Shear Bond Strengths of Two Composite Core Materials After Using All-in-One and Single-Bottle Dentin Adhesives

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Purpose: The purpose of this study was to compare the shear bond strengths of 2 composite core materials after using all-in-one and single-bottle dentin bonding materials.

<u>Material and methods</u>: The occlusal surfaces of 100 extracted, intact human third molars were ground to expose a flattened area of dentin and polished with 600-grit silicon carbide paper. Specimens were divided into 5 main groups (n = 20). Three all-in-one (AQ Bond, One-Up Bond, Xeno-CF Bond) and 2 single-bottle adhesives (Single Bond, One-Step Plus) were used. Each group was further divided into 2 subgroups. Ti-Core and Built-it F.R. core materials were applied using a translucent plastic ring (diameter: 3 mm, height: 5 mm). After storage in 37°C water for 24 hours, shear bond strengths were measured using a Universal testing machine with a crosshead speed of 0.5 mm/min. Debonded dentin surfaces were examined with SEM. Two-way analysis of variance (ANOVA) and multiple comparison (Tukey) tests were used for statistical analysis of data.

<u>Results:</u> Two-way ANOVA revealed that the type of core material did not significantly influence the shear bond strength (p > 0.05), whereas there were significant differences in shear bond strength among the types of bonding agents (p < 0.0001). Shear bond strengths for single-bottle adhesive systems were significantly higher than those for all-in-one adhesive systems (p < 0.05). Furthermore, the interaction of these 2 parameters was not significant (p > 0.05). The fracture modes were predominantly adhesive for all-in-one adhesives and cohesive for single-bottle adhesives.

<u>Conclusion</u>: Bonding of composite core materials with the newly developed all-in-one dentin adhesives produced lower shear bond strengths as compared with single-bottle adhesives. J Prosthodont 2005;14:97-103. Copyright © 2005 by The American College of Prosthodontists.

INDEX WORDS: composite core materials, all-in-one adhesives, single-bottle adhesives

A FOUNDATION or core restoration is often required after tooth fracture or extensive dental caries removal. Because the core becomes an integral part of the load-bearing structure of the tooth, it should provide a satisfactory form for resistance and retention of the coronal restoration and also should possess sufficient strength to resist occlusal forces.¹⁻⁴ Some of the most common types of core materials used are silver amalgam, glass ionomer, resin-reinforced glass ionomer, and com-

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Copyright © 2005 by The American College of Prosthodontists 1059-941X/05 doi: 10.1111/j.1532-849X.2005.00018.x posite resin.⁵⁻⁹ Because of the many treatment alternatives available, there is often much confusion choosing the most stable material or set of materials for a given procedure.

An ideal core buildup material should have physical properties similar to those of tooth structure. Cast gold and amalgam have many material properties approximating those of tooth structure, and both materials have a long record of clinical success when used for core buildups. In comparison, composite possesses some inferior physical properties, including polymerization shrinkage, water absorption, and relatively high thermal expansion.¹⁰⁻¹² Despite these clear disadvantages, however, the use of composites for core buildups is becoming more and more popular because of their esthetics, strength, rapid setting reaction, and ability to bond to the tooth structure.¹³⁻¹⁵

The effective use of composite as a core buildup material has required significant enhancements in dentin bonding technology. Dentin adhesives

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are currently available as three-, two-, and singlestep systems depending on how the 3 cardinal steps of etching, priming, and bonding to tooth substrates are accomplished or simplified.¹⁶ In order to make the clinical use of bonding systems easier and faster, some of them combine priming and bonding agent in 1 solution ("single-bottle" adhesives).¹⁷ With these systems, however, etching of the dentin is still necessary to remove the smear layer and to expose the collagen fibers of the dentinal matrix.¹⁸ When dentin is excessively dried after etching, the collagen fibril network collapses and occludes the retentive interfibrillar microspaces in the demineralized matrix. This will impede the penetration of monomers and the formation of a resin-dentin interdiffusion zone (hybrid layer).^{19,20} On the other hand, several reports suggest that resin infiltration may be incomplete even when the moist bonding technique is used.21,22

Recently, single application bonding systems (all-in-one bonding systems), which combine the function of the self-etching primer and bonding agent, have been developed. Theoretically, the acidic adhesive dissolves the smear layer incorporating it into the mixture, and demineralizes the superficial dentin, then hardens after light irradiation. The rationale behind the action of this self-etching primer adhesive is the reduction of discrepancies between the depth of demineralization and the depth of resin infiltration, since both processes occur simultaneously.²³

To date, there have been few studies of shear bond strength that compare all-in-one and singlebottle adhesive systems, especially when coupled with composite core materials. The aim of this study was to evaluate the shear bond strength of 2 composite core materials to dentin, which were bonded with single-bottle and all-in-one adhesive systems.

Materials and Methods

A total of 100 caries-free human third molars were cleansed of gross debris and stored in distilled water. The occlusal enamel was first removed by sectioning the crown perpendicular to the long axis of the tooth using a low-speed saw (Isomet, low speed saw, Buehler Ltd., Lake Bluff, IL) with water lubrification. The roots were removed by a second dentin section made as close as possible to the pulp floor, approximately 0.5 mm below the cemento–enamel junction. Dentin surfaces were ground perpendicular to the long axis using 240-grit SiC paper under running tap water to create flat surfaces. The prepared dentin surfaces were examined under a stereoscopic microscope (Nikon SMZ10, Tokyo, Japan) at $10 \times$ magnification to ensure it was free of enamel. The distance between the cemento-enamel junction and the flat occlusal surface was kept between 2.5 and 3 mm. Each tooth was embedded in self-curing acrylic resin using a cylindrical plastic mold. The occlusal surface of the teeth was at the same level of the embedding medium to form 1 flat surface. Final finish was accomplished by grinding on wet 600-grit SiC paper. Before bonding, the specimens were stored in distilled water at 37° C.

The specimens were randomly divided into 5 main groups of 20 specimens. The general compositions of the materials used in this study are shown in Table 1. Before the bonding procedures, a piece of polyethylene tape with a circular hole 4 mm in diameter was positioned on the dentin surface of each specimen to control the area of the bond.

In the first group, before application of Single Bond (3M Dental Products Division, St. Paul, MN), 35% H₃PO₄ (Ultra-Etch, Ultradent, South Jordan, UT) was used for 20 seconds, then the specimens were rinsed for 15 seconds and dried gently enough to leave the dentin surface moist. Using a fully saturated brush tip for each coat, 2 consecutive coats of Single Bond were applied and dried for 2 to 5 seconds, then light cured for 10 seconds.

In the second group One-Step Plus (Bisco Dental Products, Schaumburg, IL) was applied. After etching with 35% H_3PO_4 (Ultra-Etch, Ultradent) for 15 seconds, the specimens were dried gently for 2 to 3 seconds. Then 2 consecutive coats of One-Step Plus bonding material was applied, gently air dried, and light cured for 10 seconds.

In the third group all-in-one AQ Bond (Sun Medical Co., Ltd., Moriyama, Japan) was applied. After the application of the first coat for 20 seconds, the second coat was applied and then dried. The specimens were light cured for 10 seconds.

In the fourth group, in which One-Up Bond (Tokuyama Corp., Tokyo, Japan) was used, 1 drop of One-Up Bond A and 1 drop of One-Up Bond B were mixed together and applied in 2 consecutive coats until a glossy dentin surface was obtained. Specimens were light cured for 10 seconds.

In the fifth group, Xeno-CF Bond (Sankin Kogyo, Tokyo, Japan) was applied to the dentin surface and then light cured for 10 seconds.

All 5 groups were divided into 2 subgroups of 10 specimens each. A translucent plastic ring, 4 mm in diameter with 5 mm of height, was placed over the dentin surface. In the first subgroup, Ti-Core (Essential Dental Systems, S. Hackensack, NJ) base and catalyst were mixed in equal parts into a homogenous mass on a mixing pad with a plastic spatula provided by

Table 1.	Materials	Used
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Materials Manufacturer		Composition		
Bonding agents -Single Bond	3M Dental Products Division,	Hema+BisGMA+dimethacrylate+methacrylate		
(single-bottle bonding)	St. Paul, MN	functional copolymer+polyitaconic acids		
-One-Step Plus	Bisco Dental Products,	Biphenyl dymetacrylate, hydroxyethyl		
(single-bottle bonding)	Schaumburg, IL	methacrylate, acetone, glassfrit		
-ÀQ Bond (all-in-one bonding)	Sun Medical Čo., LTD., Moriyama, Japan	1. Base: Methacrylate monomers; approx. 30%, 4-Meta, UDMÁ, 2-hyroxyethyl methacrylate (HEM)		
		2. Acetone/water: less than 70%. AQ sponge: 1-polyurethane foam:approx 80%. 2-sodium p-toluensulfinote approx 20%		
-One-Up Bond	Tokuyama Corp., Tokyo,	MAC-10 adhesive monomer		
(all-in-one bonding)	Japan	Methacrylate		
		Fluoroaluminosilicate glass fiber		
-Xeno-CF Bond	Sankin Kogyo, Tokyo,	Pyrophosphate ester, UDMA,		
(all-in-one bonding)	Japan	fluoride-releasing phosphazene monomer, photo sensitizer, microfiller particles		
Core Materials		1 7 1		
-Ti- Core	Essential Dental Systems, S. Hackensack, NJ	Fluoridated hybrid dilled, autocuring composite		
-Build-It F.R.	Jeneric/ Pentron Inc., Wallingford, CT	BISGMA, UDMA, HDDMA, silane treated glass fillers, chopped glass fibers, stabilizers, UV absorber Dual-cure composite		

the manufacturer. Ti-Core material was then inserted in the plastic ring with a Centrix syringe (Mark IIIp C-R Syringe, Centrix Inc., Milford, CT). The second subgroup was prepared identically to the previously described subgroup, but restored with Build-it F.R. (Jeneric/Pentron, Inc., Wallingford, CT) core material instead of Ti-Core. Build-it F.R. core material, which was delivered in an auto-mix cartridge, was injected into the plastic ring and light cured for 40 seconds from facial, lingual, and occlusal aspects.

The cores were allowed to set for 1 hour before the translucent plastic rings and adhesive tape were removed. The specimens were stored in distilled water at 37°C for 24 hours before being tested.

The shear bond strength testing was performed with a Universal testing machine (Instron, Losenhausen Machinenbau A.G. Düsseldorf, Germany) using a loading cell of 5000 N, applied at a crosshead speed of 0.5 mm/min. A chisel-shaped rod was aligned in the crosshead so that the force delivered to the specimen was immediately adjacent and parallel to the dentin surface. Each specimen was continuously loaded until fracture occurred. The shear bond strength values were calculated (in MPa) by dividing the force at which bond failure occurred by the bonding area. A two-way analysis of variance (ANOVA) design was used to examine the effect of adhesive system, core material, and the interaction of these 2 parameters on shear bond strength. Multiple comparisons were performed using Tukey's tests. Statistical significance was set at the 5% level.

The debonded dentin surfaces were examined with SEM (JEOL JSM model 6400, Tokyo, Japan) at magnification of 1500×. After debonding, a trough 3 mm in diameter and 2 to 3 mm in depth was cut around the debonding area with a No. 330 bur using high-speed hand piece. The specimens were then sectioned with a diamond wheel and low speed saw (Isomet) with water coolant to free the samples. The samples were washed with tap water, then placed in an ultrasonic cleaner (T-14B, L&R Mfg. Co., Kearny, NJ) with 1 part alcohol and 2 parts distilled water for 10 minutes, followed by another 10 minutes in 100% distilled water. All the samples were left to dry in a desiccator for 24 hours and then sputter coated with gold–palladium before SEM observation.

Results

The mean shear bond strengths and their standard deviations with each adhesive system for both core materials are presented in Table 2. Two-way ANOVA revealed that the type of core material did not significantly influence the shear bond strength (F = 2.663; p > 0.05), whereas there were significant differences in shear bond strengths among the types of bonding agents (F = 9.250; p < 0.0001). Furthermore, the interaction of these 2 parameters was not significant (F = 0.174; p > 0.05), indicating that the effect of different types of bonding agents on the shear bond strength does not vary depending on the type of core materials used (Table 3).

Subgroup 1 Subgroup 2 Ti-Core Build-it F.R. $Mean \pm SD$ $Mean \pm SD$ Group 1 Single Bond $8.45 \pm 1.46^{\text{A1}}$ 9.45 ± 2.27^{A1} $8.39\pm2.45^{\rm A1}$ $9.25 \pm 2.04^{\rm A1}$ Group 2 One-Step Plus 6.85 ± 1.70^{B1} 7.10 ± 1.20^{B1} Group 3 AQ Bond 7.18 ± 1.70^{B1} Group 4 One-Up Bond $6.78 \pm 1.33^{\text{B1}}$ Group 5 Xeno- \overline{CF} Bond 6.10 ± 1.31^{B1} $6.60 \pm 1.79^{\mathrm{B1}}$

Table 2. Means and Standard Deviations of Shear Bond

Comparisons within each column at each core material are presented by upper-case letters and comparisons of values in each row at each dentin adhesive are presented with numbers. Mean with same letters or numbers were not significantly different by Tukey test (p > 0.05).

Pair-wise comparisons with Tukey test revealed that the mean shear bond strengths for single-bottle adhesive systems (Single Bond, One-Step Plus) were significantly higher than those for all-in-one adhesive systems (AQ Bond, One-Up Bond, Xeno-CF Bond) independent of the type of the core material used (p < 0.05). The differences between the means of single bottle adhesive systems and the all-in-one adhesive systems were not statistically significant (p > 0.05).

The SEM views of the debonded dentin surfaces showed no obvious differences between the composite core materials, and revealed that the failure type was not dependent on the type of the core material used (Figs 1-4). Single-bottle adhesive systems exhibited predominantly cohesive failure in the bonding layer. The debonded dentin surface was partially covered by a thin resin layer, and remnants of the bonding layer were seen on the dentin surface (Figs 1 and 2). On the other hand, the failures for all-in-one adhesive systems were predominantly at the dentin/bonding layer interface indicating an adhesive type of failure. On the debonded dentin surface dentinal tubules were very apparent (Figs 3 and 4).

 Table 3. Two-Way ANOVA: Influence of Bonding

 Agent and Core Material; Dependent Variable: Shear

 Bond Strength

	Degrees of Freedom			F-Test	P-Value
Bonding agent	4	116.040	29.010	9.250	0.000
Core material	1	8.352	8.352	2.663	0.106
Bonding agent-core material	4	2.180	0.545	0.174	0.951

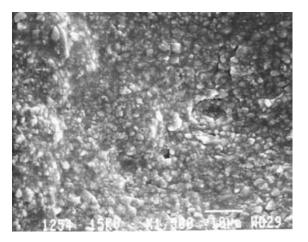


Figure 1. SEM micrograph of debonded dentin surface of Single Bond-Ti-Core group. Cohesive failure at adhesive layer–core material interface shows remnants of bonding layer on the dentin surface (original magnification $1500 \times$).

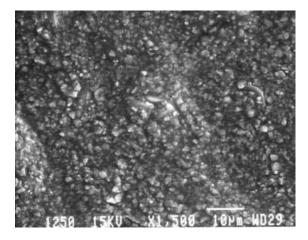


Figure 2. SEM micrograph of debonded dentin surface of Single Bond-Build-it F.R. group represent cohesive type of failure (original magnification 1500×).

Discussion

In this study, the latest dentin adhesive systems, including all-in-one bonding systems (AQ Bond, One-Up Bond, Xeno-CF Bond) and single-bottle bonding systems (Single Bond and One-Step Plus) were chosen. Furthermore, the composite core materials used in this study were selected according to the differences in their filler systems and polymerization modes.

The mean shear bond strength of Ti-Core material with single-bottle adhesives (8.39 MPa, 8.45 MPa) was similar to that reported in a

Strength Values (MPa)

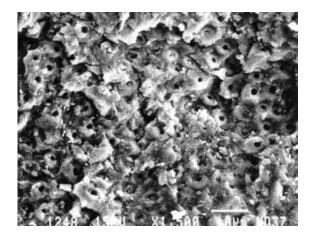


Figure 3. SEM micrograph of debonded dentin surface of AQ Bond-Ti-Core group. Adhesive type of failure was observed on dentin surfaces. On the debonded dentin surface dentinal tubules were very apparent (original magnification $1500 \times$).

previous study by Al Wazzan²⁴ (9.81 MPa), but greater than the value reported by Cohen et al²⁵ (1.59 to 6.01). The variations may be explained by differences in bonding methodology, storage environment, and testing techniques. For each bonding system used in this study, the shear bond strength values of the dual cured core material Build-it F.R. were slightly higher than the values of the chemically cured Ti-Core material. This difference, however, did not reach statistical significance (p > 0.05).

O'Keefe and Powers²⁶ concluded that the different polymerization modes between compos-

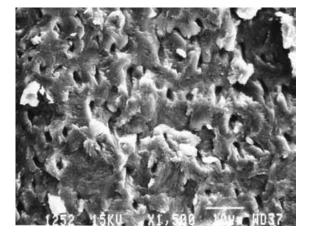


Figure 4. SEM micrograph of debonded dentin surface of AQ Bond-Build-it F.R. group represent adhesive type of failure (original magnification 1500×).

ite core materials and dentin adhesives have little influence on the bond strength. Hagge and Lindemuth²⁷ revealed variations in the shear bond strengths between the multi-step and singlebottle adhesive systems and a composite core material but could not demonstrate any relationship between different bonding systems and polymerization modes. In the present study, regarding the used core materials and dentin adhesives of different polymerization modes, similar results were obtained.

The shear bond strengths ranged from 8.39 to 9.45 MPa for single-bottle adhesives and from 6.10 to 7.18 MPa for all-in-one adhesives. Miyazaki et al²⁸ reported higher values for the singlebottle adhesive system Single Bond (18.1 MPa) and the all-in-one adhesive system One-Up Bond (13.7 MPa). Werner and Tani²⁹ also found higher values for several all-in-one dentin adhesives including AQ Bond (19.0 MPa) and One-Up Bond (11.5 MPa) bonding agents. Another study showed more similar shear bond strength values for One-Up Bond.³⁰ Assuming that as a consequence of variations commonly found in tooth substrates and differences in the methods employed, studies determining the bond strength to tooth are important mainly for their relative values, and numerical comparisons are not always possible.²³

The use of all-in-one bonding systems that do not require rinsing and serve simultaneously as conditioner, primer, and adhesive is a recent approach to the simplification of bonding techniques. Although a single-step adhesive is desirable in a clinical situation, the combination of acids and hydrophilic and hydrophobic monomers into a single solution may compromise the function of each one of these components. Nevertheless, the results of this study showed that all-in-one bonding systems have lower shear bond strength values than the single-bottle systems. This result could be attributed to the insufficiency of dentin conditioning with all-in-one system.³¹ For single bottle adhesives, phosphoric acid etching can completely remove the smear layer. Since the smear plugs were removed and tubule orifices were demineralized, thicker resin tags were formed.³² Conversely, some studies have shown that all-in-one bonding systems have the ability to dissolve the smear layer and to form a relatively thick hybrid layer.³³ Furthermore, it was also clearly demonstrated that the bonding agent wets and penetrates the etched dentin surface very well. Therefore, it was

suggested that the lower bond strengths of the all-in-one bonding systems compared with 2-step systems are not a result of insufficient wetting by the adhesive.³⁴

The SEM observation of adhesive failure for allin-one systems indicated a bond to dentin lower than the cohesive strength of the adhesive. Similar types of failure were also demonstrated in studies performed by Toledano et al,³³ Bouillaguet et al,³⁵ and De Munck et al.³⁶ A possible explanation may be inadequate resin penetration into the dentinal surface and the low degree of polymerization of the resin at the dentin-resin interface. The lack of additional solvent-free resin layers, the low resin concentration, and/or low viscosity of some of these self-priming adhesives could result in insufficient polymerization.^{33,34} Consequently, attempts were made to increase the thickness of the adhesive layer. Recent studies have suggested that application of a second adhesive layer after light curing of the first layer may improve the bonding of unfilled all-in-one adhesives.37,38 Another approach to increase the saturation of the resindentin interface is filler addition. Controversial results were reported in previous studies about the failure type of the all-in-one adhesives. Miyazaki et al²⁸ and Fritz and Finger³⁴ reported cohesive failure within the adhesive layer, indicating the inherent strength of the adhesive polymer might be the weak link. On the other hand, the debonded surfaces for single-bottle adhesives exhibited a cohesive failure through the bonding layer, indicating that the bond to dentin exceeded the cohesive strength of the adhesives.

Conclusions

Based on the results of this in vitro study, the shear bond strengths of single-bottle adhesives were higher than the values of all-in-one systems. Thus, the use of single-step dentin bonding systems should be preferred for the composite core build up rather than all-in-one dentin adhesive systems.

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