

# Push-out bond strength and intratubular biomineralization of a newly developed hydraulic root-end filling material premixed with dimethyl sulfoxide as a vehicle

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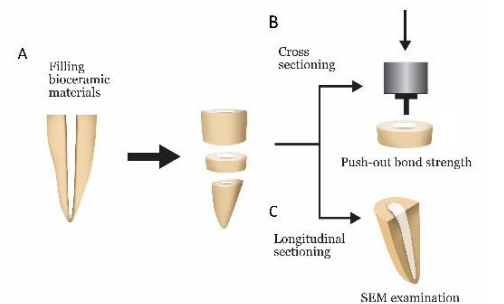
## INTRODUCTION

Hydraulic bioceramic cements have been used successfully in root-end fillings due to their favorable sealing ability. Despite its favorable sealing ability, hydraulic bioceramic material presents less-than-ideal working properties, since the cement resulting from the mixture of powder with liquid is difficult to handle. As a result, a method of mixing the powder with a non-aqueous vehicle to produce an injectable, ready-to-use form was developed. This process results in greater plasticity and higher flow, as well as improved handling characteristics compared to conventional bioceramic materials. However, little information exists regarding the effect of the vehicle on the bonding of the material to dentin.

Recently, an injectable hydraulic bioceramic cement premixed with dimethyl sulfoxide (DMSO) was developed (Endocem MTA Premixed; Maruchi, Wonju, Korea). In the present study, we aimed to evaluate the bonding ability to root canal dentin of the DMSO-containing root-end filling material in comparison to a widely used conventional powder-liquid cement (ProRoot MTA). To evaluate the bonding, we investigated push-out bond strength and biomineralization in the dentinal tubules.

## MATERIALS & METHODS

The root canal of a single-rooted premolar was filled with either ProRoot MTA or Endocem MTA Premixed ( $n = 15$ ). A slice of dentin (2 mm) was obtained from each root. Using the sliced specimen, the push-out bond strength was measured, and the failure pattern was observed under a stereomicroscope. The apical segment was divided into halves; the split surface was observed under a SEM, and intratubular biomineralization was examined by observing the precipitates formed in the dentinal tubule. Then, the chemical characteristics of the precipitates were evaluated with EDS analysis. The data were analyzed using the Student  $t$ -test followed by the Mann-Whitney  $U$  test ( $p < 0.05$ ).



$$\text{Push-out bond strength (MPa)} = \text{maximum load (N)} / \text{adhesion area of root filling (A)} \text{ (mm}^2\text{)}$$

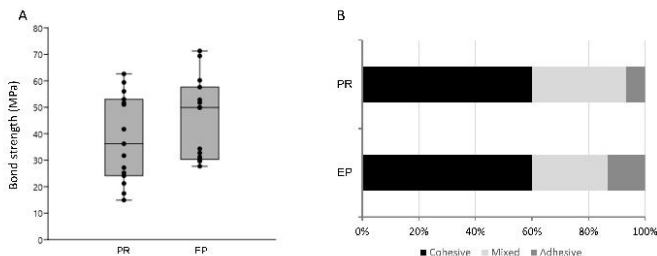
$$A = \pi (r_1 + r_2) \times \sqrt{(r_1 - r_2)^2 + h^2}$$

$r_1$ : smaller radius of canal diameter  
 $r_2$ : larger radius of canal diameter  
 $h$ : thickness of the root section

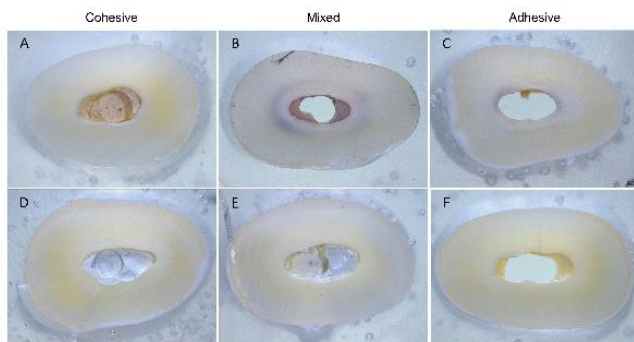
**Figure 1.** Illustration of the experimental procedure. (A) The tooth filled with the tested materials was sectioned horizontally to obtain a sliced specimen and an apical segment. (B) Push-out bond strength was measured with the sliced specimen using a universal testing machine. (C) The apical segment was sectioned longitudinally, and the intratubular biomineralization was observed under SEM.

## RESULTS

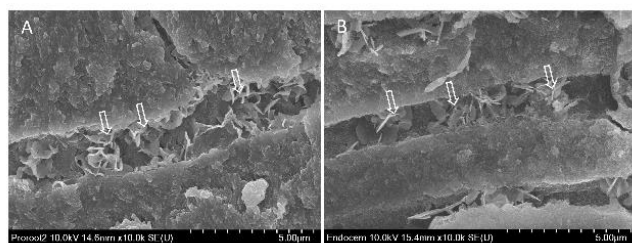
No significant difference was observed between the 2 tested groups in push-out bond strength ( $p > 0.05$ ) (Figure 2A). Cohesive failure was predominant in both groups, followed by mixed and adhesive failures (Figure 2B and Figure 3). In both groups, small flake-shaped precipitates were observed along dentinal tubules (Figure 4). As shown by the EDS analysis of the crystal seeds visible in the tubules, the mass percentage of calcium and phosphorus was around 2:1, similar to hydroxyapatite (Figure 5).



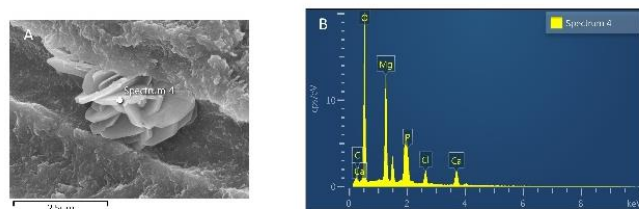
**Figure 2.** Push-out bond strength and the failure patterns of the tested materials. (A) Bar chart showing the mean bond strength of the 2 tested material groups. (B) Failure mode distribution according to filling material. PR, ProRoot MTA; EP, Endocem MTA Premixed.



**Figure 3.** Failure mode analysis using a stereomicroscope at  $\times 30$  magnification. (A-C) Representative images of the ProRoot MTA group. (D-F) Representative images of the Endocem MTA Premixed group.



**Figure 4.** Representative SEM images of intratubular biomineralization. (A) ProRoot MTA-filled root canal. (B) Endocem MTA Premixed-filled root canal. Arrows indicate the flake-shaped intratubular precipitates.



**Figure 5.** EDS analysis of the chemical characteristics of intratubular precipitate. (A) A SEM image showing the precipitate (white cross). (B) Representative graph of EDS analysis of the precipitate. (C) Semi-quantitative chemical composition showing the calcium/phosphorus ratio of the crystalline area denoted with a white cross.

## CONCLUSIONS

This study showed that Endocem MTA Premixed, a newly developed premixed bioceramic root-end filling material, showed similar push-out bond strength to the widely used material ProRoot MTA. Consequently, the null hypothesis was accepted. Endocem MTA Premixed also exhibited biomineralization in dentinal tubules. Considering its ease of use and enhanced handling characteristics, Endocem MTA Premixed has potential for use as an acceptable root-end filling material for endodontic surgery.

## Acknowledgement

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