

# The Effect of Etching Technique on the Retention of Adhesively Cemented Prefabricated Dowels

Fiorella L. Potesta, DDS, MS,<sup>1,2</sup> James C. Broome, DDS, MS,<sup>3</sup> Sandra J. O'Neal, DMD, MS,<sup>4</sup> Daniel A. Givan, DMD, PhD,<sup>5</sup> & Lance C. Ramp, DMD, PhD<sup>5</sup>

<sup>1</sup>Private practice, Orlando, FL

<sup>2</sup>Formerly Assistant Professor, Department of Prosthodontics, School of Dentistry, University of Alabama at Birmingham, Birmingham, AL

<sup>3</sup>Associate Professor, Department of Prosthodontics, School of Dentistry, University of Alabama at Birmingham, Birmingham, AL

<sup>4</sup>Professor of Dentistry and Chair, Department of Prosthodontics and Biomaterials, School of Dentistry, University of Alabama at Birmingham, Birmingham, AL

<sup>5</sup>Assistant Professor, Department of Prosthodontics, School of Dentistry, University of Alabama at Birmingham, Birmingham, AL

#### Keywords

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#### Correspondence

Fiorella Potesta, UAB—Prosthodontics, 1919 Seventh Ave. S, R 606, SDB, Birmingham, AL 35294. E-mail: fpotestak@aol.com

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## Abstract

**Purpose:** To determine if etching technique influences the bond strength of resin cement to root canal dentin.

**Materials and Methods:** Fifty-five extracted teeth were endodontically treated, dowel space prepared, and divided into five groups. Each group was treated with different etchant consistencies: acid gel, semi-gel, low-viscosity gel, liquid, and a self-etching primer. After dowel cementation, four sections were removed from each root and a push-out test was performed.

**Results:** Significant effects were found for etching procedure and for location within the root canal. The apical segment produced the lowest bond strength. Self-etching primer showed the highest bond strength.

**Conclusions:** The consistency of etchant material influenced the bond strength of a prefabricated dowel in the canal.

Dowels and cores are often used in the restoration of endodontically treated teeth that have suffered excessive loss of coronal tooth structure. In such cases, cementation of a dowel inside the root canal is used to provide retention for the final restoration<sup>1</sup>; however, it has been shown that root preparation for dowel insertion can result in additional loss of tooth structure.<sup>2</sup> This can lead to catastrophic root fracture under long-term clinical use.<sup>3,4</sup> In recent years, the choice of materials in the preprosthetic restoration of endodontically treated teeth has changed. Metal dowels (cast or prefabricated) are more rigid than dentin, which makes the dowel the stiffest element in the restoration of an endodontically treated tooth. In function, load may be transferred to the canal walls, which may result in root fracture.<sup>5-8</sup> Adhesively cemented prefabricated dowels exhibit a modulus of elasticity similar to dentin, leading to a better distribution of forces.<sup>9</sup> Bonding within the root canal is the most unfavorable situation for the clinical use of the current dentin-enamel bonding system. Various factors contribute to the difficulty of this procedure. These include limited visualization of the depth of material penetration into the canal and variability in root structure.<sup>10</sup> Formation of the hybrid layer and resin tags is considered the most important factor in resin retention to dentin and is an essential mechanism of adhesion between dentin bonding and the dentin substrate. Etching is the principal factor in the demineralization of the dentin and smear layer dissolution. Different factors, such as the etching time, acid concentration, and liquid versus gel formulation of the product, can affect the demineralization pattern of the dentin and subsequently the bond strength of the adhesive. Numerous studies have been performed regarding dentin substrate and bonding system<sup>11-13</sup>; however, no studies have compared the effect of different types of etchants in the canal on bond strength to root canal dentin.

The purpose of this in vitro study was to evaluate the bond of resin cement to root canal dentin and the effect of various etchant materials on the morphology of root canal dentin.

## **Materials and methods**

Fifty-five extracted human single-rooted teeth were obtained from local surgeons. The teeth were cleaned of tissue and debris, steam autoclaved for two cycles, and stored in saline prior to use. Steam autoclaving has been shown to be effective in sterilizing extracted teeth without affecting the bond strength of adhesives to dentin.<sup>14,15</sup> Radiographs were taken in buccolingual and mesiodistal directions. Teeth with multiple canals and irregularly shaped canals were eliminated from the study. Fiftyfive teeth were selected based on similarities in canal shape, size, and location. Teeth included in the study had a root length of at least 16 mm from the cemento-enamel junction (CEJ) to the anatomical apex. The crowns were removed from the root at the level of the CEJ using a low-speed diamond wheel (Brasseler, Savannah, GA), and the crowns were discarded. Endodontic treatment was performed on all teeth; lateral condensation was performed on all teeth using Roth's eugenol-based cement (Roth International Ltd., Chicago, IL) and gutta percha. A 0.04 tapered size 45 master gutta percha cone was placed to working length in the canal, and nonstandardized accessory points and a finger spreader were used to complete the lateral condensation. Following storage in normal saline for 72 hours, each canal was prepared to a standard depth of 13 mm, leaving at least 3 mm of gutta percha at the apex. Peeso reamers were used to remove 10 mm of gutta percha, starting with size 2 up to size 4. Following the reamers, a number 2 preshaping and finishing drill (D.T. Light-Dowel System, Bisco, Inc., Schaumburg, IL) was used to finalize preparation of the canal space to a standard depth of 13 mm.

Ten teeth per group were randomly assigned to five experimental groups as detailed in Table 1.

Custom endodontic dowel replicas were fabricated using flowable composite (Aelite-Flo, Bisco, Inc.) to avoid failures at the dowel–cement interface and to simulate the effect of the pressure generated when the dowel was inserted into the canal. For groups 1-4, acid was injected into the canal with a tuberculin syringe and allowed to sit for 15 seconds, rinsed with water from an air/water syringe for 10 seconds, and air dried. Paper points were used in all groups after etching to dry the canals. All-Bond 2 (Bisco, Inc.) was used as the adhesive for these groups. Two coats of Primer B were applied to the canal with a brush and lightly air-dried. Following cleaning with an alcohol wipe, Primer B was also applied to the dowel. Cement (Hi-X, Bisco, Inc.) was mixed and loaded into a unit-dose needle tip

Table 1 Experimental groups

Group	Etchant material
1	37% phosphoric acid gel (Scotchbond Etching Gel, 3M ESPE, St. Paul, MN)
2	37% phosphoric acid low-viscosity semi gel (Bisco, Inc, Schaumburg, IL)
3	37% phosphoric acid low-viscosity gel (Bisco, Inc).
4	37% phosphoric liquid acid, (Concise Etching Liquid, 3M ESPE)
5	Single step bonding system (Panavia F, Kuraray Medical, Inc., New York, NY)

(Needle Tube, Centrix, Shelton, CT). Cement was injected into the canal, starting at the apical end and withdrawing back toward the orifice. For group 5, one-step bonding system Panavia F was used for etching, priming, and bonding. After placement of the cement, the composite dowel replica was gently seated into the canal, and firm pressure was maintained on the dowel for 10 seconds. Excess cement was removed with a brush.

#### **Push-out test specimen preparation**

The push-out test is an extrusion test for determining bond strength to dentin.<sup>16</sup> This test applies a constant vertical force to the specimen, evaluating bond strength between luting material and root dentin. Seventy-two hours after cementation and storage in normal saline solution, four sections were removed from each root starting at the cervical root end. The roots were sectioned at right angles to their long axis using a custom-made holding device. Sections were 2-mm long (Fig 1). The resulting four root sections allowed evaluation of the bond strength of dowel cementation to dentin at standardized levels within the root.

Using a caliper, the diameter of the cemented dowel canal on the cervical and apical sides of each root section and the length of each root section were measured. The bonded surface area was determined using the formula for a truncated cone. All sectioned specimens were stored in normal saline for 24 hours before testing. A push-out test was performed on different sections of the root using a Model 858 Mini Bionix (MTS Systems Corporation, Eden Prairie, MN), a pushing device with two different diameters according to the level of the tooth to be tested (Fig 2), and a custom-made mount. The sections of cemented dowels were pushed from the tooth at a rate of 0.5 mm/min using a 100 N cell. Results were reported in MPa. Data were analyzed using a one-way ANOVA for each variable and two-way ANOVA for a combination of both variables. A dowel hoc Scheffé test was used to analyze differences among each group between levels and etching techniques.

#### **SEM** specimen preparation

One tooth from each experimental group was evaluated using SEM Model SX- 30E (ISI, Milpitas, CA) to determine the adaptation of the adhesive and cement to the root canal dentin by observing resin tag formation. These specimens were placed into hydrochloric acid (37.2%) for 36 hours to remove the organic and mineral components of the dentin. The remaining polymer specimens were mounted on scanning electron microscope (SEM) stubs and sputter coated with gold palladium alloy (Model Hummer X, Anatech Ltd., Hayward, CA). This allowed for visual examination of resin tags. An overall picture of remaining dowel was taken at  $12 \times$  magnification. Then each dowel was divided into four sections representing the four levels of interest (Fig 2). Each section was examined at higher magnification, and a representative section was photographed at  $1000 \times$  magnification.



**Figure 1** (A) Pipette tip in a block; (B) application of the etchant; (C) rinsing of etchant; (D) penetration of etchant after rinsing.



Figure 2 Root after section. Four root sections were divided in (left to right): cervical level; under cervical level; upper apical level; and apical level.

## Results

#### **Push-out test**

The mean values for the push-out test considering the effect of adhesive technique are shown in Table 2. Scheffé analysis showed significant differences within the groups. Group 5 showed the highest bond strength, and group 2 showed the lowest bond strength. Group 1 demonstrated the second highest bond strength. Group 3 showed a statistically significant difference when compared to the other four groups. Groups 2 and 4 did not show any statistically significant difference when compared to each other, although they did show a significant difference as compared to groups 1, 3, and 5 (Table 2, Fig 3).

Table 2 Mean values for bond strength of the five experimental groups

Groups	Count	Mean (MPa)	SD	SE
1 (gel etchant)	40	4.494	2.783	0.440
2 (semi-gel etchant)	40	1.725	1.895	0.300
3 (low-viscosity gel etchant)	40	3.148	2.128	0.336
4 (liquid etchant)	40	1.838	0.939	0.148
5 (single step)	40	6.363	2.987	0.472



Figure 3 Mean values at failure for the five experimental groups.

As shown in Table 3 and Figure 4, the bond strength was compared within each level and showed a statistically significant difference between the four levels, with the highest bond strength at the cervical level (level A) and the lowest at the apical level (level D). As seen in Table 3, level A showed a mean of 5.6 MPa, which is statistically significant when compared to the other three levels. The second highest bond strength was found in level B with 4.0 MPa. A statistical significance exists when compared to levels A, C, and D. Level C shows a mean of 3.0 MPa at failure load and is also statistically significant when compared to the other levels. Level D, which is the apical level, shows the lowest bond strength (1.5 MPa), and has a statistically significant difference.

#### **SEM** evaluation

Using SEM, the interface between adhesive and root dentin was observed. Resin tag formation was different among the four levels. The analysis of group 1 showed resin tags with varying density along the endodontic preparation. Resin tags were longer at the cervical level and shorter and less dense at the apical level (Fig 5). In group 2, SEM analysis demonstrated the presence of few irregular resin tags over the dowel surface. In group 3, resin tags were longer at the cervical level and shorter and less dense at the apical level. Group 4 showed resin tags with varying density along the endodontic preparation, and in group 5, resin tags were longer at the cervical level and shorter and less dense at the apical level.

# Discussion

Bonding in root canal is probably the most unfavorable situation for the clinical use of a bonding system. This study showed higher bond strength at the cervical level and lower bond strength at the apical level. These results are similar to

Tab	le 3	Mean	value	tor	bond	strength	at	different	levels
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Tooth section	Count	Mean (MPa)	*	SD	SD
Level A (cervical)	50	5.597	А	3.124	0.442
Level B	50	4.004	В	2.680	0.379
Level C	50	2.960	С	2.109	0.298
Level D (apical)	50	1.494	D	1.522	0.215

\*Different letters represent statistical significance (p < 0.05).



Figure 4 Bond strength at four different levels. Level A shows the highest bond strength, and level D shows the lowest.

those found by Kanca and Sandrik<sup>17</sup> and Mannocci et al<sup>18</sup> who demonstrated differences in density of resin tag formation between the coronal area and apical area when observed with SEM evaluation.

Clinically, the inability to visualize the flow of the material within the canal decreases the control over the material, creating areas where the smear layer cannot be removed. Consequently, collagen fibers are not exposed, and the dentin becomes impermeable to the flow and proper bonding of endodontic dowels. The differences in bond strength within the canal correlate with the findings of Pashley et al<sup>15</sup> who found that dentinal tubule numbers and diameters are lower toward the apex, leading to lower bond strengths in that region.

Access and visibility play an important role in the bonding procedure. It is very difficult to control moisture and surface coverage by the etchant and adhesive inside the canal. Moisture control is done by the use of air-drying or by the use of some kind of drying device such as cotton pellet, foam pellet, or paper points. The importance of moisture control is related to the fact that most adhesive systems require the presence of moisture on the dentin surface, which will allow the adhesive to penetrate into the collagen fibril mesh and the dentinal tubules after acid-etch treatment.<sup>17</sup>

Another important factor is how to deliver the etchant and the adhesive to the deepest portion of the canal space and effectively cover the whole surface. Etchants come in a syringe presentation, where the needle tip is not long enough to reach the apical portion of the tooth. Furthermore, the viscosity of the gel does not allow the etchant to flow along the canal wall, leaving unetched areas. It is very difficult to determine if remaining etchant is left inside the canal space after rinsing. Therefore, it is recommended that thorough rinsing be done to avoid this possible situation. Removal of residual etchant is very important, because it may interfere with the chemistry and penetration of the adhesive, resulting in a low pH environment and, consequently, inhibition of polymerization of the material. It can also cause improper resin tag formation and poor bond strength. Williamson et al<sup>10</sup> evaluated the effect of rinsing technique and canal size on etchant removal. The influence of rinsing technique and canal sizes on the operator's ability to achieve neutral pH within the canal was evaluated by measuring the pH of the canal with a paper point and applying it to pH measuring strips. They concluded that the commonly used technique of rinsing etchant from the canal with an air/water syringe is ineffective and may have adverse consequences for achieving adhesive bonding. Our findings for bond strength are related to those obtained in the previous study. Bond strength values were lower at the apical level, and this finding could be correlated to the fact that rinsing etchant from the canal with an air/water syringe is ineffective.



Figure 5 Scanning electron microscope (SEM) evaluation at different levels for group 1, 37% phosphoric acid gel. (A) Level A (cervical level)—resin tags long and dense; (B) level B—resin tags are long and dense; (C) level C—resin tags are short and less dense; (D) level D (apical level)—resin tags are short and less dense. Resin tags show similarity for all groups.

Ferrari et al<sup>19</sup> reported the differences in the bonding mechanism (resin tag formation) between the use of a brush and a microbrush as a carrier of adhesive into the root canal space. They reported that the length of resin tags were significantly different between the two groups studied. The group where a brush was used as a carrier showed uniform tag formation in the coronal and middle third but was less evident in the apical third. In the group where a microbrush was used, there was uniform resin tag formation in all thirds of the root canal space. The reason for these findings is how the shape of the microbrush allows it to go deeper in the canal and deliver more adhesive in the apical third. In our study, we examined the use of different etchant consistencies with the purpose of comparing their effects. All groups showed a difference in bond strength at various levels. The viscosity of these gel etchants did not allow the etchant to flow along the canal walls; however, in group 4 there was no difference in bond strength within the levels. But it shows low bond strength when compared to the gel etchants. Baharav et al<sup>20</sup> compared the efficacy of liquid and gel acid etchants and found a similar penetration when they were combined with mechanical agitation. In the study, when mechanical agitation was not used, the gel-type etching agent produced wider and deeper penetration. A limitation of this study was that it was done on a flat visible surface. This has little clinical relevance because there is no mechanical agitation of the etchant when it is applied inside the canal. This rationale may explain why in our study, gel etchants showed better penetration than liquid etchants; however, liquid etchants flowed better and created an even bonding mechanism along the root canal.

Self-etching adhesives are becoming extremely popular among clinicians, and it is believed they behave in a similar fashion to conventional total etch adhesive systems. They do not require separate rinsing of the phosphoric acid, and strict moisture control is eliminated, because water is an essential component of these adhesives. These adhesives are more acidic than conventional adhesives by virtue of their increased concentrations of low pH acidic resin monomers. Self-etching primers are also being tested for use to bond fiber dowels inside the canal space, due to the simplicity of the technique. In a study by Foxton et al,<sup>21</sup> it was reported that no differences were found in the microtensile bond strength values at the coronal and apical third of root canal dentin when using a dual cure self-etching primer adhesive resin and a dual-cure resin composite luting agent. Our findings for bond strength were different than those of Foxton et al.21

The effect of etchant consistency was also evaluated in this study. Our expectation was that the bond strength would increase near the apex when liquid etchants were used, due to their improved flow properties; however, this was not completely supported by our results. Although liquid etchants flowed better and created a more even bond strength within the canal, they did not have good penetration into the dentin. This was confirmed by the density and size of the resin tags seen in the SEM evaluation.

## Limitations of the study

There are many factors that could have influenced the results of this study. First is the anatomical and histological variation between teeth, including dentinal tubule numbers and diameters that are lower toward the apex, leading to lower bond strengths apically.<sup>15</sup> In this study, every tooth was evaluated with a radiograph, and all the teeth had similar characteristics; however, each tooth had variable morphology, including undercuts, various diameters, and canal shapes. During the push-out tests, mechanical retention was found due to the undercuts, causing misleading results. Even though all the canals are prepared prior to dowel placement, there is always a possibility that the minor undercuts create some mechanical retention. This in turn could lead to bond strength values that are artificially elevated. Second, the diameter of the canal in relation to the dowel diameter can create various thicknesses of cement. The presence of small cement thickness allows for more even distribution of the transmitted load. It also decreases the amount of polymerization shrinkage of the resin material and the associated stress of shrinkage. Sometimes due to diverse root canal anatomy clinicians must deal with different cement thicknesses at various levels, which is a factor in considering reduced bond strength. A study by Bruggemann et al<sup>22</sup> compared the effect of dentin age and acid-etching time and found differences between younger dentin and older dentin. When etchant was applied for 15 seconds, there was statistically significant difference in the bond strength between young and old dentin; however, when etchant was applied for 30 seconds, no difference was observed in bond strength. In this in vitro study, teeth were selected randomly irrespective of the age of the patient. Based on Bruggemann et al's<sup>22</sup> results, this could be a possible source of variability of the results. The differences between the specimens used in this study showed a high coefficient of variation in the data; to improve this, we would have to have a higher number of specimens, and they would have to be more uniform.

## Conclusion

Within the limitations of this study, it can be concluded that consistency of etchant material did influence the bond strength of a prefabricated dowel in the canal and that the penetration of the etchant material inside the canal is insufficient, creating a difference in bond strength along the root canal dentin, being stronger at the cervical level and weaker at the apical level. Of clinical significance, this study showed that the gel etchants gave a better result over liquid etchants, mechanical pumping action during etching procedures is recommended to improve bonding at the apical level, and self etchant systems can be considered an option for bonding inside the canal.

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