Effect of Irrigation with Different Solutions on Push out Bond Strength of Root Canal Obturation Materials

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Abstract
Evaluate push out bond strength (PBS) of endodontic obturation materials after washing by NaOCl, EDTA, and MTAD. Materials and methods: 30 mandibular premolars were utilized. Root canal instrumentation of all samples was achieved by ProTaper universal files to size F4. Samples were distributed to 3 groups; each group involved 10 samples as the following: Group1- samples were lastly washed by 5 ml of 5.25% NaOCl for 5 minutes. Group2- samples were lastly washed by 5 ml of 17% EDTA for 5 minutes. Group3- samples were lastly washed by 5 ml of MTAD for 5 minutes. All samples were filled by single cone technique with AH Plus sealer, and F4 ProTaper gutta-percha cone. 3 sections of 2 mm thickness were transected from every sample at 3, 7, and 11 mm from root apex, which represented apical, middle, and coronal sections consecutively. PBS was tested by universal digital testing device. Failure modes were assessed by stereomicroscope at 40 X magnification. Lastly, collected data was analyzed statistically. Results: MTAD group revealed significant greatest mean of PBS followed by EDTA group, and NaOCl group revealed significant least mean of PBS. In all groups, greater mean of PBS was in coronal section followed by middle section while, lesser mean of PBS was in apical section though, the differences among them were not significant within the same group. Mixed and cohesive failures were most dominant modes of failure in MTAD, and EDTA groups. While, in NaOCl group adhesive, and mixed failures were most dominant. Conclusions: last washing with MTAD is better than that with EDTA, and NaOCl in terms of greatest PBS.

Introduction:
Shaping, and cleaning of root canal systems are considered to be the most important phase of endodontic treatment (1). Mechanical preparation of roots canals causes disruption of dentin matrix. This leads to formation of layer from inorganic, and organic materials that is called smear layer, this layer may also involves microorganisms, and their by-products (2). The thickness of the smear layer is ranged
from 0.5 to 2 microns (3). Adhesion of endodontic obturation materials to dentin is basic to success the root canal treatments (4). Despite the thickness of smear layers is very tiny; the presence of smear layers may prevent sealer diffusion to dentin tubules and decrease sealer adhesion to the dentin of the root canal walls (5). Thus, elimination of smear layers is promoted to increase adhesion of sealer to the dentin of the root canal system. Consequently, both apical sealing ability, and bond strength to the radicular dentin of endodontic filling materials are improved. Lastly, successful root canal treatment is achieved (4). There are many endodontic irrigation solutions that are utilized to eliminate smear layer (6). Irrigation of roots canals with different solutions leads to alterations in the structural and chemical characteristics of dentin, which can modify its solubility, and permeability properties (7). Accordingly, adhesion of endodontic filling materials to radicular dentin is affected (8). Sodium hypochlorite (NaOCl) is the main irrigation solution that is used in root canal treatment (3). It has clear antimicrobial activity, and solvent ability of the organic material of the smear layer (9). Ethylene diamine tetra acetic acid (EDTA) is a chelating agent that is commonly used as irrigation solution in endodontic therapy (10). It is usually utilized in concentration of 17% (11) and has the capacity to dissolve the inorganic part of the smear layers (12). MTAD is a biocompatible and active root canal irrigation solution. It is mix of tetracycline isomer, citric acid, and detergent, which is Tween 80 (2). This irrigation solution has an efficient antimicrobial property, and high capacity for smear layer elimination without occurrence damage to the radicular dentin (13, 14). The aim of the existing research is to estimate PBS of endodontic obturation materials after the last washing by NaOCl, EDTA and MTAD. The study hypothesis was that there would be no differences in PBS of endodontic obturation materials after the last washing by various solutions, which involved NaOCl, EDTA, and MTAD.

Materials and Methods:
The present research was agreed by university committee of researches ethics with number (UoM.Dent/ H.L.7/ 20). 30 lower premolars were gathered and saved in distiller water. Teeth were radiographed to disregard those which had more than one canal, obstructed canal, tortuous canal, resorption defects, and open apex. Traditional occlusal access opening was achieved for each tooth. Barbed broach (Dentsply Maillefer, Switzerland) was utilized for extirpation of pulp. Size 10 K-file (Dentsply Maillefer, Switzerland) was inducted into canal of root, till file tip could be visualized at apical foramen. 1 mm was detracted from prior length to determine the working length. Crowns of gathered premolars were crosscut by sectioning diamond disk (GEBR.BRASSELER GmbH & Co. KG, Germany) for working length adjusting to 15 mm. Root canals of all samples were instrumented by rotary ProTaper universal files (Dentsply Maillefer, Switzerland) to F4 finishing file according to manufacturer recommendations. Each ProTaper file was banished after using for instrumentation of 4 samples. Through root canal preparation, every sample was rinsed by 2 ml of 2.5% NaOCl before beginning of preparation and after each file use. Samples were distributed haphazardly to 3 groups; each group contained 10 samples as the following:

Group 1– In this group root canals of the samples were lastly washed by 5 ml of 5.25% NaOCl (Chloraxid, CERKAMED Medical Company, Poland) for 5 minutes.

Group 2– In this group root canals of the samples were lastly washed by 5 ml of 17% EDTA (PD, Switzerland) for 5 minutes.

Group 3– In this group root canals of the samples were lastly washed by 5 ml of MTAD (Dentsply Tulsa Dental Specialties, Tulsa, OK) for 5 minutes. Dryness of canals was done by F4 proTaper paper point (Dentsply Maillefer, Switzerland). F4 ProTaper gutta-percha cone (Dentsply Maillefer, Switzerland) was inducted into canal of root to whole working length then, tug-back was
checked. Single cone obturation technique was utilized in the current study with AH Plus sealer (Dentsply Detrey GmbH, Germany), and F4 ProTaper gutta-percha cone. Later, glass ionomer cement (Cavex, Germany) was used to seal all the samples coronally. Then all samples were kept for 7 days in 100% humidity, and temperature of 37 centigrade for permitting the whole setting of sealer (3-4). After that, three horizontal sections of 2 mm thickness were transected from every sample at 3, 7, and 11 mm from root apex, which represented apical, middle, and coronal sections consecutively. Transecting process was done by sectioning diamond disk, which was applied vertically to long axis of root with abundant water irrigation for cooling, and thickness of sections was adjusted by using of digital caliper (Mitutoyo, Japan). Both coronal, and apical surfaces of each section were observed by stereomicroscope (Motic, China) at 20 X magnification to check that there were no voids in the sealer, and the root canal had round shape see Fig. (1). If there was any void in the sealer, and\ or the canal was not round in shape, the sample of this section was omitted from the study, and replaced by another sample, which would be prepared in the same manner as mentioned above. After that, each section was fixed over the center of acrylic cylindrical block that had central circular hole of 2 mm in diameter along its height. The dimensions of cylindrical block were 16 mm diameter, and 10 mm height. The sections were fixed in apical to coronal direction for obviating any objection that was related to the roots canals tapering through the achievement of push out test. The endodontic obturation material was loaded by cylindrical plunger of stainless steel that was attached to upper portion of universal digital testing device (TERCO, MT, 3037, Sweden). Plungers with different diameters were used to place plunger tip nearly covering for the whole endodontic obturation material without contacting the walls of root canal. The test was achieved with cross head speed 0.5 mm/minute. Greatest load in which failure occurred was recorded, and PBS was computed by dividing highest failure load on surface area of adhesion for root canal obturation material according to following formula (15-16):

\[
PBS = \frac{F}{A}
\]

Where, PBS= push out bond strength in Mpa, F= maximum failure load in Newton, A= surface area of adhesion in mm². The surface area of adhesion was calculated as the following:

\[
A = \frac{1}{2} (C_1 + C_2) H
\]

\[
A = \frac{1}{2} (\pi D_1 + \pi D_2) H
\]

\[
A = \frac{1}{2} \pi H (D_1 + D_2)
\]

\[
as \ (H = 2 \ mm) \ therefore \ A = \pi (D_1 + D_2)
\]

Where, C1= Circumference of the canal from the apical aspect, C2= Circumference of the canal from the coronal aspect, H= Thickness of section (2 mm), \( \pi = 3.14 \), D1= Diameter of the canal from the apical aspect in mm, and D2= Diameter of the canal from the coronal aspect in mm. Diameter of the canal was measured by Motic Image software (Motic, China) that was connected to the digital stereomicroscope.

After the data gathering, statistical analysis of data was accomplished with descriptive statistics, one way analysis of variance, and Tukey test at \( P \leq 0.05 \), by using of SPSS Program version 19.0 for window software (IBM, Armonk, New York).

Also, all the sections were interspected by stereomicroscope at 40 X magnifying to identify failure mode, which would be under one of the following classifications:

1. Adhesive failure (occurred at the interface between endodontic filling material and dentin wall of the root canal).
2. Cohesive failure (occurred within the endodontic filling material). (3) Mixed (included both adhesive and cohesive failure).

Results:
The results of the current study demonstrated that NaOCl group revealed the least mean of PBS with different root sections (Coronal, Middle, and apical) followed by EDTA group whereas, MTAD group revealed the greatest mean of PBS with different root sections. One way analysis of variance showed
significant differences among the experimental groups at different root sections as p value < 0.05. Tukey test showed that each experimental group in this study differed significantly with the other groups at different root sections see Table (1). Also, means of PBS for different sections (Coronal, Middle, and apical) with in the same group were compared, and similar results were noted in all experimental groups where, the greater mean of PBS was in the coronal section followed by middle section while, the lesser mean of PBS was in the apical section. Although, one way analysis of variance showed non-significant differences among different sections within the same group in all experimental groups as p value > 0.05 see Table (2). The three modes of failure are illustrated in Fig. (2). The percentages of failure modes for all experimental groups at the different sections are listed in Table (3). These results displayed that mixed, and cohesive failures were the most dominant modes of failures in the MTAD, and EDTA groups whereas, adhesive, and mixed failures were the most dominant modes of failure in the NaOCl group.

Discussion:

Adhesion of obturation materials to radicular dentin is fundamental for succeeding endodontic therapy (4) as; this adhesion inhibits fluid leakage and decreases the possibility of filling detachment from the root canal walls during restorative treatment or during masticatory functions (17-18). Numerous factors affect the adhesion of endodontic obturation materials to dentin, these factors involve natures of radicular dentin in particular tooth or even in different sites of the same root (19-20), the absence or presence of smear layer, type of irrigation solutions, type of sealers, and their reaction with dentin (21). The existence of smear layer prevents the diffusion of endodontic obturation materials to dentin tubules, and irregularities of the canal system also, it prevents whole adaptations of endodontic filling materials to the walls of dentin (22-23). Thus, elimination of the smear layer by irrigating materials increases the bonding of endodontic filling materials to the walls of dentin (4, 10). Many irrigating solutions have been newly utilized to improve bonding between the obturation materials, and the root canal walls (24). Therefore, in the existing study PBS of endodontic obturation materials after washing with various solutions (NaOCl, EDTA, and MTAD) was assessed. Numerous ways have been exploited for checking bond strength involving shear, micro-tensile, and push out bond strength tests (4, 25). Shear, and micro-tensile bond strength tests do not replicate clinical situations and efforts to thoroughly duplicate them have produced complex models, which are difficult to replicate and explain (19). From the other hand, PBS test can be easily reproduced and interpreted (26). Furthermore, it is more effectual, and permits endodontic filling material to be estimated even in low bond strength (27). Also, it creates fracture parallel to dentin sealer interface (28), and produces more clinically dependable and effective results, which better instantiates bond strength of endodontic filling material (26). Consequently, PBS test was utilized in the current study. In existing research for purpose of standardizations, mandibular premolars with mature apices, and straight single canal were used only. Premolars crowns were crosscut for working length adjusting to 15 mm. Furthermore, the last used files in whole groups were F4 ProTaper finishing files, which have 6% apical tapering, and ISO 40 tip diameter. Moreover, the samples of all groups were obturated by single cone technique with AH Plus sealer, and F4 gutta-percha cone. Also, three horizontal sections of 2 mm thickness were crosscut from every sample at 3, 7, and 11 mm from root apex, which represented apical, middle, and coronal sections consecutively. The thickness of every section was adjusted by digital caliper. The thickness of 2 mm was used to avoid premature detachment of the endodontic obturation materials. The sections were inspected under stereomicroscope at 20 X magnification from apical and coronal aspects to investigate that there were no
voids in the sealer, and the root canal was round in shape. If there was any void in the sealer and or the canal was not round in shape, the sample of this section was excluded from the study. In the current study MTAD group showed the significant highest mean of PBS, and the most dominant modes of failures in this group were the mixed and cohesive failures. These results can be clarified by the high efficacy of MTAD solution to eliminate smear layer (24). MTAD efficacy for eliminating smear layer can be elucidated by collaborated action of doxycycline, citric acid, and detergent (Tween 80) that are existed in MTAD (9). The blend of the doxycycline (broad spectrum antibiotic), and citric acid, which acts as a chelation agent, secures elimination of the smear layer (29). The existence of detergent diminishes surface tension of MTAD (30). Accordingly, the penetration capacity of MTAD into dentinal tubules are improved (9, 24), and the effect of MTAD to eliminate smear layer is promoted even at the apical segment of root canal (31). Therefore, once MTAD is utilized as irrigating solution, elimination of smear layer is efficient, and diffusion of sealer into dentin tubules during the obturation is improved (9). Hence, mechanical locking of sealer with dentin tubules happens, and adhesion area is enlarged (32). Consequently, adhesion and bond strength are increased. On the other hand, the presence of Tween 80 (detergent) in MTAD increases dentin wettability and inter tubular permeability (33). AH Plus sealer displays the maximum push out bond strength in the slightly moist condition, where this condition gives ideal substrate to bond the sealer (34-35) owing to covalent bonds formation between open epoxide rings of sealer, and exposed amino groups of dentinal collagens (35). In this study canals were dried by paper point, and in MTAD group Tween 80 as the constituent of MTAD increases dentin wettability and inter tubular permeability. All that could supply slightly moist condition, which is ideal condition for bonding AH Plus sealer, and this also, can illustrate the greatest bond strength of MTAD group. In the existing study EDTA group showed high mean of PBS also, the most dominant modes of failures in this group were the mixed and cohesive failures. These results can be elucidated by EDTA efficiency to eliminate smear layer, which is linked with its chelating ability (9). Supremacy of MTAD on EDTA in the existing study can be explained by higher efficacy of MTAD for eliminating smear layer as a compare with EDTA (9, 36-38). NaOCl group showed the significant lowest mean of PBS in this study, and the most dominant modes of failures in this group were the adhesive and mixed failures. These results can be explained by that NaOCl as a single irrigating solution is not efficient to remove the smear layer (39), where its physicochemical properties work only on the organic part of the smear layer (40). On the other hand NaOCl can cause dissolution of the dentinal collagen (41). Accordingly, it decreases bonding strength between AH Plus sealer, and radicular dentin. Where the bonding of AH Plus sealer depends on covalent bonds formation between its open epoxide rings and amino groups of dentinal collagens (27, 35). Also, NaOCl liberates oxygen, which inhibits the polymerization progression consequently; adhesion is adversely affected, and bond strength is decreased (18, 42). Similar results to the results of the existing study were proved by Farag et al., (24) who revealed that samples which were finally rinsed with MTAD had greater PBS than samples which were finally rinsed with EDTA and NaOCl. Other comparable results were recorded by Mozyeni et al., (43) who concluded that final rinsing by MTAD significantly improved PBS as compare with final rinsing by combination of NaOCl, and EDTA.

Andраби, and his colleagues reported that NaOCl failed to eliminate the smear layer. MTAD is more effective than EDTA for removing smear layer although, the difference between them was not significant at coronal, and middle thirds, but at apical third MTAD was significantly better (44). Kour et al., (9) detected that MTAD is more efficient in elimination of smear layer than EDTA, and higher depth of sealer diffusion was observed in MTAD samples as compare with EDTA samples. These results can
support the results of this study. Jain et al., (45) revealed that samples of MTAD group exhibited lower PBS than the samples of EDTA group and these results controvert results of the current study. This can be ascribed to that Jain, and his colleagues prepared the root canals by different instrumentation system also, they used different protocol in irrigation, where they rinsed the canals with MTAD, and EDTA only for 30 seconds, and finally they rinsed the canals with normal saline. Furthermore, Jain, and his colleagues filled the canals by different obturation techniques, which was cold lateral compaction technique. In the existing study, results displayed that there was diminution in bond strength from coronal to apical direction, and these results were similar to the results of numerous studies (24, 46-48).

This can be clarified by the diminution of root canal diameter and anatomical variations in the apical third restrict flow of the irrigating solutions, thus the elimination of smear layer is affected (49). Incomplete elimination of smear layer diminishes the diffusion of sealer into tubules subsequently, bond strength is decreased (50). Also, the reduction of dentinal tubules density apically (42, 51, 52), and inhomogeneous hybridization of dentin in the apical part adversely affect the bond strength (51,52).

**Conclusions**

Under the conditions of existing research, it can be deduced that, bonding strengths of endodontic obturation materials can be affected by types of irrigating solutions, and the last washing with MTAD is better than the last washing with EDTA, and NaOCl in terms of greatest PBS.

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**Fig. (1):** Stereomicroscopic views of prepared section for PBS test. (A): coronal view. (B): apical view.

**Fig. (2):** Stereomicroscopic views illustrate modes of failure. (A) Adhesive failure. (B) Cohesive failure. (C) Mixed failure.
Table (1): Comparisons of PBS means for different experimental groups at each root section alone.

<table>
<thead>
<tr>
<th>Root sections and experimental groups</th>
<th>Mean (MPa)±SD</th>
<th>F-value</th>
<th>P-value*</th>
<th>Tukey test**</th>
</tr>
</thead>
<tbody>
<tr>
<td>MTAD Coronal</td>
<td>5.46±0.38</td>
<td>64.763</td>
<td>0.000</td>
<td>A</td>
</tr>
<tr>
<td>EDTA Coronal</td>
<td>4.32±0.36</td>
<td></td>
<td></td>
<td>B</td>
</tr>
<tr>
<td>NaOCl Coronal</td>
<td>3.22±0.27</td>
<td></td>
<td></td>
<td>C</td>
</tr>
<tr>
<td>MTAD Middle</td>
<td>5.34±0.32</td>
<td>89.658</td>
<td>0.000</td>
<td>A</td>
</tr>
<tr>
<td>EDTA Middle</td>
<td>4.10±0.25</td>
<td></td>
<td></td>
<td>B</td>
</tr>
<tr>
<td>NaOCl Middle</td>
<td>3.17±0.41</td>
<td></td>
<td></td>
<td>C</td>
</tr>
<tr>
<td>MTAD Apical</td>
<td>5.22±0.36</td>
<td>97.673</td>
<td>0.000</td>
<td>A</td>
</tr>
<tr>
<td>EDTA Apical</td>
<td>3.94±0.45</td>
<td></td>
<td></td>
<td>B</td>
</tr>
<tr>
<td>NaOCl Apical</td>
<td>2.94±0.23</td>
<td></td>
<td></td>
<td>C</td>
</tr>
</tbody>
</table>

*P<0.05 means significant differences among the different experimental groups at each root section alone.

**Different letters vertically indicate significant difference. Comparisons were done for the different experimental groups at each root section alone. SD= standard deviation.

Table (2): Comparisons of PBS means for different root sections at each experimental group alone.

<table>
<thead>
<tr>
<th>Experimental groups and root sections</th>
<th>Mean (MPa)±SD</th>
<th>F-value</th>
<th>P-value*</th>
<th>Tukey test**</th>
</tr>
</thead>
<tbody>
<tr>
<td>MTAD Coronal</td>
<td>5.46±0.38</td>
<td>4.321</td>
<td>0.254</td>
<td>A</td>
</tr>
<tr>
<td>MTAD Middle</td>
<td>5.34±0.32</td>
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<tr>
<td>MTAD Apical</td>
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<td></td>
<td>A</td>
</tr>
<tr>
<td>EDTA Coronal</td>
<td>4.32±0.36</td>
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<tr>
<td>EDTA Middle</td>
<td>4.10±0.25</td>
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<td>B</td>
</tr>
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<td>EDTA Apical</td>
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<td></td>
<td></td>
<td>B</td>
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<tr>
<td>NaOCl Coronal</td>
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<tr>
<td>NaOCl Middle</td>
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<td>NaOCl Apical</td>
<td>2.94±0.23</td>
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<td></td>
<td>C</td>
</tr>
</tbody>
</table>

*P>0.05 means non-significant differences among the different root sections at each experimental group alone.

**Similar letters vertically indicate non-significant difference. Comparisons were done for the different root sections at each experimental group alone. SD= standard deviation.
Table (3): Percentage of Failure modes for the different experimental groups at different root sections

<table>
<thead>
<tr>
<th>Groups</th>
<th>Root Sections</th>
<th>Failure Modes %</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>Adhesive</td>
</tr>
<tr>
<td></td>
<td>Coronal</td>
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</tr>
<tr>
<td></td>
<td>Middle</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>Apical</td>
<td>0</td>
</tr>
<tr>
<td>MTAD</td>
<td>Coronal</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Middle</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>Apical</td>
<td>30</td>
</tr>
<tr>
<td>EDTA</td>
<td>Coronal</td>
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<tr>
<td></td>
<td>Middle</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>Apical</td>
<td>60</td>
</tr>
</tbody>
</table>

References

15. Lopes GC, Ballarin A, Baratieri LN. Bond strength and fracture analysis between
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