Shear bond strength of self-etch and total-etch bonding systems at different dentin depths

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Abstract: The purpose of this study was to evaluate the dentin shear bond strength of four adhesive systems (Adper Single Bond 2, Adper Prompt L-Pop, Magic Bond DE and Self Etch Bond) in regards to buccal and lingual surfaces and dentin depth. Forty extracted third molars had roots removed and crowns bisected in the mesiodistal direction. The buccal and lingual surfaces were fixed in a PVC/acrylic resin ring and were divided into buccal and lingual groups assigned to each selected adhesive. The same specimens prepared for the evaluation of superficial dentin shear resistance were used to evaluate the different depths of dentin. The specimens were identified and abraded at depths of 0.5, 1.0, 1.5 and 2.0 mm. Each depth was evaluated by ISO TR 11405 using an EMIC-2000 machine regulated at 0.5 mm/min with a 200 Kgf load cell. We performed statistical analyses on the results (ANOVA, Tukey and Scheffé tests). Data revealed statistical differences ($p < 0.01$) in the adhesive and depth variation as well as adhesive/depth interactions. The Adper Single Bond 2 demonstrated the highest mean values of shear bond strength. The Prompt L-Pop product, a self-etching adhesive, revealed higher mean values compared with Magic Bond DE and Self Etch Bond adhesives, a total and self-etching adhesive respectively. It may be concluded that the shear bond strength of dentin is dependent on material (adhesive system), substrate depth and adhesive/depth interaction.

Descriptors: Dentin; Adhesives; Dental Etching.

Introduction

The mechanism for adhesive bonding to dental substrates, especially to dentin, has been extensively studied in the last few decades. The establishment of effective interlocking occurs when the adhesive penetrates into the intratubular and intertubular dentin so that resin penetration into the conditioned dentin results in the formation of intratubular resin tags and a “hybrid layer.”[1] Micromechanical attachment is the most important mechanism of resin adhesion to dentin. However, there are several factors that can affect hybrid layer and resin tag formation, such as acid-etching, the moisture conditions of the substrate, and adhesive and dentin depth.[2]

During dentin acid-etching, the mineral content of the dentin surface is removed, and the collagen fibrils remain supported by water.[3] The moist bonding technique prevents the collagen fibers from collapsing

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and provides stability to the collagen, thus enabling greater infiltration of adhesive monomers.[4] However, some studies have found that the resin monomer infiltration may be incomplete even when the "wet" bonding technique is used,[5] suggesting that another factor affect hybridization, the composition of adhesive systems.

After decades of evolution, adhesives may include different formulations and, consequently, their bond values may vary in relation to dental substrate.[6] Currently there is a tendency to simplify bonding procedures by mixing the prime and bond functions. In addition, acid monomers are currently used in primers, which introduced the self-etching adhesive concept.[7]

Due to different factors that affect the bond strength of composite resin and dentin, it is important to evaluate the influence of the adhesive system, the type of acid-etching and the dentin depth as they relate to the shear bond strength of the dentin-composite resin interface at the same dentin locations.

Materials and Methods

This study was approved by the Ethics Research Committee of Ribeirão Preto School of Dentistry (University of São Paulo, Brazil). Forty noncarious extracted human third molars had their roots removed 2 mm beyond the enamel/cementum junction and their crowns bisected longitudinally in a mesiodistal direction with a nº 3072 diamond bur in a water-cooled high speed turbine to create two similar halves (buccal and lingual). The half surfaces were set in 12 mm high PVC cylinders (Tigre S/A, Joinvile, Brazil) as holders (1.5 cm diameter, 1.0 cm high) using cold-cured acrylic resin (Clássico, São Paulo, Brazil), and a polytetrafluoroethylene (Teflon) mold developed for this experiment was used to place their central region in the third part of the ring. The teflon mold produced a step-off of 0.5 mm between the dental surface and the acrylic resin, which was needed to access the most superficial desired dentin for the experiment.

The two groups (40 buccal and 40 lingual) were then randomly reorganized into four groups (n = 10) corresponding to the four adhesive systems tested with a microhybrid resin composite. The systems were the following: Adper Single Bond 2 (ASB2) / Z-250, Adper Prompt L-pop (APLP) / Z-250, Magic Bond DE (MBDE) / Fill Magic and Self Etch Bond (SEB) / Fill Magic. The adhesives, the composition and the bonding procedures are shown in Table 1, according to manufacturer's provided information.

Table 1 - Dentin adhesive systems tested.

Adhesive systems	Components	Bonding procedures
Adper Single Bond 2 (3M ESPE, Sumaré, Brazil) Lot: 7LH	Ethanol, water, Bis-GMA, 5 nm silane treated colloidal silica, 2-hydroxyethyl methacrylate, glycerol 1, 3 dimethacrylate, methacrylate functional copolymer of polyacrylic and polyitaconic acids and diuretane dimethacrylate	After etching, rinsing and drying, apply 2 coats of adhesive while massaging in over the entire surface for 15 s. Dry gently to evaporate the solvent. Light-cure for 10 s
Adper Prompt L-Pop (3M ESPE, Sumaré, Brazil) Lot: 287452	A: Phosphate mono and di-hema, dimethacrylate, camphorquinone, substituted aromatical amine and substituted phenol B: water, hydroxyethyl methacrylate, methacrylate polycarbonic acid and substituted phenol	Mix A and B blisters, according to the manufacturer's recommendation; the adhesive must be massaged in over the entire surface for greater effect while applying pressure for 15 s; reapply until the appearance of a glossy surface; remove excess solvent until a fine film is formed. Light-cure for 10 s
Magic Bond DE (Vigodent S/A, Rio de Janeiro, Brazil) Lot: 001/07	HEMA, dimethacrylates, neopentyl fluoride, acrylate fluoride, adhesive monomer (MEP), highly dispersed silicon dioxide, photoinitiators and stabilizers in an alcoholic solution	After etching, rinsing and drying, apply adhesive and gently massage in for 10 s; apply a stream of air. Avoid very fine coats with a strong stream of air. Light-cure for 20 s. The surface must be glossy; if not, repeat the application
Self Etch Bond (Vigodent S/A, Rio de Janeiro, Brazil) Lot: 002/07	1: Hema, copolymer, adhesive monomer (MEP), dimethacrylate, alcohol, water, photoinitiators and stabilizers 2: Adhesive monomer (MEP), HEMA, bis-GMA, alcohol, dimethacrylates, microfiller, photoinitiators and stabilizers	1. Apply the primer gently, massaging the cavity for 20-30 s. Do not wash. Gently apply a stream of air 2. Apply the bond, apply a stream of air and light-cure for 20 s

To obtain the required dentin circular shape area with 3 mm diameter, the surfaces were wet abraded in a horizontal polisher (model PFL, Fortel Ind. e Com. Ltda., São Paulo, Brazil) with 150, 320, 400 and 600 grit abrasive papers (Norton, Ind. Com. Ltda., São Paulo, Brazil), to expose and standardize the required bond area, the so-called "superficial dentin."

For all groups, the wet bonding technique was applied and the excess moisture was gently removed using an absorbent paper, gently applied to the dentin surface.[8]

The adhesive was applied and then light-cured, according to manufacturer's recommendations using a photocuring unit (Ultraled XP; Dabi Atlante S/A, Ribeirão Preto, Brazil), with a light output not less than 500 mW/cm². The specimens were individually fixed in a metallic clamping device (developed at the Houston Biomaterials Research Center and manufactured at the Precision Workshop at Ribeirão Preto School of Dentistry of University of São Paulo, Brazil) thereby keeping the dentin surface parallel to a flat base. A split-bisected polytetrafluoroethylene jig was positioned on the tooth/resin block surface, thus providing an inverted conical centralized cavity (3-mm diameter at base and 5-mm high) with the smaller diameter corresponding to the demarcated 3-mm-diameter-bonding site. The composite resins were inserted into the matrix in three steps, each polymerized for 20 s and applied in a conical shape 4-mm high. As the matrix cavity filled, the specimen was removed from the clamping device.

After 24 hours of storage in distilled water at 37°C, the specimens (combined PVC ring/dentin surface/composite resin cylinder) were tested for shear bond strength using an Emic universal testing machine (MEM-2000 Model, São José dos Pinhais, Brazil) at a crosshead speed of 0.5 mm/min and a 200 Kgf load cell. After testing, fractured specimens were observed with an optical microscope (Nikon, Model 86786, Tokyo, Japan) at a magnification of 20x to assess failure modes, which were classified as adhesive, cohesive or mixed. Adhesive failure occurred at the specimen/adhesive interface, cohesive failure occurred in the material or the substrate with no damage to the interface and mixed failure simultaneously involved the interface and the material or substrate. After the analysis, the 10 identified specimens were again stored in distilled water and refrigerated.

To evaluate the influence of dentin depth on shear bond strength, the samples were identified and abraded 0.5 mm four times until reaching a depth of 2 mm, measured by digital calipers. After each 0.5 mm abrasion the same dentin surface preparation protocol described above was performed, and the specimens were stored again in distilled water and were refrigerated. A schematic illustration of specimen preparation and details about the experiment are presented in Figure 1.

The adhesive shear bond strength values were recorded in kgf/cm² and were converted into MPa. Means and standard deviations were calculated, and data were analyzed by ANOVA. Multiple comparisons were done using the Tukey test.

Results

Table 2 reveals the analysis of variance that showed a statistically significant difference ($p < 0.01$) between the adhesive systems and dentin

Table 2 - Analysis of variance - original values.

Source of variation	SS	df	MM	F	p value
Depth	913.8391	4	228.4598	8.16	0.0035%*
Adhesive	10697.5645	3	3565.8547	127.36	0.00001%*
Adhesive x depth	768.2980	12	64.0248	2.29	1.0027%*
Residual I	5039.5483	180	27.9975		
Residual II	4513.4688	180	25.0748		
Total variation	22519.6250	399			

*Significant level at 1%.

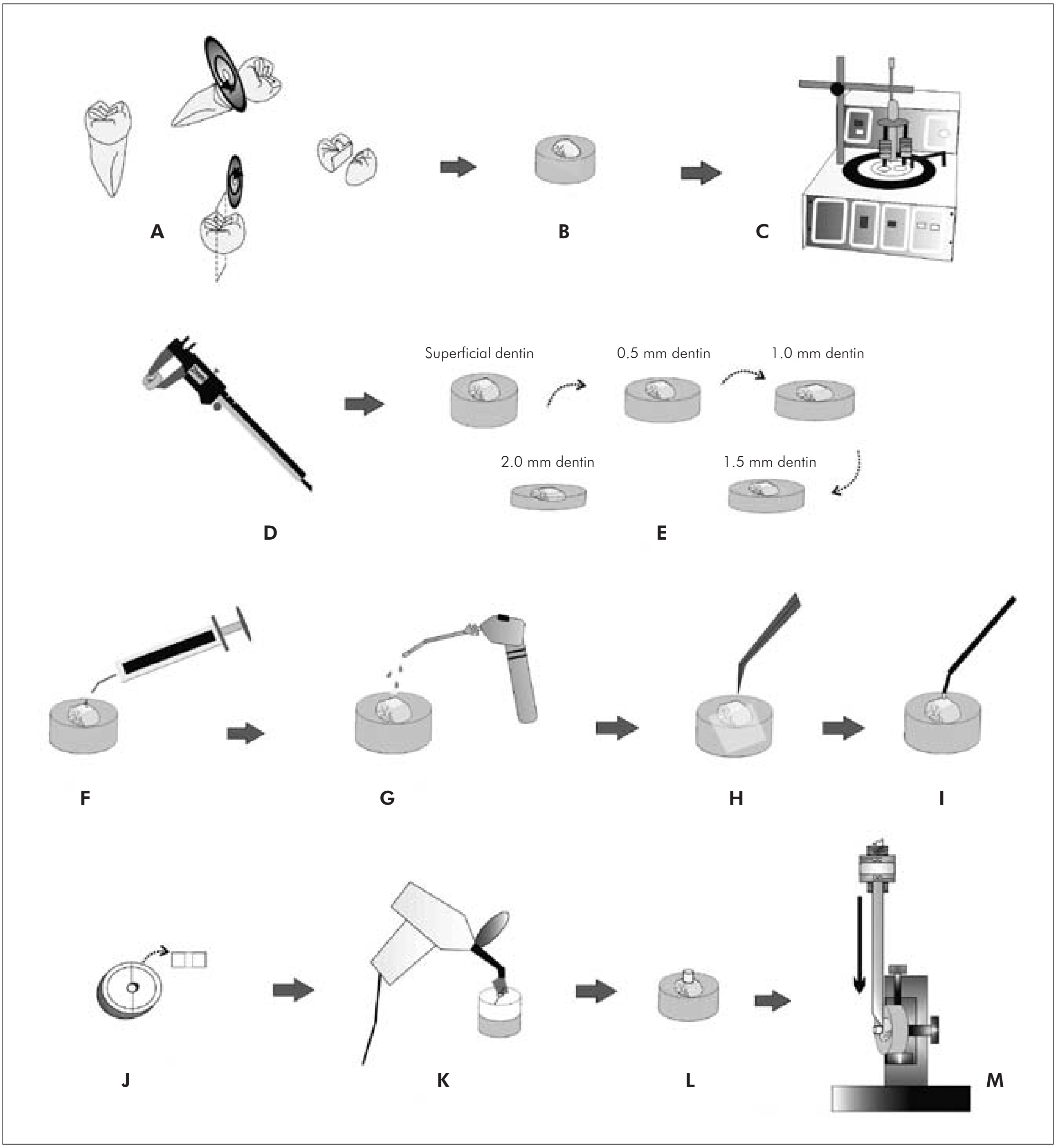

Figure 1 - Flowchart of the study shear bond strength test. **A**: Tooth sectioning; **B**: Tooth section embedding; **C**: Abrading and polishing of dentin; **D**: Measuring of dentin depth; **E**: Specimens at different depths; **F**: Acid etching; **G**: Washing; **H**: Drying with absorbent paper; **I**: Applying adhesive systems; **J**: Metallic deposits to build up the specimens; **K**: Light-curing; **L**: Finished specimen; **M**: shear bond strength test.

depths, as well as an adhesive x dentin depth interaction. The means of the variation factors are given in Tables 3 and 4. Figure 2 shows the interaction between the adhesive and dentin depth factors.

Discussion

The results from this study reveal that dentin shear bond strength depends on adhesive and dentin depth. Consequently, shear bond strength also de-

Table 3 - Means for adhesive variation.

Dental adhesive	Mean MPa (SD)	Critical value
Adper Single Bond 2	18.7612 (± 7.24)[a]	1.94189
Magic Bond DE	10.1532 (± 4.72)[b]	
Adper Prompt L-Pop	12.9430 (± 6.10)[c]	
Self Etch Bond	4.40270 (± 2.67)[d]	

Different letters indicate statistical difference.

Table 4 - Means for depth variation.

Dentin depth (mm)	Mean MPa (SD)	Critical value
Superficial	13.67225 (± 7.4)[a]	2.30717
0.5	12.39263 (± 7.92)[a]	
1.0	11.94600 (± 7.82)[a]	
1.5	10.47975 (± 6.98)[b]	
2.0	9.33450 (± 6.41)[b]	

The same letters indicate statistical similarity.

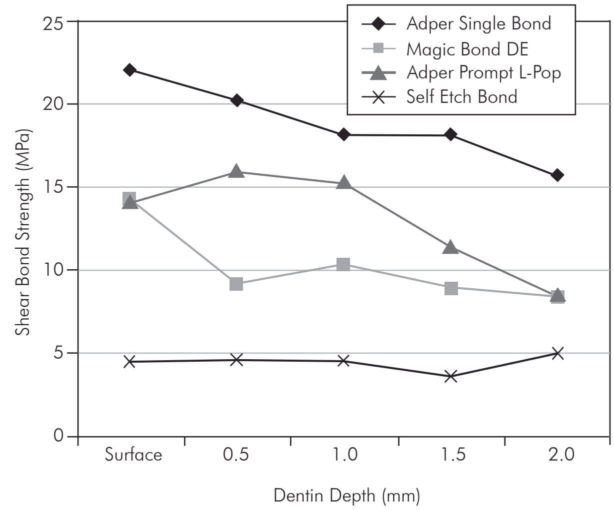

Figure 2 - Illustration of the adhesive x dentin depth interaction.

pends on the adhesive and dentin depth interaction.

It is important to consider the composition and the substrate treatment by adhesive factor and, therefore, self or total etching. Different studies report that the chemical composition of adhesive systems determines clinical success.[9] Polyacrylic acid in ASB2 and APLP adhesives promotes chelation with calcium and the formation of hydrogen bridges with dentin components; it may be the significant factor resulting in higher shear bond strength values.

Another component that may be responsible for the high bond strength values is the 5 nm silica nanofiller incorporated at 10% weight in ASB2 adhesive. These particles may have a role in the formation of a resin film that stabilizes the hybrid layer.[10] The intermediate layer of the adhesive filler promotes an elastic zone that improves the capacity to accommodate contractile forces during composite resin polimerization.[11] In addition, the nanofillers in ASB2[12] are smaller, which improves its wettability and penetration compared with bigger fillers such as the silic dioxide in the MBDE and the microfillers in SEB.

Regarding adhesive and substrate treatment, there are potentially similar bond strength results between self and total etching adhesives.[13] On the other hand, one study[14] showed that conventional adhesive demonstrates better performance than the self-etching adhesive when applied according to the original prescription and with previous acid etching.

The results of this study showed that SEB (Vigodent), a two-step self-etching adhesive, had the lowest shear bond strength values, while APLP (3M), a one-step self-etching adhesive, demonstrated a better performance than the total etching adhesive, MBDE.

In this case, in addition to adhesive composition, which has already been discussed, the efficacy of self-etching adhesives may be an important factor responsible for the higher mean bond strength values of APLP compared to SEB. The PLP is at pH 1.0 and is thus considered, in terms of etching aggressiveness, a strong self-etching adhesive.[15] SEB is at pH 5, according to the manufacturer information, and is thus considered to be mild self-etching. These results suggest that low bond strength values for SEB may be related to the weak etching of the dentin substrate. The incomplete infiltration of some self-etching adhesives may occur because of the etching potential reduction of acid monomers in the hybrid layer base or because of noncured hydrophilic components due to bonding area deterioration when these self-etching adhesives are used.[16] However, one study demonstrated that a smear layer removal step after etching and before adhesive application could produce a more durable and realistic bond to dentin.[17] On the other hand, some studies[18] have also shown that APLP produces an etching effect that approached that of acid etching with phosphoric acid at 35%, suggesting its efficacy and adhesive potential.

The preservation of hydroxyapatite in the hybrid layer may serve as a receptor for additional chemical bonding because the calcium has chemical bonding potential with the monomers usually present in self-etching adhesives, in addition to protecting collagen against hydrolysis.[19]

Regarding dentin depth, the bond strength values decreased as depth increased. The statistical analysis revealed significant differences between two groups of dentin depth, one including the superficial, 0.5 mm and 1.0 mm depths, and the other including the 1.5 mm and 2.0 mm depths.

Similar studies have been performed describing the inverse relationship between dentin porosity (which increases with depth) and shear bond strength;[20] these studies show morphologic structural variation of dentin that affects dentin bonding.[21-23]

As depth increases, there is an increase in dentin tubules and minor hybridization with a greater number of tags or larger tags. Because the tags can contribute approximately one-third of the shear bond strength,[24] minor adhesive resistance is expected at deep dentin because it is an area that has less solid dentin and thus lower bonding values regardless of the type of adhesive used.[22]

In general, bond strength decreases as depth increases; however, these values demonstrated several points of variation. This may be explained by the morphology and structural features in the same dental areas of the dentin substrate.[20] These substrate features include moisture variation and tubule orientation beyond the degree of tubular obstruction due to calcification by the stimuli that the tooth has experienced, and these differences can explain why the same adhesive system may demonstrate different bond strength values when applied to different regions of dentin substrate.[25] Thus, it is important to diagnose each tooth individually to choose the appropriate treatment.

The adhesive and dentin depth interaction revealed statistical differences, consistent with a study[26] that concluded that bond strength is affected by the adhesive system, the substrate or both. Some studies[27] have observed that bond strength is affected by dentin depth depending exclusively on adhesive composition.

The dental surface variation did not influence shear bond strength. A comparison of the results obtained in this study with those of other studies may be difficult because a large amount of studies are conducted to evaluate only one dental surface, usually the buccal, and do not evaluate the dental surface as a variation factor.[28]

Conclusion

Based on these findings, it may be concluded that bond strength is affected by adhesive and dentin depth. The dental adhesive systems had a significant influence on shear bond strength. The ASB2 demonstrated the highest mean values and the SEB had the lowest for all dentin depths evaluated. The dentin depth adversely affected the bonding mechanism. The dental surface did not affect shear bond strength at the dentin-resin interface.

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