

Original Article

Effect of MTAD on the shear bond strength of self-etch adhesives to dentin

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ABSTRACT

Background: As the use of different irrigants to eliminate residual debris and smear layer in the field of endodontic is unavoidable, by considering the effect of irrigants on the bond strength of resin composite restorations, this study was designed to evaluate the effect of a mixture of a tetracycline isomer, an acid, and a detergent (MTAD) on the shear bond strength of two self-etch adhesives, Clearfil SE Bond and Adper Prompt L- Pop to dentin.

Materials and Methods: The crowns of 80 extracted premolars were transversally sectioned to expose dentin. Flat dentin surfaces were wet abraded with 320-grit abrasive paper and randomly assigned to eight groups according to two self-etch adhesive and four dentin surface treatments: direct application over smear layer (no treatment), etching with 35% phosphoric acid for 15s, 1 min 5.25% NaOCl/1 min MTAD and 20min 1.3% NaOCl/5min MTAD. Shear bond strength was tested 24 h after storage in distilled water at 37°C in incubator. Data were analyzed using one-way ANOVA followed by duncan post-hoc ($\alpha=0.05$).

Results: Phosphoric acid etching prior to SE Bond application significantly decreased the shear bond strength to dentin ($P<0.05$). Application of MTAD clinical protocol (20min 1.3% NaOCl/5min MTAD) did not significantly decrease the shear bond strength of self-etch adhesives to dentin ($P=0.745$).

Conclusions: Based on the results of present investigation, it seems that the use of clinical protocol of 1.3% NaOCl as a root canal irrigant and a 5-min application of MTAD as a final rinse to remove the smear layer has no adverse effect on the shear bond strength of self-etch adhesives to dentin.

Key Words: Adhesives, bond strength, smear layer, MTAD

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INTRODUCTION

The effective cleaning and shaping of root canals and appropriate three dimensional seal are the main goals of endodontic treatments.^[1] Different methods of cleaning and shaping, produce a smear layer which

can cover the root walls and dentin tubules.^[2,3] This layer consists of organic and inorganic substances and is capable to prevent the penetration of intracanal medications into the dentinal tubules. This prevention can pose some devastating effects on the close adaptation of root canal filling materials and the root canal walls.^[4] As a consequence, the use of irrigants to eliminate residual debris and smear layer is mandatory. These irrigants should have the capacity to flush out all residual debris, lubricate root canal walls and dissolve the organic materials. In the best condition, these materials show high antibacterial capacity.^[5] According to all mentioned features, there are a wide range of irrigants like EDTA,

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NaOCl, chlorhexidine and iodine potassium iodide (IKI) which should be chosen based on clinicians' experience. From all common irrigants, it has been declared that a mixture of a tetracycline isomer, an acid, and a detergent (MTAD) can fulfill all goals and is effective in removing a smear layer.^[6-8] This material was introduced for the first time by Torabinejad *et al* in 2003. This material was a mixture of tetracycline isomer (doxycycline), an acid (citric acid) and a detergent [tween 80].

Coronal seal plays an important role in the final success of endodontically filled teeth.^[9] So, teeth restoration immediately after root canal treatment can dramatically decrease the coronal leakage. Other side, the use of resin composites has been suggested for the restoration of endodontically treated tooth.^[10] But some researchers have indicated that the use of intracanal irrigants can change the dentinal properties and consequently affect the composite resin bond strength.^[5,9]

Therefore, achieving predictable bond to dentin has long been a goal and challenge in restorative dentistry. Current dentin adhesives employ two different approaches to achieve the goal of micromechanical retention between resin and dentin.^[11,12] The first method is total-etch or etch and rinse technique, attempts to remove the smear layer completely via acid-etching and rinsing. The second approach, the self-etch technique, aims at incorporating the smear layer as a bonding substrate.

To assess the effect of different irrigants on resin composites bond strength, we can mention J Nascimento Santos study in 2006 which showed that different irrigants pose different effects on bond strength.^[1] Also in the study by Yurdagüven *et al*, it was concluded that in order to use MTAD as an irrigant, it is better to use etch and rinse approach instead of self-etch and significant difference was observed in the microtensile bond strength after using MTAD as an irrigant.^[9]

Because of using different irrigants specially MTAD as an effective material in the field of endodontic and their effect on the bond strength of resin composite restorations, this study was designed to evaluate the effect of MTAD on the shear bond strength of two self-etch adhesives, Clearfil SE Bond and Adper Prompt L-Pop to dentin.

MATERIALS AND METHODS

Eighty human premolars were stored in 0.1%

Chloramine T. Only caries-free, unrestored teeth with no cracks on their crowns were selected. Teeth were sectioned approximately 1 mm below the cemento-enamel junction (CEJ) to expose deep-coronal dentin surface. All specimens were mounted in poly methyl methacrylate, using a Teflon mold leave the flat surface exposed. The sides with the flat bonding surfaces were polished with 320-grit abrasive papers.^[13] The teeth were randomly divided into eight groups ($n=10$). Before bonding, the dentin surfaces were prepared in the following manners:

- Groups 1 and 5: No treatment was applied (control groups).
- Groups 2 and 6: The bonding surface was etched for 15 seconds with 35% phosphoric acid (3M ESPE, St. Paul, MN, USA).
- Groups 3 and 7: The bonding surface was irrigated with 5.25% NaOCl solution (Merck, Darmstadt, Germany) for 1 min, followed by 1-min application of MTAD.
- Groups 4 and 8: The bonding surface was treated with MTAD, using clinical protocol (20 min rinsing with 1.3% NaOCl, followed by 5-min application of MTAD).

After treatment, all prepared bonding surfaces were rinsed with distilled water and gently dried using oil free air syringe. Then all prepared surfaces, treated with Clearfil SE Bond (Kuraray Co Ltd., Osaka, Japan) bonding agent in groups 1, 2, 3 and 4 and with Adper Prompt L-Pop (3M ESPE) in groups 5, 6, 7 and 8 according to the manufacturers' instructions. After the dentin bonding agents were polymerized, a 2-mm inner diameter (2-ID) and 3-mm height Teflon tube was attached to the flattened dentin surfaces. The tube was subsequently filled with Z100 (3M) composite resin and light cured with an Optilux 401 curing light (Kerr. Demetron, Danbury, CT) from the top of the mold for 40s. The bonded specimens were stored in distilled water at 37°C for 24 hours, followed by 500 thermal cycles between 5 and 55°C baths. Shear bond strength was measured using a Universal Testing Machine (Dartec, England) at a crosshead speed of 1 mm/min. The mode of failure was assessed with a stereomicroscope (MBC-10, Number N9116734, SF-100B, Lomo, Russia) at 28× magnification. Shear bond strength data was statistically analyzed using one-way ANOVA followed by Dunnett's post-hoc Duncan's post-hoc. All statistical tests were run at 5% level of significance ($\alpha=5\%$).

One specimen from each group was sectioned through the resin-dentin interfaces using a low-speed rotary cutting machine. The cut surface of specimen was polished with 2000-grit abrasive and etched with 35% phosphoric acid for 15s to remove the inorganic substrate and rinsed under a stream of water. The specimens were then immersed in a 5.25% sodium hypochlorite solution for 30 min to dissolve the organic substrate and water-rinsed. The specimens were dehydrated by placement in ascending concentrations of ethanol and then gold-coated (BAL-TEC SCD 005 Sputter coater, GA, USA). The tooth-resin interfaces were observed with a Scanning Electron Microscope (Philips XL30, Philips, the Netherlands).

Table 1: Mean shear bond strength (MPa) and standard deviations for Clearfil SE Bond and Adper Prompt L-Pop

Surface treatment	Clearfil SE Bond	Adper Prompt L-Pop
Control group (no treatment)	27.05±5.01	21.29±3.41
Acid etch	19.90±3.54	16.23±3.77
NaOCl/MTAD	24.78±7.59	20.52±5.09
MTAD (clinical protocol)	21.16±7.10	20.08±2.39

Table 2: Mode of failure for dentin bonding specimens (n=10)

Surface treatment	Clearfil SE Bond			Adper Prompt L-Pop		
	A	C	M	A	C	M
Control group (no treatment)	0	1	9	1	5	4
Acid etch	5	1	3	3	1	6
NaOCl/MTAD	1	3	6	1	3	6
MTAD (clinical protocol)	1	4	5	1	3	6

A: Adhesive failure, C: Cohesive failure, M: Mixed failure

RESULTS

Shear bond strength results

The means and standard deviations of the shear bond strength of Clearfil SE Bond and Adper Prompt L-Pop are presented in Table 1. The respective modes of failure are listed in Table 2.

The mean shear bond strength without phosphoric acid etching was 27.05 MPa for Clearfil SE Bond and 21.29 MPa for Adper Prompt L-Pop. When the phosphoric acid etching was performed, mean bond strength was 19.90 MPa for Clearfil SE Bond and 16.23 MPa for Adper Prompt L-Pop. When the data for shear bond strength were statistically analyzed, it was concluded that for Clearfil SE Bond, phosphoric acid pretreatment significantly decreases the shear bond strength to dentin ($P<0.05$), but for Adper Prompt L-Pop, there was no significant difference between two groups (etched and non-etched) ($P>0.05$). Duncan's post-hoc showed that there were no significant differences between the experimental surface treatment groups (NaOCl/MTAD, MTAD clinical protocol) and the control groups (no treatment).

Scanning electron microscopic observations

Figures 1-4 show the interaction of resin-dentin in various groups.

Figure 1 shows typical images of the dentin-resin interfaces in the cross-sectioned specimens treated by self-etch adhesives (Groups 1 and 5).

Figure 2 shows the interfaces in the specimens with etching (Groups 2 and 6). Phosphoric acid etching yielded thicker hybrid layers and distinct resin tags.

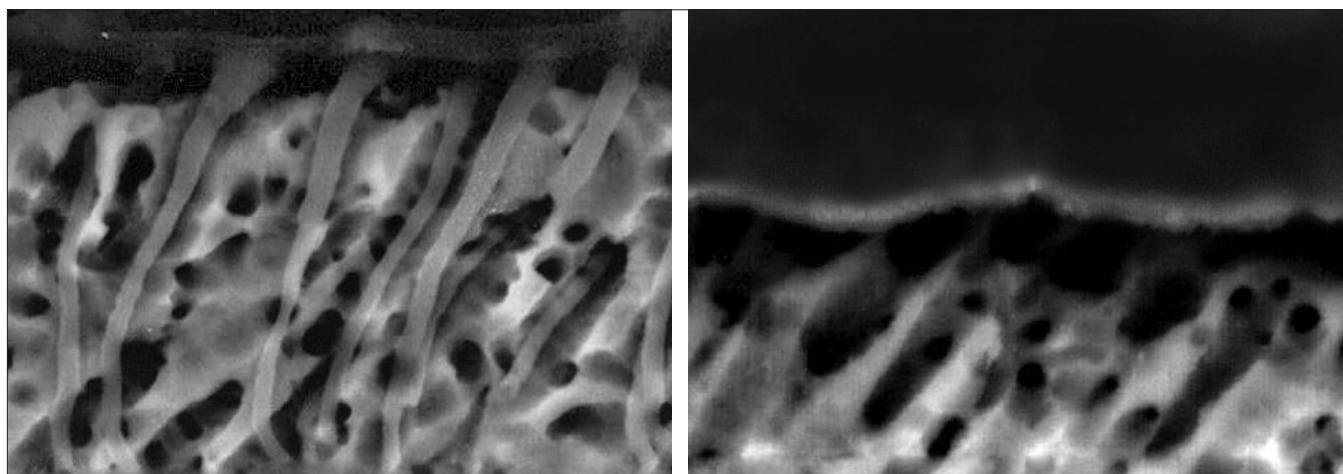


Figure 1: SEM images of the dentin-resin interfaces in the cross-sectioned specimens without any treatment. Left: Clearfil SE Bond. Right: Adper Prompt L-Pop. A hybrid layer 1-2 μ Clearfil (SE Bond) and 2-3 μ (Adper Prompt L-Pop) was created at the interface in both groups

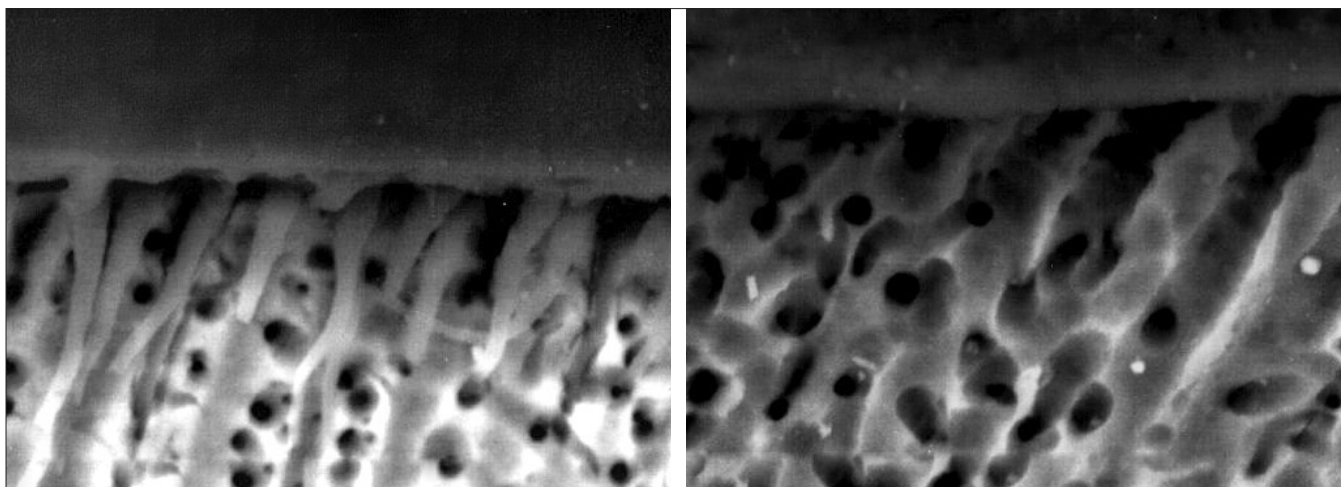


Figure 2: SEM images of the dentin-resin interfaces in the specimens with phosphoric acid etching. Clearfil SE Bond, Adper Prompt L-Pop. Thick hybrid layers and resin tags observed

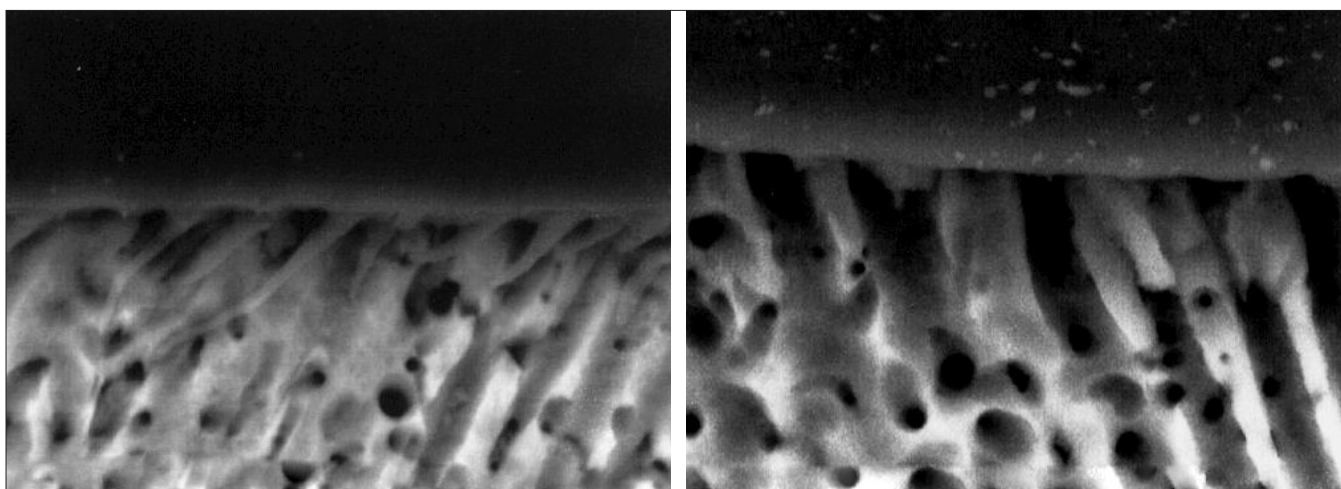


Figure 3: SEM images of the dentin-resin interfaces in the specimens with NaOCl/MTAD rinsing. Clearfil SE Bond, Adper Prompt L-Pop. A hybrid layer 2-3- μ thick was created at the interface in both groups

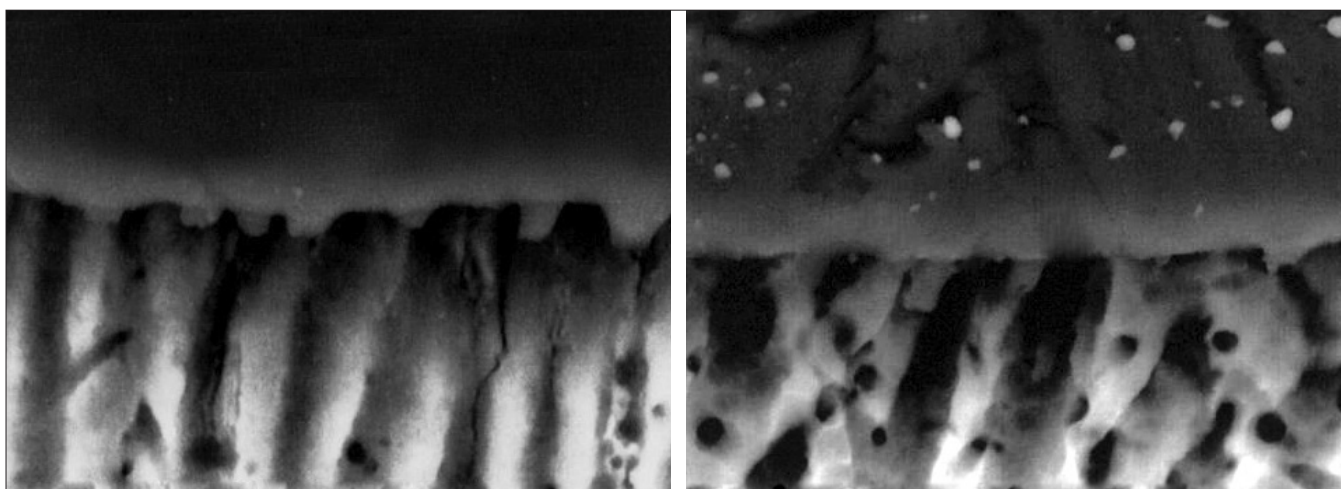


Figure 4: SEM images of the dentin-resin interfaces in the specimens treated by MTAD clinical protocol. Clearfil SE Bond, Adper Prompt L-Pop. The hybrid layer is approximately 2-3- μ thick

Figures 3 and 4 show the interfaces in the specimens with MTAD pretreatments (Groups 3, 5 and 7, 8).

DISCUSSION

Different studies have shown that common techniques in root canal cleaning and shaping are capable to produce a smear layer which can prevent the penetration of intracanal medicines into dentinal tubules. So it is necessary to remove this layer in order to provide an appropriate adaptation between root canals and intracanal medicines.^[4] It has been reported that various chemical agents pose an adverse effect on the dentin structure which can ultimately change the properties of different adhesives;^[5] on the other hand stable adhesion between resin composite and tooth is essential for clinical success of restorations as adhesions failure in margins, allows leakage of oral fluid and bacterial invasion.

In the present study, to survey the effects of MTAD on bond strength, some specimens were exposed for 1 min to 5.25% NaOCl before 1 min exposure to MTAD. Some specimens were exposed for 20 min to 1.3% NaOCl before a 5 min exposure to MTAD as recommended for performing root canal therapy.^[14]

In all mentioned specimens, a reduction was seen in shear bone strength but this reduction was not significantly difference with control group for each adhesive.

MTAD is an intracanal irrigant with mixture of doxycycline, citric acid and tween 80. Doxycycline is among the strongest anti-collagenase in tetracycline antibiotic group.^[15] Barkhordar *et al* concluded that 100 mg/ml solution of doxycycline, because of its acidic capacity is effective to remove smear layer from root surface.^[16] It has been reported that doxycycline's molecules can react with dentin calcium molecules^[17] and this reaction can act as a barrier to dentin, which preclude the penetration of monomer molecules into dentinal tubules and ultimately reduce the bond strength of adhesives to dentin.

Because of the presence of citric acid in MTAD, it has a high acidic capacity (PH \approx 2).^[6] This acidic property of MTAD, acts as an etchant agent that can effectively remove the mineral contents of smear layer. Garcia-Godoy *et al* in their study declared that the use of MTAD can cause dentin matrix to collapse^[18] and this condition, limits the penetration of adhesives into dentin collagen matrix that eventually reduce the bond strength of adhesives to dentin.

In the study by Yurdaguvan *et al* in 2009, aiming at evaluation of the effect of intracanal irrigants on the micro tensile bond strength of dentin adhesives, it was concluded that the mean micro tensile bond strength in self-etch adhesives under the effect of MTAD, showed a significant reduction in compare to control group. Also it seems that etch and rinse adhesives are better choice than self-etch adhesives when MTAD is a selective intracanal irrigants.^[9]

The results of Machnick study in 2003 showed that, tooth under root canal treatments which were initially rinsed with 1.3% sodium hypochlorite for 20 minutes and then with MTAD for 5 minutes, do not need acid etching prior to the use of adhesive, it means that by this technique the need for etching step was eliminated.^[19]

All mentioned and present studies show the effect of MTAD on the dentin structure. As the use of MTAD on dentin before self-etch adhesives, increase the dentin demineralization depth and consequently increase the difference between the depth of demineralization and resin penetration, this will uncover some parts of collagen network beneath hybrid layer, which can cause a reduction in bond strength.

As by increasing the usage time of MTAD, the demineralization depth increase, bond strength of specimens, rinsed with MTAD for 5 minutes was higher than specimens rinsed with MTAD for 1 minute.

In our study, etching of the dentin with phosphoric acid prior to the application of Clearfil SE Bond significantly decreased the bond strength. Along with this decrease in bond strength, an increase in the number of adhesive failures was observed. The acidic adhesive monomer in the self-etching adhesives decalcifies the dentin and simultaneously infiltrates into the dentin, so that it is able to diffuse into the deepest area of the decalcified dentin. The presence of adhesive monomer with adequate concentration facilitates the infiltration and diffusion of the bonding resin, subsequently applied and it can create a dense and firm hybrid layer after polymerization of the bonding resin. In the specimens etched with phosphoric acid prior to self-etch primer, a thick hybrid layer was formed at the resin-dentin interfaces but its appearance showed a porous structure.^[20]

Several authors have suggested that incomplete infiltration of the demineralized collagen network by the bonding resin^[20,21] is the underlying cause for this decrease in bond strength. Phosphoric acid

can decalcify a deeper area of the dentin than self-etching primers because its acidity is stronger than the primers. It has been reported that the bonding agent cannot infiltrate the whole of the dentin layer, decalcified by an etchant, and thus non-resin-impregnated dentin remains between the hybrid layer and the intact dentin.^[22] Since we employed the dry bonding technique, collapse and shrinkage of the collagen network should have occurred.^[23,24] Any collapse, even partial, may hinder efficient resin infiltration, leading to porous zone, in particular at the bottom of the hybrid layer.^[25,26] Nevertheless, the water-containing primer of Clearfil SE Bond must be able to re-expand this network at least in part.^[27]

Unlike with etch and rinse adhesives, not all hydroxyapatite were removed from the hybrid layer in dentin as the demineralization by mild self-etch adhesives (pH \approx 2) is restricted both in depth and in extent.^[28] Some researchers have pointed out that the functional monomers in self-etch adhesives can chemically interact with hydroxyapatite, and this chemical interaction provide better resistance against micro- and nano-leakage.^[29-32] The lack of chemical interaction between the functional monomer 10-MDP in Clearfil SE Bond and hydroxyapatite may also account for a lower bonding effectiveness, as hydroxyapatite is no longer available throughout the hybrid layer after phosphoric acid etching.^[28]

Our results indicate that pretreatment with phosphoric acid decreases the shear bond strength of Adper Prompt L-Pop to dentin, however, this difference was not significant ($P>0.05$).

Although the present study investigates the effect of MTAD on the shear bond strength of self-etch adhesives, it is recommended that other studies should be conducted to survey the effect of MTAD in longer duration.

CONCLUSIONS

Based on the results of the present investigation, it seems that the use of clinical protocol of 1.3% NaOCl as a root canal irrigant and a 5-min application of MTAD as a final rinse to remove the smear layer has no adverse effect on the shear bond strength of self-etch adhesives to dentin.

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