Comparative evaluation of push-out bond strength of three retrograde filling materials in teeth with root apices resected at 90°: Mineral trioxide aggregate Angelus, Zirconomer, and Bioactive bone cement

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Abstract
Background: Surgical endodontic dissatisfaction is frequently caused by an inadequate seal at the apex. The retrograde filling material utilized should prevent any contaminants from seeping into the periradicular tissue.

Aim: (1) To evaluate push-out bond strength of mineral trioxide aggregate (MTA) Angelus, Zirconomer, and Bioactive bone cement as root-end filling material, (2) To compare push-out bond strength of three different retrograde filling materials with a universal testing machine.

Materials and Methods: At the cementoenamel junction, 36 removed upper anterior teeth were sliced horizontally. The samples were resected 3 mm at the apical end at a 90° angle along the long axis of the tooth, following the standard protocols. With an ultrasonic tip S12 90ND, a 3 mm root-end cavity was made. The teeth were then divided into three groups at random: Group 1: MTA Angelus, Group 2: Zirconomer, and Group 3: Bioactive bone cement. With the help of small pluggers, each material was compacted in the root-end cavity. Acrylic resin was used to embed the specimens. Each specimen's apical section was sliced into 2-mm thick slices perpendicular to the long axis. A universal testing equipment was used to apply a compressive push-out load, and the push-out bond strength was determined in megapascals.

Results: Zirconomer showed higher push-out bond strength when placed in the retrograde cavity preparation, followed by MTA Angelus and then Bioactive bone cement.

Conclusion: Zirconomer showed superior resistance to dislodgment when compared to MTA Angelus, followed by Bioactive bone cement.

Keywords: Periapical infection; push-out bond strength; retrograde filling

INTRODUCTION

Bacterial infection is the most common source of apical periodontitis, and the microbes in the infested area are a significant determinant in deciding whether the apical lesion heals or persists. Apical surgery is advised when traditional root canal treatment fails. This technique necessitates resection of the root end, which removes the infected area of the root where microbes that are resistant to canal disinfection are present in the form of a biofilm.[1]

Root-end resection is a crucial part of endodontic surgery because it helps to remove “anatomical variations, resorptive flaws, perforation defects, ledges, canal obstructions, and detached instruments from the root.”

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Date of submission : 08.05.2022
Review completed : 12.06.2022
Date of acceptance : 01.07.2022
Published : 17.08.2022

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How to cite this article: Chaudhari PS, Chandak MG, Jaiswal AA, Ikhar A. Comparative evaluation of push-out bond strength of three retrograde filling materials in teeth with root apices resected at 90°: Mineral trioxide aggregate Angelus, Zirconomer, and Bioactive bone cement. J Conserv Dent 2022;25:605-9.
Resecting the apical 3 mm of the root has been shown to eradicate “98% of apical ramifications and 93% of lateral canals” that contributes to periapical diseases.[3] Owing to the limited access, retrograde cavities made with burs are frequently not deep enough and ill-aligned along the long axis of the tooth. Inadequate root-end cavity preparation leads to inadequate root-end filling, and as a result, a poor prognosis.

Unfortunately, conventional retrograde materials such as “amalgam, composite resins, glass-ionomer cements (GICs), and Cavit” – have varying degrees of “biocompatibility, sealing ability, and moisture resistance.”[3]

As a result, “mineral trioxide aggregate (MTA)” was introduced in 1998 to address these deficiencies. MTA is a hydrophilic powder mostly made up of portland cement which hardens when exposed to moisture and generates “calcium (Ca) hydroxide and Ca silicate hydrate gel.” MTA has a strong marginal adaptation[4] and its retentive quality improves with time. Newer formulations of GIC, which are original materials, have been introduced, such as Zirconomer and Zirconomer Improved, which contained ceramic and zirconia reinforced GIC which were able to address the problems of amalgam and tooth-colored restorative materials. Polymethyl methacrylate (PMMA) bone cement, a PMMA polymer, is a newly introduced material that has been widely employed in orthopedic surgery, mostly for prosthesis fixation and also for stabilizing compressive vertebral fractures or filling bone defects. Despite its well-known polymerization shrinkage feature, which causes it to expand in volume during polymerization before contracting slightly, this cement exhibits exceptional adaptation to cavity margins.[5]

To withstand dislodging forces, an ideal retrograde material should be able to adhere to the dentin walls of the root-end preparation. Several studies have found that during the mastication process, these dislodging forces increase.[6] Material qualities and the surface of root-end preparation strongly affect the bond strength of retrograde filling material. The filling material – dentin interface must be kept intact in static situations and also during function and surgical procedures.[7] Therefore, the aim of the present research was the evaluation of “push-out bond strength” of MTA Angelus, Zirconomer, and Bioactive bone cement after ultrasonic retrograde cavity preparation.

**MATERIALS AND METHODS**

For this investigation, 36 anterior teeth were chosen for sample preparation. Crowns were eliminated, and the length of the teeth was standardized to 16 mm (from root apex to coronal reference point). Instrumentation of the canals was done with ProTaper Universal instruments until size F3 using an electric endo motor. After a change of each file, irrigation of root canals with 2 ml of 1% NaOCl was carried out during instrumentation. For smear layer removal, root canals were dried and filled with 17% ethylenediaminetetraacetic acid (pH 7.2) for 3 min. The canals were flushed and shaped, then dried with paper points before being obturated with gutta-percha employing the single-cone technique and AH-Plus sealer. MD-Temp white was used to seal the access cavities. The roots were then kept for 1 week at room temperature with 100% humidity. In all three groups, root resection was performed by removing 3 mm with a diamond disc under continual irrigation with normal saline solution at a 90° angle to the tooth’s long axis. With a “diamond-coated ultrasonic surgical tip S12 90ND,” a 3 mm deep root-end cavity was made. A periodontal probe was used to measure the depth of the root-end cavity.

The specimens were allocated into three groups (n = 12) at random.

- **Group I:** The retrocavities were filled with MTA Angelus (Angelus, Londrina, Brazil)
- **Group II:** The retrocavities were filled with Zirconomer (Conventional GIC, SHOFU, Japan)
- **Group III:** The retrocavities were filled with Bioactive bone cement (Surgical Simplex P, Stryker)

**Mineral trioxide aggregate Angelus**

MTA powder was blended in a 3:1 powder liquid ratio with liquid supplied by the manufacturer to form a thick putty-like consistency. To fill the retro voids, MTA was transferred through an MTA carrier.

**Zirconomer**

Zirconomer (Conventional GIC, SHOFU, Japan) was manually mixed using a glass slab and a plastic spatula at a specified powder-to-liquid ratio of 2:1.

**Bioactive bone cement**

**Preparation of Bioactive bone cement**

The optimal concentration of MTA and silane coupling agent required for changing bone cement without affecting its handling qualities are determined in preliminary studies.[8]

**Powder modification**

A mass ratio of 0.4 mg MTA was combined with 0.6 mg bone cement (60:40) until all of the MTA particles were miscible in the polymer powder.

**Liquid modification**

One drop of silane coupling agent (Monobond-S) was added to 1 mL of monomer liquid and stirred together. Under ambient circumstances at room temperature, the
produced powder and liquid of the modified bone cement were mixed in a 2:1 ratio.

Using small pluggers, each material was condensed into the designated cavity. Until the push-out test, all specimens were stored in moist gauze saturated in normal saline for 7 days at 37°C in an incubator.

**Push-out test**

Acrylic resin was used to embed the specimens. With a diamond blade at a speed of 150–200 rpm, the apical section of each specimen was cut perpendicular to the long axis into slices of 2 mm.

The conical frustum formula was used to compute the bonding surface:

\[ \text{Area} = \pi \left( R_1 + R_2 \right) \sqrt{\left( R_1 - R_2 \right)^2 + h^2} \]

With apical radius \( R_1 \) – larger radius, coronal \( R_2 \) – smaller radius, and \( h \) – thickness of a slice.

A universal testing equipment was used to apply a compressive push-out load. By dividing the load at failure by the bonding surface, the push-out bond strength was estimated in megapascals.

**Statistical analysis**

For each study group, the mean and standard deviation were calculated from the samples. The one-way analysis of variance (ANOVA)/least significant difference test was used to compare mean values. The tested groups were analyzed using IBM SPSS statistical software version 18 (IBM. SPSS Inc. USA).

**RESULTS**

The overall assessment of push-out bond strength at the dentin – retrograde filling material interface revealed that the Zirconomer group had the strongest bond, followed by MTA Angelus, and then Bioactive bone cement.

The ANOVA results revealed that the type of retrograde filling material had a highly significant influence \((P < 0.001)\) on the push-out bond strength of Zirconomer subgroups when compared to Bioactive bone cement and MTA Angelus, and the statistical analysis of data revealed that the push-out bond strength of Zirconomer subgroups was highly significant when compared to Bioactive bone cement and MTA Angelus \([P < 0.001]; \) Table 1].

**DISCUSSION**

Adhesiveness to intraradicular dentin is of paramount importance among the physicochemical properties owing to the necessity of the retrograde filling material to be in close proximity with well adaptation to the dentin walls when mechanical load is applied to teeth in function or operative and surgical techniques.

The type of material utilized, the burs used for cavity preparation, and the pH value of the environment are the factors which play a prime role in adhesion of retrograde filling.

Because there is a paucity of information on the bond strength of both Zirconomer and Bioactive bone cement, this is the first study to compare the push-out bond strength of these three materials: MTA Angelus, Zirconomer, and Bioactive bone cement.

When compared to Zirconomer, the results of this study revealed that MTA Angelus has a lesser push-out bond strength. In Zirconomer, the glass component is treated to finely regulated micronization to obtain ideal homogeneous particle size, resulting in improved mechanical properties such as increased strength. The homogeneity of the glass particles adds to the material’s durability and strength to sustain the occlusal load. When hydroxyapatite (HA)/ZrO\(_2\) particles were incorporated into the GIC matrix, there was a homogeneous distribution of these particles in the matrix, and mechanical properties were found to perform superior to HA GICs. It’s an excellent retrograde filling material due to its superior strength and endurance, as well as chemical bonding. However, more in vivo research is required to back up our preliminary findings. Aggarwal et al. found a substantial difference in bond strength between the tested groups, with MTA having the lowest bond strength. They attributed this to MTA’s extended maturation phase, which they linked to the creation of a passivating trisulfate coating over hydrated crystals, simulating a terrible clinical condition.

When Bioactive bone cement and Zirconomer were compared in terms of push-out bond strength, it was observed that Bioactive bone cement was less resistant to dislodgment forces than Zirconomer. However, no previous research has looked at the strength of these two materials’ push-out bonds.

| Group A: Bioactive bone cement | Group B: MTA Angelus | Group C: Zirconomer |
|-------------------------------|---------------------|---------------------|
| Mean                          | 9.32                | 10.28               | 14.20               |
| SD                            | 0.55                | 0.66                | 0.77                |
| SE                            | 0.15                | 0.19                | 0.22                |
| Minimum                       | 8.55                | 8.99                | 13.05               |
| Maximum                       | 9.94                | 10.98               | 15.45               |

MTA: Mineral trioxide aggregate, SE: Standard error, SD: Standard deviation
MTA angelus specimens had a higher push-out bond strength when compared to Bioactive bone cement in this study, but the difference was not significant. MTA has been reported to be unable to chemically adhere to root dentin. MTA releases calcium (Ca) and hydroxyl ions, which combine with phosphate in a bodily fluid to produce interfacial deposits. The spaces between the MTA and root dentin are obliterated as a result of these deposits, increasing MTA frictional resistance. Its push-out bond strength may have improved as a result of this. Ca ions’ bioavailability inside these materials stimulates the production of bone-associated proteins through Ca channels and large amounts of Ca ions could activate adenosine triphosphate, which is important in the mineralization process. The current findings are in line with those of Rashid et al. who discovered 10.60 MPa for MTA Angelus after a push-out test utilizing apical portions of human tooth roots. The use of ultrasonic retrotips has revolutionized root-end preparation. The use of an ultrasonic device has enhanced surgical techniques by allowing easier access to the root-end cavity. In addition, because the retrotip’s design does not provide a beveled cavity outline, the exposed dentinal tubules are reduced, apical leakage is reduced, and debris is reduced, allowing for more close contact of the material with the walls of the cavity and improved bond strength.

After retrograde filling with MTA Angelus, Zirconomer, or Bioactive bone cement, the samples were left in 100% humidity at 37°C for 1 week. When compared to those studied after 1 day of storage, the bond strength values between root dentin and MTA increased significantly after being allowed to set for 7 days. Furthermore, Nikhade et al. study found that increasing incubation times enhanced bond strength for Ca silicate-based composites.

In our research, Bioactive bone cement had a bond strength that was comparable to MTA, the gold standard material for retrograde filling. The bone cement is low in cytotoxicity, has a quick 15-min setting time, and its characteristics are unaffected by moisture or blood contamination. The creation of a biologically active bone-like apatite layer on an artificial material’s surface in the bodily environment is a must for it to display bioactivity. Apatite development can be driven by the release of Ca ions from the modified bone cement into the bodily fluid, and a catalytic effect of Si-OH groups generated on the surface of the material, according to Miyazaki et al. “The addition of MTA powder to bone cement gives Ca ions, whereas the addition of silane to the liquid component provides Si-OH group owing to the hydrolysis of alkoxysilane after exposure to the body environment, resulting in heterogeneous HA nucleation.” Because chemical bonding can be produced with PMMA, adding silane to bone cement increases its mechanical characteristics. In addition, the modest expansion of MTA during setting compensates for any PMMA polymerization shrinkage, resulting in increased sealing and strength of modified bone cement. This could explain why Bioactive bone cement and MTA Angelus have comparable bond strengths.

When compared to MTA Angelus and Bioactive bone cement, Zirconomer showed higher retention. Because Zirconomer has a faster setting time, it would be advantageous because it would allow for the quick and safe insertion of the final material in the retrograde cavity, as well as a shorter period during which the cement could be washed out. Although this material requires more research into its other physical qualities for making it suitable as retrograde filling material.

### CONCLUSION

In comparison to MTA Angelus and Bioactive bone cement, Zirconomer showed the highest push-out bond strength within the study’s limitations. Push-out bond strength was comparable between MTA Angelus and Bioactive bone cement groups. As a result, Bioactive bone cement might be contemplated as a potential retrograde filling material.

### Financial support and sponsorship
Nil.

### Conflicts of interest
There are no conflicts of interest.

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