# In vitro comparison of shear bond strength of a flowable composite resin and a single-component glass-ionomer to three different pulp-capping agents

## Porównanie in vitro wytrzymałości na ścinanie wiązania płynnej żywicy kompozytowej i jednoskładnikowego szkło-jonomeru z trzema materiałami do pokrycia miazgi

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## Abstract

**Background.** Various materials are used for vital pulp capping and the bond strength of restorative materials to these pulp-capping agents significantly affects the success rate of vital pulp therapy.

**Objectives.** The aim of this study was to determine the shear bond strength of a flowable composite resin and a single-component glass-ionomer to mineral trioxide aggregate (MTA), calcium-enriched mixture (CEM) cement and Biodentine™ as pulp-capping agents.

**Material and methods.** Seventy-two cylindrical acrylic resin blocks, with a central hole 4 mm × 2 mm, were prepared. Mineral trioxide aggregate, CEM cement and Biodentine were placed in the cavities (n = 24 in each group) and incubated for 24 h. The blocks were subdivided into the composite resin and glass-ionomer subgroups. Cylindrical plastic molds, measuring 3 mm in height and diameter, were used to place the restorative materials on the samples. The shear bond strength test was performed at a strain rate of 1 mm/min in a universal testing machine. The samples were evaluated under a stereomicroscope at ×25 magnification for fracture modes. The data was analyzed with the one-way analysis of variance (ANOVA) and Tukey tests.

**Results.** The maximum and minimum mean shear bond strength values were recorded in the Biodentine–composite resin (4.77 MPa) and MTA–glass-ionomer (2.20 MPa) groups, respectively. There were significant differences in the mean shear bond strength values of MTA, CEM cement and Biodentine to the composite resin and glass-ionomer ($p < 0.001$).

**Conclusions.** A composite material may be preferable for definitive filling after pulp capping with Biodentine.

**Key words:** shear bond strength, mineral trioxide aggregate, composite resin, glass-ionomer cement, Biodentine

**Słowa kluczowe:** wytrzymałość wiązania na ścinanie, agregat trójtlenków mineralnych, żywica kompozytowa, cement szkło-jonomerowy, Biodentyna

## Introduction

Vital pulp therapy consists in placing a biocompatible material on the exposed pulp of the teeth with an open apex.[1] Various materials are used for pulp capping, including calcium hydroxide, mineral trioxide aggregate (MTA) and newer silicate-based cements, such as BioAggregate®, EndoSequence®, Biodentine™, etc.[2]

Mineral trioxide aggregate is a hydrophilic cement composed of calcium oxide, silica and bismuth oxide. Several successful clinical applications have been reported for MTA.[1] Long-term studies (over 3 years) have reported that the success rate of vital pulp therapy with MTA is higher than in the case of calcium hydroxide (78% vs 60%).[2] These favorable outcomes for direct pulp therapy with MTA have been confirmed in recent systematic reviews.[3] Despite the various reported advantages, MTA has also some disadvantages, including potential for discoloration, difficult handling, long setting time, high cost, unavailability of a solvent, and difficulty with its removal after setting.[4]

Biodentine is a new calcium silicate-based cement with high purity, which has drawn attention as a substitute for dentin in composite resin restorations, direct pulp capping and endodontic treatment.[5,6] Biodentine consists of tricalcium silicate, calcium carbonate (as a filler), zirconium oxide (as an opacifier), and a water-based liquid containing calcium chloride. Calcium chloride serves as a water-reducing agent and decreases the initial and final setting times. The incorporation of calcium chloride into the liquid not only results in the acceleration of the setting time of Biodentine, but also improves the handling properties and strength of the cement.[5] In previous studies, Biodentine has exhibited better sealing ability, higher compressive strength, shorter setting time, lower microleakage, better antimicrobial properties, less toxic effects, and better biocompatibility, bioactivity and biomineralization compared to MTA.[7]

Calcium-enriched mixture (CEM) cement is a newly introduced material in endodontics. It is a water-based cement with clinical properties similar to those of MTA; however, its chemical properties are different.[8] This new material is recommended for direct pulp capping and pulpotomy in deciduous and permanent molars.[9]

One of the most important issues in vital pulp therapy is the ability to seal the pulp-capping agents, because keeping them intact affects the prognosis of the therapeutic procedure. Composite resins are common restorative materials, especially in the esthetic zones. A proper bond between the composite resin and the pulp-capping agent can distribute stresses beyond the bonded area on the tooth surface, decrease microleakage and increase the strength of the remaining tooth structure.[10]

In cases with an insufficient amount of enamel around the access cavity, resin-modified glass-ionomer (RMGI) is considered a suitable restorative material for the reconstruction of the crowns of the teeth that have undergone pulp therapy.[11] Glass-ionomers bond to the tooth structure

chemically, exhibit proper bio-compatibility, no polymerization shrinkage and no free monomers, and have dimensional stability in the presence of moisture, as their advantages.[12] At present, newer types of glass-ionomer cements have been introduced to overcome the disadvantages of old cements, including sensitivity to water and low translucency, and preserve their advantages, such as fluoride release and adhesion, at the same time.[13] A proper bond between the restorative material and the pulp-capping agent results in the distribution of stresses on all bonded surfaces, ensures the vitality of the pulp and its sealing, and improves the prognosis of vital pulp therapy.[7,14]

Cantekin and Avci evaluated the shear bond strength of a methacrylate-based and a silorane-based composite resin and a glass-ionomer to MTA and Biodentine.[7] They reported that the highest bond strength was related to the methacrylate-based composite resin bonded to Biodentine.[7] Later on, Doozaneh et al. reported that the bond strength of a self-adhering flowable composite resin to CEM cement and MTA was higher than in the case of improved RMGI with an additional application of an adhesive.[15] Also, in 2018, Elmi et al. concluded that irrespective of the type of adhesive system, the shear bond strength of a composite resin to CEM cement is higher than that of RMGI.[16]

The purpose of the present in vitro study was to determine the shear bond strength of a composite resin and a single-component glass-ionomer to MTA, CEM cement and Biodentine as pulp-capping agents.

## Material and methods

A total of 72 cylindrical acrylic resin blocks (Acropars®; Marlic Medical Ind. Co., Tehran, Iran) were prepared for the purpose of this in vitro study. A cavity, measuring 4 mm in diameter and 2 mm in depth, was prepared at the center of each cylinder. Mineral trioxide aggregate (ProRoot® MTA; Dentsply Sirona Inc., York, USA), CEM cement (Yektazist Dandan, Tehran, Iran) and Biodentine (Septodont, Saint-Maur-des-Fossés, France) were used according to the manufacturers' instructions. Mineral trioxide aggregate was mixed at a powder-to-liquid ratio of 3:1.[4] The liquid and powder of CEM cement were mixed according to the manufacturer's instructions to achieve a proper consistency. Biodentine was prepared in an amalgamator by adding 5 drops of Biodentine liquid to the capsule containing its powder in 30 s. Then, the prepared materials were placed in the cavities at the center of the acrylic blocks.

The blocks were divided into 3 groups according to the material used: MTA, CEM cement and Biodentine (n = 24 in each group). The acrylic blocks were incubated at 37°C and 100% relative humidity for 24 h for the complete setting of the materials.[7] As the acid-etching procedure affected the compressive strength and surface microhardness, after 24 h, surface changes occurred, which enhanced bonding.[17] Then, each group was divided into 2 subgroups

– Grandio Flow® composite resin and Ionoseal® glass-ionomer (VOCO GmbH, Cuxhaven, Germany).

The blocks receiving the composite resin were acid-etched with 35% phosphoric acid for 15 s to avoid over-etching, which decreases the shear bond strength.[18] Afterward, they were rinsed with water for 30 s, followed by drying with an oil-free air stream for 5 s. At the next stage, the adhesive Solobond® M (VOCO GmbH) was applied on the specimen surfaces. It was applied twice and dried with an air flow for 5 s in order to evaporate its solvent. The next step was light-curing (LED D; Guilin Woodpecker Medical Instrument Co. Ltd., Guilin, China) for 15 s.

Cylindrical plastic molds, measuring 3 mm in diameter and height, were used to place the flowable composite resin.[19] The molds were filled with the Grandio Flow composite resin and placed on the prepared surfaces of the samples before setting, followed by light-curing for 20 s from the top, based on the manufacturer's instructions. Then, the plastic molds were gently detached from the composite resin molds, which was followed by light-curing for 20 s from the sides. Similar plastic molds were used for the Ionoseal glass-ionomer. The glass-ionomer was placed within the transparent molds put on the samples, followed by light-curing from the top for 20 s. Then, the plastic molds were gently separated from the glass-ionomer samples, which was followed by light curing from the sides for 20 s. Next, the samples were stored at 37°C and 100% relative humidity for 24 h.

Subsequently, the samples were transferred to a universal testing machine (Walter+Bai AG, Löhningen, Switzerland), equipped with a chisel-shaped head measuring 5 mm in width. A perpendicular force was applied at the restorative material–pulp-capping agent interface at a crosshead speed of 1 mm/min to detach the composite resin and glass-ionomer from the endodontic materials and to draw a graph. Before carrying out statistical analyses, the resultant data, recorded in N, was divided by the surface area of the samples (7.06 mm$^2$) in order to determine the bond strength in MPa. Finally, all samples were evaluated under a stereomicroscope (trinocular zoom stereo microscope SMP-200; HP Inc., Palo Alto, USA) at ×25 magnification to evaluate the fracture modes (cohesive, adhesive or mixed).

The data was analyzed with the one-way analysis of variance (ANOVA), *t*-test and post hoc Tukey tests.

The significance level was assumed at *p* < 0.05. The tables were drawn using the software IBM SPSS Statistics for Windows, v. 20 (IBM Corp., Armonk, USA).

## Results

As shown in Table 1, the maximum and minimum mean shear bond strength values were recorded in the Biodentine–composite resin (4.77 MPa) and MTA–glass-ionomer (2.20 MPa) groups, respectively. There were significant differences in the mean shear bond strength values of MTA, CEM cement and Biodentine to the composite resin and glass-ionomer (*p* < 0.001). Since the differences in the mean shear bond strength value of MTA, CEM cement and Biodentine to the composite resin and glass-ionomer were significant, the post hoc Tukey tests were used. The mean shear bond strength value of MTA to the composite resin was 3.19 MPa, which was significantly higher than those in the MTA–glass-ionomer and CEM cement–glass-ionomer groups (*p* < 0.001). On the other hand, the mean shear bond strength value of the MTA–composite resin group was lower than those in the CEM cement–composite resin and Biodentine–composite resin groups, which was statistically significant (*p* < 0.001). However, despite the lower mean shear bond strength value in the MTA–composite resin group compared to the Biodentine–glass-ionomer group, the difference was not statistically significant (*p* = 0.88).

Based on the data in Table 2, the shear bond strength values for the CEM cement–composite resin group was

Table 1. Mean shear bond strength values of MTA, CEM and Biodentine to composite resin and glass-ionomer

| Pulp-capping agent | Restorative material | Number | Mean bond strength [MPa] | *SD* |
|---|---|---|---|---|
| MTA | composite resin | 12 | 3.19 | 0.23 |
| | glass-ionomer | 12 | 2.20 | 0.27 |
| CEM cement | composite resin | 12 | 4.00 | 0.47 |
| | glass-ionomer | 12 | 2.36 | 0.39 |
| Biodentine | composite resin | 12 | 4.77 | 0.30 |
| | glass-ionomer | 12 | 3.35 | 0.38 |

*SD* – standard deviation; MTA – mineral trioxide aggregate; CEM – calcium-enriched mixture.

Table 2. Two-by-two comparisons of the shear bond strength value between the study groups

| Study groups | MTA –composite 3.19 | CEM cement –composite 4.00 | Biodentine –composite 4.77 | MTA –glass-ionomer 2.20 | CEM cement –glass-ionomer 2.36 | Biodentine –glass-ionomer 3.35 |
|---|---|---|---|---|---|---|
| MTA–composite | – | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 | *p* = 0.88 |
| CEM cement–composite | *p* < 0.001 | – | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 |
| Biodentine–composite | *p* < 0.001 | *p* < 0.001 | – | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 |
| MTA–glass-ionomer | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 | – | *p* = 0.87 | *p* < 0.001 |
| CEM cement–glass-ionomer | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 | *p* = 0.87 | – | *p* < 0.001 |
| Biodentine–glass-ionomer | *p* = 0.88 | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 | *p* < 0.001 | – |

higher compared to all other groups except for the Biodentine–composite resin group and the differences, whether higher or lower, were significant ($p < 0.001$). The Biodentine–composite resin group had a higher shear bond strength value than all other 5 groups and the difference was statistically significant ($p < 0.001$).

Also, the mean shear bond strength value in the MTA–glass-ionomer group was lower compared to other groups and this difference was not significant only with regard to the CEM cement–glass-ionomer group ($p = 0.87$); however, the difference was significant in the case of the other groups ($p < 0.001$).

The mean shear bond strength value in the CEM cement–glass-ionomer group was not significantly different only from that in the MTA–glass-ionomer group ($p < 0.87$); however, the difference with regard to other groups was significant ($p < 0.001$). The comparison of the mean shear bond strength values between the Biodentine–glass-ionomer and other groups showed no significant difference between this group and the MTA–composite resin group ($p = 0.88$); however, the differences between this group and other groups were significant ($p < 0.001$).

## Discussion

After vital pulp therapy, the treated tooth requires a suitable restoration. Recently, composite resins have been widely used for this purpose, but in some areas, where there is not enough enamel around the preparation site, RMGIs can be a good alternative.[20] The bond strength of restorative materials to pulp-capping agents plays a crucial role in the coronal sealing, and consequently the success of vital pulp therapy.[7] A proper bond between the restorative material and the pulp-capping agent also distributes stresses on the bonded surface area of dentin.[14]

Therefore, in the present study, the shear bond strength test was used to evaluate the adhesive properties of restorative materials (composite resin and glass-ionomer) to 3 pulp-capping agents (MTA, CEM cement and Biodentine). The shear bond strength of different restorative materials to MTA has been evaluated in previous studies.[11,15] However, only a few studies on the bond strength of different restorative materials to CEM cement and Biodentine are available.[11,15,16,21]

The results of the present study showed that the highest shear bond strength between the pulp-capping agent and the restorative material was in the Biodentine–composite resin group. Furthermore, Biodentine exhibited higher shear bond strength to the composite resin and glass-ionomer compared to that of MTA and CEM cement. This result is in agreement with Cengiz and Ulusoy's study.[22] Such a difference might be explained by the fact that, unlike MTA, in which only distilled water is used for setting, the liquid of Biodentine contains a mixture of distilled water, calcium chloride and a water-soluble monomer.[23]

Calcium chloride accelerates the setting reaction, and the water-soluble monomer serves as a water-reducing agent and decreases the amount of water in the material, increasing the strength of the material.[23,24]

In the present study, the surfaces of the pulp-capping agents were not polished; therefore, they had some indentations, resulting in the greater penetration of the composite resin and glass-ionomer. The use of a bonding agent in the composite resin groups could result in higher bond strength. Composite resin is a hydrophobic material and forms a better bond with surfaces that have low water content. The bonding agent used in the present study was of the total-etch type (Solobond® M; VOCO GmbH), which creates a better bond with drier surfaces. Considering the presence of a water-reducing agent in Biodentine, the water content of the material decreases, resulting in better conditions for bonding with composite resin.

The exact mechanism of bonding restorative materials to Biodentine has not been elucidated. Since Biodentine and MTA have a similar chemical structure, it is probable that their water absorption is also similar. Etchants, during shorter than usual times, might cause the selective elimination of the matrix around crystal structures, leading to successful bonding through micromechanical interlocking.[17]

The majority of studies on various adhesive systems applied on MTA have shown favorable shear bond strength obtained with the use of total-etch adhesive systems; it has been demonstrated that phosphoric acid creates deeper and more retentive microscopic pores compared to self-etch adhesive systems.[17] In the present study, the samples receiving composite resin were acid-etched with 35% phosphoric acid. Since no resin structure is present in MTA and CEM cement, it can be claimed that their bonding to restorative materials is completely mechanical.

Altunsoy et al. evaluated the shear bond strength of 2 different types of composite resin to MTA, CEM cement and Biodentine.[25] The results showed that the lowest bond strength was related to Biodentine–composite resin, contrary to the results of the present study. They used a self-adhesive flowable composite resin, without etching and dentin bonding. In addition, in their study, the specimen surfaces were polished with abrasive paper for 1 min, which resulted in a decrease in bond strength.[25]

Cantekin and Avci evaluated the shear bond strength of a methacrylate-based and a silorane-based composite resin as well as a glass-ionomer to MTA and Biodentine.[7] They reported that the highest bond strength was related to methacrylate-based composite resin bonded to Biodentine. The results of their study are consistent with those of the present study. It should be pointed out that in the study by Cantekin and Avci, contrary to the present study, the composite resins were packable; in the present study, a flowable composite resin was used. In addition, the glass-ionomer used in the study by Cantekin and Avci was a type of conventional self-cured glass-ionomer.[7]

Ajami et al. compared the shear bond strengths of composite resin and glass-ionomer to MTA, CEM cement and white MTA (WMTA) mixed with $Na_2HPO_4$ (NAMTA) as pulp-capping agents.[11] The highest bond strength was recorded in the NAMTA–composite resin group. It should be pointed out that some properties of NAMTA are different from those of MTA. The study carried out by Ajami et al. was different from the present one with regard to the methods and the materials used. In their study, all samples were sandblasted and a packable composite resin was used in association with a one-step self-etch adhesive system.[11]

Based on the results of the present study, the minimum shear bond strength value between the pulp-capping agent and the restorative material was recorded in the MTA–glass-ionomer group. When a restorative glass-ionomer is placed on MTA, one of the following reactions might occur:

– the $COO^-$ group in polyacrylic acid might react with calcium in MTA to produce calcium salts;
– the MTA hydrated silicate gel might be compressed by the glass-ionomer hydrated silicate gel to create byproducts.[26]

Given the high percentage of metallic oxides in MTA and the porous surface topography of MTA, it is expected that glass-ionomer will form a relatively strong bond with MTA.[27] The glass-ionomer used in the present study was Ionoseal, which is a single-component material. Since no etching or bonding were used in the glass-ionomer groups, possibly this material exhibited lower bond strength compared to the composite resin bonded to the pulp-capping agents. However, since in the present study, the surfaces of the pulp-capping agents were not polished and had some inherent porosity as well as considering the fact that the glass-ionomer used in the present study was flowable and exhibited proper adaptation to the surface, the restorative material formed a relatively strong bond to the pulp-capping agents.

In the study by Cantekin and Avci, in which the bond strength of Biodentine and MTA to a silorane-based composite resin, a methacrylate-based composite resin and a self-curing conventional glass-ionomer was evaluated, the lowest bond strength was recorded in the MTA–glass-ionomer group,[7] which is consistent with the results of the present study.

In the study by Ajami et al., in which the shear bond strength of composite resin and glass-ionomer to MTA, CEM cement and NAMTA was evaluated, the lowest bond strength values were recorded in the CEM cement–glass-ionomer group,[11] which does not agree with the results of the present study. It should be pointed out that such a discrepancy in the results might be attributed to the differences in the methodologies, including the use of sandblasting and the use of polyacrylic acid as a conditioner before the application of RMGI.

In the present study, the fracture mode in all samples was cohesive within the pulp-capping agents. Recent studies have demonstrated that the fracture mode between MTA and dentin was cohesive within MTA. However, the number of cohesive fractures decreased and the number of adhesive fractures increased over time.[28] In addition, the researchers pointed out that when CEM cement was used as a root-end filling material, the fracture mode was cohesive in the push-out test.[29] In order to carry out a successful restorative procedure with 2 different materials, there should be a proper bond between the 2 materials. Generally, a bond is favorable when the fracture occurs within the material rather than at the bonded interface (i.e., a cohesive fracture is better than adhesive failure).[19]

Since in the present study, the failure mode in all samples was cohesive within the pulp-capping agents, the results indicate a favorable bond between the restorative material and the pulp-capping agent. In the evaluation of failure modes, especially in the shear bond strength tests, one important issue should be taken into account. The tendency toward a cohesive fracture might be attributed to the uneven distribution of stresses within the bonded materials, resulting in early failure before the bonded surface is affected.[30] This is an inherent problem with the shear bond strength test, in which a lot of tensile force is applied to the area below the force application point, with simultaneous compressive stresses at the point opposite the force application point.[31]

## Conclusions

Within the limitations of an in vitro study, the composite resin exhibited stronger bonds to all the evaluated pulp-capping agents compared to the glass-ionomer. Among the pulp-capping materials, Biodentine exhibited a higher bond strength value to the flowable composite resin and the glass-ionomer. Therefore, to achieve a proper bond, the use of a composite resin on Biodentine is recommended.

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