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Original Article

Push-out Bond Strength of Calcium Enriched Mixture Exposed to Alkaline Environment

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Abstract

Statement of Problem: Calcium hydroxide which is commonly used as an intracanal medicament, changes the pH of dentin and periradicular tissues to an alkaline pH. In some clinical situations, endodontic reparative cements like calcium enriched mixture cement are used after calcium hydroxide therapy. However, the alkaline pH may affect the physical properties of this cement.

Objectives: This study was designed to evaluate the effect of alkaline pH on the push-out bond strength of calcium enriched mixture.

Materials and Methods: 80 root slices were prepared from single-rooted human teeth and their lumens were instrumented to achieve a diameter of 1.3mm. Calcium enriched mixture (CEM) was mixed according to the manufacturer's instruction and introduced into the lumens of root slices. The specimens were then randomly divided into 4 groups (n = 20) and wrapped in pieces of gauze soaked in synthetic tissue fluid (STF) buffered in potassium hydroxide at pH values of 7.4, 8.4, 9.4, or 10.4. The samples were incubated for 4 days at 37°C. The push-out bond strengths were then measured using a universal testing machine. Failure modes were examined under a light microscope at ×20 magnification. The data were analyzed using one-way analysis of variance and Tukey's post hoc tests.

Results: The greatest (1.41 ± 0.193 MPa) and lowest (0.8 ± 0.06 MPa) mean push-out bond strengths were observed after exposure to pH values of 7.4 and 8.4, respectively. There were significant differences between the neutral group and the groups with pH of 8.4 (p = 0.008) and 10.4 (p = 0.022). The bond failure was predominantly of cohesive type for all experimental groups.

Conclusions: Under the condition of this study, alkaline pH adversely affected the Push-out bond strength of CEM cement.

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Introduction

Root end filling materials are designed to prevent the egress of microorganisms and their byproducts into the periradicular tissue. Many materials have been used for closure of root end cavities-like gutta-percha, zinc oxide eugenol-based cements, composite resin, glass ionomer cement, gold foil, polycarboxylate cements, amalgam, and mineral trioxide aggregate (MTA) [1-3]. Unfortunately most of these materials have shown deficits in their biocompatibility, leakage, solubility, handling characteristics, moisture sensibility, and/or price [3,4].

Recently a new endodontic material has been introduced into the market named calcium enriched mixture (CEM). The calcium compounds of this material are different from other calcium containing cements like MTA. It consists of different calcium components like calcium oxide, calcium phosphate, calcium carbonate, calcium silicate, calcium sulphate, calcium hydroxide, and calcium chloride. CEM cement is hydrophilic cement which is set in an aqueous environment, and forms hydroxyapatite with its endogenous and exogenous ion sources. It also shows good handling characteristics and forms an effective seal as a root end filling material [5,6].

The pH value of the periapical tissue may change to acidic or alkaline in different situations. The pH of human abscess has been measured as low as 5.0 [7]. On the other hand, pre-treatment with calcium hydroxide causes the periradicular tissue to become alkaline [8].The increase or decrease of pH value of the periapical tissues may potentially affect the physical and chemical properties of the root end filling materials. It has been shown that tensile strength [9], surface hardness [10], sealing ability [11] and push-out bond strength [12] of MTA have been affected by acidic environment. Recent studies have shown the effect of acidic environment on the compressive strength and push out bond strength of CEM cement. Acidic environment also adversely affected the push-out bond strength of CEM [13] but not its compressive strength [14].

Regarding alkaline pH, it has been reported that pH of 10.4 caused a lower surface hardness, higher porosity and dehydrated structure of white MTA [15]. However, one study on the compressive strength of CEM cement in an alkaline pH showed that the lower pH (9.4 vs. 10.4) values significantly improve the strength of this cement [14].

To the best of our knowledge, there is no information regarding the effect of alkaline environment on the bond strength of CEM cement to the intra-radicular dentin. Therefore, this study was conducted to evaluate the push-out bond strength of CEM cement after an exposure to a range of alkaline pH.

Materials and Methods

Freshly extracted human teeth including single rooted mandibular premolars or maxillary incisors that were either intact or contained only small caries lesion were used in this study. Teeth with cracks or internal resorption were excluded from the study. After removing the crowns, the mid-root dentin was sectioned perpendicularly to the long axis of the root in order to achieve 80 dentin disks with the thickness of 1.5±0.2 mm. The lumens of the root slices were drilled with #2 to 5 Gates-Glidden burs (Dentsply, Maillefer, Ballaigues, Switzerland) to obtain 1.3-mm diameter standardized cavities. CEM cement (biunique dent, Tehran, Iran) was mixed following the manufacturer's instructions and placed inside the lumens of the root slices. The specimens were then randomly divided into 4 groups (n = 20). In group A, the root slices were wrapped in pieces of gauze soaked in synthetic tissue fluid (STF) that was prepared as follows: 1.7 g of KH_2PO_4, 11.8 g of Na_2HPO_4, 80.0 g of NaCl, and 2.0 g of KCl in 10 L of distilled water (pH, 7.4). In groups B, C, and D, the specimens were wrapped in pieces of gauze soaked in STF buffered in potassium hydroxide at pH values of 8.4, 9.4, and 10.4, respectively. Each group was placed in a separate container. Alkaline-soaked pieces of gauze were replaced every day with fresh ones to ensure a sufficient alkaline environment within the containers. The specimens were kept in an incubator for 4 days at 37° C.

Push-out Test

The push-out bond strengths were measured using universal testing machine (Zwick/Roell Z020 Zwick,

Table1: Mean push out bond strength values (MPa), standard deviation and mode of failures in all experimental groups

		Failure Mode		
pH	**Mean±SD**	**Adhesive**	**Cohesive**	**Mixed**
7.4	1.41±0.19	0%	70%	30%
8.4	0.8±0.06	4.76%	57%	33.3%
9.4	1.19±0.13	20%	60%	20%
10.4	0.87±0.10	9.09%	68%	22.7%

CombH & Co,Germany). The CEM cement was loaded with a 0.7-mm diameter cylindrical stainless steel plunger at a cross head speed of 1 mm/min. The maximum load applied to the CEM cement was recorded in newtons before the dislodgement occurred. To calculate the bond strength in MPa, the recorded value in newtons was divided by the area in mm^2 using the following formula: $2\pi r \times h$, where π is the constant 3.14, r is the root canal radius, and h is the thickness of the root slice in millimeters. The slices were then examined under the light microscope (Dinolit, Taiwan) at ×20 magnification to determine the mode of the bond failure. Each sample was evaluated for one of the three failure modes: adhesive failure that occurred at the CEM cement and dentin interface, cohesive failure that happened within CEM cement, and mixed failure mode (Figure 1). The data were analyzed by using one-way analysis of variance and Tukey's post hoc tests.

Results

The mean push-out bond strength ±standard deviations and mode of failures of experimental groups is shown in Table 1.

The greatest and lowest mean push-out bond strength values were observed after exposure to pH of 7.4 and 8.4, respectively. A significant difference was found between the experimental groups (p = 0.005).

Tukey's post hoc test showed significant differences between the pH value of 7.4 and both of 8.4 (p=0.008) and 10.4 (p = 0.022). Inspection of the specimens revealed that the bond failure was predominantly of cohesive type for all the experimental groups (Table 1).

Discussion

Various methods have been used for evaluating the adhesion of dental materials to the dentin, including shear, tensile and push-out bond strength tests. The push-out test has been shown to be efficient and reliable [16].

In some clinical situations, CEM cement might be exposed to an alkaline environment. The pH of the pulp, dentin, cementum and periodontal ligament of the vital or necrotic teeth with complete or incomplete root formation varies between 6.4 - 7 [17]. In the presence of calcium hydroxide as an intra-canal medicament, the pH of the dentin is increased up to 12.2 [17]. In the present study, the influence of alkaline pH on the push-out strength of CEM cement was evaluated for the first

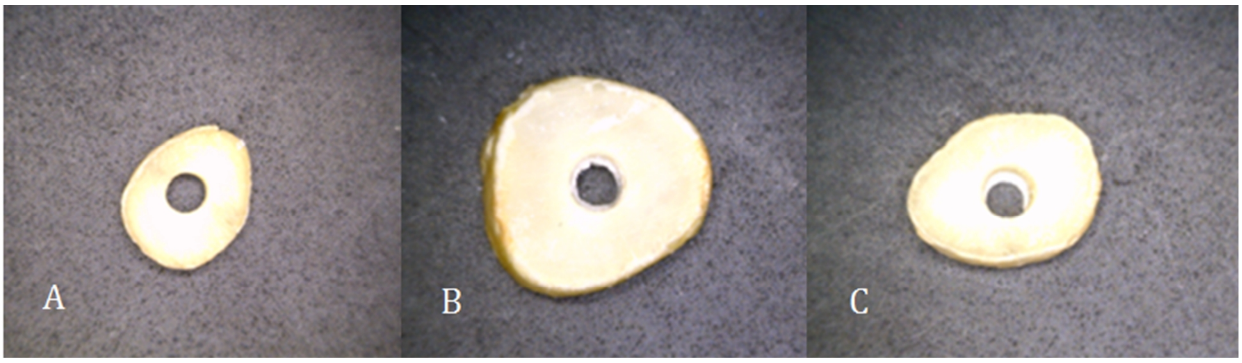

Figure 1: Various failure modes; A) Adhesive failure; note the clean canal wall, B) Cohesive failure within CEM, C) Mixed failure; note the CEM residual inside the canal

time. Based on the results of the present study, pH values of 8.4 and 10.4 had an adverse effect on the bond strength between CEM and the dentin. The alkaline pH of 9.4 also decreased the bond strength, although its difference with neutral pH was not significant. It seems that push-out strength of CEM is very pH sensitive as it decreased when pH raised from 7.4 to 8.4 and then increased in the pH of 9.4 and again decreased in pH of 10.4. The study on the push-out bond strength of MTA in the presence of alkaline pH also showed pH sensitivity, as the bond strength increased in pH of 8.4 and then decreased in pH values of 9.4 and 10.4 [18]. Therefore in the clinical situations in which the pH value of the circumpulpal dentin may vary between 8-12.2 after calcium hydroxide therapy [8], it is hard to anticipate the exact effect of alkaline pH on the bond of CEM and MTA to the dentin. The push-out bond strength of MTA in the presence of alkaline pH showed that the greatest and lowest mean push-out bond strengths were observed after the exposure to pH values of 8.4 and 10.4, respectively [18]. This is different from the present study as the lowest bond strength was observed in the pH of 8.4; somehow similar to our study, the pH value of 10.4 adversely affected the push out bond strength of CEM cement. In other words; both MTA and CEM were adversely affected when exposed to higher alkaline pH values. Xu *et al.* found that an alkaline accelerator influences the early hydration of Portland cement. In addition they revealed a breakdown in calcium silicate gel during the hydration of Portland cement in the presence of high concentration of NaOH [19]. Similar mechanisms may affect the hydration of CEM cement in the alkaline environment.

On the other hand, a study on the effect of different pH values on the compressive strength of CEM cement concluded that the alkaline environment significantly increased the compressive strength of CEM cement [14].These conflicting results can be attributed to the fact that push-out test and compressive strength are of different natures. In addition, in the push-out test, the thickness of the material subjected to the test is about 1.5 mm but in the compressive strength test, this thickness is about 6 mm. Moreover, in the experimental model used in the present study, both dentin and CEM cement were exposed to different alkaline pH values. Therefore; not only the bond between CEM and dentin, but also the hydration and setting of CEM cement may have been influenced by alkaline pH. Furthermore, in the push-out test, the thickness of the material that has been subjected to the test is about 1.5 mm but in the compressive strength test, this thickness is about 6 mm. So, the difference in the results of these two studies could also be attributed to the difference in the thickness of CEM cement.

A study on the push-out bond strength of CEM cement in the presence of acidic environment reported a decrease in the higher acidic environment [13]. The present study also showed a decrease in the push-out bond strength of CEM cement in the presence of alkaline pH. Thus it seems that the optimum bond strength for CEM cement would be expected in the neutral pH.

In this study, the bond failure observed in all the experimental groups was mostly of cohesive type although some samples showed mixed or adhesive bond failure. Shokouhinejad *et al*. [20] and Sobhnamayan *et al*. [13] also reported the same results. On the other hand, Rahimi *et al*. in a study on the effect of blood contamination on the retention characteristics of MTA and CEM cement showed that the bond failure of both CEM cement and MTA was predominantly of the mixed type; this finding is not in agreement with the results of the present study [21].

Conclusions

Regarding the limitation of this *in vitro* study, it was concluded that push-out strength of CEM is very pH sensitive in the presence of alkaline pH. While pH of 9.4 did not affect the bond strength, adverse effects were observed in the pH values of 8.4 and 10.4. Therefore, the application of CEM cement after calcium hydroxide therapy is not recommended.

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