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ORIGINAL RESEARCH

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Comparison of Shear Bond Strength of Calcium-enriched Mixture Cement and Mineral Trioxide Aggregate to Composite Resin

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ABSTRACT

Aim: Adhesion of composite resin and pulp capping biomaterials remarkably influences treatment outcomes. This *in vitro* study aimed to compare the shear bond strength of composite resin to calcium enriched mixture (CEM) cement, mineral trioxide aggregate (MTA) and resin modified glass ionomer (RMGI) with or without acid etching.

Materials and methods: A total of 90 cylindrical acrylic blocks containing a central hole, measuring 4 mm diameter and 2 mm height were prepared. The blocks were randomly divided into three experimental groups based on being filled with CEM, MTA or RMGI. Samples in each group were then randomly divided into two subgroups, i.e. with or without phosphoric acid etching. Placing composite resin cylinders on the samples, shear bond strengths were measured using a universal testing machine. Failure modes of the samples were evaluated under a stereomicroscope. Data were analyzed using two-way ANOVA and Tukey tests.

Results: Shear bond strengths in the etched and nonetched samples were not significantly different ($p = 0.60$). There was a significant difference in shear bond strength values of the three experimental materials ($p < 0.001$) and RMGI showed the highest strength values ($p < 0.001$); no significant difference was observed between MTA and CEM ($p = 0.51$). The interaction of the type of material and surface etching was statistically significant ($p < 0.001$). All of the samples showed cohesive failure mode.

Conclusion: Acid etching of MTA, CEM and RMGI do not improve the shear bond strength of these materials to composite resin. Besides, shear bond strength values of MTA and CEM to composite resin, are favorable due to their cohesive mode of failure.

Clinical significance: When MTA and CEM biomaterials are used in vital pulp therapy, it is advisable to cover these materials with RMGI. In addition, if it is not possible to use RMGI, the surface etching of MTA and CEM biomaterials is not necessary prior to composite restoration using total-etch adhesive resin.

Keywords: CEM cement, Composite resin, MTA, RMGI, Shear bond strength.

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INTRODUCTION

Introduction of gray mineral trioxide aggregate (MTA) in 1993 has opened new horizons in endodontics/dentistry.[1,2] MTA as a hydrophilic cement is mainly composed of calcium oxide, silica and bismuth oxide.[3] In a short time, MTA gained numerous clinical applications in endodontic procedures and is recommended for management of internal/external root resorption, as an apical barrier for teeth with necrotic pulps/open apices, as a root-end filling, for repair of root/furcation perforations and particularly for vital pulp therapy [direct pulp capping (DPC) and pulpotomy].[4] Mente et al demonstrated that long-term (>3 years) pulp vitality after DPC with MTA is more effective than calcium hydroxide (78% *vs* 60%).[5] This favorable result for DPC with MTA was confirmed with a recent systematic review; however, the review revealed that pulpotomy treatment provides a more predictable outcome than DPC.[6] Interestingly, Eghbal et al demonstrated complete dentinal bridge formation and healthy pulps after treatment with MTA pulpotomy in all (n = 12) mature permanent teeth with established irreversible pulpitis; their success measure was based on histological examination.[7] Although MTA is a biocompatible material and provides a very suitable seal, it has some drawbacks such as unpredictable antibacterial effect and prolonged setting time.[4,8] A wet cotton pellet should be placed over MTA at least for ≈4 hours prior to placement of final restoration at the second visit.[8]

Recently, a new endodontic biomaterial, calcium enriched mixture (CEM) cement, has been introduced.[9] CEM cement is a tooth-colored water-based cement with similar clinical applications as MTA, but different chemical composition.[10,11] Sealing ability and cytotoxicity of CEM cement are comparable to those of MTA[9,12] but unlike MTA, it has the ability to promote hydroxyapatite formation even in normal saline solution.[13] CEM has exhibited favorable treatment outcomes for management of internal/external root resorption,[14,15] management of nonvital teeth with open apices,[16] repair of furcal perforations,[17,18] root-end filling,[19] direct pulp capping [20] and pulpotomy in primary and permanent molars.[15,21]

Composite resins have gained popularity in restorative dentistry as a result of their favorable esthetic results.[22] Apart from proper bonding of composite resins to dental substrates, the bond strength between composite resins and the pulp capping biomaterials is important for the quality of fillings and success of restorations.[23] Moreover, proper bonding of composite resins to pulp capping biomaterials produces the adhesive joint, which is capable of spreading stress relatively evenly over the entire region of the bond.[24] Therefore, the aim of the present study was to evaluate the shear bond strength of composite resin to MTA and CEM with/without acid etching; resin modified glass ionomer (RMGI) was used in the control group as a material with appropriate bond strength.

MATERIALS AND METHODS

A total of 90 cylindrical acrylic specimens with 6 mm diameter and 4 mm height were used. Holes with a 4 mm diameter and 2 mm height were created in each acrylic specimen. The specimens were randomly divided into three experimental groups of CEM, MTA and RMGI ($n = 30$), respectively. Each group was then randomly subdivided into two equal subgroups ($n = 15$) of surface treatment with or without acid etching.

In group 1, CEM (BioniqueDent, Tehran, Iran) powder and liquid was mixed in a mortar to achieve a thick cream consistency. The prepared mixture was then placed inside the holes using a spatula, covered by a wet cotton pellet and placed in a moist environment for 24 hours to enable complete setting. In the CEM first subgroup, the surfaces of material were etched with 35% phosphoric acid gel (3M ESPE Dental Products, St Paul, MN, USA) for 15 seconds, rinsed for 30 seconds and dried with an air spray (oil- and water-free) for 5 seconds. At the next stage, Adper Single Bond adhesive resin (3M ESPE Dental Products, St Paul, MN, USA) was applied in two coats according to manufacturer instructions. The air spray was used for 2 to 5 seconds to evaporate the solvent. The adhesive resin was then light-cured for 20 seconds at a light intensity of 400 mW/cm^2 using Astralis 7 light-curing unit (Ivoclar Vivadent, FL-9494 Schaan, Liechtenstein). Transparent molds with a diameter and height of 3 mm were then placed on the prepared samples. The molds were packed using composite resin (Z250, Filtek™, 3M ESPE Dental Products, St Paul, MN, USA) and light-cured. The same procedures were repeated in the CEM second subgroup, the only difference being that acid etching was not carried out.

In group 2, MTA (Dentsply, Tulsa Dental, OK, USA) was used, and all of the procedures were the same as those in CEM subgroups.

In group 3, RMGI (GC, Fuji II LC, Tokyo, Japan) was used. RMGI powder was mixed with its liquid on a glass slab using a plastic spatula according to manufacturer instructions. The mixture was placed inside the molds and light-cured for 40 seconds. The rest of the procedures were the same as described for CEM subgroups.

Shear bond strength tests were carried out by mounting the specimens on a universal testing machine (Hounsfield Testing Equipment, Model H5K-S, Tinius Olsen Ltd, Surrey, England) and subjecting them to a shearing force at a strain rate of 1mm/min using a 0.5 mm wide chisel. The shear bond strength was calculated and expressed in MPa. All of the specimens were then evaluated under a stereomicroscope (Nikon, Tokyo, Japan) at ×25 in order to determine failure modes. The failure modes were classified as adhesive (failure at composite resin-experimental material interface) and cohesive (failure inside the experimental material).

In order to prepare specimens for scanning electron microscope (SEM) evaluation, two discoid specimens were prepared from each material (total of 6 specimens). One specimen from each material was prepared with 35% phosphoric acid gel and the second was prepared without acid application. The specimens were evaluated under SEM (Tescan, Vega II XMU, Brno, Czech Republic) after gold sputtering.

For statistical analysis, two-way ANOVA was used. Tukey's post hoc test was used for paired comparison of the experimental groups. Statistical significance was set at $p < 0.05$.

RESULTS

Table 1 shows the means and standard deviations (SD) of shear bond strength values in the subgroups. Results of

Table 1: Statistical indices of shear bond strength in the experimental groups

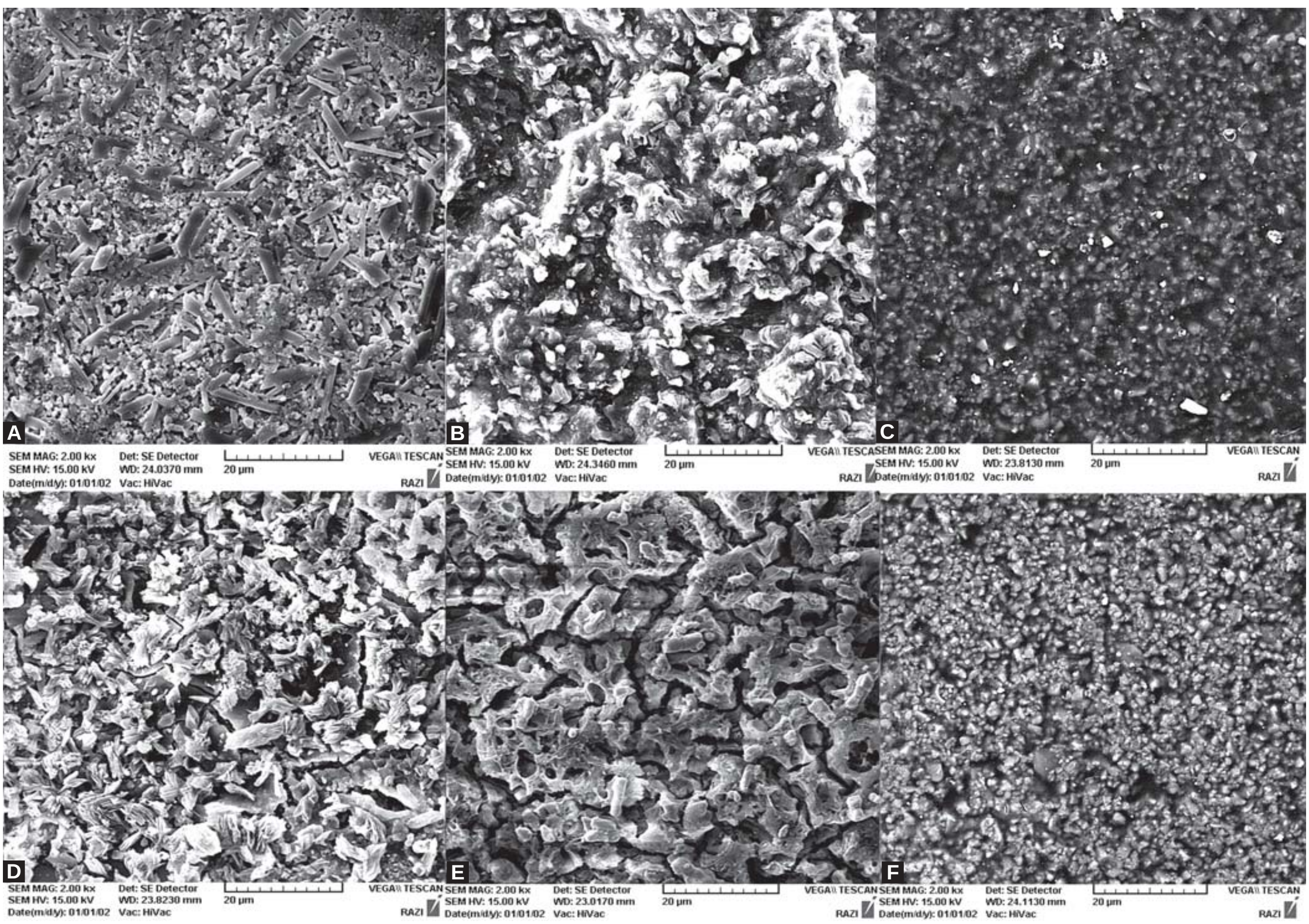
		Unetched				*Etched*			
		Mean	*SD*	*Minimum*	*Maximum*	*Mean*	*SD*	*Minimum*	*Maximum*
Materials	CEM	1.91	0.62	0.92	2.66	3.24	1.06	1.39	4.81
	MTA	2.76	1.26	0.58	4.93	4.65	2.38	2.16	12.34
	RMGI	16.71	5.73	8.88	27.40	12.47	3.74	8.02	21.59

two-way ANOVA showed a statistically significant difference ($F_{2,81}$ = 134.95, $p < 0.001$) in mean bond strength values of the materials. The bond strength of composite resin to RMGI was significantly higher than that of composite resin to MTA and CEM ($p < 0.001$). However, there were no significant differences in bond strength values of MTA and CEM to composite resin ($p = 0.51$). The differences in bond strengths in relation to surface preparation method were not statistically significant ($F_{1,81}$ = 0.26, $p = 0.60$). The interaction of material type and surface preparation method on bond strength was statistically significant ($F_{2,81}$ = 8.82, $p < 0.001$). Microscopic evaluation of modes of failure indicated that all of the failures were cohesive.

Figures 1A to F show SEM images of unetched and etched surfaces of the experimental materials. In unetched CEM specimens, the surface matrix contains numerous irregular needle-like crystals. Selective loss of matrix in the periphery of crystal structures in etched CEM specimens is visible. In unetched MTA specimens, the uncrystallized surface matrix contains globular irregular needle-like crystals and numerous canaliculi. In etched MTA specimens, selective loss of matrix around crystal structures has produced layered crystals with a honeycomb appearance. In unetched RMGI specimens, the crystals have sharp edges with no evidence of matrix loss. In etched RMGI specimens, the etching process has resulted in deepening of the matrix, loss of sharp edges and reduction in size of the crystals.

DISCUSSION

Composite resin restorations are required following pulp capping procedures in areas in which esthetics is of concern. Therefore, bonding between composite resin and the pulp



Figs 1A to F: Scanning electron micrographs of unetched (A-C) and acid-etched (D-F) specimens in the experimental subgroups A, D) CEM; B, E) MTA and; C, F) RMGI (Mag ×2000) capping biomaterial, has an important role in quality of fillings and treatment outcomes.[23] The most commonly used method for evaluation of adhesive properties of restorative materials is bond strength test.[23] Therefore, shear bond test was used in this study to evaluate the adhesive properties of two pulp capping biomaterials (CEM, MTA) and RMGI, with/without surface etching, to composite resin.

The results showed that the bond strength of composite resin to MTA and CEM pulp capping agents with/without surface etching were comparable, and were significantly weaker than RMGI. Furthermore, the failure modes in all of the specimens were cohesive, which is an indication of the low cohesive strength of CEM, MTA and RMGI compared to their high bond strength values.

The bond strength of RMGI to composite resin has been previously evaluated;[25] the results indicated cohesive failure of RMGI which is similar to our obtained results. This may be attributed to the presence of air bubbles inside RMGI, which act as stress concentration points.[26] Besides, the higher bond strength of composite resin to RMGI is attributed to the strong chemical bond between RMGI and composite resin.[27,28] Various factors are involved in the chemical bond of RMGI to composite resin, including: (i) unsaturated double bonds in the air-inhibited layer of RMGI, (ii) unpolymerized hydroxyethyl methacrylate (HEMA) on the surface of RMGI, which increases the wettability of the bonding agent, resulting in increased bond strength after polymerization, and (iii) unsaturated suspended methacrylate in the polyacid chain, which can form covalent bonds with composite resin ingredients during RMGI polymerization.[28]

The bond strength of pulp capping biomaterials (MTA and CEM) to composite resin depends on their chemical/physical characteristics. In the present study, the shear bond strength of composite resin to MTA and CEM was higher with acid etching compared to without acid etching, although the difference was not statistically significant. Recently, the effect of acid etching on MTA characteristics has been evaluated under SEM; it was reported that the surface gel-like amorphous structures and needle-like crystals are removed during acid etching,[29] which was in agreement with our findings. The selective removal of matrix from the periphery of crystals without significant loss of MTA and CEM results in a spongy appearance, which can produce an ideal surface for bonding with resin materials.[29]

MTA and CEM biomaterials are hydrophilic cements which set in the presence of moisture.[10] The main ingredients of MTA powder are Portland cement, Gypsum and bismuth oxide. However, it has been shown that CEM differs chemically from MTA; it has been shown that phosphorous is the major component of CEM,[11] whereas in MTA, this element is close to the threshold detection limit.[3,10,11] Hydroxyapatite precipitation was found on CEM, even stored in normal saline.[13] Recent studies have revealed that failures of the bond between gray MTA and dentin are usually cohesive inside MTA. However, as the length of the time interval between placement of MTA and the final restoration increased, the possibility of cohesive failure is reduced and the failure mode is more likely to be adhesive.[30] Researchers have shown that when CEM was used as a root-end filling material, the failure mode of fracture was cohesive in push-out bond strength tests.[31] The findings of this study reveal a favorable bond strength between pulp capping biomaterials (MTA and CEM) and composite resin. To accomplish a successful restorative procedure when two different materials are used, there should be an appropriate bond between the two materials.[32]Generally, the bond is acceptable when fracture occurs inside each material rather than in the bonded interface (i.e. cohesive rather than adhesive).[34]

The surface preparation technique did not have a significant effect on the shear bond strength of composite resin to experimental materials. In regards to etching/not etching the materials used in this study, there is a controversy in relation to RMGI and a lack of evidence exists for CEM and MTA. A study has shown that acid etching of RMGI decreases shear bond strength of composite resin bonded to its surface because acid etching removes the air-inhibited surface layer which has an important role in bonding to composite resin.[28] However, some other studies have not reported any significant differences in shear bond strength values of composite resin to etched and unetched RMGI,[25,33,34] which is consistent with the results of this study.

In the present study, interaction of the experimental material and the surface preparation method on shear bond strength of composite resin was evaluated, and the highest bond strength was recorded in the RMGI unetched subgroup. This may be attributed to the high cohesive strength of light-cured glass-ionomer, presence of a chemical bond between this cement and composite resin, and the presence of an air-inhibited surface layer which has an important role in establishing a bond to composite resin when RMGI surface is not etched.

It is suggested that the effect of different chemical and mechanical surface treatment methods on the bond strength of composite resin to CEM and MTA be evaluated in future studies.

For more insights and an improved perceptive of the adhesion mechanism of adhesive systems to MTA/CEM, more research studies are desirable.

CONCLUSION

Taking into account the limitation of this *in vitro* study, surface etching of MTA and CEM biomaterials are not necessary prior to composite resin restoration using total-etch adhesive resin during vital pulp therapy.

CLINICAL SIGNIFICANCE

When MTA and CEM biomaterials are used in vital pulp therapy, it is advisable to cover these materials with RMGI. In addition, if it is not possible to use RMGI, the surface etching of MTA and CEM biomaterials is not necessary prior to composite restoration using total-etch adhesive resin.

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