# Shear Bond Strength of Calcium Enriched Mixture Cement and Mineral Trioxide Aggregate to Composite Resin with Two Different Adhesive Systems

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#### Abstract

**Objective:** Immediate restoration after vital pulp therapy is essential in order to create and maintain effective coronal seal.

Purpose of Study: The aim of this study was to evaluate the shear bond strength of recently used pulp capping materials: white mineral trioxide aggregate (MTA), and calcium enriched mixture cement (CEM) to composite resin with the use of etch-andrinse and self-etch adhesive systems and compare them with the bond strength of commonly used resin modified glass ionomer (RMGI) cement.

**Materials and Methods:** Forty specimens from each test material were fabricated, measuring 4 mm in diameter and 2 mm in depth. The specimens of each material were divided into 2 groups of 20 specimens according to the adhesive system (Single Bond vs. Clearfil SE Bond) used for bonding of resin composite. The shear bond strength values were measured at a crosshead speed of 1.0 mm/min and fractured surfaces were examined. Data were analyzed using two-way ANOVA and a post hoc Tukey's test (P<0.05).

**Results:** Analysis of data showed a significantly higher bond strength for RMGI compared to MTA and CEM (P<0.001); however, no significant differences were observed in the bond strength values of MTA and CEM (P=0.9). Furthermore, there were no significant differences in relation to the type of the adhesive system irrespective of the type of the material used (P=0.95) All the failures were of cohesive type in RMGI, MTA and CEM.

**Conclusion:** Bond strength of RMGI cement to composite resin was higher than that of MTA or CEM cement irrespective of the type of the adhesive system.

**Keywords**: Mineral trioxide aggregate; Composite resin; Bond strength

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## INTRODUCTION

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The primary objective of restorative dentistry is to restore and preserve dental health through proper restorative treatment modalities in order to protect the pulp and restore its function [1]. This aim is achieved by vital pulp therapy in open apex teeth to allow apexogenesis and normal development of the root and by pulp capping in mature teeth in order to avoid root canal therapy and the subsequent extensive restorations [2,3].

It is important to note that the success of all vital pulp capping procedures is directly related to the control of pathogenic agents [4]. A pulp exposed in a sterile environment can repair itself and form a dentinal bridge [2,3]. However, in the presence of bacteria, pulp disease and finally pulp necrosis are inevitable. Therefore, a proper bond between the pulp capping agent and the restorative material is of utmost importance because in the absence of a proper seal bacteria will penetrate into the pulp and the pulp capping procedure will fail [4].

Calcium hydroxide is commonly used for pulp capping. Some of the disadvantages of calcium hydroxide as a pulp capping material include gradual degradation, tunnel-like defects in the dentinal bridge and low sealing ability [5,6]. Furthermore, dentine primer is applied as a routine procedure prior to bonding strategies. Some of these primers contain acetone or alcohol that may affect the properties of calcium hydroxide. A previous study showed that calcium hydroxide treated with alcohol or acetone based primers had the highest erosion values and the lowest compressive strength values. Water based primer had the least effect on calcium hydroxide values. Therefore, it should be covered with a stronger material such as RMGI with proper sealing ability [7].

Mineral Trioxide Aggregate has been suggested as a choice material for retrograde filling, perforation repair, pulp capping, pulpotomy and as an apical barrier for root canal treatment of immature teeth with open apices [8,9]. Recently a new dental cement has been suggested, which, in addition to proper biocompatibility similar to MTA, has good handling and chemical properties and a more favorable color. This new cement has applications similar to those of MTA and is called Calcium Enriched Mixture (CEM) due to different calcium compounds in its chemical composition [10,11]. Recent research has shown that after setting of CEM, hydroxyapatite is produced [13]; its anti-bacterial activity is similar to that of calcium hydroxide and significantly higher than those of MTA and Portland cement [14].

Because of the great importance of the bond strength between the pulp capping material and the final restorative material, this was an issue of concern. Tunc et al. as well as, Beyrak et al. reported that the etch-and-rinse system provides higher shear bond strength between MTA and composite resin than self-etch systems [15,16]. Recently, Oskoee et al. evaluated the bond strength of MTA and CEM to composite resin using etch-and-rinse adhesive system with and without acid etching procedure [17]. However, there is scant information on the adhesion of composite resin-based materials to MTA and CEM using various bonding strategies. Therefore, the aim of this study was to evaluate the shear bond strength of recently used pulp capping materials: MTA and CEM to composite resin with the use of etchand-rinse and self-etch adhesive systems and compare them with the bond strength of commonly used RMGI cement as the control group.

### MATERIALS AND METHODS

The materials used in this study were RMGI (GC Fuji II LC, GC Corporation, Tokyo, Japan), ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN, USA) and the new endodontic cement, Calcium Enriched Mixture (BioniqueDent Tehran, Iran).

Forty specimens from each test material were prepared by using cylindrical acrylic blocks. Each block had a central hole measuring 4 mm in diameter and 2 mm in depth [17,18].

RMGI powder was mixed with the liquid at a ratio of 3.2:1 for 25 seconds according to the manufacturer's instructions. Then, the central holes of the acrylic blocks were filled with the

mixture and light-cured for 20 seconds with a QTH light-curing unit (Litex 680A, Dentamerica, 18320 Bedford Circle, City of Industry CA91744, USA) with a light intensity of 600 mW/cm<sup>2</sup>.

MTA was mixed with distilled water according to the manufacturer's instructions. The central holes of the acrylic blocks were filled with MTA, flattened with a spatula, covered with a wet cotton pellet and sealed with temporary filling material (Cavit; ESPE America Inc., Norristown, PA, USA). Then, the specimens were stored at 37°C with 100% relative humidity for 48 hours to allow complete setting.

CEM powder was mixed with the manufacturer's recommended liquid at a ratio of 3:1, placed in the central holes of the acrylic blocks, flattened with a spatula, covered with a wet cotton pellet and sealed with a temporary filling material. Then, the specimens were stored at 37°C and 100% relative humidity for 50 minutes for the setting reaction to be completed. After the removal of the temporary material, MTA and CEM surfaces were not rinsed or polished [18]. The specimens of each material were divided into 2 groups of 20 each.

#### **Group One:**

Material surface was etched for 15 seconds with 35% phosphoric acid etching gel (Scotchbond<sup>TM</sup> Etchant, 3M Dental Products, St Paul, MN, USA) and rinsed with water for 10 seconds; excess water was removed by cotton pellets, without desiccating the surface. Single Bond (3M ESPE) was then applied in 2 consecutive coats, gently air-dried for 5 seconds (keeping the air syringe 2 cm from the surface), and light-cured for 10 seconds. Resin composite (Gradia Direct, Shade A2, GC Corporation, Tokyo, Japan) was filled into a cylinder-shaped plastic mold with an internal diameter of 3 mm and a height of 2 mm. Upon filling, the mold was placed on the prepared surface of the sample and the composite was condensed. Then, molds were light cured for 20 seconds from top and the lateral sides.

#### **Group Two:**

Material surface was dried for 10 seconds (keeping the air syringe 2 cm from the surface) to ensure a dry surface. Clearfil SE Bond (Kuraray Medical Inc., Tokyo, Japan) primer was applied and agitated for 20 seconds, gently air-dried for 5 seconds and light-cured for 10 seconds after applying the bonding agent according to the manufacturer's instructions. Composite bonding procedures were done similar to group one. The polymerized specimens were stored in 100% relative humidity at 37°C for 24 hours. For shear bond strength testing, the samples were secured in a holder placed on the plate of the universal testing machine (Hounsfield Test Equipment – model: H5K-S, Perrywood Business Park, Honey Corckland, Salfords, Redhill, Surrey, UK) and then underwent a shearing force with a knifeedge blade at a cross-head speed of 1.0 mm/min. Shear bond strength was calculated in MPa by the peak load at failure divided by the specimen surface area.

### Statistical Analysis

Two-way ANOVA was used to determine the effect of the adhesive systems and materials and the cumulative effect of these two factors (the adhesive systems and materials). Post hoc comparisons were carried out using the Tukey's test. Statistical significance was set at P<0.05.

#### RESULTS

Analysis of data with two-factor ANOVA demonstrated statistically significant differences in the mean bond strength values in relation to the type of the material used (F  $_{(2,82)}$  = 91.63, P<0.001); however, there were no significant differences in relation to the type of the adhesive system (F  $_{(1,82)}$ =0.004, P=0.95).

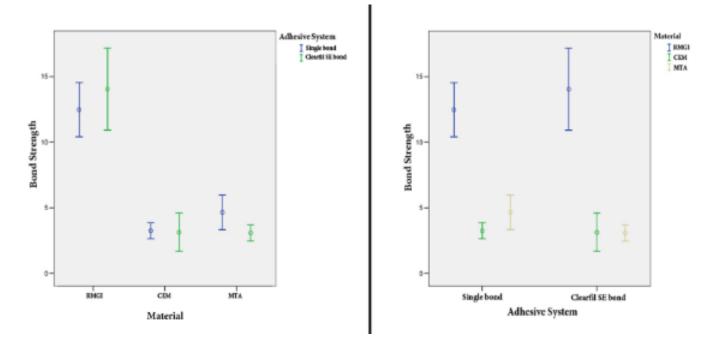


Fig1. Diagram of two-way ANOVA

In addition, the cumulative effect of the two factors was not significant (F  $_{(2.82)} = 1.80$ , P=0.17) (Figure 1).

Two-by-two comparison of the groups with a post hoc Tukey's test showed a higher bond strength with RMGI compared to MTA and CEM (P<0.001); however, the differences between MTA and CEM bond strength values were not significant (P=0.9) (Table 1). All the failures in the current study were of the cohesive type inside the materials under study.

#### DISCUSSION

The results of the current study showed that the bond strength of RMGI, irrespective of the adhesive type used, was significantly higher than that of CEM and MTA; however, no significant differences were observed between MTA and CEM bond strength values.

The higher bond strength of RMGI might be attributed to the chemical bond between composite and RMGI, which has been confirmed in previous studies [19,20].

Material	Adhesive System	Bond strength (MPa)	
		Mean	SD
RMGI	Single Bond	12.47 <sup>A</sup>	3.74
	Clearfil SE Bond	14.03 <sup>A</sup>	5.65
MTA	Single Bond	4.65 <sup>B</sup>	2.38
	Clearfil SE Bond	3.08 <sup>B</sup>	1.10
CEM	Single Bond	3.24 <sup>B</sup>	1.06
	Clearfil SE Bond	3.13 <sup>B</sup>	2.52

Different capitals mean statistically significant differences.

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RMGI has a liquid resin component, which is composed of polyacrylic acid (209-30%); HEMA (30-35%), UDMA (<10%) and comphorquinone (<1%); the photoinitiated reaction of the liquid is accomplished after the liquid is mixed with fluoroaluminosilicate glass powder; an acid-base reaction follows, which completes the reaction between the liquid and the powder [21].

Due to the presence of the resin component, especially HEMA, there is a possibility of copolymerization of unreacted resin double bonds during the setting reaction with the adhesive systems [19], which can increase the bond strength of this material compared to MTA and CEM; MTA and CEM do not have any resin components in their chemical composition and are composed of mineral oxides and silicates. The bonding mechanisms of MTA and CEM are probably micromechanical and the result of penetration and interlocking of the adhesive systems into the surface pores and irregularities due to their chemical composition. The polymerization reaction of composite resins is inevitably associated with polymerization shrinkage [22]. The polymerization shrinkage of the available composite resins has been reported to range from 2.9% to 7.1%, depending on the test used; the polymerization shrinkage can result in shrinkage stresses up to 7 MPA [22,23].

At present, no composite resin without polymerization shrinkage is available in the market and research is underway to produce composite resins with low or no polymerization shrinkage [24]. In the current study, the mean bond strength of RMGI to composite resin with the use of self-etch and etch-and-rinse systems 14.03±5.65 adhesive was and 12.47±3.74 MPa, respectively, which were higher than the polymerization stresses produced during composite resin polymerization. Other studies, too, have reported higher bond strength of RMGI to composite resin compared to polymerization stresses produced during composite resin polymerization, which is an important fact from a clinical viewpoint [25,26]. The bond strength of RMGI to composite resin is higher than the polymerization stresses produced during composite resin polymerization. Also, MTA and CEM have longer setting times despite their superiority to calcium hydroxide for pulp capping procedures [8,12]. Thus, according to the results of the current study, it is suggested that both these cements be covered with RMGI, followed by composite resin restoration. Previously, Yesilyurt et al. reported that conventional glass-ionomer comment could be used for this purpose 45 minutes after initial setting of MTA [18]; however, its bond strength to composite resin was influenced by its low cohesive strength [27].

Another important consideration in the current study was the fact that the type of the adhesive did not influence the bond strength of the materials to composite resin. According to several previous studies, acid etching does not have any effect on improving the bond strength of RMGI to composite resin with the etch-andrinse adhesive systems [25,26,28].

Etching and rinsing procedures in the etchand-rinse adhesive systems result in the preferential dissolution and detachment of filler particles from the RMGI surface, producing a honey-comb appearance on the cement surface [27].

The same pattern has been reported subsequent to acid etching of MTA surface [29]. This process degrades the cement surface and reduces the cohesive strength of the material [27].

Contrary to the results of the current study, previous studies on the bond strength of compomer and composite resin to MTA with the use of etch-and-rinse adhesive systems have reported higher bond strengths compared to the use of self-etch adhesive systems [15,16].

All the failures in the current study were of the cohesive type inside the materials, which

might reflect their low cohesive strength compared to the bond strength.

Therefore, the bond strength values recorded reflect the cohesive strength of the etched cements rather than the actual bond strength at the material-composite resin interface. McCarthy et al. evaluated the mechanical and bond strength properties of a commercially available RMGI and conventional GI cements. They evaluated the ratios of the mechanical strength tests to the bond strength tests at 1 and 24 hours. The results indicated a close association between the diametral tensile and bond strength tests; the diametral tensile strength was found to be approximately twice the value of the bond strength for both cements at 1 hour and 24 hours. This finding may allow prediction of bond strength on the basis of mechanical properties [30].

Placement of a restoration immediately after VPT is recommended in order to create and maintain an effective seal, which is essential for success of VPT.

Composite resin is recommended for this purpose due to lower loads applied to the pulp capping biomaterial. Furthermore, due to the possible inadvertent effects of acid etching/irrigation process of etch-and-rinse adhesive systems on setting time of pulp capping biomaterials and possibility of dislodging/dissolving them, we suggest future studies to focus on bonding properties of freshly mixed pulp capping biomaterials using various self-etch adhesive systems.

# CONCLUSION

Based on the results of this in-vitro study, it can be concluded that the bond strength of RMGI cement to composite resin is higher than that of MTA or CEM cement irrespective of the type of the adhesive system.

Furthermore, the bond strength of RMGI to composite resin is higher than the polymerization stresses produced during composite resin polymerization and MTA and CEM have longer setting times despite their superiority to calcium hydroxide for pulp capping procedures. Therefore, it can be suggested that both these cements be covered with RMGI, followed by composite resin restoration.

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