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# Micro-Shear Bond Strength between Mineral Trioxide Aggregate Paste Fast Setting and Different Composite Materials

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## Abstract

This study aimed to estimate the micro-shear bond strength between mineral trioxide aggregate (MTA) "fast setting" premix paste (ENDOCEM™, Maruchi, Wonju, Korea) and different composite materials (EverX™ Flow GC, Japan and Bulk Fill™ Flowable A2, 3M ESPE, USA). A total of 40 resinous molds were prepared for application of Premix MTA paste, then the restorative material was applied with different times of restoration. The total samples were divided into 2 groups according to the time of application of restorative materials (group A after 5 min and group B after 24 h), then each group was divided into 2 subgroups according to the restorative materials used (A1 and B1 subgroups with EverX™ Flow, GC, Japan, and A2 and B2 subgroups with Bulk Fill™ Flowable A2, 3M ESPE, USA). The mean micro-shear bond strength increased with time elapsed between the application of the MTA and the placement of the restorative materials. After 24 h, Subgroup B2 had the highest bond strength, followed by Subgroup B1. After 5 min, Subgroup A1 had a higher bond strength than Subgroup A2. The properties of the calcium-silicate- materials (CSMs) improved after waiting 24 hrs, which contradicts the manufacturer's recommendation to allow only 3 min for the initial setting. Incorporating the 10-MDP monomer in the dental adhesive enhanced integration at the interface between the adhesive and the restorative resin, as reflected in the mixed mode of fracturing.

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## Introduction

The most conservative procedure for treating inflamed pulp tissue is to preserve the vitality of the pulp tissue through vital pulp therapy (VPT) [1,2]. This technique involves the application of bioactive and biocompatible materials to preserve the vitality of the pulpal tissue in addition to reducing the inflammation and inhibiting the microleakage of bacteria [3,4]. Previously, calcium hydroxide has been used in VPT [5], but several studies have reported that calcium hydroxide does not adhere to the wall of the dentin, resorbs over time and provides an inadequate seal, resulting in a defective dentinal bridge [6,7]. Calcium silicate has been used as an alternative in VPT to overcome the disadvantages of calcium hydroxide. One of these calcium-silicate-based materials is mineral trioxide aggregate, which has several advantages over calcium hydroxide, such as its biocompatibility, antibacterial effectiveness, endogenous stimulation of bioactive molecules and better sealing ability. These properties make MTA the gold standard material in vital pulp therapy [8]. The main disadvantages of traditional MTA are its difficulty of application and the discolouration of the tooth structure over time [9]. Also, due to long setting time of MTA, a second visit to the dentist is needed to finish the restoration, which means further chair-side time, additional treatment costs and an increased treatment failure rate [3,9]. In addition, uncooperative patients mostly avoid the second visit, especially children [10]. To address this, a new calcium silicate material has been introduced that offers a reduced chance of tooth discolouration and a shorter setting time than traditional MTA [9,11-13].

According to the manufacturer, ENDOCEM™ Premix MTA paste (Maruchi, Wonju, Korea) is an injectable MTA with a fast (3 minutes) initial setting time. ENDOCEM Premix MTA resists discoloration, making it an ideal choice for aesthetic dental treatments, and it contains zirconium oxide, making it an ideal radiopaque material. Also, being based on pure tricalcium silicate, it has


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better physical properties than the powdered types of MTA [14].

## Material and Methods

### Specimen Preparation

Three-dimensional (3D) printed resinous moulds, designed using exocad WebView software, were produced. Each mould was 7.5 x 3 cm and 5 mm thick. A 3-mm-diameter, 2-mm-thick metal disk was used to make 10 cylindrical holes (2 mm deep, 3 mm in diameter) in each of four moulds (Figure 1). All the holes (40 holes) were filled with ENDOCEM™ Premix MTA. To provide a standard surface, a bond brush was first applied to the MTA, after which a glass slab was sited over the specimens.

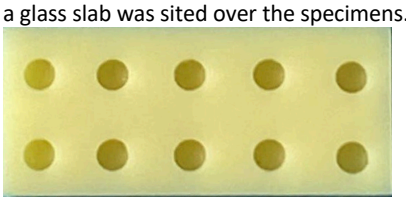

Figure 1. A 3D printed resinous mold.

These samples were separated into two groups according to the time elapsed since the holes were filled with them MTA (i.e., 5 min and 24 h). Both groups were further distributed among subgroups based on the restorative materials used ($n$ = 10) (Figure 2).

Plastic rings, 2 mm high by 3 mm in diameter, were fabricated to be positioned over the MTA during the restorative procedure.

### Restorative Material Placement Over Fast Setting Calcium Silicate Materials (CSMs)

The holes in the moulds were filled with the ENDOCEM™ Premix MTA, which was then covered with a moist sponge at 100% humidity. The specimens were separated into 2 groups (A and B) according to the time of the start of the restorative procedure after MTA placement. In Group A, the restorative procedure began 5 min after MTA placement, (air drying then) a one-step, (a universal / multi-mode adhesive) agent being placed over the MTA in two layers, depending on the instructions of the manufacturer, then a LED lamp used for curing (Eighteeth Curing Pen E1, China). The samples were further divided into subgroups according to the restorative material used, with Subgroup A1 having EverX™ Flow (GC, Japan) applied (2 mm thick, 3 mm in diameter), aided by the plastic ring fabricated for this purpose, then being cured by LED lamp, and Subgroup A2 having Bulk Fill Flowable A2 (3M ESPE, USA) applied, then being cured by LED lamp. In Group B, the restorative procedure began 24 h after the MTA placement (the specimens were incubated in 37°C distilled water for 24 h), after air drying, a two layers of the one-step, (a universal / multi-mode adhesive) agent placed over the MTA, depending on the instructions of manufacturer, then cured by LED lamp. These samples were then similarly subdivided, with Subgroup B1 having EverX™ Flow applied (same thickness and diameter) and Subgroup B2 having Bulk Fill™ Flowable A2 applied, both subgroups then cured by LED lamp.

All the specimens (Groups A and B) were then placed in 37°C distilled water for 24 hr. After that, the plastic rings were carefully detached using a #12 surgical blade.

### Micro-Shear Bond Strength Testing

Before assessment the bonding strength, the resinous moulds were sectioned into 10 pieces to separate each hole from the rest. A universal testing machine (Laryee, China) was employed to test the micro-shear bond strength, using a 0.5 mm/min crosshead jig speed and a 50 kg weight cell. The fracture load was documented in Newtons and then recalculated to give the megapascals by dividing the fracture load by the area of the filling material that was deboned. The deboned areas of the samples were inspected by using Stereomicroscope (SZX16 Olympus, Tokyo, Japan) at 40x of magnification to determine the failure mode. The failure mode was categorized as cohesive (within the MTA or the filling materials), adhesive (at the line between the materials) and mixed (a mix of the cohesive and adhesive modes).

### Statistical Analysis

For the statistical analysis, we used a one-way analysis of variance (ANOVA) and Tukey's post hoc test, employing Jamovi v.1.6 statistical software (Jamovi Project, https://www.jamovi.org) ($\alpha = 0.05$).

## Results

Table 2 presents the descriptive statistics for the micro-shear bond strength of the different restorative materials and the ENDOCEM™ Premix MTA for all specimens. The mean micro-shear bond strength increased with time elapsed between the application of the MTA and the placement of the restorative materials. After 24 h, Subgroup B2 had the highest bond strength, followed by Subgroup B1. After 5 min, Subgroup A1 had a higher bond strength than Subgroup A2.

Anova test revealed significant differences as shown in Table 3, so LSD was performed (Table 4).

The difference was statistically significant between group A1 and B1, A1 and B2. A2 and B1, A2 and B2. Non-significant differences are present between groups A1 and A2 and B1 and B2.

Table 5 revealed the mode of fracture between the ENDOCEM™ MTA, bonding agent, and restorative materials that were shown in the electronic microscope.

## Discussion

We allowed different setting times (5 min and 24 h) for one-step, self-etch 7th generation adhesive (Single Bond Universal) and ENDOCEM™ Premix MTA before applying one of two restorative materials (EverX™ Flow or Bulk Fill™ Flowable). We found the micro-shear bond strength to be statistically higher in the 24 h group than the 5 min group. This may be due to the setting of the materials via hydration, early bonding (before 24 h) weakening the adhesion because the material continued to hydrate and expand, the best bonding occurring after complete setting.[15] With time, more calcium ions are released, inducing apatite precipitation, which would likely contribute to the sealing of, and integration with, the tooth structure, these ions playing a key role in forging chemical bonds with the adhesives and forming bioactive layers [15,16]. Conversely, freshly set materials may contain unreacted components and be highly alkaline, which could interfere with resin polymerization if the bonding is done too early [17,18].

The results showed no significant differences within the subgroups (5 min and 24 h). This could be explained by a mechanism of micromechanical bonding first, during which the adhesive resin penetrates the surface irregularities and pores of the CSM (i.e. physically interlocking polymerization), followed by chemical bonding via functional monomers, especially the 10-MDP monomer, reacting with calcium ions from the CSM surface, forming stable MDP–calcium salts. This complex would be hydrolysis-resistant and would strengthen the interface. In a third phase, a bioactive layer of calcium and hydroxide ions reacting with phosphate ions may form a hydroxyapatite layer that acts as a natural bridge between the resin and the CSM [19-21].

The restorative materials used in the subgroups showed no distinct effects. This may be attributed to covalent bonding between the adhesive and the resin composite via crosslinking monomers from the adhesive. The Bulk Fill Flowable gave better results, but these were not statistically different and were possibly due to the highly thixotropic nature of the materials enhancing its adaptation and flowability.

Our micro-shear bond strength values were ≈3–7 MPa, while in other studies, Biodentine was found to have values of ≈10–15 MPa and MTA ≈5–10 MPa. These differences may be due to the different materials used, the different setting reactions and ions released from the CSM, different bonding between the chemical structures (related to the functional 10-MDP monomer), and different adhesion techniques. Also, different moisture levels will have affected the results, excess moisture possibly diluting the adhesive, over drying reducing

infiltration; a slightly moist surface has been found to be optimal [17,18,21].

In terms of fracture mode, there was no adhesive failure in any sample, possibly because of the superior ionic bond between the CSM (calcium ions and silanol groups) and the functional 10-MDP monomer in the adhesive [17,18]. Also, ENDOCEM™ is a premixed injectable MTA with an overall fast setting time and an initial setting time of about 3 min. This gives this material enhanced mechanical properties and bonding surfaces.

Cohesive failure was higher in the CSM allowed to set for 5 min, possibly because of the hydration reaction with water and nucleation of the CSM needing more time to mature and finally set [22]. There was no cohesive failure in the resin restorative materials, probably because they were engineered to have high compressive and tensile strength [23].

Mixed adhesive–cohesive failure was higher in those samples that had the restorative applied after 24 h. This may be due to maturation of the CSM enhancing ion release, and the better mechanical properties of the resin's internal composite structure being stronger than the bond interface so that when stress was applied, failure occurred in the weakest part. The EverX™ Flow resin composite with Fibers enhanced the mechanical properties [4,22,24].

## Conclusion

1- Incorporating the 10-MDP monomer in the dental adhesive enhanced integration at the interface between the adhesive and the restorative resin, as reflected in the mixed mode of fracturing.

2- Emplacing the adhesive after the initial CSM setting period is not recommended because it will not fully mature and will be more sensitive to cohesive failure.

3- The properties and surface integration of both the Bulk Fill Flowable and EverX™ Flow resin composites were good.

4- The properties of the CSM improved after waiting 24 h, which contradicts the manufacturer's recommendation to allow only 3 min for the initial setting.

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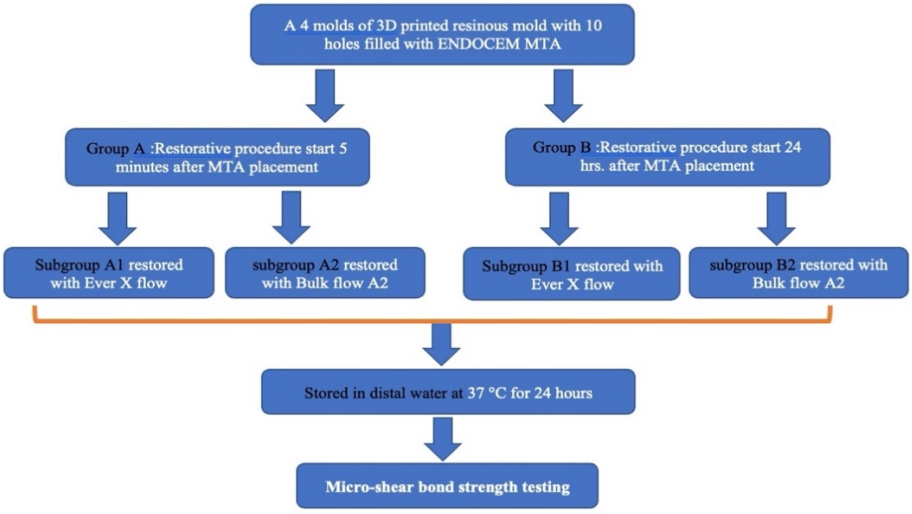


Figure 2. Specimens grouping.

Table 1. Components of used materials in this study.

| Materials | Manufacturer | Components |
|---|---|---|
| Endocem MTA Premixed Regular | Maruchi, Wonjusi, Gangwon, Korea | Tricalcium Silicate, Dodecacalcium Heptaaluminate And Dimethyl Sulfoxide |
| Single Bond Universal | 3M ESPE, U.S.A. | MDP Phosphate Monomer, Dimethacrylate Resins, HEMA, Vitrebond Copolymer, Filler, Ethanol, Water, Initiators, Silane |
| Filtek Bulk Fill Flowable Restorative | 3M ESPE, U.S.A. | Bisgma,Tegdma,Bisemaand Procrylat Resins, Ytterbium Trifluoride Fillers, Zirconia /Silica |
| Ever X Flow | GC, Japan | Bis-MEPP, TEGDMA and UDMA. short E-glass fibres and particle fillers, mostly barium glass. |

Table 2. Micro-shear bond strength for all subgroups.

| | N | Mean | Std. Deviation | Minimum | Maximum |
|---|---|---|---|---|---|
| A1 (5 min) Ever X flow (mpa) | 10 | 3.769 | 0.68205 | 2.49 | 4.89 |
| A2 (5 min) flow A2 (mpa) | 10 | 3.36 | 0.51672 | 2.38 | 4.11 |
| B1 (24 hrs) Ever X flow (mpa) | 10 | 5.962 | 0.65307 | 5.36 | 7.21 |
| B2 (24 hrs) Bulk flow A2 (mpa) | 10 | 6.743 | 0.9258 | 5.39 | 8.12 |

Table 3. Summary of Anova test.

| | Sum of Squares | df | Mean Square | F | p-value |
|---|---|---|---|---|---|
| Between Groups | 81.616 | 3 | 27.205 | 53.984 | 0.000 |
| Within Groups | 18.142 | 36 | 0.504 | | |
| Total | 99.758 | 39 | | | |

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Table 4. Summary of LSD test.

| Multiple Comparisons | | | | |
|---|---|---|---|---|
| LSD | | | | |
| (I) group | | Mean Difference (I-J) | Std. Error | p-value |
| A1 (5 min) Ever X flow (mpa) | A2 (5 min) flow A2 (mpa) | 0.409 | 0.31747 | 0.206 |
| | B1 (24 h) Ever X flow (mpa) | -2.193* | 0.31747 | 0.000 |
| | B2 (24 h) Bulk flow A2 (mpa) | -2.974* | 0.31747 | 0.000 |
| A2 (5 min) flow A2 (mpa) | A1 (5 min) Ever X flow (mpa) | -0.409 | 0.31747 | 0.206 |
| | B1 (24 h) Ever X flow (mpa) | -2.602* | 0.31747 | 0.000 |
| | B2 (24 h) Bulk flow A2 (mpa) | -3.383* | 0.31747 | 0.000 |
| B1 (24 hrs) Ever X flow (mpa) | A1 (5 min) Ever X flow (mpa) | 2.193* | 0.31747 | 0.000 |
| | A2 (5 min) flow A2 (mpa) | 2.602* | 0.31747 | 0.000 |
| | B2 (24 h) Bulk flow A2 (mpa) | -0.781* | 0.31747 | 0.019 |
| B2 (24 hrs) Bulk flow A2 (mpa) | A1 (5 min) Ever X flow (mpa) | 2.974* | 0.31747 | 0.000 |
| | A2 (5 min) flow A2 (mpa) | 3.383* | 0.31747 | 0.000 |
| | B1 (24 h) Ever X flow (mpa) | 0.781* | 0.31747 | 0.019 |
| ***. The mean difference is significant at the 0.05 level.** | | | | |

Table 5. Mode of fracture.

| Mode of fracture | Group A1(5min) Ever X flow | Group A2(5min) flow A2 | Group B1(24h) Ever X flow | Group B2(24h) Bulk flow A2 |
|---|---|---|---|---|
| Cohesive Within MTA | 9 | 9 | 2 | 6 |
| Adhesive | 0 | 0 | 0 | 0 |
| Cohesive Within composite material | 0 | 0 | 0 | 0 |
| Mixed mode of failure | 1 | 1 | 8 | 4 |