

Use of Self-Etching Adhesives to Seal Resected Apices

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The purpose of this study was to evaluate the sealing ability of four self-etching adhesives placed over blood-contaminated/uncontaminated resected root apices without root-end preparation. Extracted maxillary incisors and canines were randomly divided into four groups of 10 teeth each. After canal preparation and resection of the apex, four self-etching adhesives (Clearfil SE Bond, One-Up Bond F, Unifil Bond, and ABF) were applied over the control and contaminated surfaces. The roots were then subjected to 15 cm of water pressure to simulate periapical microleakage stress. Data were analyzed using a two-way repeated measures, ANOVA. Positive and negative controls responded as expected. Statistical analysis indicated that there were no significant differences in the sealing effectiveness among the adhesives applied to contaminated or uncontaminated groups. All contaminated groups had significantly higher leakage ($p < 0.003$) than their uncontaminated pairs.

Currently, root-end preparations are made into the resected root-end using ultrasonics with magnification. The root-end preparations must be filled with a restorative material, such as Super-EBA, MTA, or others (1–3). This is a technique-sensitive procedure that requires a dry field for ideal working conditions. One possible way to simplify the procedure would be to seal the resected root surface, without a preparation, with one of the recently developed self-etching adhesives.

The complexity of dentin-bonding procedures and their sensitivity to contamination by blood (4, 5) has limited their use to well-isolated sites. However, several studies have shown that dentin surfaces contaminated by blood can be decontaminated relatively easily (5). If adhesive resins (6) are to have a practical use in sealing resected apices, the resins must be able to tolerate some degree of blood contamination (7). The recent introduction of self-etching adhesives may provide endodontists with new options. Contemporary dentin adhesives are classified into three-step, two-step, and single-step systems depending on how the three cardinal steps of etching, priming, and bonding to dentin are accomplished

(8). The three-step systems require acid etching, rinsing, priming, and application of an adhesive. The two-step systems are subdivided into self-priming adhesives that require a separate etching step and the self-etching primers that require an additional bonding step. The single-step adhesive systems contain a mixture of acidic monomers capable of acid-etching dentin, mixed with hydrophilic monomers that can both prime and bond dentin, all in a single application. The purpose of this *in vitro* study was to evaluate the sealing ability of four self-etching adhesives placed over blood-contaminated and uncontaminated resected root apices using a fluid-filtration method (9). The null hypothesis is that there is no effect from blood contamination on the ability of self-etching adhesives to seal resected roots.

MATERIALS AND METHODS

Forty-four, single-rooted, extracted, human, maxillary incisors and canines were used in this study. After decoronation, the root canals were cleaned and shaped using Flexofiles (DENTSPLY/Maillefer, Tulsa, OK) and 5.25% NaOCl (Clorox; Clorox Co., Oakland, CA). The canals were instrumented to an ISO #50 file 1-mm short of the apical foramen. One milliliter of 5.25% NaOCl was used as an irrigant after each file. The canals were flared using a 1-mm step-back technique to an ISO #70 file and flushed with 5 ml of 5.25% NaOCl and air dried. The apical 3 mm of the roots were resected 90 degrees to the long axis of the root using a multipurpose bur (DENTSPLY/Maillefer) in a high-speed hand-piece with water irrigation. Forty-four teeth were randomly divided into four groups of 10 teeth each and two positive and two negative control teeth. Four commercially available self-etching adhesives were used: group 1: Clearfil SE Bond (Kuraray Medical Co., Ltd., Osaka, Japan); group 2: One-Up Bond F (Tokuyama, Tokyo, Japan); group 3: Unifil Bond (G-C International, Tokyo, Japan); group 4: an experimental antibacterial, fluoride-containing, self-etching primer adhesive system, ABF (Kuraray Medical Co., Ltd.).

The adhesives were mixed according to the manufacturers' instructions. The resected surface of the root was treated with double applications of the self-etching adhesives, but only the second application was light-cured. The two positive controls did not have bonding agents placed over the resected root surface, whereas the two negative controls had their resected root ends sealed with Zapit cyanoacrylate (Dental Ventures of America, Inc., Corona, CA). After light-curing the resins, the sealed roots were

TABLE 1. Sealing ability of self-etching adhesives on uncontaminated and blood-contaminated root apices

Material	Fluid Leakage ($\mu\text{l min}^{-1} \text{cm H}_2\text{O}^{-1}$) Mean \pm SD (n = 10)	
	Uncontaminated	Contaminated
Clearfil SE Bond	0.116 \pm 0.084 ^a	0.480 \pm 0.312 ^b
One-Up Bond F	0.164 \pm 0.147 ^a	0.315 \pm 0.246 ^b
Unifil Bond	0.208 \pm 0.135 ^a	0.522 \pm 0.194 ^b
ABF	0.105 \pm 0.097 ^a	0.390 \pm 0.198 ^b

Groups identified with the same superscript letters are not significantly different ($p > 0.05$).

* Difference between paired means was statistically significant at $p < 0.003$.

stored in 37°C water for 24 h. Following the 1-day storage, the cervical end of the root was cemented to a 0.7- \times 0.7- \times 0.2-cm block of Plexiglas using Zapit. An 18-gauge, stainless-steel tube, 2-cm long, penetrating the Plexiglas but ending flush with the upper surface, was centered over the root canal just before cementation with Zapit. This tube provided access to the root canal to allow for testing of the adhesives using the fluid-filtration technique (10). The sealed root was attached to a device (Flodec, De Marco Engineering, Geneva, Switzerland) that permitted quantitation of minute amounts of fluid movement under clinically relevant filtration pressure (11, 12). The roots were submerged in water during the fluid-filtration tests to prevent any evaporation of water across the exposed root surfaces. Measurements of fluid flow were made for a period of 2 to 5 min with the filtration pressure held constant at 15 cm of water.

After testing the uncontaminated specimens, the resin seal was removed with the same multipurpose bur used to resect the apex, taking care to remove no more than 0.5 mm of dentin. This removed the cured adhesive and permitted each specimen to serve as its own control for paired statistical evaluation. Human blood was then placed on the freshly resected surface and allowed to clot for 5 min. A double application of the same adhesive system was placed on the resected and blood-contaminated surface, but only the second application was light-cured. Quantitative measurements of fluid flow were again taken after 24-h storage in 37°C water.

RESULTS

No leakage was detected in the negative controls at any time. The positive controls allowed a mean leakage of 52.2 $\mu\text{l min}^{-1} \text{cm H}_2\text{O}^{-1}$. A two-way repeated measures, mixed model analysis of variance of the results indicated that there was a significant difference ($p < 0.0001$) between the leakage of contaminated and uncontaminated groups (Table 1). However, within the uncontaminated group, there was no clear significant difference among the four adhesives in their ability to seal the resected root surfaces ($p = 0.47$). There was no statistically significant interaction between the contaminated/uncontaminated groups and the adhesive materials ($p = 0.316$). With every material, the resin seals made to blood-contaminated resected dentin leaked significantly more ($p < 0.003$) than the resin seals made to uncontaminated dentin as determined by the least squares means test (Table 1). There were no significant differences between the adhesives in their ability to seal uncontaminated dentin. Although all adhesives applied to blood-contaminated dentin allowed more fluid leakage, none of the means were statistically different from each other.

DISCUSSION

Previous studies of the ability of restorative materials to seal root-end fillings in resected roots using fluid filtration have been performed at higher, nonphysiologic pressures (13, 14). Recent work by Camps et al. (15) indicated that pressures above 100 cm H₂O underestimate the hydraulic conductance of the specimen using this technique, making comparison of their results with those of this study invalid. Although blood contamination generally lowers the sealing ability of resins to dentin, a study of the sealing ability of bonding agents to resected root dentin revealed that blood contamination did not adversely affect the sealing ability of the resins (16).

All of the adhesives used in this study are self-etching, that is, they are intrinsically acidic enough to etch through smear layers into the underlying dentin. They also are self-priming, because they all contain hydroxyethyl methacrylate mixed with acidic monomers. The instructions call for no rinsing; instead, a gentle air stream is used to evaporate the solvent before the application of a separate adhesive layer that is light-cured. Only One-Up Bond F, among the adhesives that were used, is classified as an all-in-one adhesive.

One-Up Bond F is a new all-in-one adhesive that comes in two bottles. When one drop of each material is mixed, the solution turns a reddish-pink color. The manufacturer's instructions call for the mixture to be applied to the dentin surface without agitation for 20 s followed by light-curing for 10 s. This adhesive system is much simpler than the others, because no evaporation of solvent is required and no separate adhesive needs to be applied. This is an advantage because air blasts to bony wounds may stimulate bleeding, excessively thin the adhesive, or produce air emboli. Light-curing changes the color of the solution from red to colorless, confirming that the material was light-cured. Because there was no significant difference among the four adhesive systems in their ability to seal dentin, the selection of which adhesive to use should be based on simplicity of use. Clearly, One-Up Bond F was the simplest adhesive to place.

During microendodontic surgery, a certain degree of bevel often is required to facilitate access to the root end. Previous studies have reported that a significant increase in leakage occurred as the amount of bevel increased from 0 to 45 degrees (17). The study by Gilheany et al. (17) suggested that the increase in leakage was caused by the increase in the number of exposed dentinal tubules as the bevel angle became progressively larger. Because the angle of root resection was not varied between groups, the present study did not evaluate the prevention of microleakage from dentinal tubules. Previous work by Rud et al. (18) demonstrated that the introduction of a bonding agent into the dentinal tubules may help prevent apical leakage.

The reaction of periapical tissues to One-Up Bond F has not yet been determined. Animal studies on periapical wound healing should be conducted before the material is used in endodontic treatment. The long-term success of such treatment requires that the resin film remain impermeable to any underlying bacteria, bacterial products, or necrotic tissue and that the bond be durable for many years. If this or other resin adhesives promote cementogenesis over the material (19, 20), the result would be both an adhesive seal and a biologic seal. Further long-term studies should be performed on periapical healing adjacent to One-Up Bond F, because it is easy to use and seems to seal well, even during contaminated conditions.

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References

- Oynick J, Oynick T. A study of a new material for retrograde fillings. *J Endodon* 1978;4:203-6.
- Torabinejad M, Hong CU, Lee SJ, Monsef M, Pitt Ford TR. Investigation of mineral trioxide aggregate for root-end filling in dogs. *J Endodon* 1995;21:603-8.
- Tang HM, Torabinejad M, Kettering JD. Leakage evaluation of root end filling materials using endotoxin. *J Endodon* 2002;28:5-7.
- Pashley EL, Tao L, Mackert JR, Pashley DH. Comparison of *in vivo* vs. *in vitro* bonding of composite resin to the dentin of canine teeth. *J Dent Res* 1988;67:467-70.
- Xie J, Powers JM, McGuckin RS. In vitro bond strength of two adhesives to enamel and dentin under normal and contaminated conditions. *Dent Mater* 1993;9:295-9.
- Reeh ES, Combe EC. New core and sealer materials for root canal obturation and retrofilling. *J Endodon* 2002;28:520-3.
- Vignaroli PA, Anderson RW, Pashley DH. Longitudinal evaluation of the microleakage of dentin bonding agents used to seal resected root apices. *J Endodon* 1995;21:509-12.
- Haller B. Recent developments in dentin bonding, *Am J Dent* 2000;13:44-50.
- Derkson GD, Pashley DH, Derkson ME. Microleakage measurement of selected restorative materials: a new *in vitro* method. *J Prosthet Dent* 1986;56:435-40.
- Ciucchi B, Bouillaguet S, Holz J, Pashley D. Dentinal fluid dynamics in human teeth, *in vivo*. *J Endodon* 1995;21:191-4.
- Christiansen RL, Meyer MW, Visscher MB. Tonometric measurement of dental pulpal and mandibular marrow blood pressures. *J Dent Res* 1977;56:635-45.
- Held D, Thron HL. Etudes sur la circulation de l'os. I. La pression tissulaire de la moelle. *Arch Sei Physiol* 1962;16:166-77.
- Yatsushiro JD, Baumgartner JC, Tinkle JS. Longitudinal study of the microleakage of two root-end filling materials using a fluid conductive system. *J Endodon* 1998;24:716-9.
- Wu MK, Kontakiotis EG, Wesselink PR. Long-term seal provided by some root-end filling materials. *J Endodon* 1998;24:557-60.
- Camps J, Giustiniani S, Dejou J, Franquin JC. Low versus high pressure for *in vitro* determination of hydraulic conductance of human dentine. *Arch Oral Biol* 1997;42:293-8.
- Miles DA, Anderson RW, Pashley DH. Evaluation of the bond strengths of dentin bonding agents used to seal resected root apices. *J Endodon* 1994;20:538-41.
- Gilheany PA, Figdor D, Tyas MJ. Apical dentin permeability and microleakage associated with root end resection and retrograde filling. *J Endodon* 1994;20:22-6.
- Rud J, Munksgaard EC, Andreasen JO, Rud V, Asmussen E. Retrograde root filling with composite and a dentin-bonding agent. 1. *Endod Dent Traumatol* 1991;7:118-25.
- Andreasen JO, Munksgaard EC, Fredebo L, Rud J. Periodontal tissue regeneration including cementogenesis adjacent to dentin-bonded retrograde composite fillings in humans. *J Endodon* 1993;19:151-3.
- Fogel H, Peikoff M. Microleakage of root-end filling materials. *J Endodon* 2001;27:456-8.