Does Adding Various Accelerators to Mineral Trioxide Aggregate Have a Negatively Effect on Push-Out Bond Strength?

Alper İlker  Evren Sarıyılmaz  Fatih Çakıcı
Ordu University Faculty of Dentistry, Department of Endodontics, Ordu, Turkey

Significance of the Study
- Mineral trioxide aggregate (MTA) is widely used in dentistry due to its satisfactory properties such as its biocompatibility and mechanical properties. However, the long setting time of up to 4 h is one of the main drawbacks of its use. Previously, a variety of accelerators were added to the MTA mixture to overcome these drawbacks. This study reveals that the push-out bond strength could be affected by the type of accelerator.

Keywords
Accelerators · Calcium chloride · Disodium hydrogen orthophosphate · KY jelly · Mineral trioxide aggregate · Push-out bond strength

Abstract
Objective: This study compares the effect of the white mineral trioxide aggregate (WMTA) accelerators, including disodium hydrogen orthophosphate (Na2HPO4; 2.5 wt%), calcium chloride (CaCl2; 5 and 10 wt%), and KY jelly, on the push-out bond strength of WMTA. The null hypothesis was that the WMTA accelerators would not affect the push-out bond strength of WMTA. Materials and Methods: Slices (2-mm-thick) were obtained from 75 human mandibular molar distal roots. The slices were enlarged up to size 6 Gates-Glidden burs to obtain a 1.5-mm canal diameter. The slices were randomly divided into 4 experimental groups and a control group (n = 15 in each group). Freshly prepared WMTA mixture was placed into the root slices and stored at 37°C in a 100% humidified atmosphere for 60 days. The force required to dislodge the WMTA cement from the root slice was determined using a universal testing machine. Results: Push-out bond strength of 5- and 10-wt% CaCl2, and 2.5-wt% Na2HPO4 WMTA groups was significantly lower than in the KY-jelly and control groups (p < 0.05). The mean push-out bond strength of KY jelly was lower than in the control group but not statistically significant. Conclusion: The addition of KY jelly to WMTA did not have an adverse effect on the push-out bond strength of WMTA, in contrast to the other accelerators, including Na2HPO4 and CaCl2, which reduced the push-out bond strength.

Introduction
The principal ingredients of mineral trioxide aggregate (MTA) powder are tricalcium silicate, tricalcium aluminate, tricalcium oxide, and silicate oxide. Trace amounts of a few other mineral oxides, which have vari-
ous effects on the chemical and physical properties of MTA, are also present [1]. The use of MTA is indicated in many endodontic procedures, including vital pulp therapy [2], pulpotomy [3], repair of furcation [4] or root perforations [5], and retrograde filling [6]; this is on account of its excellent biological, chemical, and physical properties [7–9].

Sealing ability is one of the key factors for the success of MTA because effective sealing blocks leakage to the inside of the tooth and thus prevents reinfection [10]. Reyes-Carmona et al. [11] showed that an interfacial layer with a tag-like structure formed at the MTA and dentin surface, and that this biomineralization process could enhance the push-out bond strength of MTA.

The main drawback of MTA is the long setting time of up to 4 h after mixing, thus necessitating multiple treatment sessions [12]. Various accelerators have been added to shorten the setting time and overcome this drawback [13, 14]. Disodium hydrogen orthophosphate (Na₂HPO₄) [15], calcium chloride (CaCl₂) [14], and KY jelly have been reported to significantly reduce the setting time [13, 16]; however, previous studies also revealed that the accelerator additives can have adverse effects on the physicochemical properties of MTA [15, 17].

The main purpose of this study was to compare the effects of the MTA accelerators Na₂HPO₄, CaCl₂, and KY jelly on the push-out bond strength of MTA. The null hypothesis was that the MTA accelerators would not affect the push-out strength of MTA.

**Materials and Methods**

The study protocol was approved by the Ordu University Clinical Research Ethics Committee (2016/82). This study was performed in accordance with the World Medical Association Declaration of Helsinki and written informed consent was obtained from all participants. Seventy-five single-canal distal roots of human mandibular molar teeth extracted due to periodontal disease were used in this study. Organic and inorganic remnants on teeth were removed with a periodontal curette, and the teeth were then stored in distilled water. Teeth were excluded if they had a distal curvature was > 10° according to the Schneider method [18], the root that had > 1 canal, a calcified canal, internal or external resorption, fractures, or extensive root caries. An ISO size 10 K-file was inserted into every distal root canal, and then radiographs were taken to determine the degree of root canal curvature; if the curvature was > 10° according to the Schneider method, the tooth was also excluded. Afterward, the crowns and mesial roots of the remaining teeth were removed by using a water-cooled diamond fissure bur (Maillefer, Ballaigues, Switzerland). A 2-mm-thick slice from the middle third of each distal root was obtained by using a water-cooled, low-speed diamond saw (Mecatome T180, Presi, France). Each slice was enlarged by using size 2–6 Gates-Glidden burs to obtain a canal diameter of 1.5 mm. Afterwards, the teeth were immersed for 3 min in 2.5% NaOCl and 2 min in distilled water. EDTA was not used in the irrigation protocol so as to preserve the inorganic texture of the dentin [19]. Furthermore, Lee et al. [20] have shown that EDTA might disrupt the hydration of MTA. The slices were randomly divided into 5 groups (n = 15).

Exact amounts of white MTA (WMTA; ProRoot MTA; Dentsply Tulsa Dental, Tulsa, OK, USA, lot No. 0000161264) and the tested accelerators were weighted using an analytical balance with 10⁻⁴ g accuracy (Precisa XB 220A, Precisa Instruments, Dietikon, Switzerland) and then mixed. After that, distilled water or KY jelly was added to the previously prepared mixture using a pipette with a precision range of 5–10 μL (Eppendorf Research® plus, Hamburg, Germany). The experimental groups described below were prepared according to previous studies by using the method described above [21].

Group 1 (WMTA + 5-wt% CaCl₂) consisted of 1.6 g WMTA, 0.08 g CaCl₂ (Merck, Darmstadt, Germany), and 0.63 mL of distilled water. Group 2 (WMTA + 10-wt% CaCl₂) consisted of 1.6 g WMTA, 0.16 g CaCl₂, and 0.66 mL of distilled water. Group 3 (WMTA + 2.5-wt% Na₂HPO₄) consisted of 1.6 g WMTA, 0.04 g Na₂HPO₄ (Merck), and 0.615 mL of distilled water. Group 4 (WMTA + KY jelly) consisted of 1.6 g WMTA and 0.6 mL KY jelly (Johnson and Johnson, Markham, ON, Canada). Group 5, the control group, consisted of 1.6 g WMTA and 0.6 mL distilled water.

Freshly prepared cement was immediately placed into the root slices using an MTA carrier, and then condensed using a plugger. Excess WMTA was removed from the surface of the slices with a spatula. Afterwards, all samples were visualized under a ×10 microscope to identify any cracks, defects, or gaps between the material and dentin walls. If any of these were identified on a sample, it was discarded and a new one was prepared. The specimens were then wrapped in gauze moistened with distilled water and stored at 37 °C in a 100% humidified atmosphere for 60 days, as previously reported.

**Push-Out Tests**

A universal testing machine (Autograph AGS X; Shimadzu Co., Japan) was used to measure the force required to dislodge the cement from the root slice. Specimens were placed on a metal holder with a 1.7-mm-diameter hole to allow free motion of the material and dentin walls. If any of these were identified on a sample, it was discarded and a new one was prepared. The specimens were then wrapped in gauze moistened with distilled water and stored at 37 °C in a 100% humidified atmosphere for 60 days, as previously reported.

**Statistical Analysis**

The data were analyzed using SPSS v16.0 (SPSS Inc., Chicago, IL, USA). The Shapiro-Wilk test revealed that the data were not normally distributed (p < 0.05). Differences between the groups were assessed by the Kruskal-Wallis test and Mann-Whitney U test. The level of significance was set at 0.05.
Results

The mean values of push-out bond strength and the standard deviation for each group are presented in Table 1. Group 1 (5-wt% CaCl₂), group 2 (10-wt% CaCl₂), and group 3 (2.5-wt% Na₂HPO₄) showed significantly lower push-out bond strength than groups 4 (KY jelly) and group 5 (control) \((p < 0.05)\); no significant differences in push-out strength were found between groups 1, 2, and 3 \((p > 0.05)\) or between groups 4 and 5 \((p > 0.05)\).

Discussion

The push-out test which was used in this study is frequently performed for evaluating the strength of calcium-silicate-based materials [22]. Chen et al. [23] suggested that the pin diameter should be <0.85 times the filler diameter, but not so small as to puncture the filler material. They also claimed that the push-out bond strength formula is only suitable for a specimen thickness >0.6 times the filler. In this study, these ratios were 0.8 and 1.33, respectively.

Conditions for keeping specimens until they are tested is another key factor for push-out tests, especially in MTA studies. Reyes-Carmona et al. [11] reported that keeping MTA in PBS until the push-out test was conducted could enhance the push-out strength due to biomineralization. They also explained that tag-like structures were formed in the dentinal tubules at specimens immersed in PBS, but that such structures were not seen in the specimen in contact with the wet cotton pellet. We wrapped specimens in gauze moistened with distilled water. The purpose of our study was to evaluate the effect of accelerators on the push-out bond strength of MTA, and these accelerators may affect the formation of tag-like structures. This should be tested in further studies.

The smear layer is a loosely adherent layer (with a thickness of 1–5 μm) that covers the root dentin surface after root canal instrumentation. It is made of organic and inorganic material, i.e., dentin shavings, necrotic pulp remnants, bacteria and their products. EDTA and NaOCl should be used in succession to achieve effective smear layer removal. It has been shown that the removal of the smear layer enhances the push-out strength of bone to the root dentin of the root canal filling, which consists of gutta percha and root canal sealer. On the contrary, removal of the smear layer has been shown to reduce the push-out strength to bone to root dentin containing calcium silicate cements such as MTA [24]. The authors of that study suggested that the smear layer could be involved in mineral interactions between the cement and the root dentin [24]. In addition, Lee et al. [20] demonstrated that EDTA disrupts the hydration of MTA by chelating calcium ions released from the tricalcium complex in the MTA.

The push-out bond strength appears as a mean (standard deviation). Groups that do not share the same superscript letter are significantly different \((p < 0.05)\). The values were rounded to 1 decimal place.

### Table 1. Push-out bond strength values for each group

| Specimens, n | Push-out bond strength, MPa |
|-------------|-----------------------------|
| Group 1 (5%-wt CaCl₂)\(^a\) | 15 | 3.9 (1.8) |
| Group 2 (10%-wt CaCl₂)\(^a\) | 15 | 4.9 (2.1) |
| Group 3 (2.5%-wt Na₂HPO₄)\(^a\) | 15 | 5.6 (5.1) |
| Group 4 (KY jelly)\(^b\) | 15 | 9.3 (2.3) |
| Group 5 (distilled water)\(^b\) | 15 | 10.4 (3.0) |

The push-out bond strength appears as a mean (standard deviation). Groups that do not share the same superscript letter are significantly different \((p < 0.05)\). The values were rounded to 1 decimal place.
KY jelly is a water-based lubricant that contains chlorhexidine [28] and it was previously used as an MTA accelerator [17]. Kogan et al. [17] reported that KY jelly significantly lowered the compressive strength of the MTA mixture. Similarly, we observed that KY jelly decreased the push-out bond strength values, although this difference was not statistically significant. Unfortunately, no previous study on the effect of KY jelly on the push-out bond strength of MTA exists for comparison.

MTA, due to its hydrophilic particles, requires the presence of water to set [29]. Bentz [30] reported that chemical structure shrinkage occurs when Portland cement is hardened in a dry environment; however, in a moist environment, Portland cement hydration concludes with expansion due to crystal growth and the probable swelling of hydration products. Similarly, Hawley et al. [31] reported that both gray MTA (approx. 2.2–3%) and WMTA (approx. 0.6–1%) also expand during setting, at different expansion rates. On the other hand, accelerators (calcium chloride, calcium nitrite/nitrate, and calcium formate) have been shown to alter the expansion rate in a statistically non-significant manner [13]. Taken together, possible alteration of the expansion rate of MTA due to accelerators might affect the push-out bond strength of MTA to root dentin. This has not been confirmed, however, and further studies are needed to test this hypothesis.

**Conclusion**

The type of accelerator may affect the push-out bond strength of MTA. Our results show that the addition of KY jelly accelerator to WMTA did not have an adverse effect on push-out bond strength of WMTA, but that the accelerators Na$_2$HPO$_4$ and CaCl$_2$ reduced it.

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**Disclosure Statement**

We have no conflicts of interest to declare.

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