

Effect of adhesive application prior to bracket bonding with flowable composites

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ABSTRACT

Objective: To evaluate the effect of applying an adhesive, prior to different flowable composite resins, on the shear bond strength (SBS) of orthodontic brackets to acid-etched human enamel.

Materials and Methods: One hundred metal brackets were bonded to 35% phosphoric acid-etched human premolars using four flowable composites (Admira Flow, Tetric Flow, Filtek Supreme, Transbond Supreme) and a conventional orthodontic composite resin (Transbond XT). For each experimental group, half of the specimens were bonded in combination with Transbond XT adhesive. After 24 hours of storage, an SBS test was performed. Adhesive remnant index scores were determined after the failure of brackets. Two-way analysis of variance and Student-Newman-Keuls multiple comparison tests were performed at $P < .05$.

Results: When using an adhesive system, the five composites performed equally. The application of adhesive had a significant effect on SBS. Without adhesive, SBS decreased in all groups, except for Transbond Supreme LV. All the test groups exhibited similar bracket failure modes.

Conclusions: When using an adhesive system, flowable composite resins exhibited similar SBS. Transbond Supreme was the only resin performing similarly with or without adhesive system application. (*Angle Orthod.* 2011;81:716–720.)

KEY WORDS: Flowable composite resin; Shear bond strength; Adhesive; Bracket

INTRODUCTION

For many decades, the most popular bonding system in orthodontics has been based on the acid-etching technique introduced by Buonocore¹ in 1955 and modified for orthodontic purposes by Newman^{2,3} and Rietz et al.⁴ during the 1960s. The essential step in this technique is preconditioning the enamel surface with a 37% orthophosphoric acid solution or gel for approximately 30 seconds to dissolve the minerals of the enamel. Actually, bonding systems have been simplified and improved to provide increased long-term strength and promote the durability and reliability of orthodontic brackets. Two main strategies are used to create durable enamel bonding: (1) the acid etched/

composite technique with an intermediate thin layer of resin (also called primer or bonding agent) to penetrate the etched enamel, which has been widely adopted in contemporary orthodontic practice to increase bond strength between the tooth and composite resin,⁵ and (2) self-etching systems or acidic primers combining the etching and priming steps. This minimizes the time working during bonding and eliminates possible damage to the gingival tissue.⁶

Filled dental restorative materials are also used as orthodontics composite resins.⁷ These materials consist of an organic diacrylate (BIS-GMA), a coupler (silane), and a higher percentage content of inorganic filler (quartz, silica). Traditional dental composite resins are densely loaded with reinforcing filler particles for strength and wear resistance when small and highly packed filler particles protect the polymer matrix in the composite from food bolus wear.⁸

The goal of current orthodontic research is to improve the bonding procedure by minimizing the time of working during bonding and debonding without jeopardizing the ability to maintain a clinically useful bond strength. In this way, flowable composite resins merit great attention because of two of their clinical handling characteristics, which have not existed for composites until very recently: (1) no stickiness and (2)

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Table 1. Compositions and Manufacturers of Tested Materials^a

Restorative Material	Manufacturer	Composition
Transbond XT	3M ESPE, St. Paul, Minn	Primer: Bis-GMA, TEGDMA Adhesive: silane-treated quartz, Bis-GMA, dichlorodimethylsilane reaction product with silica; 77% quartz (silica) filler
Transbond Supreme LV	3M ESPE, St. Paul, Minn	Combination of silica and zirconia Nanofillers, glass filler (65% wt), Bis-GMA, UDMA, TEGDMA
Tetric Flow	Ivoclar-Vivadent, Schaan, Liechtenstein	Dimethacrylate and triethylene glycol dimethacrylate (Cavifil: 31.5% wt; syringe: 35% wt); inorganic filler, particles: barium glass, ytterbium trifluoride, Ba-Alfluorosilicate glass, highlydispersed silicon dioxide, and spheroid mixed oxide (64.6% wt); catalysts and stabilizers
Admira Flow	Voco, Cuxhaven, Germany	Bis-GMA, di-UDMA, TEGDMA; filler type: Ba-Al-silicate glass, Si O ₂ ; filler content (vol%/wt%) 50.5/64
Filtek Supreme XT	3M ESPE, St. Paul, Minn	Nanofiller, particle size 20–75 nm, silaned 65–75 wt.%, Bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate 5%–15%, diurethane dimethacrylate 5%–15%, pyrogenic SiO ₂ , silaned 5%–15%, Bis-GMA 1%–10%, triethyleneglycol dimethacrylate <5%, water <2%, and camphorquinone

^a PENTA indicates penta-acrylate ester; TEGDMA, triethylene glycol-dimethacrylate; Bis-GMA, bisphenyl glycidyl methacrylate; Bis-EMA, bisphenol A ethoxylated dimethacrylate; UDMA, urethane dimethacrylate; BHT, butylated hydroxyl toluene; TCB resin, carboxylic acid modified dimethacrylate.

fluid injectability⁹ because flowable composites were created by retaining the same small particle sizes of traditional hybrid composites but by reducing the filler content and allowing the increased resin to reduce the viscosity of the mixture.¹⁰

Recently, flowable composites have been applied for orthodontic use by many clinicians.⁸ However, there are still few studies available on the bonding characteristics of flowable composites and the effect of these materials on the enamel surfaces during debonding.^{11–16} Thus, if a flowable composite without the intermediate low-viscosity resin can guarantee clinically acceptable bond strength to acid-etched enamel, it would be advantageous for orthodontic bracket bonding by reducing the number of steps during bonding and the potential for error through contamination during the bonding procedure. However, although eliminating the placement of a bonding agent after a separate etching process would result in a comparable number of steps to using a one-step etch/bond agent, there would be a cost advantage.

Accordingly, the aim of the present study was to evaluate the effect of applying an adhesive, prior to different flowable composite resins, on the shear bond strength (SBS) of orthodontic brackets to acid-etched human enamel.

MATERIALS AND METHODS

A total of 100 sound-extracted human premolars were stored in a 0.5 chloramine T solution at 48°C for a

maximum of 6 months after extraction. Exclusion criteria included previously restored premolars and premolars with enamel defects or cracking and delamination of the enamel. The premolars were mounted in a self-cure acrylic block. The buccal crown surface of each premolar was rinsed and dried after a 15-second polish with fluoride-free pumice slurry.

One hundred stainless steel metal premolar brackets with a micro-etched base (3M Unitek, Monrovia, Calif) were directly bonded to the acid-etched enamel. Four flowable composites (Tetric Flow, Ivoclar-Vivadent, Schaan, Liechtenstein; Transbond Supreme LV, 3M; Admira Flow, Voco, Cuxhaven, Germany; Filtek Supreme XT, 3M) and an orthodontic bonding system (Transbond XT, 3M) were examined (five experimental groups; N = 20). The components and manufacturers of these composites are shown in Table 1.

The buccal enamel surface was etched with 37% phosphoric acid gel (Etch-37, Bisco, Schaumburg, Ill) for 30 seconds, rinsed for 15 seconds, and dried with oil-free and moisture-free air for 20 seconds until the enamel had a faintly white appearance. For each experimental group, half of the specimens (N = 10) were bonded with a Transbond XT primer (3M) according to the manufacturer's instructions. Composites were applied to the bracket base, and the bracket was positioned on the tooth and pressed firmly with a Hollenback carver to expel the excess adhesive. In both groups, each bracket was subjected to a 300-g compressive force using a force gauge (Correx Co, Berne, Switzerland) for 10 seconds, after which excess

Table 2. Mean (SD) Shear Bond Strength Values (MPa) Obtained for Each Tested Group^a

	Without Adhesive	With Adhesive
Transbond XT	5.7 (2.2) _{2b}	14.6 (1.5) _{1a}
Admira Flow	6.8 (1.4) _{2b}	12.7 (0.7) _{1a}
Tetric Flow	11.2 (1.6) _{1b}	15.1 (2.5) _{1a}
Filtek Supreme XT	6.0 (2.6) _{2b}	13.3 (1.4) _{1a}
Transbond Supreme LV	14.5 (1.2) _{3a}	13.3 (1.5) _{1a}

^a Within the same column, groups with the same subscript number are not statistically significant ($P > .05$). The subscript letter indicates differences between subgroups without adhesive and with adhesive.

bonding resin was removed using a sharp scaler. Then, the composite was light cured for 20 seconds from the occlusal edge and 20 seconds from the gingival bracket edge.

The bonding adhesives were all light cured with a curing light (XL300; 3M/Unitek Dental Products), with a light intensity of 1000 mW/cm² measured with a built-in radiometer, which was calibrated every 10 minutes to ensure consistent light intensity.

The bracket teeth were immersed in sealed containers of deionized water and placed in an incubator at 37°C for 72 hours to permit adequate water absorption and equilibration, and an SBS test was performed. The specimens were secured in a jig attached to the base plate of a universal testing machine (Instron Corp, Canton, Mass). A chisel-edge plunger was mounted in the movable crosshead of the testing machine and positioned so that the leading edge was aimed at the enamel-composite interface before being brought into contact. A crosshead speed of 0.5 mm/min² was used.

After debonding, each specimen was examined under a stereoscopic zoom microscope (SMZ800, Nikon Corporation, Tokyo, Japan) to identify the location of the bond failure. The residual composite remaining on the premolar was assessed by using the remnant index (ARI), where each specimen was scored according to the amount of material remaining on the enamel surface as follows: 0 = no adhesive remaining, 1 = less than 50% of the adhesive remaining, 2 = more than 50% of the adhesive remaining, and 3 = all adhesive remaining with a distinct impression of the bracket base.

Descriptive statistics including means (MPa) and standard deviations (SD) were calculated for the SBS analysis. A two-way analysis of variance (ANOVA) and Student-Newman-Keuls multiple comparison tests were used to determine the statistical significance of any difference in mean SBSs among the 10 groups. The ARI was analyzed for percentage and frequency of fracture type, and a chi-square test was used as the statistical test. Significance for all statistical tests was

Table 3. Frequency Distribution of the Adhesive Remnant Index (ARI) Scores (%) for the 10 Groups^a

	Without Adhesive				With Adhesive			
	0	1	2	3	0	1	2	3
Transbond XT	30	30	30	10	10	40	30	20
Admira Flow	20	40	30	10	10	20	50	20
Tetric Flow	10	40	40	10	0	50	30	20
Filtek Supreme XT	20	40	30	10	10	40	40	10
Transbond Supreme LV	20	20	40	20	20	30	40	10

^a There were no statistically significant differences in ARI score among the adhesives. 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, and 3 = all adhesive left on tooth surface.

predetermined at $P < .05$. All of the statistical analyses were performed using SPSS 12.0 for Windows (SPSS, Chicago, Ill).

RESULTS

Shear Bond Strengths

The descriptive statistics, including the mean and SD values for each of the 10 groups, are presented in Table 2. Data were analyzed using two-way ANOVA, and Student-Newman-Keuls multiple comparison tests were performed at $P < .05$. The application of adhesive ($F = 25.06$, $P < .001$) had a significant effect on SBS. The power of the multiple ANOVA analysis for SBS was about .82.

When using an adhesive system, the five composites performed equally. Without adhesive, SBS decreased in all groups, except for Transbond Supreme LV.

Adhesive Remnant Index

Table 3 shows the ARI scores for the resins. According to statistical analysis (chi-square analysis) of the ARI scores, all of the test groups exhibited similar bracket failure modes ($P > .05$). Regardless of the adhesive, bond failure occurred partly at the bracket-adhesive (resin) interface but mainly within the adhesive (resin).

DISCUSSION

The bond strength was not measured under oral conditions, where mechanical impact and biochemical changes may result in adhesive material fatigue and inadvertent debonding forces.¹⁷ Nevertheless, in vitro shear debonding forces are an acceptable methodology to determine future in vivo comparative conditions.^{13,17}

When using an adhesive system, the four flowable composites performed similarly to the conventional

orthodontic composite resin, similar to that observed in some previous studies.^{17,18} With increased flowability, flowable resins can flow easily onto a bracket base and into an etched tooth structure and thus are expected to enhance the level of mechanical retention.¹⁸

Without adhesive, the SBS decreased in all groups, except for Transbond Supreme LV. A review of the published literature indicates that many researchers have achieved similar SBS values,^{17,19} indicating that certain flowable composites of thinner viscosity may bond to enamel adequately without the requirement of an intermediate bonding resin. Penetration of liquids into narrow capillaries, such as the microporosities of etched enamel, is influenced by properties of the liquid, such as viscosity and the surface free energy of the capillary wall.¹⁹ In addition, viscosity of the restorative resin has been described as a parameter that influences the penetration of restorative resins into acid-etched enamel.¹⁹ Transbond Supreme LV contains a dimethacrylate polymer that modifies the rheology of the material and provides a flow-on-demand handling characteristic, allowing the material to flow under pressure yet hold its shape after placement until light cured.²⁰ This rheology control allows the adhesive to be moderately filled (65% by weight) with a combination of agglomerated and nonagglomerated nanoparticles, composed of the following: (1) 75-nm diameter nonagglomerated/nonaggregated silica nanofiller, (b) 5- to 10-nm diameter nonagglomerated/nonaggregated zirconia nanofiller, and (3) loosely bound agglomerated zirconia/silica nanocluster, consisting of agglomerates of 5–20 nm primary zirconia/silica particles. The cluster particle size ranged from 0.6 to 1.4 μm .²⁰

The bond strengths of the four flowable composites without adhesive systems were greater than the 5.9 to 7.8 MPa considered by Reynolds and Von Fraunhofer¹⁷ to be adequate for routine clinical use. Moreover, according to Pickett et al.,²¹ the mean bond strengths recorded in vivo following comprehensive orthodontic treatment are significantly lower than bond strengths recorded in vitro. Although mean SBSs were within the acceptable range, they would be considered marginal, at the lowest acceptable level. It should be stated that with the exception of Tetric Flow and Transbond Supreme LV, the bond strengths of the materials were approximately 50% of the bond strengths obtained when the bonding agent was included. Although the bond strengths observed without adhesive made it into the minimally acceptable range, they were observed in vitro, under meticulous control of the technique of etching, rinsing, drying; therefore, a direct relationship to bonding in the oral environment should be approached with caution and discussed. It would be helpful to the orthodontic clinician to know the

percentage reduction in SBS that was observed with each material in this study when the step of bonding agent placement was eliminated to allow the clinician to make conclusions about the acceptability of this.

On the other hand, Transbond XT showed 5.7 MPa. This orthodontic composite resin has a higher filler concentration (77% quartz-silica filler)²² than the other flowable composites used. The influence of filler concentration on the viscosity remains a clinically important issue²³ because charged particles in the composite resin limit the free flow of adhesive into the enamel pores, inhibiting the formation of resin tags.^{24,25}

The ARI developed by Årtun and Bergland²⁶ has been used by many investigators to help standardize bond failure analysis. The ARI may oversimplify the very complex issues of bond failure analysis, but it does allow for statistical analysis and cross-study comparisons.¹⁵ According to optical microscopic observation, debonding occurred mainly within the adhesive (cohesive type; ARI score 1–2). Therefore, the bond failure patterns for the flowable composites were potentially favorable for enamel preservation. The enamel fractures and damage tend to increase with an ARI score of 5; in other words, the fracture occurred at the enamel-adhesive interface.

CONCLUSIONS

- The application of adhesive has a significant effect on the SBS of the flowable composites.
- When using an adhesive system, flowable composite resins exhibit similar SBS. Transbond Supreme was the only resin performing similarly with or without adhesive system application.

REFERENCES

1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surface. *J Dent Res*. 1955;34:849–853.
2. Newman GV. Bonding plastic orthodontic attachments to tooth enamel. *J N J Dent Soc*. 1964;35:346–358.
3. Newman GV. Epoxy adhesive for orthodontic attachments: progress report. *Am J Orthod*. 1965;51:901–912.
4. Retief DH, Dreyer CJ, Gavron G. The direct bonding of orthodontic attachments to teeth by means of an epoxy resin adhesive. *Am J Orthod*. 1970;58:21–40.
5. Rider M, Kenny B, Tanner AN. The effect of enamel bonding agent on in vitro composite bond strength. *J Dent*. 1977;5:295–302.
6. Chu CH, Ou KL, Dong DR, Huang HM, Tsai HH, Wang WN. Orthodontic bonding with self-etching primer and self-adhesive systems. *Eur J Orthod*. In press.
7. Bayne SC, Taylor DF, Heymann HO. Protection hypothesis for composite wear. *Dent Mater*. 1992;8:305–309.
8. Elaut J, Asscherickx K, Vande Vannet B, Wehrbein H. Flowable composites for bonding lingual retainers. *J Clin Orthod*. 2002;36:597–598.

9. D'Attilio M, Traini T, Di Iorio D, Varvara G, Festa F, Tecco S. Shear bond strength, bond failure, and scanning electron microscopy analysis of a new flowable composite for orthodontic use. *Angle Orthod.* 2005;75:410–415.
10. Tecco S, Traini T, Caputi S, Festa F, de Luca V, D'Attilio M. A new one-step dental flowable composite for orthodontic use: an in vitro bond strength study. *Angle Orthod.* 2005;75:672–677.
11. Lalani N, Foley TF, Voth R, Banting D, Mamandras AH. Polymerization with the argon laser: curing time and shear bond strength. *Angle Orthod.* 1999;69:525–534.
12. Meehan PM, Foley TF, Mamandras AH. A comparison of the shear bond strengths of two glass ionomer cements. *Am J Orthod Dentofacial Orthop.* 1999;115:125–132.
13. Park SB, Son WS, Ko CC, García-Godoy F, Park MG, Kim HI, Kwon YH. Influence of flowable resins on the shear bond strength of orthodontic brackets. *Dent Mater J.* 2009;28:730–734.
14. Ozel E, Korkmaz Y, Attar N, Karabulut E. Effect of one-step polishing systems on surface roughness of different flowable restorative materials. *Dent Mater J.* 2008;27(6):755–764.
15. Ryou DB, Park HS, Kim KH, Kwon TY. Use of flowable composites for orthodontic bracket bonding. *Angle Orthod.* 2008;78:1105–1109.
16. Uysal T, Sari Z, Demir A. Are the flowable composites suitable for orthodontic bracket bonding? *Angle Orthod.* 2004;74:697–702.
17. Reynolds IR, Von Fraunhofer JA. Direct bonding of orthodontic attachments to teeth: the relation of adhesive bond strength to gauze mesh size. *Br J Orthod.* 1976;3:91–95.
18. Frankenberger R, Lopes M, Perdigão J, Ambrose WW, Rosa BT. The use of flowable composites as filled adhesives. *Dent Mater.* 2002;18:227–238.
19. Asmussen E. Penetration of restorative resins into acid etched enamel: viscosity, surface tension and contact angle of restorative resin monomers. *Acta Odontol Scand.* 1977;35:175–182.
20. Cinader DK, Darell SJ. Transbond™ Supreme LV low viscosity light cure adhesive: suitable for indirect bonding. *Orthod Perspect.* 2009;16:24–26.
21. Pickett KL, Sadowsky PL, Jacobson A, Lacefield W. Orthodontic in vivo bond strength: comparison with in vitro results. *Angle Orthod.* 2001;71:141–148.
22. James JW, Miller BH, English JD, Tadlock LP, Buschang PG. Effects of high-speed curing devices on shear bond strength and microleakage of orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 2003;123:555–561.
23. Ostertag AJ, Dhuru VB, Ferguson DJ, Meyer RA Jr. Shear, torsional, and tensile bond strengths of ceramic brackets using three adhesive filler concentrations. *Am J Orthod Dentofacial Orthop.* 1991;100:251–258.
24. Hocevar RA. Direct bonding metal brackets with the Concise-enamel system. *J Clin Orthod.* 1977;11:473–482.
25. Roberts MW, Moffa JP, Jenkins WA. Clinical evaluation of three acid-etch composite resin systems: two-year report. *J Am Dent Assoc.* 1978;97:829–832.
26. Årtun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. *Am J Orthod.* 1984;85:333–340.