An Alternative Adhesive Strategy to Optimize Bonding to Root Dentin

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Abstract

Introduction: This study examined the hypothesis that the shear-bond strengths of AH Plus (AH) and Hybrid Root Seal (HRS) to root dentin might be optimized by using a powdered dentin-reinforced bonding procedure. **Methods:** The surfaces of root halves obtained from extracted human premolars were ground (800-grit silicon carbide paper), treated (5.25% NaOCl 17% EDTA for 5 minutes followed by distilled water) and randomly allocated into two groups according to the sealer and then into three subgroups according to bonding procedure. Dentin particles with a maximum size of 25 μm were incorporated into the adhesive of Clearfil Liner Bond 2V, and groups were created as follows: no adhesive, adhesive alone, and with powdered dentin reinforced adhesive; 3 $\times$ 3 mm high buildups were created using sealer and allowed to set (37°C, 100% humidity, 72 hours). Shear tests were performed (1 mm/min). Data were calculated as MPa and analyzed (two-way analysis of variance, Tukey test). **Results:** A significant difference was found between the groups (P = .000). Adhesive or reinforced adhesive had a negative effect on the shear-bond strength of AH, but they significantly increased the shear-bond strength of HRS (P = .000). HRS showed a similar bond strength with either adhesive alone or adhesive reinforced with powdered dentin. AH group was characterized by mixed failure, whereas the predominant failure type of the HRS group was cohesive failure within the sealer. **Conclusions:** Reinforcing adhesive resin with powdered dentin may be considered an alternative for optimizing the bonding of methacrylate-based sealers to root dentin and might affect the shear bond strength. *(J Endod 2011;37:1427–1432)*

Key Words

Adhesion, *in vitro* test, powdered dentine, root dentine, self-etch adhesive

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Resin-based sealers have gained popularity with the recent growing interest in adhesive endodontics (1). However, several factors make adhesion to the root canal system a challenge, such as chemical substances used during the biomechanical preparation (2), volumetric changes that occur in resin-based sealers during polymerization (3), debonding of the sealer because of polymerization shrinkage stresses (4), and various geometric factors (5). Studies on alternative strategies to improve bonding to root dentin determined that adhesive systems were effective in optimizing bond strengths to radicular dentin (6, 7) and in improving sealing ability (8).

Self-etching adhesives with acidic monomers have recently gained popularity. These systems are easy to apply and are characterized by reduced technique sensitivity. Because the acidic functional monomers are not rinsed off, the potential exists for further demineralization (9). Maeda et al (10) and Iwasa et al (11) incorporated powdered dentin to self-etching primers in order to neutralize continuous etching activity (12) and to test possible pH changes that occur during the reactions (13). They concluded that dentin has a strong buffering capacity (10) and a strong modulation effect against the acidity of self-etching adhesives (11).

Fillers have also been added to adhesives in order to improve bond strengths by reinforcing the hybrid layer and reducing polymerization shrinkage (14). The lack of shrinkage stress relief is one of the major problems associated with endodontic bonding because of the high cavity configuration-factor (C-factor) (5, 15). Filler may also give the bond a gel-like consistency (16), which results in a thicker bonding-resin layer (16, 17). Adhesives with thicker bonding-resin layers may act as stress breakers, confirming the elastic bonding concept and resulting in a superior bond (18).

The term “monoblock,” literally meaning a single unit, has recently become a familiar term in the endodontic literature (19). Achieving a mechanically homogenous unit with root dentin using adhesive materials should be the goal of successful endodontic treatment although this is not always possible in the clinical conditions. Monoblocks in endodontically treated teeth and potential negative outcomes of different interfaces with various elastic modules have been explained (20). Incorporating powdered dentin into adhesives has the potential to help create a more dentin-like adhesive interface between the sealer and the root dentin, thus supporting the monoblock concept (20) and a strong ionic interaction between the root dentin and the adhesive material to enhance the bond strength.

Thus, the purpose of this study was to test the hypothesis that shear-bond strengths of an epoxy-resin-based sealer AH Plus (AH) (Dentsply, De Trey, Konstanz, Germany) and a 4-methacryloxyethyl trimellitate anhydride (4-META) containing the self-etching, self-adhesive sealer, Hybrid Root Seal (HRS) (Sun Medical, Tokyo, Japan), to root dentin might be optimized by incorporating a powdered dentin-reinforced, self-etching bonding procedure.

Materials and Methods

The materials used in the study are listed in Table 1.

Preparation of Root Surfaces

Forty-two extracted human premolar teeth that had been stored in distilled water for a maximum of 2 months were used. Soft-tissue covering surfaces were scaled using periodontal curettes (Dufflex, Rio de Janeiro, RJ, Brazil). The crowns

TABLE 1. Ingredients, Manufacturers, and Batch Number of the Materials Used in This Study

Sealer	Ingredients	Manufacturer	Batch
AH Plus	Paste A: Bisphenol-A, epoxy resin, Bisphenol-F, calcium tungstate, zirconium oxide, silica, iron oxide pigments. Paste B: Dibenziyldiamine, Aminoadamantane, Tricylodecane-diamine, calcium tungstate, zirconium oxide, silica, silicone oil	De Trey, Dentsply, Konstanz, Germany	1003001934
Hybrid Root SEAL	Liquid: 4-META, monofunctionalmethacrylate monomer, multifunctional macrylate monomers and photo-initiators; Powder: mixture of zirconiumz oxide filler, SiO_2 filler, and polymerization initiators	Sun Medical, Shiga, Japan	SW1 (SM1, ST1)
Liner Bond 2V	Primer A: 10-MDP, 2-HEMA, water, photoinitiator, accelerators. Primer B: HEMA, water, accelerator. Bond A: 10-MDP, hydrophobic aliphatic dimethacrylate, colloidal silica dl-camphorquinone, accelerators, others Bond B: BisGMA, HEMA, dibenzoyl peroxide, hydrophobic aliphatic dimethacrylate colloidal silica, MDP, dimethacrylates, photoinitiator, accelerator, and microfiller	Kuraray, Tokyo, Japan	Primer A: 00163A; Primer B: 00160A; Bond A: 002618; Bond B: 00070A

4-META, 4-methacryloyloxyethy trimellitate anhydride; Bis-GMA, bisphenol A-glycidyl methacrylate; HEMA, hydroxyethyl methacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate.

were removed and the roots longitudinally sectioned using an Isomet saw (Buehler, Lake Bluff, IL) under water cooling to obtain two root halves from each tooth (n = 84) (Fig. 1*A* and *B*). Each half was embedded horizontally in a self-curing polymethyl-methacrylate resin (Vertex Dental; Dentimex, Zeist, Netherlands), keeping the root surfaces exposed (Fig. 1*C*). Bonding surfaces were polished with 800-grit silicon carbide paper and were treated with 5.25% NaOCl and 17% EDTA for 5 minutes followed by distilled water. Samples were then randomly allocated into two groups according to sealer type (AH or HRS, n = 42).

Preparation of Powdered Dentin–reinforced Adhesive

Ten extracted noncarious third molar teeth were longitudinally sectioned, and the enamel layers were removed. Two mesh layers (FRITSCH GMBH, Idar-Oberstein, Germany) with 52-μm and 25-μm diameter holes on the top and bottom, respectively, were used to obtain powdered dentin. Exposed dentin surfaces were abraded with a diamond bur under water cooling above the mesh layer. Powdered dentin pieces of a maximum size of 25 μm were sieved through the mesh layer, collected into a container, dried, and then incorporated into the adhesive from a commercial bonding system (Clearfil Liner Bond 2V Bond A and B, Kuraray, Tokyo, Japan) according to the filler weight percentage (20 wt%) (21). Scanning electron microscope–energy dispersive x-ray (Zeiss EVO/LS10; Bruker, Bremen, Germany) analysis of the powdered dentin is shown in Figure 2. A motorized mixer (Magnetic Stirrer MK 418; NUVE, Ankara, Turkey) was used to constantly stir throughout the mixing procedure.

Preparation of Test Samples

The following three subgroups were created for each sealer (n = 14): no adhesive, adhesive alone, and powdered dentin-reinforced adhesive. Clearfil Liner Bond 2V was used as the adhesive according to manufacturer instructions and was cured for 10 seconds using a light-curing unit with a minimum intensity of 700 mW/cm^2 (Bluephase; Ivoclar Vivadent, MV, Lichtenstein, Germany); 3 × 3 mm-high buildups with a constant surface area of 3.45 mm^2 were created at the coronal one third of the roots using the sealers, which were mixed according to manufacturer instructions at room temperature (Fig. 1*D*). Polyethylene tubes were used to create the buildups. The samples were allowed to bench set for 2 hours to ensure that the initial

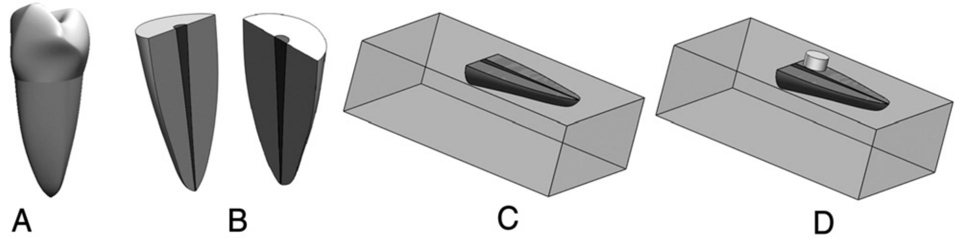

Figure 1. A schematic of the experiment protocol.

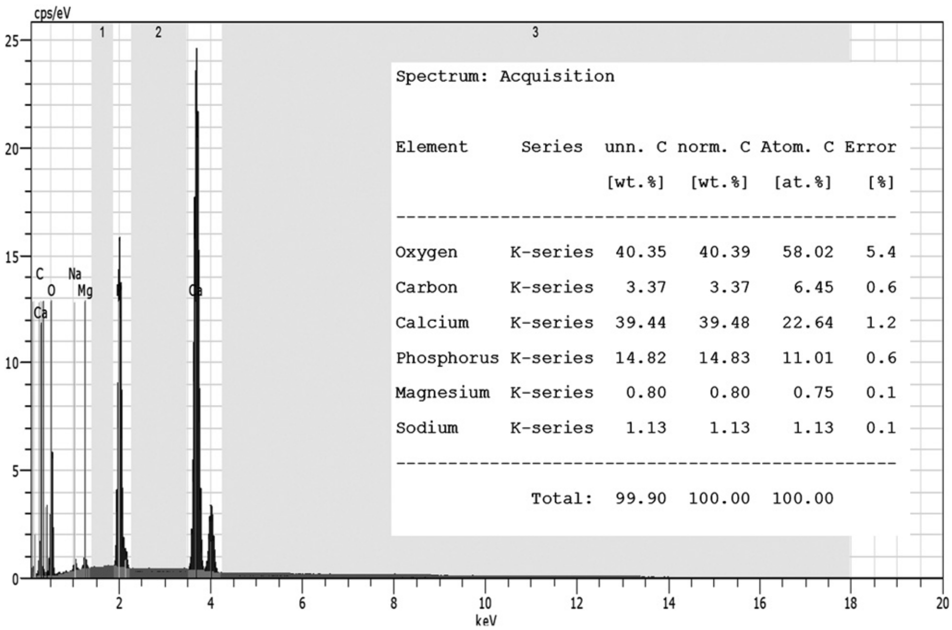

Figure 2. Scanning electron microscope–energy dispersive x-ray analysis of the powdered dentin used in the study as a filler.

setting reaction had occurred. Specimens were stored (100% humidity, 37°C, 72 h) and later retained in a universal testing machine (Instron, Canton, MA) to apply shear stress at a crosshead speed of 1 mm/min.

Shear Bond Strength Test

A custom-made constructed attachment was used to enable an easy and accurate fixation and for testing the samples. This custom fabrication allowed the chisel to travel parallel to the root dentin surface and contact the sealer cylinder at its interface with the dentin. The maximum load (N) for specimen debonding was recorded and divided by the contact surface area to determine the shear-bond strength in mega pascals (MPa). After testing, the fracture modes were examined under a dissecting microscope with ×22 magnification (SZ-TP Olympus; Tokyo, Japan). Failed interfaces from representative samples of each group were sputter-coated with gold and evaluated under scanning electron microscopy. Data were analyzed using two-way analysis of variance and Tukey tests.

Results

The mean shear bond strength measurements and failure patterns of the tested samples are shown in Table 2. A significant difference was found between the group means (P = .000). Although both adhesive alone and powdered dentin-reinforced adhesive negatively affected the shear-bond strength of AH, they significantly increased the shear-bond strength of HRS (P = .000). AH showed greater bond strength than HRS when both sealers were applied without adhesive. When the sealers were applied with adhesive (either with or without powdered dentin), HRS showed significantly greater bond strength ($P < .05$). AH samples showed primarily (66%) "mixed failure" without adhesive and 57% "adhesive failure" when used with adhesive (Table 2). HRS samples showed predominantly (85%–90%) "cohesive failure" inside the sealer when used with adhesive. Figure 3 depicts cohesive failure inside the sealer in a randomly selected sample from the HRS group. Figure 4 displays high magnification of adhesive with dentin particles. A representative sample from the AH group showed "mixed failure" (Fig. 4*C*).

Discussion

The shear-bond strength test has proven to be a feasible and reproducible method that provides homogenous results with considerably low variability in bond strength when compared with the push-out test (22). Therefore, in this study, the shear-bond strength test was used. The hypothesis of this study was partially rejected because the results suggested that use of an adhesive layer reduced the bond strength of AH regardless of the presence of powdered dentin reinforcement. A previously published study showed that the use of dentine bonding agents significantly improved the adhesion of an epoxy resin-based sealer to root canal dentine, and optimal results were obtained with the self-etching system (23). Regarding bonding of AH to root dentin, it has been reported that covalent bonds occur between the epoxide rings and the exposed amino groups in the collagen network (24). The application of a decalcifying agent improved the

TABLE 2. Mean Shear Bond Strength Values and Failure Patterns of the Tested Samples

Groups	Without adhesive		With adhesive		Adhesive + powdered dentin	
AH	$2.06 \pm 0.41^{a\alpha}$	34% C 66% M	$0.45 \pm 0.20^{c\alpha}$	57% A 43% M	$1.03 \pm 0.26^{b\alpha}$	70% A 30% M
HRS	$0.41 \pm 0.20^{a\beta}$	100% M	$1.32 \pm 0.59^{b\beta}$	85% C 15% M	$1.55 \pm 0.50^{b\beta}$	90% C 10% M

Values with different letters in the same line and different symbols in the same column are significantly different at $P < .05$.

A, adhesive failure; C, cohesive failure inside the sealer; M, mixed failure.

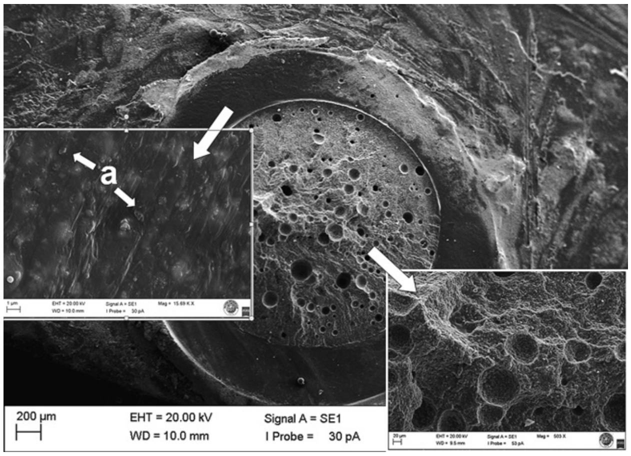

Figure 3. Cohesive failure inside the sealer in a randomly selected sample from the HRS group. Voids are visible within the sealer in the high-magnification image ×500 (*white arrow*). The powdered dentin pieces (a) are detected within the adhesive layer in the high magnification image (×1,500).

bond strength and sealing ability of AH based on the ability of the material to bond to the organic phase of root dentin, most likely in the collagen network (25). In the present study, AH showed a mean bond strength of 2.06 MPa in the absence of adhesive. These results are in accordance with those of Neelakantan et al (25) (eg, middle third, 2.7 MPa) although the irrigation protocols differ and testing methods are not the same. The bond strength of AH decreased when used with an adhesive and increased when powdered dentin was included, confirming findings on the ability of AH to bond to the organic phase of root dentine (25).

Fourth-generation methacrylate resin-based sealers such as HRS were designed with the intention to combine self-etching primer and a moderately filled flowable composite into a single product (26, 27). The acidic monomer (4-META) of this material is reported to be capable of diffusing through the demineralized surface to promote the formation of a hybrid layer after polymerization (28). In contrast, HRS has been reported as unable to etch beyond thick smear layers into the underlying intact radicular dentin in the absence of adjunct EDTA as an irrigant (26, 29). In the absence of EDTA, mild etching adhesives cannot etch through thick smear layers, and HRS promotes a superficial decalcification of dentin at approximately 2-μm deep (29). Therefore, in the present study, the final root dentin surface treatment was performed using 17% EDTA.

The results from this study indicate that AH shows greater bond strength compared with HRS when both are applied in the absence of adhesive ($P < .05$), confirming the results from a previous study (30). The low bond strength of HRS was explained by incomplete polymerization inside the canal and high C-factor (31). The lack of relief of shrinkage stresses created in deep, narrow canals (32) causes endodontic bonding procedures to be more complicated. Shrinkage stresses are greater in root canal sealers than in highly filled resin composites (5). Although methacrylate resin–based sealers present with significant volumetric shrinkage during polymerization, epoxy resin–based sealers do not shrink (33). In this study, flat root dentin surfaces were used; thus, the effects of incomplete sealer polymerization and high C-factors were eliminated (5).

Adhesive resin use either with or without powdered dentin reinforcement significantly increased the adhesive performance of HRS (Table 2), differing from results published in a recent study (27). In previous studies, powdered dentin was used for its buffering effects against the acidity of self-etching primers and self-etching adhesives (10, 11). In the present study, powdered dentin was incorporated in

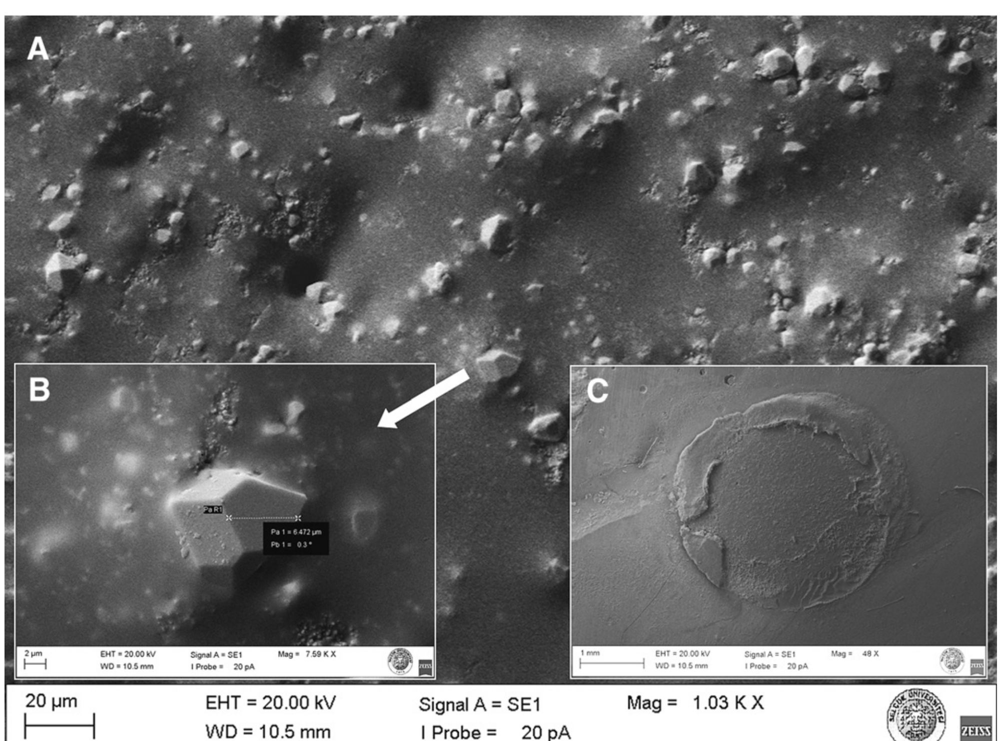

Figure 4. (*A*) High magnification of adhesive reinforced with powdered dentin particles (×1,000). (*B*) The powdered dentin pieces of approximately 6.472–25-μm diameters are detected within the adhesive. (*C*) A representative sample from the AH group shows a mixed failure pattern.

the adhesive resin to improve the mechanical properties of the adhesive layer and to create a more dentin-like adhesive interface. Therefore, the pH of the system primer (pH = 2) was held constant. The predominant adhesive mechanism of methacrylate resin-based sealers to radicular dentin is micromechanical (27). Because of the limited aggressiveness of the sealer, HRS might not etch a thick smear layer. The adjunct use of a dual-cured self-etching adhesive for simultaneously etching and priming dentin before the application of a methacrylate resin–based sealer was found to create more definitive hybrid layers and higher tensile bond strength to dentin (6). Primer treatment with a pH of 2 might have contributed to the increased adhesive performance of HRS in the present study. Furthermore, the addition of powdered dentin to the adhesive slightly increased the bond strength (not significantly, Table 2). The presence of an insoluble precipitate has been reported to show a chemical interaction between functional monomers and dentin powders comprised of hydroxyl apatite (HA) and type 1 collagen (10, 11). A balance between the acidity and chemical reactivity of the functional monomer as well as the buffering capacity of dentin created a strong bond between root dentin and the methacrylate resin-based sealer (10, 11). Our findings confirm the results shown in a recent study indicating that weak acids attach to the HA surface and leach minimal calcium but bind strongly to the mineral surface and remain even after rinsing; strong acids also bind to the calcium ions in HA but cause greater demineralization and tend to debond from the dentinal hard tissues by forming more soluble calcium salts (34).

The failure analysis of the samples indicated a predominance of cohesive failures within the sealer for HRS, thus confirming previous studies (26, 31). When the resistance to dislocation increases, the disruption of the sealer-dentin interface becomes less probable and failure is more likely to occur within the sealer itself (26). In this situation, HRS may provide a more impermeable seal. Sealing performance by this material has been justified (31, 35).

The AH group was characterized by mixed and adhesive failures (Table 2). In fact, 34% cohesive failures were observed for the AH group when self-etching adhesive was not used. In this study, the smear layer was removed using EDTA. This may explain why cohesive failures were observed in samples with AH in the absence of dentine bonding agents as previously claimed by Gogos et al (23).

Creating a monoblock unit with the support of a dentin-like interface using powdered dentin-reinforced adhesive was a second aim of this study. No interface analysis was performed, and the bond strength values were material dependent. Therefore, this topic requires further evaluation. The potential effects of contamination from powdered dentin during the experiments were disregarded. Further research is warranted to test the long-term adhesive stability of sealers applied in combination with powdered dentin-reinforced adhesive in order to improve clinical safety and reduce dentin particle size.

Conclusions

The bonding performance of Hybrid Root Seal was improved by using a self-etching adhesive either in the presence or absence of powdered dentin. The adhesive resin use negatively affected the shear bond strength of AH Plus. Within the limitations of this *in vitro* study and in view of the results, it may be speculated that powdered dentin is a potential alternative to reinforce adhesive resins by optimizing the bonding between methacrylate resin-based sealers and root dentin.

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The authors deny any conflicts of interest related to this study.

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