



ORIGINAL RESEARCH

Evaluation of the effect of MTAD in comparison with EDTA when employed as the final rinse on the shear bond strength of three endodontic sealers to dentine

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Abstract

The purpose of the study was to evaluate the effect of MTAD in comparison with EDTA as a final rinse on the shear bond strength of Kerr, Apexit and AH plus. Dentine surfaces of 135 extracted human non-carious maxillary premolars were divided into three groups ($n = 45$). Groups I and II were conditioned with 1.3% NaOCl for 20 min, followed by a final rinse of 17% EDTA for 1 min and MTAD for 5 min respectively. Group III was treated with distilled water. Each group was further divided into three subgroups ($n = 15$). Subgroup 1: Kerr, Subgroup 2: Apexit and Subgroup 3: AH plus. Three-millimetre-long sections of polyethylene tubes were filled with freshly mixed sealer and placed on the dentine surfaces. The bonding between the sealer and dentine surface was evaluated using shear bond testing. The values were statistically evaluated using one way ANOVA followed by Tukey's test. Significant difference was found among the bond strength of the sealers and irrigating regimes. AH plus showed superior bond strength among the tested sealers, when EDTA was employed as the final rinse ($P < 0.05$). MTAD as a final rinse affected the bond strength of AH plus and Apexit, which were significantly lower than the control group.

Introduction

One of the major goals of root canal treatment is to seal the root canal system three dimensionally. A sealer along with gutta-percha is used to achieve fluid impervious apical seal. The sealer serves as a lubricant when inserting the gutta-percha point, as a filling material to fill the irregularities of preparation, and is necessary because gutta-percha does not bond spontaneously to the dentinal walls of the prepared canal (1). During root canal preparation, a smear layer is formed on the walls of the canal (2), which consists of dentine debris, including pulp remnants, bacteria and endotoxins (3). The smear layer prevents the penetration of the sealer into dentine tubules, which increases the potential for microleakage (4). Various methods to remove the smear layer has been extensively studied and one of the most effective methods is by employing a combination of NaOCl and EDTA (5).

A new endodontic irrigant containing a mixture 3% doxycycline, 4.25% citric acid and detergent (Tween 80) is commercially available as Bio pure MTAD (Dentsply Tulsa Dental, Tulsa, OK, USA) and is an effective irrigant for the removal of smear layer (6). The recently revised MTAD protocol for clinical use is an initial irrigation for 20 min with 1.3% NaOCl and followed by a 5 min final rinse with MTAD (7).

The property of smear layer removal using MTAD is attributed to the citric acid. After the final rinse, the manufacturer recommends that the MTAD should be left inside the canal where it is neither removed nor inactivated. This is done for sustained antibacterial activity. As MTAD is neither inactivated nor removed prior to the obturation, there is a possibility of MTAD interacting with dentine, which could have a bearing on the bonding ability of certain resin-based sealers (8). In addition, there have been no reports comparing the effect of MTAD with

Table 1 Root canal sealer tested

Sealers	Code	Composition	Manufacturer
Kerr	KE	Powder Zinc oxide – 42% Stabyelite resin – 27% Bismuth subcarbonate – 15% Bismuth sulphate – 15% Liquid Eugenol – 100%	Sybron Co, Ltd, Romulus, MI, USA
Apexit	AP	Calcium salts (hydroxide, oxide, phosphate), hydrogenised colophony, disalicylate, bismuth salts (oxide, carbonate), silicon dioxide and alkyl ester of phosphoric acid	Ivoclar Vivadent, Schaan, Liechtenstein
AH Plus	AH	Paste A Bisphenol-A epoxy resin Bisphenol-F epoxy resin Calcium tungstate Zirconium oxide Silica Iron oxide pigments Paste B Dibenzyl diamine Aminoadamantane Tricyclodecane-diamine Calcium tungstate Silica Silicone oil	Dentsply De Trey GmbH, Konstanz, Germany

EDTA when employed as final rinse on the sealer – dentine shear bond strength, of commonly employed endodontic sealers. Hence this study was conducted to test the null hypothesis that there is no difference in the shear bond strength of three commonly used endodontic sealers to dentine, when either EDTA or MTAD was used as the final rinse.

Materials and methods

One hundred and thirty-five freshly extracted human maxillary first premolars were scaled to remove all adhering soft tissue and debris, washed under running tap water, placed in distilled water and refrigerated at 4°C. The coronal two-thirds were removed with a low speed diamond saw and the exposed dentine surfaces were employed. The teeth were fixed with cold cure acrylic resin to a plastic cylindrical ring (2 cm in diameter and 2.5 cm deep). The rings were filled with auto polymerising polymethyl methacrylate resin (PMMA) mixed in accordance with the manufacturer's instructions to embed the tooth with its coronal surface exposed. After the PMMA had set, the coronal surface of tooth was ground with wet waterproof polishing paper through grades 240, 320, 400 and 600 on a Handimet grinder (Buehler, Lake Bluff, IL, USA) to get a flat, superficial layer of dentine. Three strokes in two directions perpen-

dicular to each other for every grade served to standardise the surface preparation of the dentine. The smear layer was not disturbed.

The prepared teeth were divided into three groups of 45 teeth each: Group I was comprised of teeth that were irrigated with 1.3% NaOCl for 20 min followed by 17% EDTA as a final rinse for 1 min. Group II was comprised of teeth that were irrigated with 1.3% NaOCl for 20 min followed by MTAD as a final rinse for 5 min while Group III was irrigated with distilled water (control). Each group was then divided into three subgroups ($n = 15$) according to the sealer that was used: Subgroup I: Kerr, Subgroup II: Apexit and Subgroup III: AH plus (Table 1).

Polyethylene tubes were cut to form 3-mm-high cylinders. These cylinders were used to apply the sealers on to the dentine with a constant surface area of 3.45 cm². To restrict the sealer to a particular area on dentine, adhesive Teflon tape with a hole corresponding to the size of the cylinder's contact area was affixed to the dentine (9). This hole provided a window to the predetermined area. Because of the difference in the composition of sealers, different methods and times had to be used to ensure complete setting (10). The specimens with zinc oxide eugenol-based sealers (e.g. Kerr Sybron Co, Ltd, Romulus, MI, USA) were first placed in a humidior that was kept for 3 h at room temperature and transferred to an incubator at 37°C. The epoxy-type sealer (AH plus,

Table 2 Mean shear bond strength of three endodontic sealers comparing 17% EDTA and MTAD as final rinse

Groups	Subgroups	Mean shear bond strength (MPa \pm SD)
(I) NaOCl/EDTA	Kerr	0.131 \pm 0.02
	Apexit	1.121 \pm 0.41
	AH plus	6.472 \pm 1.02
(II) NaOCl/MTAD	Kerr	0.128 \pm 0.01
	Apexit	0.32 \pm 0.08
	AH plus	2.01 \pm 0.47
(III) Distilled water	Kerr	0.129 \pm 0.02
	Apexit	0.81 \pm 0.21
	AH plus	3.70 \pm 1.08

Dentsply De Trey, GmbH, Konstanz, Germany) was left overnight in a conditioned temperature and humidity room (22°C and 25%). After their initial set, they were transferred to an incubator at 37°C. Calcium hydroxide-based sealer (Apexit, Ivoclar Vivadent, Schaan, Liechtenstein) was mixed with a slightly moistened spatula and set at room temperature before being transferred to a humidifier at 37°C (11). All the specimens were then stored for a period of 1 week. The specimens were removed from the incubator; air dried, and then engaged perpendicularly at their bases on a universal testing machine (Model 4411, Instron, Warren, MI, USA) at a crosshead speed of 0.5 mm min⁻¹. The probe was positioned so that the chisel would travel parallel to the dentinal surface and contact the sealer cylinder at its interface with this surface. The shear force required to separate the cylinder from the dentine was recorded in Newtons (N) for each specimen, then divided by the contact surface area to determine the shear bond strength in Mega Pascals (MPa). After testing, the fracture modes were examined in a dissecting microscope with 20 \times magnification.

Statistical analysis

One-way ANOVA followed by Tukey's test was used to analyse the data. Significance was established at $P < 0.05$ level.

Results

The mean shear bond strength for the various groups and subgroups are illustrated in Table 2.

1. Irrespective of the final rinse employed, AH plus showed the highest bond strength (6.476 \pm 1.02 MPa), which was statistically superior to both Apexit and Kerr.

2. Based on statistical significance (Tukey's test), for the AH plus subgroup, the groups can be ranked as Group I (6.476 \pm 1.02), Group III (3.74 \pm 1.08) followed by Group II (2.05 \pm 0.47).

3. Based on statistical significance (Tukey's test), for the Apexit subgroup, the groups can be ranked as Group I (1.124 \pm 0.41), Group III (0.84 \pm 0.21) followed by Group II (0.35 \pm 0.08).

4. Kerr sealer statistically showed no difference between the three groups.

Discussion

The endodontic smear layer forms over the surface of dentinal walls when the root canals are instrumented (3). The significance of the smear layer in endodontics has been the subject of extensive debate. Certain factors would indicate removal of the smear layer prior to root canal filling. The smear layer can not only act as a reservoir or substrate for microorganisms, but it will also obstruct the extension of sealer tags into the dentinal tubules and thereby decreases adhesion by micro-mechanical forces (12).

MTAD, a recently introduced irrigant has also been shown to be effective in the removal of smear layer. Torabinejad *et al.* proved that MTAD has superior antibacterial effect compared with NaOCl or EDTA (13). Doxycycline present in MTAD has high binding affinity for the dentine, allowing for prolonged antibacterial effect (14). The recently revised protocol for clinical use is 20 min of 1.3% NaOCl followed by a 5 min final rinse using MTAD and has been proved to remove smear layer effectively (7).

The objective of our investigation was to assess the role of the final rinse on the shear bond strength of the sealers. Hence the irrigating regimes in both the test groups were standardised to 20 min of 1.3% NaOCl, which represents the working solution, followed by a final rinse 17% EDTA for 1 min in Group I and MTAD for 5 min in Group II. Various studies have employed 17% EDTA in different working periods from 1 to 5 min (15,16); however, Calt and Serper showed that EDTA tends to have a corrosive effect on the dentine when employed for more than 1 min (17). Hence, we employed 17% EDTA as a final rinse for 1 min in Group I.

Leakage studies are much more common than adhesion studies in evaluating the fluid impervious apical seal of various endodontic sealers. However, it has been shown, on the teeth, that leakage study methods may provide varying results (18). In addition, leakage studies do not reveal which of the two interfaces, dentine–sealer or gutta-percha–sealer, is leaking. They also do not furnish any insight on the mechanism of how the combination of two different materials can contribute to an apical seal (19).

Adhesion of the root canal filling on the dentinal walls is advantageous for two main reasons. In a static

situation, it should eliminate any space that allows the percolation of fluids between the obturating material and the dentine wall (20). In a dynamic situation, it is needed to resist dislodgement of the filling during subsequent manipulation (21).

Adhesion tests measure either tensile bond strength, where the bond is broken by a force perpendicular to the interface between material and surface, or shear strength where the force is parallel to the interface between the material and surface. The shear test was developed for measuring the bond of endodontic sealers to dentine and gutta-percha, and has been proven to be effective and reproducible (10,22).

This test model does not replicate clinical conditions. Attempts to closely duplicate these have resulted in complicated models that are difficult to reproduce and sometimes even to interpret (23). Root dentine is not uniform and the surface of the canal walls that has been prepared during the endodontic treatment may differ widely. This is true not only between specimens, but also between sites in the same root, according to the level or even the direction of the wall proximal or faciolingual. Therefore, coronal, rather than root dentine, was used for better reproducibility (24).

The results of this study showed a statistically significant difference between groups when different irrigants were used as the final rinse, thus rejecting the null hypothesis. Group II wherein MTAD was employed surprisingly showed lowest bond strength (2.05 ± 0.47 MPa) values with AH plus despite the ability of this protocol to effectively remove endodontic smear layer. The bond strength achieved in this group was statistically inferior to the distilled water group. The results of the present study are also in disagreement to that observed by Wachlarowicz *et al.* who evaluated the effect of MTAD and EDTA final rinses on the shear bond strength of epiphany sealer to dentine (25). They found similar bond strength values with both these groups. This could be attributed to the bonding procedure involved while using Epiphany sealer, wherein a sulphonic acid contained primer was first coated onto the dentinal surface prior to the application of the sealer (26).

The difference in bond strength values between the groups cannot be attributed to the presence or absence of smear layer alone, as Group III (control) wherein distilled water was employed showed superior shear bond strength values in comparison with the MTAD group. The plausibility of a dentine-bound precipitate reported by Tay *et al.* may have a bearing on the shear bond strength in group II (8). The precipitate was reported to be formed because of the oxidation of doxycycline by NaOCl. This degradation product was found to have a high affinity for hydroxyl apatite. The potential deleterious

effect of this dentine-bound yellow precipitate on the sealing ability of endodontic sealers has not been evaluated.

Adding credence to this hypothesis is the stereomicroscopic observation in this study. The failure of the shear-tested specimens was primarily along the dentine–sealer interface (adhesive in nature) in Group II. This in contrast to Group I in which the failure of the shear-tested specimens was seen mainly through the sealer (cohesive in nature) indicating that adhesion of the AH plus sealer to dentine is stronger than the sealer's cohesive strength.

Group I showed superior results, which is in concurrence with previous studies that have employed a combination of NaOCl as a working solution followed by a final rinse of 17% EDTA. This combination is effective in the removal of smear layer thus rendering the dentine conducive for bonding (9). The superior shear bond values, which are a reflection of the sealing ability, were observed with all the three sealers tested.

Among the tested sealers Kerr sealer gave the least bond strength with statistically insignificant difference between the three irrigating groups. This was in concurrence with the study reported by Economides *et al.* that presence or absence of smear layer had no significant effect on the sealing ability of zinc oxide eugenol-based sealer (27). Irrespective of the irrigant being employed to condition dentine, Kerr sealer seems to have negligible adhesive property with dentine. On the assessment of failure pattern under stereomicroscope, it was found that all specimens exhibited mixed failure (adhesive and cohesive failure) because of the negligible adhesive and cohesive strength of the set Kerr sealer.

Eldeniz *et al.* tested three resin-based sealers and found that AH plus exhibited highest shear bond strength values to dentine with and without smear layer (9). In our study, among the various tested sealers AH plus showed highest bond strength (6.476 ± 1.02 MPa) in Group I (EDTA as a final rinse), which was significantly higher than the distilled water group wherein the smear layer was left intact. This was in concurrence to the previous study performed by Eldeniz *et al.* wherein smear layer removal enhanced the bonding ability of AH plus sealer (9).

Similar to AH plus adhesion of Apexit was significantly increased with the EDTA group, wherein statistically superior bond strength (1.124 ± 0.41 MPa) was achieved after smear layer removal in comparison with group wherein smear layer was left intact after irrigating with distilled water. Least bond strength values were obtained when MTAD was employed as the final rinse. This result suggests a negative effect of MTAD on the bonding ability of both resin-based and calcium hydroxide-based sealer.

Recently Tay *et al.* reported, red-purple staining of light-exposed, root-treated dentine when root canals were rinsed with 1.3% NaOCl as initial rinse followed by use of MTAD as final rinse (8). This reaction is of a redox nature that highly resembled the previous mechanism of tetracycline staining. This process involves the oxidation of doxycycline by NaOCl wherein 1 mol of oxygen is absorbed per mole of adsorbed tetracycline and converted to a red-purple product. This red-purple degradation product that resulted from photo-oxidation of doxycycline was found to be 4- α , 12- α -anhydro-4-oxo-4-dehydro-methylaminotetracycline (AODTC) with a high affinity for hydroxyl apatite. The conversion of dentine-bound yellow precipitate to red-purple stained dentine probably requires light exposure, as stained dentine was absent when the specimens were stored in the dark but appeared when the light-protected specimens were subsequently exposed to light (8). The test samples of Group II in our study also exhibited a similar dentine-bound yellow precipitate. The reduction in shear bond strength of both AH plus and Apexit in Group II wherein NaOCl and MTAD were employed could be attributed to this degradation product, which might interfere in the sealing ability of the sealers.

However, further studies must establish the correlation between this precipitate and its role in the setting reaction and sealing ability of endodontic sealers. Moreover the long-term effect of tetracycline degradation product at the sealer-dentine interface and its role in marginal seal and resistance to bacterial ingress has to be further evaluated.

Conclusion

1. The adhesive ability of sealers tested varied markedly according to the final rinse employed.
2. Removal of smear layer with EDTA as a final rinse enhanced the adhesive ability of both Apexit and AH plus in comparison with smear layer retained group.
3. In spite of proven smear layer removing ability of MTAD as final rinse, this group showed least bond strength values, which were statistically lower than the control group.
4. Within the limitations of this study the authors conclude that a final rinse with MTAD might have a negative effect on the bonding ability of both resin-based and calcium hydroxide-based sealers.

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