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Original

Effect of orthodontic adhesive thickness on force required by debonding pliers

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Abstract: This *in vitro* study evaluated the relationship between removal force and the thickness of three orthodontic adhesives, namely, light- and chemical-cured resin cements and a resin-modified glass ionomer cement. The thickness of each adhesive was 50, 100, 150, or 200 µm, and all adhesives were bonded on bovine incisors. Removal force was measured before (TC-0) and after 1,000 thermal cycles (TC-1000), and values were compared. At TC-0, the removal strengths for adhesive thicknesses of 50 and 100 µm were significantly lower than those for thicknesses of 150 and 200 µm (P < 0.05). At TC-1000, removal strengths for adhesive thicknesses of 50 and 100 µm were also significantly lower than those for 150 and 200 µm. Superbond Orthomite specimens showed a significant difference in removal strength between TC-0 and TC-1000 (P < 0.05) at all thicknesses. There was no significant difference in the distribution of adhesive remnant index scores at any thickness. These findings indicate that decreasing the thickness of applied orthodontic adhesive reduces the removal strength required.
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Keywords: removal strength; thickness; residual adhesive; thermal cycling.

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Introduction

The maximum bond strength of orthodontic adhesives is around 25 MPa (1-4), although bond strength depends on bonding conditions. Many reports (5-8) cite a study by Reynolds, which suggested that a bond strength of 6-8 MPa is required. Current adhesives exceed that strength and can withstand orthodontic treatment. However, because strongly bonded adhesive is ripped from the enamel surface with a bracket after treatment, enamel damage is a concern.

When a bonded bracket is removed, failure can occur at one of three interfaces: between the adhesive and enamel surface, within the bonding material itself, or between the adhesive cement and bracket. However, the interface between the adhesive cement and bracket is the usual site of failure when brackets are removed (9). Therefore, although the remaining adhesive must be removed, insufficient care during the removal process can damage the enamel surface. Additionally, the use of adhesive-removal pliers to remove adhesives may cause pain or enamel damage because of the large load applied to the teeth (9).

Table 1 Materials used in this study

Adhesive system	Chemical composition	Lot no.	Manufacturer
Transbond XT (TX)			3M Unitek
Etching gel	35% phosphoric acid	5FT	(Monrovia, CA, USA)
Light cure adhesive primer	Bis-GMA, TEGDMA	712-034	
Light cure adhesive paste	Bis-GMA, silane-treated quartz, bisphenol A bis(2-hydroxy-ethyl ether) dimethacrylate, silane-treated silica	293905	
Superbond Orthomite (SB)			Sun Medical
Red activator	65% phosphoric acid	VG4	(Moriyama, Japan)
Monomer	MMA, 4-META	EM1	
Catalyst	TBB-O	VW51F	
Polymer	PMMA	EG11	
Ortholy Glass Bond (OG)			GC
A-paste	Fluoro-alumino-silicate glass, dimethacrylate, etc	1103291	(Tokyo, Japan)
B-paste	Polyacrylic acid, silicon dioxide, distilled water, etc	1103291	
Gel conditioner	Polyacrylic acid, distilled water, silicon dioxide, glycerin, etc	0910161	

Bis-GMA: bisphenol A diglycidyl ether dimethadrylate, TEGDMA: triethylene glycol dimethacrylate, MMA: methyl methacrylate, 4-META: 4-methacryloxyethyl trimellitate anhydride, TBB-O: partially oxidized tri-*n*-butylborane, PMMA: methyl methacrylate polymer

Although grinding the remaining adhesive with a carbide bar is the optimal approach (9), it is slow and inefficient (10). Therefore, if the load applied by debonding pliers could be reduced, most of the remaining resin could be removed without pain. Mehl et al. (11) reported that the thickness of the cement film affected the retentive strength of cementation in implant-retained crowns. We hypothesized that altering the thickness of the adhesive cement layer would reduce retentive strength (ie, the removal force required). Therefore, in this *in vitro* study we evaluated the relationship between the removal force required during removal of remnant adhesive cement and the thickness of orthodontic adhesives.

Materials and Methods

Materials used

The three orthodontic adhesives used in this study were a light-cured composite resin cement (TX; Transbond XT, 3M Unitek, Monrovia, CA, USA), 4-META/MMA-TBB resin (SB; Superbond Orthomite, Sun Medical, Moriyama, Japan), and a resin-modified glass ionomer cement (OG; Ortholy Glass Bond, GC, Tokyo, Japan), as shown in Table 1. All orthodontic adhesive systems were used according to the respective manufacturer's instructions. A curing unit (Optilux 501, SDS Kerr, Danbury, CT, USA; light intensity, 800 mW/cm^2) was also used.

Tooth specimens

Freshly extracted bovine permanent mandibular incisors were collected from a slaughterhouse. The criteria for tooth selection were intact labial enamel (ie, absence of fractures caused by extraction) and absence of caries. Tooth specimens were stored at -30°C until use.

Soft tissues were removed from each tooth. After the crown was separated from the root, the pulp was extirpated and the crown was stored in distilled water at room temperature 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 force application and polished with waterproof silicon-carbide papers (#400, #600). The enamel surfaces were rinsed with water and dried in an oil-free airstream.

Surface treatment procedure

For application of TX adhesive, the enamel surface was etched with 35% phosphoric acid gel (Transbond XT Etching Gel, 3M Unitek) for 30 s, washed for 20 s, and then dried with oil-free compressed air. The bonding agent (Transbond XT Light Cure Adhesive Primer, 3M Unitek) was applied to the treated surface.

For application of SB adhesive, the enamel surface was etched with red activator for 30 s, washed for 20 s, and then dried with oil-free compressed air. A partially oxidized tri-*n*-butylborane (TBB) catalyst was added to the monomer liquid, and the polymer and activated monomer liquid were then mixed.

For application of OG adhesive, the gel conditioner was applied to the enamel surface, left for 20 s, washed for 20 s, and then dried with oil-free compressed air. The A and B pastes were then mixed for application to the enamel surface.

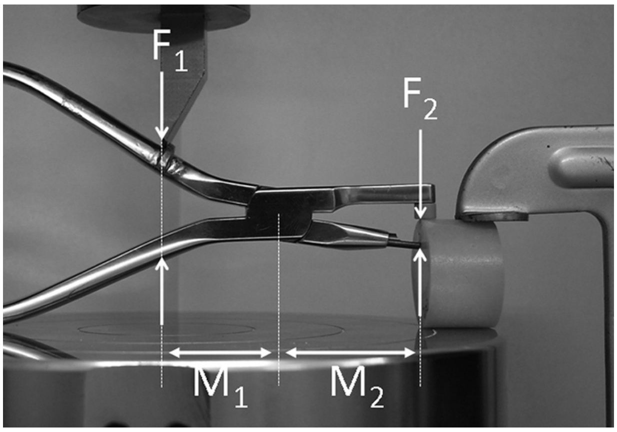

Fig. 1 Method for applying removal force.

Establishing adhesive thickness
The thickness of the adhesive layer was set to 50, 100, 150, or 200 µm for each adhesive. Masking tape that had been piled up to the required thickness and punched with a hole (diameter, 2 mm) was applied to the enamel, the surface of which had been treated as described above. The hole was filled with the respective adhesive paste and mixtures, and the outer surface was covered with a polyester matrix tape (3M ESPE, St Paul, MN, USA). Light curing was carried out for 30 s through the polyester-matrix tape, except for the SB adhesive, which was chemical-cured. After light curing and initial curing, waterproof silicon-carbide papers were used to adjust the thickness until it was as described above. Thickness was confirmed with a slide caliper (NTD12P-15, Mitutoyo, Kawasaki, Japan).

Thermal cycling
To investigate the effects of thermal changes on adhesive durability, the specimens were subjected to continuous thermal cycling for 1,000 cycles between 4°C and 60°C water baths, with a 30-s dwell time in each bath, after storing for 24 h in distilled water at 37°C.

Measurement of removal force
A universal testing machine (5567, Instron, Norwood, MA, USA) was used to measure the force required to remove the adhesive layer (Fig. 1) at a crosshead speed of 1 mm/min. Adhesives—which had been stored for 24 h in distilled water at 37°C (TC-0) or subjected to thermal cycling (TC-1000)—were removed using adhesive-removal pliers (802-1006, Tomy, Tokyo, Japan) according to the modified method of Bishara et al. (12) and Horiuchi et al. (13). The steel blade tip of the pliers was placed at the adhesive-enamel interface, and a squeezing action was applied until bond failure occurred. The relationship between the measured failure load, F_1, and the removal force, F_2, is $F_1M_1 = F_2M_2$. Thus, $F_2 = F_1M_1/M_2$, where M_1 and M_2 are the moment arms in Fig. 1. To calculate removal strength the removal force, F_2, in Newtons was divided by 3.14 mm^2 (the cross-sectional area of the adhesive bonding surface).

Assessment of residual adhesive
After calculating removal 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. Residual adhesive on each tooth was assessed using the modified adhesive remnant index (ARI), as follows: 0, no adhesive remaining; 1, <50% adhesive remaining; 2, ≥50% adhesive remaining; 3, 100% adhesive remaining (14).

Statistical analysis
All data were statistically analyzed, and descriptive statistics, including the mean and standard deviation (SD), were calculated for each group by using the SPSS software package (SPSS Inc., Chicago, IL, USA). In addition, the Kolmogorov-Smirnov and Levene tests were used to verify the normality and homogeneity of variance. Before and after each thermal cycling test, differences among groups were evaluated using the Games-Howell test. Differences between values before and after thermocycling were analyzed with Welch's *t*-test. Differences in the distribution of ARI scores were analyzed using the chi-square test. A P value less than 0.05 was considered to indicate statistical significance.

Results

The removal strengths of each adhesive are listed in Table 2. For TX, SB, and OG, removal strengths were significantly lower at thicknesses of 50 and 100 µm than at thicknesses of 150 and 200 µm ($P < 0.05$). The ARI scores for each adhesive are shown in Table 3. Chi-square analysis revealed no significant differences in ARI score distribution at any adhesive thickness. However, TX specimens with an adhesive thickness of 150 µm were more likely to have no remnant adhesive on the tooth (ARI score, 0), as compared with other thicknesses. SB specimens with adhesive thicknesses of 100 and 150 µm were also more likely to have no remnant adhesive on the tooth (ARI score, 0), as compared with other thicknesses.

The removal strengths of each adhesive after thermocycling are shown in Table 4. In the TX group, removal strengths were significantly greater at thicknesses of 150 and 200 µm than at thicknesses of 50 and 100 µm. In

Table 2 Removal strength (MPa) in relation to adhesive thickness, at 0 thermal cycles

	Thickness (µm)			
Adhesive	50	100	150	200
TX	7.6[acd]	9.4[ad]	13.3[b]	15.9[b]
	(3.2)	(2.6)	(1.2)	(4.3)
SB	8.9[ad]	9.7[a]	13.3[b]	17.4[b]
	(2.6)	(0.6)	(1.8)	(3.5)
OG	4,1[c]	4.4[c]	6.8[de]	8.0[ae]
	(0.6)	(0.8)	(0.6)	(2.4)

Identical superscript letters indicate nonsignificant differences ($P > 0.05$).
$n = 10$; parentheses indicate standard deviation

Table 3 Adhesive remnant index (ARI) scores for each adhesive

		Thickness (µm)			
		50	100	150	200
Adhesive	Score	0 1 2 3	0 1 2 3	0 1 2 3	0 1 2 3
TX		4 6 0 0	4 5 1 0	2 8 0 0	5 5 0 0
SB		7 3 0 0	5 5 0 0	4 6 0 0	8 2 0 0
OG		7 3 0 0	7 3 0 0	8 2 0 0	9 1 0 0

ARI scores: 0, no adhesive left on tooth surface; 1, <50% of adhesive left on tooth surface; 2, >50% of adhesive left on tooth surface; 3, adhesive left on tooth surface.
There was no significant difference ($P > 0.05$) in relation to thickness for any adhesive.

Table 4 Removal strength (MPa) in relation to adhesive thickness, at 1,000 thermal cycles

	Adhesive thickness (µm)			
Adhesive	50	100	150	200
TX	5.9[aegh]	8.8[bd]	12.7[c]	13.7[c]
	(1.8)	(1.5)	(1.2)	(0.9)
SB	6.1[a*]	6.7[a*]	7.9[bef*]	8.7[bf*]
	(0.6)	(0.6)	(0.3)	(1.3)
OG	3.7[g]	3.9[g]	4.7[h*]	6.5[a]
	(0.3)	(0.5)	(0.4)	(0.7)

Identical superscript letters indicate nonsignificant differences ($P > 0.05$).
Asterisks indicate a significant difference ($P < 0.05$) between 0 and 1,000 thermal cycles for the same adhesive and thickness.
$n = 10$; parentheses indicate standard deviation

Table 5 Adhesive remnant index (ARI) scores for each adhesive, at 1,000 thermocycles

		Thickness (µm)			
		50	100	150	200
Adhesive	Score	0 1 2 3	0 1 2 3	0 1 2 3	0 1 2 3
TX		6 4 0 0	6 4 0 0	5 5 0 0	4 5 1 0
SB		5 5 0 0	10 0 0 0	10 0 0 0	10 0 0 0
OG		9 1 0 0	9 1 0 0	9 1 0 0	10 0 0 0

ARI scores: 0, no adhesive left on tooth surface; 1, <50% of adhesive left on tooth surface; 2, >50% of adhesive left on tooth surface; 3, adhesive left on tooth surface.
There was no significant difference ($P > 0.05$) in relation to thickness for any adhesive.

the SB group, removal strengths were significantly lower at thicknesses of 50 and 100 µm than at 150 and 200 µm ($P < 0.05$). In the OG group, removal strengths were significantly lower at thicknesses of 50 and 100 µm than at 150 and 200 µm. For all adhesive thicknesses in SB specimens, there was a significant difference in removal strength between TC-0 and TC-1000 specimens ($P < 0.05$). In addition, there was a significant difference between TC-0 and TC-1000 in 150-µm OG specimens (Table 4). The ARI scores for all adhesives at TC-1000 are listed in Table 5. In the SB group, the 50-µm specimens were less likely to have no adhesive on the tooth (ARI score, 0) than were specimens with other adhesive thicknesses. However, there was no significant difference in ARI scores in relation to adhesive thickness for any adhesive.

Discussion

This study focused on the residual adhesive cement layer after debracketing, and demonstrated that removal strength was lower when the adhesive cement layer was thinner. Regarding the relationship between adhesive thickness and bond strength, Jost-Brinkmann et al. (15) reported that composites with macrofillers yielded the highest bond strengths and that the thickness of the adhesive layer had no effect. Mackay (16) reported that increasing the thickness of four adhesives had no significant effect on mean shear bond strengths, although strength tended to decrease. However, these previous experiments used a model in which the bracket was bonded onto the tooth. Therefore, a loaded shear force would be applied to the lateral surface of both the adhesive and bracket.

Mehl et al. (11) evaluated the effects of cement film thickness on retention of cemented implant-retained crowns and found that crown retention decreased significantly between cement films with thicknesses of 15 and 50 µm. Wakasa et al. (17) reported that during a shear bond test the change in average stress with interfacial stress was related to the thickness of the bonding area.

Regarding mechanics during loading, using three-dimensional finite element analysis, Hatamleh et al. (18) found that shear stresses increased with increasing distance and bending moment upon application of a uniform shear load at different distances from the interface. Although the blade of the pliers contacts the side of remnant adhesive when removing adhesive, the load is more concentrated when the adhesive is thinner, and the bending moment and shear force at the tooth surface are lower. Adhesives of various thicknesses were compared in the present study; removal strengths were significantly lower for adhesive layer thicknesses of 50 and 100 µm than for thicknesses of 150 and 200 µm. Our findings suggest that removal strength during adhesive removal can be significantly reduced, by 20% to 60%, by decreasing adhesive thickness to 100 µm.

We also evaluated the effect of thermal cycling on removal strength. For all materials tested, removal strength tended to be higher after thermal cycling; post-thermocycling removal strengths for SB and OG specimens with a thickness of 150 µm were significantly lower before thermocycling. The significant difference in SB specimens might be due to the physical properties of 4-META/MMA-TBB resin (19). However, there were no significant differences in the removal strength for TX specimens at TC-0 and TC-1000. Therefore, the thickness of TX should be reduced to decrease subsequent discomfort during removal.

The bonded area established in this experiment was equivalent to one-third of one bracket section. Several researchers have described residual adhesive levels after debracketing, at various bond strengths (20-23). Rikuta et al. (24) reported that an area of more than 50% of the SB, which was used to bond the metal bracket onto the tooth surface in a shear test, occurred in 90% of specimens. Algera et al. (25) reported that a remnant area of more than 50% was common. Thus, regardless of frequency, the extent of adhesive application in this study matches that remaining on the tooth surface after debracketing under actual clinical conditions. We used adhesive thicknesses of 50, 100, 150, and 200 µm because a previous study (26) reported that the thickness of residual adhesive on teeth was 102.7 ± 80 µm after debonding.

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