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In vitro assessment of push-out bond strength of cold ceramic and mineral trioxide aggregate to root dentin

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ABSTRACT

Background: To provide a continuous seal of the root canal, root-filling material should bond to the root canal dentin, ensuring the integrity of both the root-filling material and dentin remains in a static and functional state. The present study assessed the push-out bond strength of mineral trioxide aggregate (MTA) and cold ceramic (CC).

Materials and Methods: In this laboratory trial study, 20 single-rooted, extracted human teeth without caries and cracks were selected. Each tooth was mounted in cold-curing resin. Then, 3 mm slices of mid-root dentin were obtained from each tooth. The specimens were randomly divided into two groups (n = 10) and filled with MTA ProRoot and CC. All specimens were stored for 30 days in an incubator at 37°C and 100% humidity. The push-out bond strength of the test materials was measured using a cylindrical punch with a 1-mm diameter. The punch was pushed against the test specimen at a speed of 1.0 mm/min using a universal material testing machine, extruding the filling test material. The push-out force during the test was recorded, and then, the internal surface of the teeth was examined to evaluate the mode of failure. Independent t-test and Chi-square were used to analyze the data. $P < 0.05$ was considered a significance threshold.

Results: The mean push-out bond strength in the CC group was 24.58 (MPa), and in MTA ProRoot, it was 23.77. No significant difference was observed between the two groups. The most frequent mode of failure in both groups was adhesive failure.

Conclusion: The two materials have adequate push-out bond strength to root dentin, and there is no difference between the bond strength and mode of failure of the two materials.

Key Words: Dental pulp cavity, mineral trioxide aggregate, root canal filling materials

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INTRODUCTION

The main purpose of using end-root filling material is to achieve an apical seal and prevent the spread of infections within the root canal to the surrounding tissues.[1] This is accomplished by instrumenting the canals, using antimicrobials, and filling the canal voids completely.[2] Characteristics of an ideal material for filling the end of the root include the following: prevention of leakage of bacteria or their products into the periapical space, nontoxic, noncarcinogenic, compatible with host tissues, insoluble in peri-apical tissue, dimensionally stable, unaffected by moisture during setting, easy to use, radiopaque, no discoloration in periapical tissues, and inducing cementogenesis.[3,4]

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The quality of the apical seal largely depends on the properties of the root-filling material.[5] Ideally, a root-end filling material should provide a suitable apical seal and attach to the dentinal walls of the canal.[6] To provide continuous sealing, a root-filling material must be well-attached to the dentin of the root canal so that the integrity of the root-filling material and dentin material remains intact not only under static conditions but also during the healing process.[7,8]

Materials used in the past to fill the root end include gutta-percha, amalgam, super-EBA, zinc oxide eugenol cement, zinc phosphate cement, carboxylate cement, glass ionomer, composite resin, gold foil, and mineral trioxide aggregate (MTA).[9-11] MTA is formulated from commercial Portland cement, combined with bismuth oxide powder for radiopacity.[12] MTA is composed of a hydrophilic powder that hardens in the presence of moisture. When this cement is mixed with water, the resulting colloidal gel achieves its initial hardness within 4 h.[13] The formation of calcium hydroxide, in addition to coagulation necrosis and dystrophic calcification that occurs after subcutaneous placement of MTA, also justifies its high pH. MTA is a biologically active substance for bone cells and stimulates interleukin production due to its alkaline pH and the release of calcium ions and interleukin production.[14] MTA induces less inflammation and better healing than other common filling materials and is capable of regenerating the periodontal ligament (PDL) complex, especially cement, and depositing new cementum on its surface.[15]

Cold ceramic (CC) is an MTA-like root-end filling material based on calcium hydroxide. CC is in the form of powder and liquid, which is used after mixing.[16] One of the characteristics of the chemical composition of CC is its high heat tolerance, which can be resterilized by dry heat. CC obviously causes a better seal than calcium hydroxide because calcium hydroxide becomes pasty in a humid environment and does not set and is washed away by tissue fluids. The initial set time is about 15 min, and after 24 h, it is completely set.[17] An *in vitro* study has shown that it produces a better seal than amalgam. Other studies comparing the tissue reaction to MTA and CC have shown that both materials are well-tolerated.[18] Evaluation of CC showed better results regarding tissue reaction compared to amalgam and was similar to MTA.[18,19]

Ideally, the root-filling material should form a strong bond with the channel wall and resist displacement during operation. The strength of the bond to root dentin depends, in part, on the type of material used. The push-out test is widely employed to evaluate the bond strength of endodontic material and postfilling material to dentin. In this study, our aim was to evaluate the push-out bond strength in CC and compare it with ProRoot MTA.

MATERIALS AND METHODS

This is a laboratory trial study that was performed on 20 single-canal human teeth. The study protocol was approved by the Research Committees of Shahid Sadoughi University of Medical Sciences and the Ethics Committee has confirmed it (Ethics code: IR.SSU.REC.1397.171).

Twenty human single-channel central teeth with caries-free roots were selected and stored in sterile saline until use. The teeth were mounted in self-hardening acrylic (Acropars, Iran). Each tooth was cut perpendicular to the longitudinal axis using a three-axis fully automatic cutting machine (Nemo Iran Company), and cuts with a height of 3 mm were selected from the middle part of the root. Subsequently, the channel space of each tooth was drilled using Premier Gates-Glidden Dental Drills with a diameter of 1.2 mm.

After preparing the canal space, the diameter inside each tooth was remeasured and recorded with a caliper to obtain the diameter. The teeth were then immersed in a 17% ethylenediaminetetraacetic acid (EDTA) solution (Denton, USA) for 60 s, followed by 5.25% hypochlorite, and then rinsed with distilled water. The teeth were divided into two groups: ProRoot MTA (Dentsply, Tulsa, USA) and CCs.

The MTA was combined with the associated liquid (normal saline) according to the manufacturer's instructions, and the CC was combined with distilled water. Ten samples of roots were filled with ProRoot MTA, and ten other samples with CCs using a 1-mm diameter plug. When placing the material, the teeth were positioned on a glass slab, and material additions were removed with a plastic spatula. The teeth were then incubated in an incubator (NEMO, Iran) for 30 days at 100°C and 37°C. The 30-day interval in this study is clinically relevant, as it corresponds to a critical posttreatment period, allowing for the assessment of the push-out bond strength of MTA and CC after an adequate time for setting and potential changes in properties.

An aluminum metal plunger with a diameter of 1 mm and an aluminum cylinder with an approximate diameter of 1 mm was utilized to measure the push-out bond strength. Tooth parts were clamped and mounted on a universal mechanical testing machine (BONGSHIN, Korea). The central metal cylinder within the channel was calibrated to accommodate the material once it had passed through the canal. The punch was attached to a universal mechanical testing machine and applied to the material at a speed of 1 mm/min until the material came out of the tooth. The amount of force recorded and the pressure required to remove the material were calculated by determining the numerical values of force and surface using the device.

Simultaneously, the inner surface of the tooth and the extracted material were evaluated with a light microscope with ×40 magnification (Dino-Lite, Taiwan). Subsequently, a narrow groove was created around each tooth specimen, and the tooth was gently broken into two parts. The inner surface of the tooth was re-evaluated under a microscope and categorized into one of the following four groups based on the type of fracture:

1. Cohesive failure in material occurred when remnants of material were observed in the dentin walls or the surface of the extracted material was significantly worn
2. Cohesive fracture in dentin occurred when the inner surface of the tooth became wider and more irregular or dentin debris was seen on the outer surface of the test material
3. Adhesive failure was noted between the material and the dentin when no material remained on the dentin wall, and the surface of the dentin and the test material removed had not changed
4. If a combination of the above conditions occurred, the failure was categorized as the combined type.

The types of failures are indicated in Figure 1.

The obtained data were entered into the Statistical Package for Social Sciences (SPSS Inc., Chicago, IL, USA) version 24. Quantitative data were reported as mean ± standard deviation and qualitative data as frequency distribution (percentage). Independent *t*-test and Chi-square were used to analyze the data. $P < 0.05$ was considered the significance threshold.

RESULTS

The investigation and determination of push-out bond strength were evaluated according to the two groups studied in this research (CC and ProRoot MTA). Based on our data, there were no significant differences between the two groups regarding push-out bond strength ($P = 0.822$). The values are indicated in Table 1 and Figure 2.

We also assessed the types of failure between the groups. Cohesive failure occurred in only one of the samples in the CC group. Mixed failures in samples were a combination of adhesive and cohesive failures. In all samples with mixed failure, there was cohesive failure in the test material, and in none of the samples did failure occur in the dentin. Based on these data, 30% of samples in both CC and MTA groups had mixed failure. Furthermore, 60% of samples in the

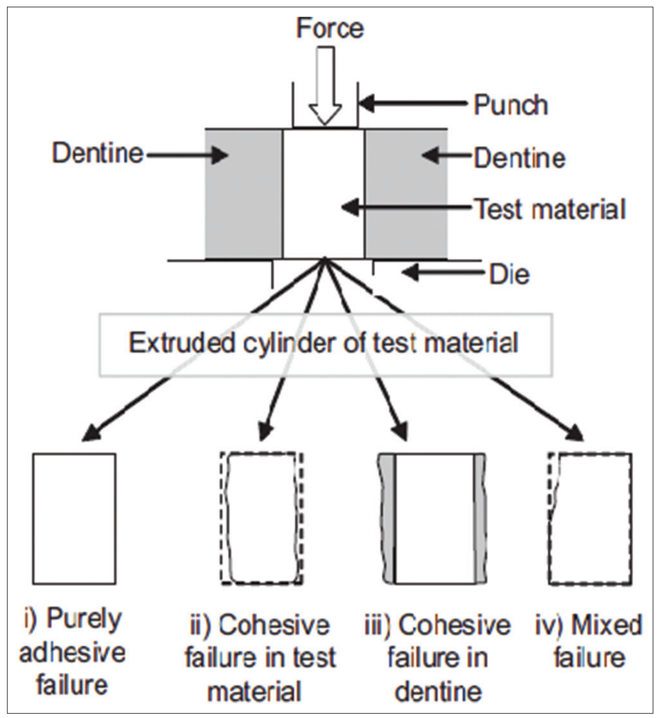

Figure 1: Different types of failure in this study.

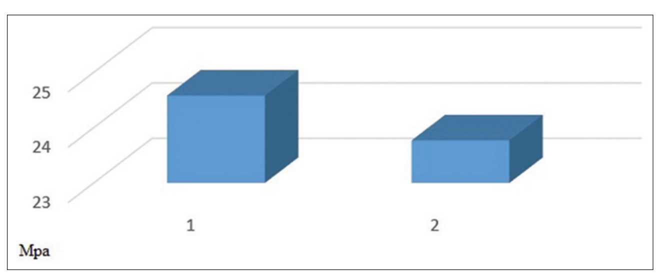

Figure 2: Different values of push-out bond strength between cases.

CC and 70% of samples in MTA groups had adhesive failure. There were also no significant differences between groups regarding the failure type [$P = 0.530$, Table 2].

DISCUSSION

The presence of a stable seal inside the root canal to prevent the penetration of irritants is a prerequisite for the success of endodontic treatments.[20] Microbes and their products have been shown to gradually penetrate along the canal filling if the root canal filling is incomplete and the canal is exposed to the oral environment, leading to treatment failure. Ideally, a retrofilling material should provide a suitable apical seal and attach to the dentinal walls of the canal.[21] In addition, the substance must have the ability to induce the regeneration of periapical tissue.[22]

MTA is a calcium-based silicate cement that causes less inflammation and promotes better healing than other common filling materials. It has the ability to regenerate the PDL complex, especially cement, and facilitates the deposition of new cementum on its surface.[23] Studies have demonstrated that the push-out test is a reliable and effective method for measuring the bond strength to dentin. Moreover, in cases involving composite resin, the push-out test has been deemed superior to the microtensile test due to premature failures occurring in the microtensile test.[24] For this reason, the push-out method was employed to measure the bond strength in this study.

In most studies, the cavity size ranged between 1 and 2 mm to ensure an adequate volume of material, considering the thickness of the surrounding dentin and standardization.[25] In this study, we prepared a sample cavity with a diameter of 1.2 mm. Hachmeister *et al*. demonstrated that increasing the thickness of the MTA plug and thereby enhancing the contact surface between MTA and dentin significantly increases displacement resistance.[26] Sections with a height of 3 mm were used in this study because the minimum thickness required for plugs in teeth to establish sufficient seal and resistance to displacement is 3 mm.[27]

Table 1: Comparison of push-out bond strength between groups

Groups	Mean±SD (MPa)	Minimum (MPa)	Maximum (MPa)	*P*
CC	24.58±10.57	9.28	46.39	0.822
MTA ProRoot	23.77±9.39	9.28	38	

CC: Cold ceramic; MTA: Mineral trioxide aggregate; SD: Standard deviation

Table 2: Comparison of different types of failure in groups

Group	Adhesive	Cohesive	Mixed	Total
CC, *n* (%)	6 (60)	1 (10)	3 (30)	10 (100)
MTA ProRoot, *n* (%)	7 (70)	0	3 (30)	10 (100)

CC: Cold ceramic; MTA: Mineral trioxide aggregate

In the assessment of failure types between groups, cohesive failure was observed in only one sample in the CC group, while mixed failures, combining adhesive and cohesive failures, were present in both CC and MTA groups. Specifically, in all samples with mixed failure, cohesive failure occurred in the test material, and no failure was observed in the dentin. Notably, 30% of samples in both CC and MTA groups exhibited mixed failure, and the majority of samples (60% in CC and 70% in MTA) showed adhesive failure. Importantly, no significant differences were found between the groups in terms of failure types.

The retention of retrofill material on the dentin wall surface and the physical properties of these materials depend on factors such as the water/powder ratio, temperature, humidity, the amount of air in the mixture, and the particle size of the material.[28] A characteristic of calcium silicate-based materials is the deposition of carbonate apatite in the presence of tissue fluids, followed by the formation of an intermediate layer and tag-like structures in dentin. A study by do Carmo *et al*.[29] found that the maturity of the intermediate layer and the bond strength in *in vitro* studies depend on the storage environment. The composition and morphology of hydroxyapatite crystals formed when materials are set depend on various factors, including ambient pH.[30-34]

The 2012 study by Formosa *et al*. utilized a 28-day storage period,[34] and the 2013 study by El-Ma'aita *et al*. employed a 7-day storage period to maximize material setting.[35] Sarkar *et al*. demonstrated that in the presence of moisture, the tensile strength between dentin and MTA increased significantly in 3 days and increased moderately in 21 days.[36] The retention time of the samples in our study was 30 days. This 30-day interval is clinically relevant as it corresponds to a critical posttreatment period, allowing for the assessment of the push-out bond strength of MTA and CC after an adequate time for setting and potential changes in properties. This duration underscores the importance of evaluating the long-term performance of root-filling materials, providing valuable insights into their stability and effectiveness in maintaining the seal of root canals. While our study duration is significant, more long-term studies have been proposed to evaluate the effect of aging on MTA bond strength. Therefore, the study time is crucial in evaluating the bond strength of materials. Research on the physical and chemical interaction between MTA and root canal walls has shown that MTA is a bioactive substance and appears to be chemically reactive to dentin through a controlled diffusion reaction between apatite on its surface and dentin, forming an intermediate layer. In addition, the chemical adhesion of MTA to dentin may be related to its superior sealing ability compared to conventional filling materials.

However, a 2006 study by Yan *et al*.[37] on the effect of irrigants on bond strength showed that, although the intensity of MTA-dentin bonding showed a tendency to decrease in the wash group with 5.25% NaOCl, the comparison with the normal saline group was not significantly different ($P < 0.05$). It was observed that the microstructure formed on the intermediate layer of the dentin wall in the NaOCl group was similar to the saline group, likely due to the high pH of 5.25% NaOCl. In this study, after rinsing with a 17% EDTA solution, the samples were washed with 5.25% sodium hypochlorite and then sterile saline to remove the smear layer.

A study has shown that the superior performance of ProRoot MTA is associated with the production of the highest amount of sediment and that effectively forms an intermediate layer with tag-like structures.[38] In a study by El-Ma'aita *et al*. in 2013,[35] which examined the effect of smear layer on the bond strength of filling materials using ProRoot MTA, they reported lower bond strength values from our study. In a 2010 study by Shokouhinejad *et al*., which examined the effect of an acidic environment on the strength of MTA ProRoot, the average bond strength was lower than in our study, which could indicate the effect of pH on bond strength.[33] On the other hand, the storage time of the samples in this study was 4 days and as mentioned, the study time can affect the bond strength of the material. In this study, as in our study, the most type of material failure was adhesive.

In our study, no difference was found between the two groups in terms of push-out bond strength and failure type, indicating that both materials demonstrated sufficient strength against displacement. We recommend that similar studies evaluating the apical microleakage of CC and MTA be conducted with longer durations and alternative methods for assessing apical microleakage.

CONCLUSION

The two materials exhibit sufficient push-out bond strength to root dentin, and there is no significant difference in bond strength or mode of failure between the two materials. The adequacy of bond strength is determined by the material's capability to establish a resilient and durable connection with the root dentin, ensuring mechanical stability and long-term performance under functional loads in clinical scenarios such as root canal treatments. Based on the findings of our study, these two root-filling materials could both be interchangeably utilized in endodontic treatments.

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Author contribution

- FM contributed in the conception of the work, conducting the study, revising the draft, approval of the final version of the manuscript, and agreed for all aspects of the work
- LAK contributed in the analysis, or interpretation of data for the work, drafting the work or revising it critically for important intellectual content, and final approval of the version to be published
- JM contributed in drafting the work or revising it critically for important intellectual content, final approval of the version to be published, agreement to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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