Evaluation of some intracanal irrigants on push-out bond strength and mode of failure of resin and non resin cements to root canal dentin (in vitro comparative study)

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Abstract
Objectives: The purpose of this study was to evaluate the effects of five intracanal irrigants [distilled water, NaOCl, NaOCl + EDTA, ozonated water and NaOCl + ozonated water] on push-out bond strength of (zinc phosphate cement and Calibra\textsuperscript{®} esthetic resin cement) and to determine the mode of failure.

Materials and methods: Seventy extracted single straight rooted teeth were selected. The crowns were sectioned at cemento-enamel junction. The sectioned roots were embedded vertically in clear acrylic resin. The root canals were prepared with K-files and Gates Glidden drills using step-back technique then the specimens were randomly assigned to five main groups according to the types of irrigant used and each main group were divided in to two subgroups and filled with zinc phosphate cement and Calibra\textsuperscript{®} esthetic resin cement. The samples were sectioned horizontally to provide 4 millimeter dentin disc from coronal third of the roots. Each sectioned specimen was tested by using computerized universal testing machine. Finally each de-bonded specimens was examined under stereomicroscope to determine the mode of failure.

Results: Two-way ANOVA tests showed that there was statistical significance difference between the two type of cement used in this study, also there was statistical significance difference between the types of irrigant; while the interaction between types of cement with types of irrigant was not statistically significant. Considering the dentine surface treatment, Paired T-test showed that irrigation with 5.25% NaOCl produce statistically significant reduction in bond strength to root canal dentin in comparison to control group, while irrigation with distilled water, 5.25 NaOCl plus 17% EDTA, 5.25% NaOCl plus 5gm/L ozonated water, 5gm/L ozonated water alone had no statistical significant affect on bond strength to root canal dentin. Comparing the cements, zinc Phosphate cement showed higher bond strength in comparison to Calibra\textsuperscript{®} esthetic resin cement.

Conclusions: NaOCl have negative effect on bond strength to root canal dentin, while distilled water, NaOCl plus EDTA, ozonated water alone or after NaOCl had no effect. Zinc phosphate cement had higher bond strength than Calibra\textsuperscript{®} esthetic resin cement. The adhesive and mixed failures were higher than the cohesive failure for all groups in general.

Keywords: Ozonated water, push-out bond strength, resin cement.

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Introduction:
Endodontic therapy comprises of two principal phases, the first phase include mechanical instrumentation to clean and shape root canals with irrigation and disinfection to prevent reinfection. The second phase is the filling phase which subsequently placed in the instrumented root canal\textsuperscript{(1)}.

Sodium hypochlorite (NaOCl) is the most common endodontic irrigant used with concentrations ranging from 0.5% to 5.25%. It presents strong antimicrobial activity and ability to dissolve necrotic pulp tissue, so it is usually chosen as suitable intra canal irrigant. However, it has been showed to be ineffective in removing the entire smear layer when used alone. Thus, the uses of chelating agents and acids have been suggested to remove the smear layer from the root canal\textsuperscript{(2)}.

Ozone is highly indicated in root canal therapy as an irrigant due to its strong disinfection property and absence of cytotoxicity. Other interesting biological characteristics include: bactericidal action, debriding effect, angiogenesis stimulation capacity and high oxidizing power\textsuperscript{(3)}.

The use of ozonated water had showed that following ozone therapy there was high metabolic activity of the associated fibroblasts indicating an increase in the healing process.

Another study assessed the effect of an ozone application on the micromechanical properties of dentin and they concluded that the application of ozone does not affect the modulus of elasticity and the Vickers’s hardness\textsuperscript{(4)}.

Ozonated water has some potential in removing the smear layer, and it has certain ability for the smear layer removal in combination with 1% NaClO, especially without erosion on the root canal walls\textsuperscript{(5)}.

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With the evolution of adhesive systems, resin cements, and restorative materials, endodontically treated teeth began to be reconstructed conservatively, and many intra radicular systems have become available for this purpose \(^6\).

Various types of cements are used for post retention. Zinc phosphate, zinc oxide eugenol and silicophosphate cements were used from the early twentieth century till 1970s when new cements were developed. At first polycarboxylate cement, next glass ionomer cements and within the last thirty years resin cements and resin modified glass ionomer cements were developed. Resin cements differ from restorative composites with lower filling structure and viscosity in their context \(^7\).

Resin type luting cements provide high bond strength to restorative materials and hard dental tissue. Moreover, their useful properties of minimal solubility, biocompatibility, and stability greatly contribute to the formation of strong tooth restoration interfaces, with minimal removal of adjacent tooth tissue \(^8\).

Materials and Methods:
Seventy extracted human permanent teeth were selected. The teeth were mechanically cleaned with ultrasonic scaler to remove hard deposit and soft tissues. Then they were stored in distilled water at room temperature after that the teeth sterilized by using 20 ml of 10% formalin (Merch, Germany) for 5 days at 37°C. The coronal portion of each tooth was sectioned at the lowest point of cemento-enamel junction using a diamond double-faced disc in a slow-speed handpiece, cooled with air/water spray. The working length was established by the direct method by subtracting 1 mm from the remaining root length determined by introducing a no. 10 K-file. Each specimen was vertically positioned in the center of cylindrical containers of 15 mm length and 20 mm diameter using dental surveyor (Dentaurm poraline, Beograd Sirbija) for equalization of the parallelism. The root canals was prepared with K-files, using the step-back technique. The apical portion was enlarged to a no. 45 master file. The coronal portion of each canal was shaped with sizes 2 to 4 Gates-Glidden drills. Then the canals were irrigated with different testing irrigants as shown in the figure 1.

Group 1(control group): The canals were irrigated by using 2 ml of distilled water (Al-Mansour Co., Iraq) for 60 seconds after each instrument. Group 2: The canals were irrigated with 2 ml of 5.25% NaOCl (Chlorox, commercial household, Iran) as in (Group1). Group 3: The canals were irrigated with 5.25% NaOCL with the same manner as in Group 1 then dried with paper point and rinsed with 1 ml of 17% EDTA (Dentaires SA Vevey, Switzerland) for 1 min and a final rinse with 3 ml of 5.25% NaOCl for 1 min. Group 4: The canals were irrigated with 2 ml of 5gm/L freshly prepared ozonated water with the same manner as (Group1). The ozonated water was prepared using ozone generator (A2z, USA) based on corona discharge which had the capacity to produce 600gm/hr of dissolved ozone according to the manufacturer instruction, and the bubbling stone of the device was immersed into 4 inch deep of 1L of distilled water for 30 seconds to produce 5gm/L of dissolved ozone according to manufacturing instruction as shown in figure 2. Chemical diagnostic test (Hanna ozone test kit, USA) was used to ensure the correct concentration of dissolved ozone in water. Group 5: The canals were irrigated with 2 ml of 5.25% NaOCL with the same manner as in Group 1, dried with paper point; then final rinse was done with 3 ml of 5gm/L freshly prepared ozonated water for 1min.

Each group (14) subdivided in to 2 subgroup and filled with zinc phosphate cement (Adhesor, Czech Republic) and Calibra\(^\text{®}\) esthetic resin (Dentsply, Switzerland) according to the manufacturer’s instructions. The quality of filling for each filled specimens was examined by periapical radiograph then the filled specimens will be stored wet environment inside incubator for 24 hr at 37°C. The block was sectioned horizontally by linear precision saw (Isomat 5000, Germany) to provide 4 mm thick dentine discs from the coronal third of the roots. The sectioned specimen was tested by using universal testing machine (Wp310, Germany) for push-out test.

![Figure 1: Sample grouping](image-url)
which calibrated at a constant speed of (1 mm/min) with a 1mm-diameter stainless steel cylindrical tip. The force needed to dislodge the filling material was transformed into bond strength, and then the failure mode of each debonded specimen was assessed under stereomicroscope (NTB-3A, Philippines) with optical magnification (4X) using digital camera connected to the computer. The data was statistically analyzed by paired T-test and two-way ANOVA.

Results:
The means and standard deviations of push-out bond strength (in MPa) for displacement of the cement from the specimens after treatment of root canal dentine with different irrigants are given in Table 1.

Two-way ANOVA tests showed that there is statistical significance difference between the two type of cement used in this study, also there is statistical significance difference between the types of irrigant; while the interaction between types of cement with types of irrigant is not statistically significant as shown in Table 2.

Comparing the cements, Zinc phosphate cement showed higher bond strength in comparison to Calibra® Esthetic resin cement. Considering the dentine surface treatment, showed that irrigation with 5.25% NaOCl produce statistically significant reduce in bond strength to root canal dentin in comparison to control group, while irrigation with distilled water, 5.25 NaOCl plus 17% EDTA, 5.25% NaOCl plus 5gm/L ozonated water, 5gm/L ozonated water alone had no statistically significant affect on bond strength to root canal dentin.

In the present study the adhesive and mixed failure were higher than the cohesive failure for all groups in general. For the control group the adhesive failure had the higher rate with no cohesive failure, also the adhesive failure was predominant for the group that irrigated with NaOCl; while mixed failure had the highest rate in both groups that irrigated with NaOCl+ ozonated water and ozonated water alone and cohesive failure was higher in group that irrigated with NaOCl+ EDTA in comparison to other groups as shown in Figure 3.

Discussion:
During irrigation, radicular and coronal dentin is exposed to the various solutions used to disinfect the endodontic space. This may cause alterations on the dentin surface and affect their interactions with materials used either for root canal obturation or for coronal restoration (8).

In this study irrigation with NaOCl showed the lowest bond strength, this result was supported by Morris et al.(9), Goldman et al.(10), Ari et al.(11), Santos et al.(12). The reasons could be its strong oxidizing agent caused degradation of organic dentin components mainly collagen, also reduces calcium and phosphorus levels and mechanical properties of dentin, such as elastic modulus, flexural strength, and

Table 1: The means and standard deviations bond strength in all studied groups

<table>
<thead>
<tr>
<th></th>
<th>Distilled water</th>
<th>5.25% NaOCl</th>
<th>5.25%NaOCl+17% EDTA</th>
<th>5gm/l ozonated water</th>
<th>5.25%NaOCl+5gm/l ozonated water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calibra®ERC</td>
<td>9.0616±0.8423</td>
<td>4.8034±0.7512</td>
<td>9.0961±0.8698</td>
<td>9.1297±1.734</td>
<td>9.0294±1.5334</td>
</tr>
<tr>
<td>ZPhC</td>
<td>7.7326±0.6797</td>
<td>4.8714±0.5696</td>
<td>7.7009±1.3542</td>
<td>7.8304±1.1643</td>
<td>7.7663±1.0786</td>
</tr>
</tbody>
</table>
Table 2: Two-way ANOVA represents effect of irrigants and type of cement on bond strength

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>SS</th>
<th>df</th>
<th>MS</th>
<th>F</th>
<th>P-value</th>
<th>F crit</th>
<th>Degree of significances</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type of cements</td>
<td>18.82933</td>
<td>1</td>
<td>18.82933</td>
<td>14.7498</td>
<td>0.000298</td>
<td>4.001191</td>
<td>Significant</td>
</tr>
<tr>
<td>Type of irrigants</td>
<td>143.3191</td>
<td>4</td>
<td>35.82977</td>
<td>28.06695</td>
<td>0.0000</td>
<td>2.525215</td>
<td>Significant</td>
</tr>
<tr>
<td>Interaction between cement and irrigant</td>
<td>5.390785</td>
<td>4</td>
<td>1.347696</td>
<td>1.055707</td>
<td>0.386357</td>
<td>2.525215</td>
<td>Not significant</td>
</tr>
</tbody>
</table>

Figure 3: The failure modes at different interfaces for the studied groups

Figure 4: Example of adhesive, cohesive, and mixed failure respectively (from left to right) of the sectioned samples under stereomicroscope
microhardness were reported after irrigation of root canals with sodium hypochlorite(15,12). Another reason may be the presence of a smear layer after treatment (14). Whereas Pelegrine et al(15) concluded that the different irrigant solutions did not affect the tensile bond strength of the fixation system used to cement the intraradicular glass fiber posts to dentin.

This study came in agreement with Cecchin et al (7), Nikaido et al(10); Barutcigil et al(17) who concluded that the use of EDTA as irrigating solution during endodontic treatment after NaOCl counteracts the deleterious effect of NaOCl. The use of EDTA counteracts the bad effect of NaOCl by its anti-oxidant power via redox reaction, allowing for free radicals polymerization without premature chains or failures. Moreover, it has the ability to remove the smear layer and the residual chlorine ions, what could benefit phosphoric acid etching and enhance resinous monomer penetration through dentinal tubules(18).

The ozonated water showed an increase in bond strength to root canal dentin which was statistically non-significant and the group that was irrigated with NaOCl and final irrigation with ozonated water as recommended by Lynch and Swift(19) doesn’t affect the bond strength to dentin by counteracting the bad effect of NaOCl. These results came in agreement with Garcia et al(20), Pithon and Santos(21). Whereas partially came in agreement with Ibraheem and Habeeb(22) who concluded that there was an increase in bond strength for ozonated water treated sample. The explanation of these results may be because Ozone is a powerful oxidizing agent but ozonated water becomes highly unstable and rapidly decomposes through a complex series of chain reactions eliminating this oxidizing effect(23). Other mechanisms of ozone are organic biodegradation and some potential in smear layer removal (22-24).

Zinc Phosphate cement showed higher bond strength in comparison to Calibra® Esthetic resin cement, the results were support the results of Sadek et al(25), Habib et al(26), Yahya et al(27); Oliveira et al(28) this is may be due to stress generated during polymerization shrinkage of resin cement, which causes its displacement from the dentinal surface as well as incomplete bonding(29). Resin cements are more “technique sensitive” than most of the other luting cements. Other factor is insufficient adhesive resin infiltration/penetration into the demineralized dentine(30). While Chan et al(31) concluded in their study greater tensile strength of the dowels secured with resin cement to root canal dentin than those secured with zinc-phosphate cement. Possible reasons for this disagreement are the lack of standard methodology, using dowel inside the root canals and the use of extracted teeth without standardization in the investigations.

The results of the failure modes came in agreement with Barutcigil et al(17), Ari et al(11), Chaharom et al(32), Morris et al(9); Sasafuchi et al(33) who showed that the failure mode of the samples treated with NaOCl mostly adhesive. For the group irrigated with NaOCl+EDTA, Barutcigil et al(17) showed that failure mode consists of cohesive, adhesive and mixed which was in agreement with the present study, while Vilanova et al(34) showed that failure mode consists of cohesive and mixed failure with no adhesive failure. The results of present study agree with the results of Ibraheem and Habeeb(22) since they concluded that ozonated water treated samples show the highest combination failures which represent the dominant type of failure.

Conclusions:
NaOCl have negative effect on bond strength to root canal dentin, while Distilled water, NaOCl plus EDTA, Ozonated water alone or after NaOCl had no effect. For the cements used in this study Zinc Phosphate cement had higher bond strength than Calibra® Esthetic resin cement. Regarding the failure modes the adhesive and mixed failures were higher than the cohesive failure for all groups in general.

References: