Effect of different chelating agents on the bond strength of a silicone-based root canal sealer to root dentin

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Abstract

Aim: The purpose of this study was to evaluate the impact of 7% maleic acid (MA), 17% ethylenediaminetetraacetic acid (EDTA) solution, 10% citric acid (CA), or 2.25% peracetic acid (PAA) on the dislodgment resistance of a silicone-based root canal sealer.

Materials and Methods: Ninety-five mandibular incisors were shaped to size R50. The specimens were randomly assigned to the following five groups (n = 15) based on the final irrigation solution: 5% sodium hypochlorite (NaOCl), 17% EDTA, 7% MA, 10% CA, and 2.25% PAA. Four specimens from each group were examined under scanning electron microscope. All the remaining canals were obturated with GuttaFlow® Bioseal. Bond strength was assessed using the push-out test. The data were analyzed statistically by two-way analysis of variance and Bonferroni tests at the significance level of P = 0.05.

Results: The roots irrigated with chelators showed statistically significantly higher bond strength than the roots irrigated with NaOCl (P < 0.05). Overall, MA showed the highest bond strength, but there was no statistically significant difference among the other chelating solutions (P > 0.05).

Conclusions: The bond strength of the GuttaFlow Bioseal to the root canal dentin may increase by the removal of smear layer. Final irrigation with the investigated chelators resulted in similar bond strength of the GuttaFlow Bioseal.

Keywords: Adhesion; chelators; push-out bond strength; root canal irrigants; silicone-based root canal sealer; smear layer

INTRODUCTION

The purpose of endodontic treatment is to completely disinfect the root canal system, and three-dimensional obturation of the root canal system to prevent reinfection. Sodium hypochlorite (NaOCl) is the most commonly used irrigant during endodontic treatment. It has antibacterial and tissue-dissolving abilities. Despite these properties, NaOCl is insufficient in eliminating the smear layer; it may be a source of nutrient for persistent infections and may lead to the failure of endodontic treatment; it may also prevent the penetration of sealer to the dentinal tubules of root canal. Similarly, the bond strengths of different sealers to dentin have been improved with the removal of smear layer.

The most commonly used irrigation solutions to remove smear layer are ethylenediaminetetraacetic acid (EDTA) and NaOCl. However, it was reported that this combination is insufficient for the complete elimination of smear layer. Maleic acid (MA), which is a mild organic acid, removes most part of the smear layer in the apical part of root canal compared to EDTA. Citric acid (CA) and peracetic acid (PAA) have similar activity to EDTA regarding the removal of smear layer. Because smear layer removal with various chelators causes modification in the structural and...
chemical composition of dentin, the microhardness and permeability properties of dentin may change,\textsuperscript{[9]} and thus the penetration ability of sealers in the dentinal tubules\textsuperscript{[7,11]} and the bonding strength of root canal obturation may change as well.\textsuperscript{[8,11,12]}

GuttaFlow\textsuperscript{®} Bioseal (GuttaFlow 3; Coltene Whaledent, GmbH + Co. KG, Langenau, Germany) is composed of bioactive glass ceramic, gutta-percha powder, zirconium dioxide, platinum catalyst, silver particles, and polydimethylsiloxane.\textsuperscript{[13]} It has similar dentin tubular penetration as AH Plus and MTA Fillapex.\textsuperscript{[14]}

To our knowledge, there is no study evaluating the effect of different chelator solutions on the dislocation resistance of GuttaFlow Bioseal root canal sealer to root dentin walls. The null hypothesis was that these chelator solutions would cause similar dentin–sealer bond strength.

**MATERIALS AND METHODS**

After ethics committee approval (protocol no: 17-KAEK-035), 95 extracted, single-rooted human mandibular anterior teeth with straight root and single canal were collected. Then, the teeth were decoronated to obtain roots of 15 mm in length. A #10 K-file was used to determine the working length. The root canals were shaped using Reciproc instruments (VDW, Munich, Germany) up to R50 file. The root canals were irrigated with 2 mL of 5% NaOCl (Werax; SDD, Izmir, Turkey). Five milliliters of distilled water was used to remove NaOCl. The samples were randomly divided into five groups (n = 15 each) according to the final irrigation solutions applied: 5% NaOCl (control), 17% EDTA (Werax; SDD, Izmir, Turkey), 7% MA (Sigma-Aldrich, Saint Louis, MO, USA), 10% CA (Merck KGaA, Darmstadt, Germany), and 2.25% PAA (Sigma-Aldrich Chemie, Steinheim, Germany).

Each 5 mL of the final irrigation solution was allowed to remain in the canal for 1 min without activation. Then, the root canals were rinsed with 5 mL of distilled water and dried using paper points.

The GuttaFlow Bioseal was prepared according to the instructions of the manufacturer and applied into the root canal using a lentulo spiral. A R50 (VDW, Germany) single-cone gutta-percha was slightly coated with the sealer and introduced into the root canal using single-cone technique.\textsuperscript{[13]} The excess gutta-percha and sealer were removed, and access to cavities was temporarily sealed (Cavit, ESPE GMBH, Seefeld, Germany). All specimens were then incubated in 100% humidity at 37°C for 3 weeks.

The specimens were cut horizontally using a water-cooled precision saw (Micracut; Metkon, Bursa, Turkey). Three 2 ± 0.01-mm-thick horizontal sections were obtained at 12, 6, and 2 mm (coronal, middle, and apical root sections, respectively) distance from the apex (n = 45 slices/group). A digital caliper (Macrorna, Simetri Teknik, Izmir, Turkey) with 0.01-mm accuracy was used to measure the final thickness of each slice. A universal testing machine (AGS-X, Shimadzu, Kyoto, Japan) at a cross-head speed of 0.5 mm/min was used to apply the push-out test. A modified hand spreader which had at least 90% of the apical diameter of the canal was placed to the universal testing machine. The maximum load at failure recorded in Newtons was used to calculate debonding values in megapascals by dividing Newtons to the bonding area of root canal filling. The bonding area was calculated as follows: \(\pi (R + r) [l h^2 + (R - r)^2]^{0.5}\) where \(h = 2 \text{ mm}, \pi = 3.14, R\) is the coronal radius, and \(r\) is the apical radius in mm.

All the fractured specimens were examined under a stereomicroscope (Zeiss Stemi 2000-C; Carl Zeiss Microlmaging, Göttingen, Germany) at \(\times30\) magnification for failure modes according to Nagas et al.\textsuperscript{[15]}

A1: adhesive failure between the sealer and root canal wall,
A2: adhesive failure between the gutta-percha and sealer,
C: cohesive failure, and M: mixed failure.

Four specimens left in each group were prepared for examination with a scanning electron microscope (SEM). Each third of the root canal space was photographed under a SEM (Jeol JSM-7001F; Tokyo, Japan).

Two-way analysis of variance was used to compare the continuous normal data among groups in terms of dislocation resistance. For post hoc comparisons between the pair-wise groups, Bonferroni test was used. Significance was set at the level of 5%. Analyses were performed using SPSS 19 (IBM SPSS Statistics 19, SPSS Inc., an IBM Co., Somers, NY, USA) software package program.

**RESULTS**

The dislodgment resistance values are presented in Table 1. The removal of the smear layer improved the dislodgment resistance of the root canal sealer. The NaOCl group had the lowest resistance to dislodgment in all regions compared to other groups (\(P < 0.05\)). The highest values were observed in the coronal thirds for all chelators, and there was a statistically significant difference between the middle and apical thirds (\(P < 0.05\)).

Regardless of the region, the NaOCl group exhibited significantly lower bond strength than the other groups (\(P < 0.05\)), whereas there were no statistically significant differences among chelators (\(P > 0.05\)).

Figure 1 depicts photomicrographs from each third. The SEM examination showed that NaOCl irrigation was not effective in removing the smear layer in all regions.
The distribution of failure types in the groups is shown in Table 2. A2, M, C, and A1 failures were the most commonly observed failure types, regardless of the type of final irritant used.

**DISCUSSION**

An ideal root canal obturation material should bond and penetrate as much as possible to the root canal wall. The push-out test is a useful test used to measure this bond strength. In a previous study, GuttaFlow Bioseal was observed to have similar dentinal tubule penetration ability to AH Plus. However, there is currently no study evaluating the dislodgment resistance of GuttaFlow Bioseal root canal sealer to root canal dentin after applying different chelators.

Similar to previous studies, this study revealed the importance of final irrigation protocols in the interaction between root dentin and sealers. The use of NaOCl as final irrigation solution led to the low bond strength values of GuttaFlow Bioseal, whereas the use of other chelators led to the high bond strength values. As there is no study to be utilized a reference to compare the result of the dislocation resistance of GuttaFlow Bioseal, comparison could not be made. Carvalho et al. reported that EDTA, PAA, or CA did not affect the dislodgment resistance of two calcium silicate-based (MTA Fillapex and TotalFill BC Sealer) and one epoxy resin-based (AH Plus) sealers. Similarly, it was reported that PAA, etidronic acid, and EDTA did not increase the dislocation resistance of iRoot® SP and AH Plus to the root canal wall. Contrary to these results, Leal et al. reported that the final irrigation protocols affected the dislodgment resistance of AH Plus. Similarly, the canals which were irrigated with NaOCl and covered with the smear layer showed lower bond strength for AH Plus than those irrigated with the other irrigants which removed the smear layer.

Akçay et al. showed that passive ultrasonic irrigation and photon-induced photoacoustic streaming activation techniques enhanced the penetration of GuttaFlow Bioseal more than the conventional syringe irrigation. Similar to the results of the above study, the smear layer in the NaOCl group [Figure 1] may have acted as a physical barrier between GuttaFlow Bioseal and dentin and thereby may have prevented chemical bonding. However, the investigated chelators removed the smear layer and enhanced the bonding strength of the sealer. RoekoSeal (Coltène Whaledent, Langenau, Germany), which is a type of silicone base, had shown no measurable bond strength to root canal dentin. The reason for this, unlike to the presented study, may be attributed to using 1-mm-thick root discs.

The combination of EDTA and NaOCl is the most commonly used irrigation protocol. However, EDTA may cause intertubular and peritubular dentinal erosion when used for >1 min. Furthermore, the cytotoxic potential of these two combinations should not be neglected, MA, which is a weak organic acid, has lower cytotoxicity and has superior smear layer removal ability than EDTA. In this study, the final irrigation with MA increased the dislodgment resistance of GuttaFlow Bioseal without a significant difference compared to EDTA. The similar smear layer removal efficiencies of EDTA and CA were observed in a previous study. The presented study revealed similar dislodgment resistance in the EDTA and CA groups. Similar to the aforementioned studies, the removal of the smear layer manifested similar dislodgment resistance of root canal filler in the EDTA and PAA groups. De-Deus et al. reported the similar smear layer-dissolving capacity of EDTA and PAA. The elimination of smear layer with the investigated chelators improved the dislodgment resistance of sealer without causing any significant difference among groups. Thus, the tested null hypothesis was accepted.

A main core material was used to strictly simulate the clinical conditions. However, previous studies showed that when the root canal sealer was used without core material, it would create a monoblock formation along the root canal walls and constitute a homogenous obturation material, it would create a monoblock formation along the root canal walls and constitute a homogenous obturation material. RoekoSeal (Coltène Whaledent, Langenau, Germany), which is a type of silicone base, had shown no measurable bond strength to root canal dentin. The reason for this, unlike to the presented study, may be attributed to using 1-mm-thick root discs.

Table 1: Push-out bond strength values (MPa, mean±standard deviation) of the experimental groups

| Groups | Coronal | Middle | Apical | Total |
|--------|---------|--------|--------|-------|
| NaOCl  | 1.7±0.65* | 1.66±0.44* | 1.7±0.63* | 1.69±0.57* |
| EDTA   | 3.58±0.75* | 2.57±0.66* | 2.42±0.51* | 2.86±0.82* |
| MA     | 4.3±1.17* | 2.57±0.36* | 2.32±0.44* | 3.06±1.15* |
| CA     | 3.35±1.1* | 2.48±0.67* | 2.27±0.73* | 2.7±0.93* |
| PAA    | 3.67±0.92* | 3.06±0.63* | 2.38±0.8* | 3.03±0.94* |
| Total  | 3.32±1.25* | 2.47±0.71* | 2.22±0.67* | 2.67±1.03* |

Different superscripts (†,‡,§) indicate statistically significant values within each column (P<0.05) and different superscripts (*,†) indicate statistically significant values within each row (P<0.05). NaOCl: Sodium hypochlorite, EDTA: Ethylenediaminetetraacetic acid, MA: Maleic acid, CA: Citric acid, PAA: Peracetic acid

Table 2: Distribution of failure modes according to root regions

| Groups | Adhesive 1 | Adhesive 2 | Cohesive | Mixed |
|--------|-----------|-----------|----------|-------|
| C      | M         | A         | C        | M     | A     |
| NaOCl  | -         | -         | 4        | 5     | 5     | -     | 1     | -     | 11    | 9     | 10    |
| EDTA   | -         | -         | 8        | 10    | 10    | -     | 1     | 7     | 4     | 4     |
| MA     | -         | 1         | 2        | 11    | 5     | -     | 2     | 13    | 10    |
| CA     | -         | -         | 8        | 12    | 10    | -     | -     | 7     | 3     | 5     |
| PAA    | -         | -         | 7        | 8     | 11    | 2     | -     | 6     | 7     | 4     |
| Total (%) | 1 (0.4) | 116 (51.5) | 7 (3.1) | 101 (44.8) |

C: Coronal, M: Middle, A: Apical. NaOCl: Sodium hypochlorite, EDTA: Ethylenediaminetetraacetic acid, MA: Maleic acid, CA: Citric acid, PAA: Peracetic acid
reported that using even plastic, soft-core materials such as gutta-percha did not affect the dislocation resistance when the punch size was equal to 70%-90% of the canal width. In accordance with this statement, the modified hand spreaders which cover at least 90% of the canal width were used in this study.

The push-out test is a reliable, easy, and widely used technique to investigate the adhesion of root canal obturation materials.\cite{6,7,12,15,16,21,22} However, there was no consensus on the variables of push-out test protocols such as slice thickness, storage time, root canal/punch diameter relation, load speed, and core material. A recently published review demonstrated that these variables influenced the bond strength of materials.\cite{23} Therefore, there is a need for further studies where all the variables are standardized.

In the CA, EDTA, and PAA groups, the most commonly found pattern was A2. Therefore, in these groups, the force required to rupture the sealer and dentin interface was higher than that needed at the gutta-percha and sealer interface. In the MA and NaOCl groups, mixed failure was the most common type of fracture mode. This result may be associated with the high bonding capacity of GuttaFlow.
Bioseal to canal dentin rather than gutta-percha. These results are similar to the results obtained by previous studies.\textsuperscript{15,22}

The dislocation resistance of the root canal filler was increased in the apical to coronal direction in all chelator groups. This may be caused by the fact that the area of the connection surface in the coronal part is larger than that of the apical part and the sealer did not adequately wet the apical dentin as much as the coronal dentin.\textsuperscript{23} Furthermore, the increase in the volume and effectiveness of the chelators from apical to coronal direction may be associated with an increase in the number of open tubules.\textsuperscript{7,24} What is more, the vapor lock effect in the apical region may prevent the penetration of irritants.\textsuperscript{24}

CONCLUSIONS

The elimination of smear layer improved the dislodgment resistance of the GuttaFlow Bioseal. Overall, the investigated chelators showed similar dislocation resistance of the sealer.

Acknowledgment

The authors thank Dr. Osman Demir for his valuable assistance with the statistical analysis.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

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