures decreased gradually thereafter.

decreased to 6.5 MPa after 8 s of heating, 6.2 MPa after 10 s, and 3.8 MPa after 12 s of heating (Table 4). The median bond strength of 40 wt% EDA was 12.9 MPa in specimens that were not heated, 4.6 MPa after 8 s of heating, 5.4 MPa after 10 s, and 3.7 MPa after 12 s of heating (Table 4). ARI scores differed significantly among heating times (Table 5).

The time course of the temperature increase for each heating time is shown in Fig. 5. The temperature increase of the pulp walls was 1.8–3.6°C after heating for 8, 10, and 12 s.

DISCUSSION

We used bovine teeth as a bonded substrate in this experiment. Bovine teeth have been used in many studies because a large and uniform bonded enamel surface can be achieved readily^{17–21}. However, the bond strength of a bracket on a bovine tooth may be somewhat lower than that on a human tooth²². Tsuruoka *et al.*¹² reported mean bond strengths of 14.5 MPa (30 wt%) and 13.1 MPa (40 wt%) for unheated EDA with stainless-steel brackets under experimental conditions similar to those of the present study. We obtained mean bond strengths of 13.8 MPa (30 wt%) and 12.7 MPa (40 wt%) for unheated EDA with ceramic brackets, and found no significant difference between values for stainless-steel and ceramic brackets. Reynolds²³ reported that a minimum bond strength of 6–8 MPa is adequate for most clinical orthodontic needs, because this strength is sufficient to withstand masticatory and orthodontic forces. The bond strengths we measured without heating would, therefore, be strong enough for use in orthodontic practice even if the bond strength of a bovine tooth may be somewhat lower than that of a human tooth. Bond

strengths after heating in 20, 30 and 40 wt% EDA were also low enough to avoid obvious enamel fractures (13.5 MPa)¹, and those of 40 wt% EDA were less than 6 MPa, the minimum bond strength suggested by Reynolds²³; however, those of 30 wt% EDA after heating for 8 and 10 s were slightly higher. Thus, 40 wt% EDA would likely be better than of 30 wt% EDA for ceramic bracket use.

Tsuruoka *et al.*¹² found cohesive failure in the adhesive and interfacial peeling between the adhesive and bracket base with the use of 40 wt% EDA. In this study, we detected no significant difference in ARI scores among microcapsule concentrations, but significant differences among heating times were found. Thus, the use of an appropriate heating time will likely ensure effective microcapsule expansion; more residue remained on the surface of 30 and 40 wt% EDA specimens heated for 10 and 12 s than on unheated specimens, and failures occurred between the adhesive and bracket base, as reported by Tsuruoka *et al.*¹².

Several studies have examined temperatures during thermal debonding^{7,24–26}. Brouns *et al.*⁷ recorded temperatures exceeding 200°C at the base of a ceramic bracket using two types of electrothermal debonding devices without cooling. The thermo-expandable microcapsules (particle sizes: 10–20 µm) used in this study expand four to five times in diameter when heated. The expanding agents they contain are volatile organic compounds, such as isobutene, pentane, petroleum ether, hexane, heptane, low-boiling-point halogenated hydrocarbon solvent, and methylsilane. The capsules are covered with a membrane polymer that consists of vinylidene chloride, acrylic acid ester, and methacrylic acid ester. When the microcapsules are heated above the softening point of the membrane polymer (80°C) and the vapor pressure of the expanding agent rises, they expand 50 to 100 times in volume. In this study, the temperature at the adherent surface of the bracket base reached the softening point at 8 s in ceramic and stainless-steel brackets, and no significant difference in temperature was detected between brackets. Thus, 8 s is an appropriate heating time associated with decreased bond strength in ceramic and stainless-steel brackets¹², although variation may result from differences in bracket thickness and shape.

Heating at 300°C may nociceptively stimulate the dental pulp. However, the actual temperature on the enamel surface was around 80–110°C, and the temperature increased by 1.8–3.6°C in the pulp wall through the enamel and dentin after heating for 8, 10, and 12 s. Zach and Cohen²⁷ reported the recovery of 85% of tissue when the pulp temperature rose to 5.5°C above body temperature in macaques. Because this value exceeds the temperature increases we observed, we believe that the nociceptive stimulation would be slight and transient, and that pulp damage will not occur with this method.

CONCLUSIONS

The use of EDA powder with 40 wt% thermo-expandable microcapsules mixed into the polymer powder of 4-META/MMA-TBB resin adhesive for ceramic bracket bonding showed sufficient bond strength before heating and significantly decreased bond strength (approximately 1/3) after heating for 8 s without pulp damage. Thus, we conclude that EDA in this condition is the most useful debracketing bond material for ceramic brackets without causing pulp damage.

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