Original Article

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Dentistry Section

The Effect of Increased Temperatures of QMix and EDTA on the Push-out Bond Strength of an Epoxy-resin Based Sealer

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ABSTRACT

Background: Bond strength of root canal sealers is mainly influenced by the surface tension of the root canal wall. Heat, or the use of a surfactant might reduce surface tension and it is known that surface tension of an EDTA solution significantly affected from temperature rise. QMix is a novel endodontic irrigant for smear layer removal containing EDTA, chlorhexidine and a detergent. There is no data regarding the influence of the temperature of the EDTA and QMix on the push-out bond strength of root canal sealers.

Aim: To evaluate effect of temperatures of QMix and EDTA on the bond-strength of AH Plus.

Materials and Methods: Sixty premolars were prepared with ProTaper F4. During preparation 3% NaOCl was performed. Specimens were divided into 4 groups as follows: 5-mL 17% EDTA at 22°C and 37°C; 5-mL QMix at 22°C and 37°C. Three specimens from each group were prepared for observation using SEM. Remaining roots were obturated and prepared for a push-out test. Data was analysed using Anova and Bonferroni tests ($p<0.05$).

Results: Samples irrigated with QMix had higher push-out bond strength values than those irrigated with EDTA ($p=0.01$), regardless of temperature. Samples irrigated with 37°C EDTA resulted in higher bond-strength values than those irrigated with 22°C EDTA ($p<0.001$).

Conclusion: Temperature of the final irrigant does affect the bond strength values of AH plus to root dentin irrigated with EDTA. Bond strength of AH Plus sealer to root canal dentin may improve with QMix.

Keywords: Adhesion, Root canal irrigants, Smear layer

INTRODUCTION

Successful endodontic therapy depends on thorough chemo-mechanical preparation and three-dimensional obturation of the root canal system, to provide a tight seal against the ingress of microorganisms and fluids into the disinfected root canal space [1]. Smear layer is an amorphous irregular layer that plugs the dentinal tubules, usually was observed after mechanical instrumentation [2,3]. This has an adverse effect on penetration of the irrigant and sealers into the dentinal tubules, thereby adversely affecting dentin bonding [4,5].

Ethylenediaminetetraacetic acid (EDTA), used usually in a concentration of 17%, dissolves the inorganic portion of dentin by chelation and is recommended for use after sodium hypochlorite (NaOCl) to complete smear layer removal [5,6]. In this way, it better prepares the dentinal wall for adhesion to the filling material [7,8]. Adhesion of root canal sealers is mainly influenced by the relative surface free energy of the intraradicular dentin surface [9,10]. It is believed that the effectiveness of irrigants is associated with their being in direct contact with the entire canal wall [5,11]. Heat, or the use of a surfactant might reduce surface tension [12-14]. It was reported that at a low or neutral pH, the surface tension of an EDTA solution significantly decreased at 37°C, compared to 22°C [14].

QMix (Dentsply Tulsa Dental, Tulsa, OK, USA) is a novel endodontic irrigant for smear layer removal containing added antimicrobial agents. It comprises EDTA, chlorhexidine (CHX) and a detergent. It has been designed as a final irrigant, and is used for 60–90 s in place of 17% EDTA. QMix is a ready-to-use clear solution, which requires no chair-side mixing. This one-step final rinse is supposed to combine the antimicrobial and substantivity properties of CHX with the smear layer removing properties of EDTA. Previous studies reported that QMix was as effective as 17% EDTA in smear layer removal [15-17], and has the push-out bond strength of epoxy-resin based sealers [17].

Push-out bond strength is the de-bonding energy between root canal wall/root canal sealers. A mechanical test performed to measure this energy, which is an effect of frictional sliding between root canal wall and root canal filling materials. In the past, the effects of different final irrigation regimens on the bond strength of canal filling materials have been investigated previously [11-13,17]. However, there is no data regarding the influence of the temperature of the final irrigant on the push-out bond strength of root canal sealers. The purpose of this study was to evaluate the effect of different temperatures of final irrigants on the bond strength of an epoxy resin–based sealer (AH Plus; Dentsply DeTrey, Konstanz, Germany). The null hypothesis was that different temperatures of irrigants might change the bond strength of AH Plus sealer.

MATERIALS AND METHODS

The present *in vitro* study was done between September and October 2014 in the Department of Endodontics, Faculty of Dentistry, University of Hacettepe, Ankara. Sixty single-rooted mandibular premolar teeth with single canals were used. The teeth were stored in distilled water until they were used. Teeth with a completely formed apex and without previous root fillings, resorptions, or calcifications were used during the study. Decoronization was done with a high-speed carbide bur under water spray to obtain approximately 16-mm specimens in length, before canal instrumentation. To check patency, #15 K File (Mani, Inc, Tochigi, Japan) was inserted until the tip of the file became visible at the major apical foramen under the microscope (Opmi-Pico; Karl Zeiss, Jena, Germany) at 8× and working lengths were determined by subtracting 1 mm from this measurement. The root canals were prepared by using ProTaper rotary instruments (Dentsply Maillefer, Ballaigues, Switzerland) to a size 40, 0.06 taper (F4) to the working length (WL). Between each instrument, the canals were irrigated with 2-mL 3% NaOCl solution. When irrigation was performed after the final file, 5-mL 3% NaOCl was used for one min in all the samples. The roots were then randomly divided into four groups (n=15) according to the final irrigation procedures [Table/Fig-1]. All the samples were irrigated for a duration of one minute. Finally; 5-mL distilled water was used for irrigation to prevent further irrigant interactions. The total final irrigation

Groups	Final Irrigation Regimes
Group 1	5-mL 17% EDTA at 22°C
Group 2	5-mL 17% EDTA at 37°C
Group 3	5-mL QMix at 22°C
Group 4	5-mL QMix at 37°C

[Table/Fig-1]: Experimental groups

volume (NaOCl + EDTA or QMix + Distilled water) was 15 mL for all specimens. The QMix and 17% EDTA solutions that were at room temperature were heated to 37°C using a heating cup that had a digital temperature display (Oushiba, OB-009 280 mL, Guangdong, China). Three specimens from each group were left for observation with the scanning electron microscopic (Quanta™ 450 FEG, FEI, Oregon, USA) to evaluate smear layer removal after final irrigation procedures. A water-cooled diamond bur was used to prepare grooves on the buccal and lingual surfaces of the teeth, and then the teeth were split along their long axis in a buccolingual direction using a hammer and chisel. For analysis with the scanning electron microscope, the samples were dehydrated and coated with gold-palladium particles (20 nm). Next, representative photomicrographs were taken from the middle third of the root canals at 2000× to evaluate the cleanliness of the canal walls. Remaining 12 specimens were dried with paper points in each group. The canals were dried with paper points. Then, all canals were obturated with an epoxy resin–based sealer, AH Plus and F4 gutta-percha cones using the single-cone technique. Root canal filling quality was evaluated with mesiodistal and buccolingual radiographs. Excessive root canal filling was removed from each specimen, and a temporary filling material (Cavit; 3M ESPE, Seefeld, Germany) was used to seal coronal access. All the specimens were stored at 37°C in 100% humidity for 2 weeks to ensure complete setting of sealer. Each root was then embedded in epoxy resin in a custom-made split ring copper mold.

Push-out Testing

After the epoxy resin had set, two horizontal sections of 1-mm thickness each were cut from the middle third of each root using a water-cooled precision saw (Isomet; Buehler, Lake Bluff, IL). A digital caliper (Teknikel, Istanbul, Turkey), an accuracy of 0.001 mm, was used to measure each slice thicknesses. This resulted in 24 horizontal sections per group with a total number of 96 horizontal sections for the four experimental groups. Apical and coronal aspects of slices were examined under microscope to verify circular shape of root canal space [18]. Then all slices were coded regarding to groups. Scanned images of coded slices were used to measure diameter of filling materials via electronic scale software (Adobe Photoshop) and to determine the diameter of suitable plungers for push-out test. A continuous load was applied at a crosshead speed of 1 mm/min from the apical to the coronal direction until bond failure occurred using a universal testing machine (Instron Corp, Canton, MA) [11,19]. Cylindrical plunger with a diameter of 0.8 mm was used, as they matched the diameter of the middle thirds [19]. The tip of plunger was situated in way that it contacted only with root canal filling material and not with dentin surface [20]. The maximum load applied to the filling material before failure was recorded in Newtons (N) and converted to megapascals (MPa) according to the following formula [11,19-21].

$$\text{Push-out bond strength } \{MPa\} = \frac{\text{Maximum load } \{N\}}{\text{Adhesion area of root canal filling } \{mm^2\}}$$

The adhesion area of each section was calculated as: $\{(\pi r_1 + \pi r_2) L\}$, where $L=\sqrt{\{r_1-r_2\}^2+h^2}$. ($\pi$ is the constant 3.14, r_1 is the smaller radius, r_2 is the larger radius, and h is the thickness of the slice in mm).

Analysis of Failure Modes

The failure modes were examined under a stereomicroscope (Olympus Corporation, Taichung, Taiwan) at 40× magnification. Each sample was categorized into 1 of 3 failure modes: adhesive failure at the sealer dentin; cohesive failure within sealer; or mixed failure in both the sealer and dentin [22].

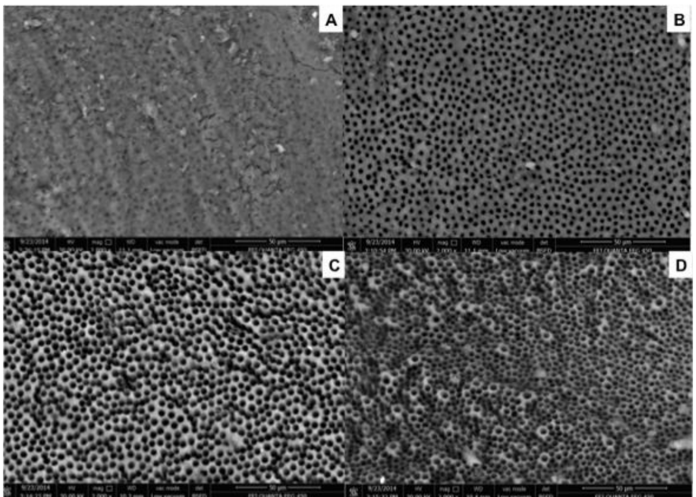
STATISTICAL ANALYSIS

Two-way analysis of variance was used to detect the effect of the independent variables - final irrigants (EDTA and QMix) and temperatures (22°C, 37°C) - and their interaction (final irrigant * temperature) on the push-out bond strength. The Bonferroni post hoc test was performed for multiple comparisons. The significance level was set at $p < 0.05$. All statistical analyses were performed using the SPSS 21.0 software (SPSS Inc., Chicago, IL).

RESULTS

Both the final irrigant (p=0.01) and their temperatures (p=0.007) affected the push-out bond strength values significantly. There appeared to be a statistically significant interaction between irrigant and temperature (p=0.001). The samples irrigated with QMix had higher push-out bond strength values than the samples that had been irrigated with 17% EDTA (p=0.01), regardless of temperature. Regardless of the irrigant, whether at a temperature of 22°C or 37°C, the push-out bond strength values were similar (p=0.17). At 22°C, the push-out bond strength of the samples irrigated with QMix was higher than that of those irrigated with 17% EDTA (p<0.001). On the other hand, there were no significant differences between the irrigants at 37°C (p>0.05).

There were statistically significant differences in push-out bond strength values among samples irrigated with EDTA at 22°C and 37°C (p=0.01). However, there were no significant differences in push-out bond strength values among samples irrigated with different temperatures of the QMix solution (p>0.05). [Table/Fig-2] shows specimens examined using a scanning electron microscope at 2000×. [Table/ Fig-3] shows the mean bond strength values (MPa) after the push-out tests. The modes of fracture are listed in [Table/Fig-4]. Cohesive failure was the most frequent type of failure in all the groups.



[Table/Fig-2]: A: 17% EDTA at 22°C, B: 17% EDTA at 37°C, C: QMix at 22°C, D: QMix at 37°C Representative scanning electron microscope images of experimental groups [2000× magnification]

Irrigant	22°C	37°C	p-value
EDTA	$2.17^a \pm 0.84$	$3.15^b \pm 0.58$	<0.001
QMix	$3.61^b \pm 1.26$	$3.51^b \pm 1.02$	0.902

[Table/Fig-3]: The mean values of push-out bond strength (MPa) and standard deviation (of experimental groups (Mean ± SD). * Different subscripts indicate a statistically significant difference in rows and columns

Failure Type	EDTA		QMix	
	22°C	37°C	22°C	37°C
Adhesive Failure	4	3	1	2
Cohesive failure	14	19	20	18
Mixed	6	2	3	4

[Table/Fig-4]: Failure pattern distribution of experimental groups tested (adhesive failure at the sealer dentin, cohesive failure within sealer, and mixed failure in both the sealer and dentin)

DISCUSSION

Root canal irrigation alters the chemical and structural composition of dentin and these changes affect dentin's permeability and solubility characteristics [23,24] as well bond strength of filling materials to the dentin surfaces [25]. To achieve optimal wettability, the surface energy of the substrate must be as high as possible, and the surface tension of the liquid contacting it must be as low as possible [9,26]. It has been reported that the application of EDTA decreased wetting of the dentin surfaces [9,26,27] or did not alter it at all [28]. Previous studies reported that the surface tension of EDTA solutions dramatically decreased when surfactant was added to them at various temperatures [14,29]. Yilmaz et al., [14] also reported that at a low or neutral pH, the surface tension of EDTA solutions significantly decreased at 37°C as compared to at 22°C. It is believed that using heat, or a surfactant, may reduce surface tension [12-14]. Therefore different temperatures used in the present study; but EDTA was used alone without any surfactant agent. Nevertheless, temperature increase of EDTA significantly enhanced the bond strength of AH Plus sealer to root canal dentin and also the bond strength of samples irrigated with QMix, which consists surfactant were found higher in both temperatures. Commercially available EDTA solutions are stored differently. Usually freshly prepared EDTA solutions are stored at room temperature. Previous studies evaluated irrigants at 22°C and 37°C [14,30], so these temperatures were used in the present study. Manufacturer advises to store QMix at room temperature. According to results of current study, there were no statistically significant differences between the QMix groups of different temperatures.

Ballal et al., [31] reported that when EDTA was used as the final irrigant, the wettability and spreading of AH plus on root canal dentin was reduced, as compared to when QMix was used as the final irrigant. The authors stated that this result might be related to the combined action of CHX and the detergent that is present in QMix. CHX has been shown to increase the free surface energy of dentin and decrease the contact angle of root canal sealers [32], while detergents have an ability to lower surface tension of solutions and improve their wettability [30,33]. It has been reported that flushing the root canal with 2% CHX after 17% EDTA enhanced the bond strength due to the presence of surface surfactant in CHX composition that increases the dentine permeability [12]. The results of present study were consistent with previous studies, sections irrigated with QMix displayed significantly higher push-out bond strengths when compared to those irrigated with 17% EDTA, at room temperature. However, Aranda Garcia et al., [17] reported that the surfactant compound of QMix did not increase the bond strength in comparison with 17% EDTA.

Evaluation of the root slices revealed that fracture modes were mainly cohesive for all groups, in the present study. This is in accordance with results from previous studies that reported a cohesive failure pattern in the AH Plus sealer group [12,19]. This may be because of the high adhesion capacity of AH Plus sealer to the canal dentin [13].

LIMITATIONS

One of the limitations of this study is that only the middle third of the canals were used, as opposed to evaluating the entire canal. The middle third was used to obtain a more circular section, and to preserve the density of the dentin tubules. It has been reported that the bond strength decreased in a coronal to apical direction [19,20], and this can be explained by the fact that the tubule density also decreases from coronal to apical, which therefore reduces sealer penetration in the apical thirds [19,34].

The temperature of the final irrigant affects the push-out bond strength values of AH plus to dentin that was exposed to EDTA.

CONCLUSION

Within limitations of this in vitro study, the null hypothesis of this study was rejected QMix displayed better push-out bond strengths of AH plus sealer compared to those of 17% EDTA, in both experimental temperatures. Using QMix as a final irrigant can improve bond strength of epoxy-resin based sealer to root canal dentin. Nonetheless, further studies are necessary to clarify the effect of the temperature of irrigants on the root dentin surface and on the degree of adhesion of root canal sealers.

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