



## The Effect of Calcium Chloride on Push-Out Bond Strength of Calcium-Enriched Mixture Cement and Mineral Trioxide Aggregate

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### ABSTRACT

**Introduction:** This *in vitro* study investigated the effect of adding 10% calcium chloride (CaCl<sub>2</sub>) on push out bond strength of calcium-enriched mixture (CEM) cement and mineral trioxide aggregate (MTA) to root canal dentin. **Methods and Materials:** A total of 120 root dentin slices with 2 mm thickness were prepared from sixty single-rooted human teeth. Dentinal discs were enlarged to achieve 1.3 mm diameter. The specimens were randomly allocated into eight groups ( $n=15$ ). Dentin discs were filled with either CEM cement or MTA with or without CaCl<sub>2</sub> and the push out test was performed after 3 and 21 days. Data were analyzed with two-way ANOVA test. The level of significance was set at 0.05. **Results:** There was an interaction effect amongst all groups ( $P=0.028$ ). After 3 days, CEM cement showed a significantly lower bond strength than other groups ( $P<0.05$ ) while MTA demonstrated significantly higher bond strength than CEM cement with or without CaCl<sub>2</sub> ( $P=0.001$ ). After 21 days, CEM cement with or without CaCl<sub>2</sub> had no significant difference with other groups ( $P>0.05$ ). However, the bond strength of MTA decreased when CaCl<sub>2</sub> was added ( $P=0.011$ ). **Conclusion:** The addition of 10% CaCl<sub>2</sub> increased the push out bond strength of CEM cement and improved it over time; while, this substance aggravated this property for MTA.

**Keywords:** Calcium Chloride; Calcium-Enriched Mixture Cement; Mineral Trioxide Aggregate; Push-Out Bond Strength

### Introduction

Mineral trioxide aggregate (MTA) is widely used for endodontic purposes such as pulp capping, root end filling and perforation repair [1, 2]. MTA has many clinical advantages and is proved to be biocompatible [3, 4]; however, due to its granular consistency, slow setting time and initial looseness, this cement has some difficulties to use for clinicians. Thus, continuous attempts has been made to improve its handling characteristic and decrease its setting time by adding different calcium compounds or hydration accelerator such as calcium

chloride (CaCl<sub>2</sub>) [5, 6], low-dose citric acid [7] and calcium lactate gluconate [8-10].

Calcium-enriched mixture (CEM) cement is another hydrophilic cement which forms hydroxyapatite with its endogenous and exogenous ion sources. The advantages of this material over MTA are better handling, lower film thickness and shorter setting time [11]. This cement has an antibacterial activity similar to calcium hydroxide but superior to MTA [11]. It has also exhibited low toxicity similar to MTA [12]. However, the level of solubility is still a controversial issue for this cement [13-15].

The idea of adding CaCl<sub>2</sub> to endodontic cements was initially tested on Portland cement. It was found that when Portland cement was mixed with CaCl<sub>2</sub> and immersed in phosphate buffered saline (PBS), the resistance of this cement to displacement forces increased compared to homologous samples without CaCl<sub>2</sub>. It has been proved that hydration accelerators can decrease the setting time of MTA while they have no adverse effect on biocompatibility of this material [9, 16]. Furthermore, these accelerators can increase the osteogenic effect and improve the mineralization of MTA [17]. A recent study showed that the compressive strength of MTA mixed with hydration accelerators decreased compared to those samples mixed with distilled water however this value increased with the elapse of time [8]. It has been found that 10% CaCl<sub>2</sub> improved the solubility, pH and setting time of CEM cement [15]. However Tabrizzadeh *et al.* [18] revealed that this substance was not able to significantly improve the marginal adaptation of this cement to the root canal walls.

Several methods have been used to evaluate the adhesion of root end filling materials to dentinal wall. Tensile, shear and push-out tests are among these test [19, 20]. Push-out test is based on shear and functional stresses and it can be simulated in clinical situations [21]. Since the push-out test creates fractures parallel to the interfacial area of dentin material interface, it is able to present a better understanding to evaluate bond strength compared to the other conventional tests [22].

The purpose of this study is to investigate the effect of adding 10 % CaCl<sub>2</sub> to CEM cement and MTA on their push-out bond strength to dentin.

## Materials and Methods

Sixty extracted single-rooted human teeth were selected. The crowns were removed and the middle third of the roots were sectioned twice transversally by using a water-cooling low-speed ISOMET diamond saw (SP1600 microtome; Leica, Nussloch, Germany) in order to obtain 120 discs with 2±0.5 mm thickness. In each section, the space of the canal was enlarged using a spherical diamond bur with two complete passes of a #5 Gates-Glidden bur (Mani, Tochigi, Japan) to obtain 1.3 mm-diameter standardized cavities. The sections were immersed in 17% EDTA (Vista Dental Products, Racine, WI, USA) for 3 min followed by 1% NaOCl for the same period of time. They were then immediately washed in distilled water and dried. The root sections were randomly divided into 8 groups of 15 samples on the basis of the materials used including MTA (Angelus, Londrina, PR, Brazil) or CEM cement (Bionique Dent, Tehran, Iran) and the

presence or absence of 10% CaCl<sub>2</sub> (Sigma Aldrich, St. Louis, MO, USA). The push-out test was performed after 3 and 21 days.

To add 10% CaCl<sub>2</sub>, 1.00 g of this substance was first dissolved in 0.66 g of liquid using a magnetic stirrer (L82, Labinco BV, Breda, The Netherlands) and this solution was then mixed with 1.00 g of either CEM cement or MTA powder. The dentin slices were placed on a metal slab with a central hole to allow for the free motion of the carrier. The cement mixture was placed into cavities with a Dovgan carrier (G. Hartzell and Son, Concord, CA, USA) and compacted with pluggers (Dentsply, Tulsa Dental, Tulsa, OK, USA). Excess material was trimmed from the surface of the specimens with a scalpel. All specimens were examined under a microscope under 10× magnification in order to discard the samples with any cracks, defects, or gaps between the material and dentin walls.

In all groups, a wet cotton pellet was placed over the cement, and the samples were stored at 37°C. Sixty specimens were stored for 3 days and the others were stored for 21 days. Gauzes were refreshed every day.

After the experimental periods, the push-out bond strength was measured using a universal testing machine (Z050, Zwick GmbH, Ulm, Germany). The dentin disks were placed on a metal slab with a central hole to allow for free motion of the plunger.

The specimens were loaded with a 0.7-mm diameter cylindrical stainless steel plunger at a speed of 1 mm/min. The maximum load applied to CEM cement before dislodgement, was recorded in Newton (N). To express the bond strength in mega Pascal (MPa), the recorded values in N was divided by the adhesion surface area of the experimental cements in mm<sup>2</sup> calculated according to following formula;  $2\pi r \times h$ , where  $\pi$  is the constant 3.14,  $r$  is the root canal radius (1.3 mm), and  $h$  is the thickness of the root slice in mm.

## Statistical analysis

Logarithmic transformation was first done to normalize the data. Then, the two-way ANOVA test was used to assess the interaction effect between the groups and time. Furthermore, Tamhane's post hoc test and *t*-test was used for multiple comparisons. The level of significance was set at 0.05.

## Results

The mean values and standard deviations of push-out bond strength in all experimental groups are shown in Table 1. There was an interaction effect amongst the groups ( $P=0.028$ ). Subgroup analysis showed that after 3 days, CEM cement showed a significantly lower bond strength than other groups ( $P<0.05$ ). The results for CEM cement+CaCl<sub>2</sub> did not show any

significant difference from those of MTA+CaCl<sub>2</sub> ( $P=0.84$ ) but it exhibited a significantly higher bond strength than CEM alone ( $P=0.007$ ) and a significantly lower bond strength than group MTA alone ( $P=0.001$ ). In other words, MTA showed a significantly higher bond strength than CEM cement with or without CaCl<sub>2</sub> ( $P=0.001$  for both groups) although it did not show any significant difference when it was mixed with CaCl<sub>2</sub>.

After 21 days, CEM cement either alone or mixed with CaCl<sub>2</sub> showed no significant difference with other groups. However, the bond strength of MTA decreased when it was mixed with CaCl<sub>2</sub> ( $P=0.011$ ).

The push-out bond strength of CEM cement both alone or with CaCl<sub>2</sub> increased significantly from day 3 to day 21 while this manner was not the same for MTA alone and MTA+CaCl<sub>2</sub> groups.

## Discussion

The present study was designed to evaluate and to compare the bond strength of MTA and CEM cement with or without CaCl<sub>2</sub> on push-out bond strength of these cements to dentine at two time intervals. Evaluation of the bond strength between these materials and dentinal wall will show the value of adhesion between them. Hence, resistance of dental materials to dislodgement forces is an important factor in the success of different endodontic procedures such as repair of perforations, apical barrier formation and root end fillings [23].

Different techniques can be used to evaluate the bond strength of a dental material to dentin including tensile, shear and push-out bond strength tests. In the present study, the push-out bond strength test was used as it is the most reliable method for evaluating the resistance of materials to dislodgement forces [24].

In the current study, after 3 days, CEM cement showed the worst results amongst the groups. However when CaCl<sub>2</sub> was added, the push-out bond strength increased and it even got better after 21 days. In general, the results for both CEM cement alone and with CaCl<sub>2</sub> improved significantly from day 3 to day 21. This trend was also the same for MTA alone and with CaCl<sub>2</sub> but the increase was not significant. Previous results have indicated that the bond strength of MTA tend to increase from 3 to 21 days [25, 26]. Rahimi *et al.* [27] also reported an increase in the bond strength of CEM cement from day 1 to day 7 when

it was mixed with normal saline. The increase in the bond strength of these materials might be attributed to the hydration and expansion process of these cement during time [28].

The present study showed that the addition of 10% CaCl<sub>2</sub> to MTA could not increase the bond strength of this material and it also got worse during the time. Consistent with our results, Almeida *et al.* [29] revealed that the presence of 10% CaCl<sub>2</sub> can negatively influence the bond strength of this material after 60 days.

Lee *et al.* [8] worked on different hydration accelerators such as CaCl<sub>2</sub>, low dose citric acid and calcium lactate gluconate solution and showed that mixing MTA with these hydration accelerators decreased the compressive strength of MTA; however, the values increased during the time [8]. These findings were partially in accordance with the results of the present study. It has also been shown that adding 2% CaCl<sub>2</sub> to MTA resulted in an initial setting time reduction from 202 to 57 min [16]. Given that the acceleration in setting time occurring in these materials is due to the penetration of the particles such as CaCl<sub>2</sub> to the cement pores, it might be assumed that this phenomenon can cause expansion and may potentially reduce the bond strength to dentin [5, 30].

The difference between the behavior of CEM cement and MTA in contact with CaCl<sub>2</sub> might be attributed to the different compositions and particle sizes of these cements. Danesh *et al.* [31] showed that the particle size of cement is an issue that directly influences its mechanical properties. CEM cement has smaller particle size and also more homogeneous nature compared to MTA. It is notable that the smaller particle size can also cause faster setting time. It has been demonstrated that adding CaCl<sub>2</sub> to CEM cement decrease its setting time to the half but this ratio was about 1 quarter for the MTA [15, 16]. In general, it may imply that CEM cement may not affect in a simple manner with this hydration accelerator.

Further investigation on other physicochemical properties of CEM cement in combination with CaCl<sub>2</sub> is needed before clinical recommendation.

## Conclusion

Calcium chloride increased the push-out bond strength of CEM cement and it improved it over time. This manner was not the same for MTA.

**Table 1.** Mean (SD) of push-out bond strength of the experimental groups

Period	CEM	CEM+CaCl <sub>2</sub>	MTA	MTA+CaCl <sub>2</sub>
3 days	-0.79 (0.46) <sup>A, a</sup>	-0.21 (0.41) <sup>BD, a</sup>	0.62 (0.48) <sup>C, a</sup>	0.09 (0.766) <sup>CD, a</sup>
21 days	0.40 (0.61) <sup>AB, b</sup>	0.49 (0.605) <sup>AB, b</sup>	0.83 (0.77) <sup>A, a</sup>	-0.080 (0.67) <sup>B, a</sup>

Within the same row, means with the same uppercase superscript letter are not statistically different ( $P>0.05$ ). Within the same column, means with the same lowercase superscript letter are not statistically different ( $P>0.05$ )

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