

Comparative evaluations of shear bond strength of mineral trioxide aggregate, Biodentine, and calcium-enriched mixture to bulk-fill flowable composite using three different adhesive systems: An *in vitro* study

Asmat Fatima, Huma Iftekhar, Sharique Alam, Rajendra Kumar Tewari, Mukhtar Un Nisar Andrabi

Department of Conservative Dentistry and Endodontics, Dr. ZADC, AMU, Aligarh, Uttar Pradesh, India

Abstract

Objective: The objective of the study was to assess the shear bond strength of bulk-fill flowable composite resin smart dentin replacement plus when bonded to mineral trioxide aggregate (MTA)-angelus, biodentine, and calcium-enriched mixture (CEM) at two different aging periods (15 min and 72 h) using three distinct adhesive systems. In addition, the study identified the specific modes of failure (adhesive, cohesive, or mixed) using a stereomicroscope and scanning electron microscope.

Materials and Methods: One hundred and twenty-six cylindrical acrylic blocks used in the study were sorted into three groups based on the bioactive substance used to fill the 3-mm diameter and 3-mm high hole in the center of each block. The groups were MTA, Biodentine, and CEM. The specimens were then divided into subgroups based on the aging interval (15 min and 72 h) of the bioactive material and the adhesive system used (two-step total-etch, two-step self-etch [SE], and one-step SE) while bonding to the restorative bulk-fill flowable composite. The shear bond strength values were measured with a universal testing machine, and the data were analyzed using two-way and one-way analysis of variance, followed by a *post hoc* test. The specimens were assessed under stereomicroscope and scanning electron microscope to characterize the mode of bond failure (cohesive, adhesive, or mixed).

Results: The study showed that the type of adhesive system and the time of bonding affected the shear bond strength of bulk-fill composite to the pulp capping agents ($P < 0.05$). For MTA, the highest bond strength was observed with two-step SE group at 15 min (18.16 ± 2.97 MPa) ($P < 0.05$). CEM exhibited the highest bond strength with two-step SE group at 72 h intervals (8.77 ± 1.76) ($P < 0.05$). The highest bond strength for biodentine group was observed with total-etch group (8.54 ± 1.35 Mpa) and two-step SE (8.19 ± 1.94 Mpa) bonded at 72 h interval ($P < 0.05$). The majority of the samples in the MTA group (29/42) and CEM group (20/42) showed a cohesive fracture, whereas Biodentine group (22/42) had an adhesive fracture in most of its samples.

Conclusion: MTA demonstrated the highest bond strength with two-step SE group at 15 min, and CEM exhibited the highest bond strength with two-step SE groups at 72 h interval. For biodentine group, the type of adhesive used did not impact the bond strength values.

Keywords: Biodentine; bond strength calcium-enriched mixture; composite resin; mineral trioxide aggregate; self-etch adhesive; total-etch adhesive; vital pulp therapy

Address for correspondence:

Dr. Huma Iftekhar,
Dr. Ziauddin Ahmed Dental College and Hospital, AMU, Aligarh,
Uttar Pradesh, India.
E-mail: huma.iftekhar@yahoo.com

Date of submission : 09.04.2024

Review completed : 01.05.2024

Date of acceptance : 08.05.2024

Published : 08.07.2024

This is an open access journal, and articles are distributed under the terms of the Creative Commons Attribution-NonCommercial-ShareAlike 4.0 License, which allows others to remix, tweak, and build upon the work non-commercially, as long as appropriate credit is given and the new creations are licensed under the identical terms.

For reprints contact: WKHLRPMedknow_reprints@wolterskluwer.com

Access this article online	
Quick Response Code: 	Website: https://journals.lww.com/jcde
	DOI: 10.4103/JCDE.JCDE_192_24

How to cite this article: Fatima A, Iftekhar H, Alam S, Tewari RK, Andrabi MU. Comparative evaluations of shear bond strength of mineral trioxide aggregate, Biodentine, and calcium-enriched mixture to bulk-fill flowable composite using three different adhesive systems: An *in vitro* study. J Conserv Dent Endod 2024;27:706-13.

INTRODUCTION

Vital pulp therapy preserves healthy pulp tissue while treating damaged or infected pulp tissue. This can be accomplished in a number of ways, including pulp capping and pulpotomy. These techniques involve removing only the affected pulp tissue and applying a biocompatible pulp capping agent to stimulate the healing and regeneration of the remaining healthy pulp tissue.^[1] The success of vital pulp therapy largely depends on selecting the appropriate pulp capping materials, as well as procedural factors such as the size and nature (traumatic, mechanical, or carious) of the exposure, and potential microbial contamination at the site.^[2] Due to their bioactive and bio-interactive properties, bioactive calcium silicate-based materials such as mineral trioxide aggregate (MTA), Biodentine, and calcium-enriched mixture (CEM) have gained popularity as a pulp capping agent in vital pulp therapy.

MTA which was introduced in 1993 by Dr. Mahmoud Torabinejad, is a powder-based material composed of tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetracalcium aluminoferrite, and bismuth oxide that hardens and sets in the presence of moisture. It also contains trace amounts of other mineral oxides that alter its chemical and physical properties.^[3] MTA has several desirable properties such as biocompatibility, low solubility, excellent sealing ability, and hard-tissue induction. MTA has the ability to stimulate reparative dentinogenesis, inducing the formation of dentin at a faster rate and with superior structural quality. Although MTA has several advantages, it also has significant drawbacks, including difficulty in handling, prolonged setting time, causing potential discoloration of teeth, low compressive strength, low flexural strength, and low modulus of elasticity.^[4]

Varieties of alternative bioactive calcium silicate-based cement have been developed to address some of the limitations of MTA. One such substitute is Biodentine, which is designed as a dentin replacement material and consists of tricalcium silicate, calcium carbonate, and zirconium oxide in its powder form, calcium chloride, and a water-reducing agent as its liquid component.^[4,5] Biodentine addresses several limitations of MTA by offering faster setting time, improved handling, reduced risk of discoloration, and better mechanical properties such as compressive and flexural strength.^[4] Another recent biomaterial is CEM cement, which is a tooth-colored water-based cement that has similar clinical applications as MTA but has a different chemical composition. When used as a pulp capping agent, CEM cement has shown comparable outcomes to MTA.^[6] In addition, CEM cement has been shown to have antibacterial properties comparable to calcium hydroxide and superior to those of MTA and Portland cement.^[7] CEM has acceptable physical properties such as lower setting time, good flow, and less

film thickness, and it is biocompatible and able to induce hard-tissue formation in vital pulp therapy.^[8]

Following the removal of the infected tissue during vital pulp therapy, it is crucial to immediately apply a pulp capping agent and then place a restorative material on top to achieve an effective coronal seal. The success of the restoration depends not only on the bond strength of the pulp capping agents to the dentin but also on the quality of the bond between the pulp capping agents and the overlying restoration.^[4] An adhesive joint between the restorative material and pulp capping agent is necessary to distribute stress evenly across the entire bond area.^[9] Therefore, selecting the appropriate restorative material and adhesive system (self-etch [SE] or etch and rinse adhesives) is crucial to ensure the success of vital pulp therapy. This selection will have a direct impact on the bond strength, durability, and longevity of the restoration.^[10]

Kayahan *et al.* suggested that the acid-etching procedure before the placement of composite restoration has the potential to reduce the compressive strength and surface microhardness of MTA.^[11] In addition, the nature of the solvent used in adhesives (acetone, ethanol, or water) and their filler content can influence the bond strength of MTA to composite resin. A restorative material requiring low condensation force should be selected when placing a coronal restoration over MTA. Among the available options, the flowable composite resin is considered a suitable choice for use with calcium-silicate cement.^[3]

Flowable resin composites were introduced in the 1990s and have better-handling characteristics, such as fluid injectability and nonstickiness. In the past, resin composites were applied in layers of 2 mm to ensure they were completely cured. However, with the technological advancements, newer “bulk-fill” resin composites such as smart dentin replacement (SDR) can be placed with a thick bulk, instead of using the incremental placement technique, due to its higher translucency and greater depth of cure.^[4]

Several studies^[12-16] have investigated the bond strength of composite resin to MTA using different adhesive systems, whereas only a few studies^[4,17,18] have examined the shear bond strength of Biodentine and CEM to composite resin using adhesive systems at different time intervals. Therefore, the present study was conducted with the aim to evaluate the shear bond strength of MTA, Biodentine, and CEM at two separate aging intervals (15 min and 72 h) when bonded to bulk-fill flowable composite resin (SDR plus) using three different adhesive systems (two-step total-etch, two-step SE, and one-step SE). In addition, the study aimed to identify the specific modes of failure (adhesive, cohesive, or mixed) using a stereomicroscope and a scanning electron microscope.

MATERIALS AND METHODS

Sample preparation

The materials used in the present study are listed in Table 1. One hundred and twenty-six cylindrical acrylic blocks were prepared using autopolymerizing acrylic resin and in the center of the cylinders, a hole with a diameter of 3 mm and a 3-mm height was created. MTA and Biodentine were mixed with distilled water, whereas CEM was mixed with its designated liquid as per the manufacturer’s guidelines, in a Dappen Dish until they reached a thick, creamy consistency. The materials were loaded onto a carrier and inserted into the prepared cavities of the acrylic cylinders. They were then compacted using a condenser and flattened using a spatula [Figure 1].

Sample grouping and subgrouping

The acrylic blocks were divided into three groups based on the type of filling material used, namely, MTA, Biodentine, and CEM, with a sample size of 42 for each group. Then, specimens in each group were subdivided according to the adhesive system used and the time interval at which the bond strength was measured [Flowchart 1]. Once MTA, Biodentine, and CEM were placed in the cavities of the acrylic resin molds, all the samples were positioned vertically inside a plastic container. The container was then sealed, and half of the samples were subjected to immediate shear bond testing while the other half were incubated at 37°C for 72 h.

The specimens in each group at both aging time intervals (15 min and 72 h) were further divided according to the adhesive system applied (two-step total-etch, two-step SE, and one-step SE) over the bioactive material (MTA, Biodentine, CEM) for bonding it to the flowable composite; SDR was then placed over the capping agents and light-cured for 20 s using the LED unit (Unicorn Denmart, New Delhi, India).

Adhesive bonding protocol

Two-step etch and rinse

Fourteen samples, i.e., seven samples from each group (15 min and 72 h incubation) were etched with 35% phosphoric acid gel (N-etch etchant gel, Ivoclar Vivadent, Schaan, Liechtenstein) for 15 s, rinsed with water for 30 s, and then dried with oil-free air syringe for 5 s. In the next step, a clean applicator tip was used to apply Tetric N Bond (IvoclarVivadent, Schaan, Liechtenstein) to the prepared surfaces of the samples. Adhesive was applied in two coats based on the manufacturer’s instructions, left for 10 s, and gently air-thinned with compressed air for 5 s.

Two-step self-etch adhesive

Similarly, 14 samples (7 samples each after 15 min and 72 h incubation) were selected and two-step SE sixth-generation adhesive, Clearfil SE Bond (Kuraray Noritake Dental Inc., Okayama, Japan) was used. It was applied to the dentin using an applicator tip and was left in place for 30 s. Excess solvent was removed by air drying with a gentle stream of compressed air for 5 s.

One-step self-etch adhesive

While, in the remaining 14 samples (seven samples each after 15 min and 72 h incubation) of each group, seventh-generation all-in-one single step SE One Coat 7.0 (Coltene, Cuyahoga Falls, OH, USA) was applied for 20 s, dried for 5 s using a gentle stream of compressed air.

After adhesive application, all the samples were light-cured for 20 s using a light-curing unit at a light intensity of 1000 mW/cm².

Assessment of shear bond strength

Shear bond testing of the samples was determined by a universal testing machine (Shanta Engineering, India) using a knife edge blade at a cross-head speed of 1 mm/min. Bond

Table 1: Composition of calcium silicate-based cement, adhesives, and composite

Brand name	Product type	Composition
MTA-Angelus (ANGELUS Dental Solutions, Brazil)	Calcium silicate-based cement	Tri-calcium silicate, bismuth oxide, di-calcium silicate, tri-calcium aluminate, calcium-sulfate dihydrate or gypsum
Biodentine (Septodont, Saint-Maur-des-Fosses, Cedex, France)	Calcium silicate-based cement	Powder: Tricalcium silicate, di-calcium silicate, calcium carbonate and oxide, iron oxide, and zirconium oxide. Liquid: Calcium chloride and hydrosoluble polymer
CEM cement (Bionique Dent, Tehran, Iran)	Calcium silicate-based cement	Calcium-silicate, calcium-phosphate, calcium-oxide, calcium salts, barium-sulfate, and zirconium
Tetric N bond, Ivoclar Vivadent, Schaan, Liechtenstein	Fifth-generation adhesive (TE 2-step)	BIS-GMA, UDMA, dimethacrylates, HEMA, phosphonic acid acrylates, SiO ₂ , ethanol
Clearfil SE Bond, Kuraray Noritake Dental Inc., Okayama, Japan	Sixth-generation adhesive (SE 2-step)	Primer: 10-MDP, HEMA, camphorquinone, hydrophilic dimethacrylate, water. Bond: 10-MDP, Bis-GMA, HEMA, camphorquinone, hydrophobic dimethacrylate, N, N-diethanol p-toluidine, colloidal silica
One Coat 7.0, Coltène, Cuyahoga Falls, OH, USA	Seventh-generation adhesive (SE 1-step)	Primer: Water, HEMA, acrylamide sulfonic acid, glycerol mono-and dimethacrylate, methacrylized polyalkenoate. Bonding: HEMA, glycerol mono-and dimethacrylate, UDMA, methacrylized polyalkenoate, camphoroquinone
SDR composite-(SDR, Dentsply, Konstanz, Germany)	Flowable composite	Matrix: Polymerization modulator, dimethacrylate resins, UDMA. Filler: Ba-B-F-Al silicate glass, SiO ₂ , amorphous, Sr-Al silicate glass, TiO

MTA: Mineral trioxide aggregate, CEM: Calcium-enriched mixture, SDR: Smart dentin replacement, TE: Total-etch, SE: Self-etch

strength was calculated in Newtons and converted into MPa by dividing the peak load at failure with the specimen surface area.

Assessment of the type of bond failure

Failure modes were determined under stereomicroscope [Figure 1a-c] (Magnus MSZ-TR, Olympus Opto Systems, Noida, India) at $\times 10$ and scanning electron (JSM-6510LV, JEOL, USA) at $\times 22$ and $\times 2500$ [Figures 2-4]. The failure types were categorized as adhesive (two flat surfaces, showing failure of filling materials/pulp capping bond), cohesive (any deficiency in the pulp capping agent surface), and mixed (combination of interfacial separation and partial cohesive failure).



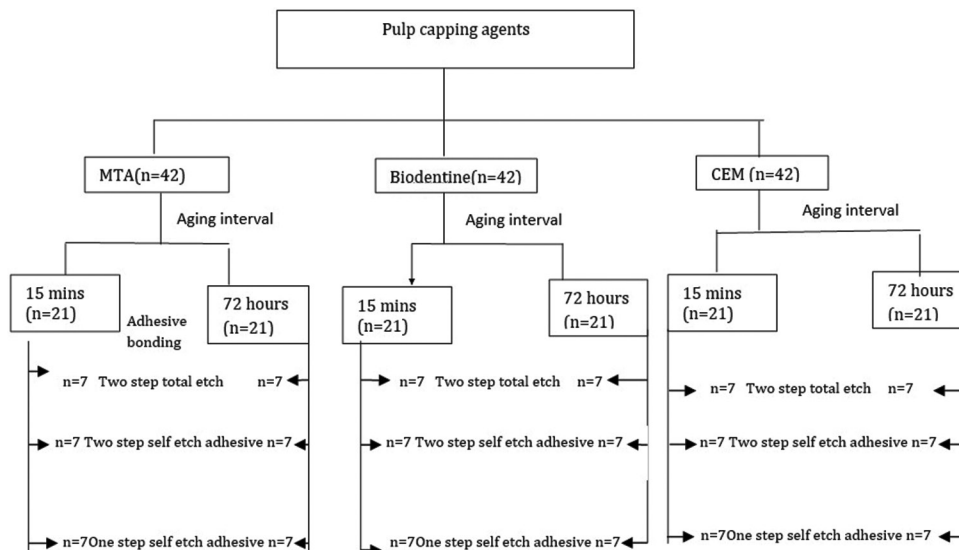
Figure 1: (a) Stereomicroscopic imaging showing cohesive failure, (b) Stereomicroscopic imaging showing adhesive failure, (c) Stereomicroscopic imaging showing mixed failure

Statistical analysis

The outcome variable in this study was shear bond strength (in MPa). Data were analyzed using paired *t*-test. Three-way analysis of variance (ANOVA) was used to study the differences of shear bond strength between three groups of MTA, Biodentine, and CEM, the two groups of time, and three groups of type of adhesive system used. Two-way ANOVA was used to analyze the differences in shear bond strength for two factors, time and etch for each group. One-way ANOVA was applied to shear bond strength values of the three groups, that is, MTA, Biodentine, and CEM and these were analyzed to test the differences between the three types of etch. The equality of variance assumption was assessed by Levene's test. If the equality of variance assumption was satisfied, then *post hoc* test was implemented through Scheffe's test. Otherwise, if the assumption was not satisfied, then Welch ANOVA was used with a Games Howell *post hoc* test. The frequency table was designed to assess the fracture modes of the adhesives bonded to MTA, Biodentine, and CEM. Since the data were not continuous but in a discrete form; Mann-Whitney *U*-test was applied to analyze the difference in the counts between the two-time categories. Kruskal-Wallis test was used to study the differences in the counts based on failure modes. The level of significance and confidence of the interval were set at 5% ($P < 0.05$) and 95%, respectively.

RESULTS

Two-way ANOVA was applied for each group which showed that both time and the adhesive system had a significant effect ($P < 0.05$) on shear bond strength values. The mean shear bond strength of MTA, Biodentine, and CEM are depicted in Graph 1. The maximum and minimum mean shear bond strength values were recorded in the MTA-two-step SE group at 15 min (18.16 ± 2.97 MPa)



Flowchart 1: Depicting grouping and subgrouping of the samples

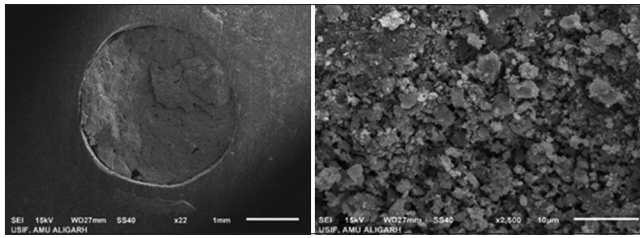


Figure 2: Scanning electron microscope imaging showing cohesive failure mode at $\times 22$ and $\times 2500$

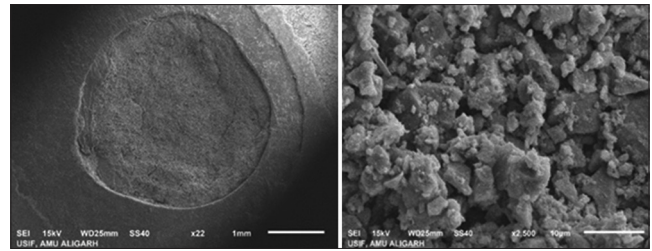


Figure 3: Scanning electron microscope imaging showing adhesive failure mode at $\times 22$ and $\times 2500$

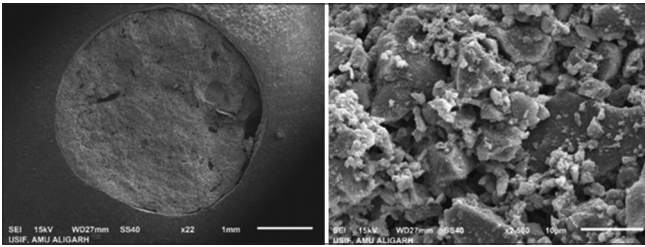
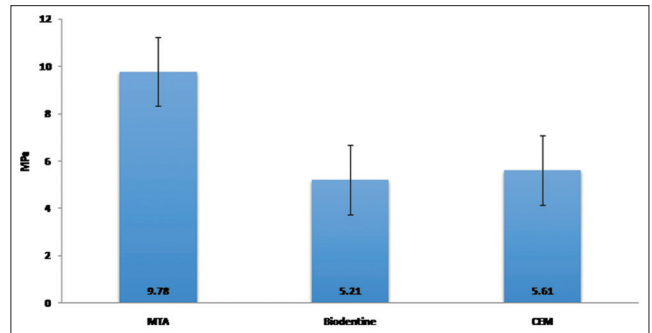


Figure 4: Scanning electron microscope imaging showing mixed failure mode at $\times 22$ and $\times 2500$



Graph 1: Mean shear bond of mineral trioxide aggregate, Biodentine, and calcium-enriched mixture. MTA: Mineral trioxide aggregate, CEM: Calcium-enriched mixture

and Biodentine-two-step total-etch group at 15 min (1.95 ± 0.76 MPa), respectively [Table 2 and Graph 2]. In the MTA group, there was a significant difference between the shear bond strength values of two-step total-etch and two-step SE ($P < 0.05$), whereas no significant differences were found between two-step total-etch and one-step SE as well as between two-step SE and one-step SE ($P > 0.05$). In the Biodentine group, no significant difference was seen in the shear bond strength values between the adhesive systems ($P > 0.05$). In the CEM group, it was found that all of the three pair-wise comparisons of adhesive systems for the shear bond strength were significant ($P < 0.05$). In MTA group, higher bond strength with all the adhesive systems was seen at 15 min, whereas Biodentine and CEM group showed higher bond strength at 72 h time interval.

Table 2: Mean and standard deviations of shear bond strength of bulk-fill flowable composite bonded with different adhesive systems to mineral trioxide aggregate, Biodentine, and calcium-enriched mixture (MPa)

Groups (n=42)	Adhesive system (n=14)	15 min (n=7), mean \pm SD	72 h (n=7), mean \pm SD	P
MTA	2-Step TE	10.52 \pm 1.52	5.53 \pm 2.46	0.002
	2-Step SE	18.16 \pm 2.97	5.12 \pm 1.55	<0.001
	1-Step SE	14.25 \pm 3.46	5.08 \pm 2.34	<0.001
Biodentine	2-Step TE	1.95 \pm 0.76	8.54 \pm 1.35	<0.001
	2-Step SE	2.84 \pm 0.86	8.19 \pm 1.94	<0.001
	1-Step SE	2.86 \pm 1.23	6.83 \pm 1.02	0.002
CEM	2-Step TE	3.04 \pm 1.93	4.42 \pm 1.55	0.221
	2-Step SE	2.09 \pm 0.79	8.77 \pm 1.76	<0.001
	1-Step SE	3.06 \pm 1.09	12.23 \pm 1.81	<0.001

MTA: Mineral trioxide aggregate, CEM: Calcium-enriched mixture, TE: Total-etch, SE: Self-etch, SD: Standard deviation

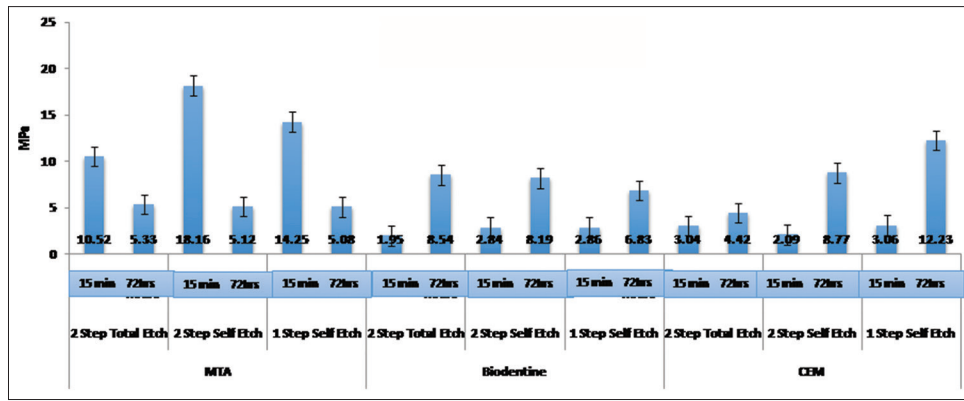
Table 3 shows the frequency of fracture modes of composite and adhesive bonded to MTA, Biodentine, and CEM. Fracture mode in the majority of the samples of MTA and CEM groups was cohesive, whereas the adhesive fracture was seen in most of the samples of the Biodentine group.

DISCUSSION

The bond strength between the biomaterial and composite resin is essential for ensuring the longevity and durability of the restoration.^[19] High shear bond strength between the bonded interface enhances adhesion and retention, ultimately reducing the risk of microleakage within the restoration.^[20] If the bond strength is weak, it can lead to microleakage, secondary caries, and ultimately, restoration failure. To achieve gap-free restoration margins, it is crucial to have bond strength values between 17 and 20 MPa.^[21] In this study, the shear bond strength between the pulp

capping biomaterial and bulk-fill flowable composite was found to range from 1.95 ± 0.76 MPa (for the Biodentine-two-step total-etch group) to 18.16 ± 2.97 MPa (for the MTA-two-step SE group after 15 min). While the effect of different restorative procedures on MTA's bond strength has been extensively studied,^[11,13-15,22] there is a scarcity of experimental data on CEM and Biodentine's bond strength to composite, with only a few studies published on the subject.^[4,12,23-25]

The present study found that regardless of the type of adhesive used, the bond strength between bulk-fill flowable composite and MTA was significantly higher (9.78 MPa) than the bond strength between the composite and



Graph 2: Mean shear bond strength of mineral trioxide aggregate, Biodentine, and calcium-enriched mixture with three different adhesive systems at 15 min and 72 h. MTA: Mineral trioxide aggregate, CEM: Calcium-enriched mixture

Table 3: Fracture modes of the adhesives bonded to materials

Group	Etch	Failure Modes	3 h	72 h
MTA	2 TE	Cohesive	5	4
		Adhesive	0	1
		Mixed	2	2
	2 SE	Cohesive	7	4
		Adhesive	0	2
		Mixed	0	1
	1 SE	Cohesive	5	4
		Adhesive	0	1
		Mixed	2	2
Biodentine	2TE	Cohesive	3	1
		Adhesive	3	5
		Mixed	1	1
	2 SE	Cohesive	1	4
		Adhesive	2	3
		Mixed	4	0
	1 SE	Cohesive	0	1
		Adhesive	4	5
		Mixed	3	1
CEM	2 TE	Cohesive	0	3
		Adhesive	6	2
		Mixed	1	2
	2 SE	Cohesive	2	6
		Adhesive	3	0
		Mixed	2	1
	1 SE	Cohesive	2	7
		Adhesive	5	0
		Mixed	0	0

Biodentine (5.21 MPa) or CEM (5.61 MPa). The possible explanation for the stronger bond between composite resin and MTA is that when the MTA surface is etched with phosphoric acid, it creates a honeycomb microstructural pattern by removing the matrix surrounding the crystals. This pattern may facilitate the formation of a strong micromechanical bond between the resin substrate and the MTA.^[26] This conclusion aligns with a previous study by Doozaneh *et al.*^[27] which also found stronger bond strength between composite and MTA than CEM. However, Ajami *et al.*^[28] found weaker bond strength between resin composite and MTA compared to CEM. In addition, Altunsoy *et al.*^[4] reported higher shear bond strength for MTA and CEM than for Biodentine, whereas

Tulumbaci *et al.*^[21] found higher bond strength for MTA than for Biodentine.

Various studies have investigated the impact of different adhesive systems on the bond strength of composite resin to MTA. Oskoe *et al.*^[29] found no significant difference in bond strength between etch and rinse and two-step SE adhesive systems. Atabek *et al.*^[12] and Tunç *et al.*^[16] reported that the two-step total-etch adhesive system had a significantly higher shear bond strength to MTA compared to the one-step SE adhesive system. Conversely, Neelakantan *et al.*^[15] demonstrated a higher bond strength of composite resin to MTA when using one-step SE adhesive systems. This can be attributed to factors such as pH or improved wetting ability due to the presence of solvents such as water and alcohol, leading to a decrease in contact angle. When the present study examined the impact of different adhesive systems, significant differences ($P < 0.001$) were observed in bond strengths between two-step total-etch and two-step SE, as well as two-step total-etch and one-step SE adhesives. The highest bond strength was noted for two-step SE adhesive (18.16 ± 2.97 MPa) system when the composite resin was placed over MTA at 15 min time intervals [Graph 2]. This result is consistent with previous research, which has shown that two-step SE adhesives have greater bond strengths than one-step SE adhesives.^[29-31] The reason for the high bond strength of two-step SE adhesives may be attributed to the fact that it involves the application of a separate, more hydrophobic adhesive resin after the hydrophilic SE primer. This increases the hydrophobicity of the interface, which leads to better sealing and improved bond durability.^[32]

In the present study, the highest shear bond strength of composite resin to Biodentine was obtained using two-step total-etch adhesive (8.54 ± 1.35 MPa). A possible explanation for the higher shear bond strength obtained with two-step total-etch adhesives could be that phosphoric acid used in the system has a low pH (0.1–0.4), which can provide greater microretention on the surface

of Biodentine than any SE adhesive.^[33,34] Elmi *et al.*^[19] reported that the type of bonding system had no effect on the bond strength of composite resin to CEM. Similar findings were reported by Jaberi-Ansari *et al.*^[17] In our study, CEM demonstrated the highest shear bond strength with one-step SE adhesive (12.23 ± 1.81 MPa).

It is important; however, to consider the results within the limitations that we did not observe a statistically significant difference in the bond strength of flowable composite to Biodentine or CEM when different adhesive systems were employed ($P < 0.05$).

The existing research does not recommend the ideal time interval for placing an adhesive composite restoration following the placement of calcium silicate cement-based pulp capping agent. There are only a few research studies that have investigated the optimal time gap between placing the calcium silicate cement and restoring the tooth with a composite restoration for achieving the highest bond strength.^[35]

In a study by Ajami *et al.*,^[20] composite restorations were placed immediately, at 2.45 h, and 3 days after mixing MTA. They found that the bond strength of composite resin was the lowest at the baseline, but it increased significantly 2.45 h after mixing MTA, with no significant changes in bond strength up to 3 days. The present study also found that higher bond strength of composite to MTA was observed at 15 min rather than 72 h, regardless of the adhesive system used. This could be due to the porous nature of MTA and the better penetration ability of dentin adhesives into the surface of MTA within this time interval. Neelakantan *et al.*^[15] and Tsujimoto *et al.*^[36] also found the highest shear bond strength values when the composite restoration was placed immediately over the MTA. However, in contrast to the findings of the present study, Atabek *et al.*^[12] placed composite over MTA after 4, 24, 48, 72, and 96 h and found that the highest bond strength was obtained at 48 h. They concluded that it is better to postpone the restorative procedures for at least 96 h after mixing MTA to allow the material to achieve its optimum physical properties.

The present study found that regardless of the adhesive systems used, the bond strength of Biodentine was higher at 72 h than at 15 min. This may be because Biodentine is a porous material that needs time for the hydrated calcium silicate gel to crystallize and achieve sufficient bulk strength to withstand the stresses during polymerization.^[37] There were statistically significant differences ($P < 0.001$) in the CEM group at both time intervals (15 min and 72 h) for the two-step SE and one-step SE groups. However, no significant difference ($P > 0.05$) was observed in the two-step total-etch adhesive group.

In the present study, we observed that cohesive bond failure was the dominant mode of bond failure in the MTA

and CEM group samples, whereas adhesive failure was seen in the majority of Biodentine group samples. These results are in line with previous studies conducted by Vanderweele *et al.*^[38] and Elmi *et al.*^[19] which reported cohesive failure in gray MTA and CEM, respectively. However, there is limited research available on the type of bond failure that occurs with Biodentine as a pulp-capping agent, with Odabas *et al.*^[39] reporting more cohesive fractures, and Deepa *et al.*^[9] reporting a mix of cohesive and adhesive fractures. Vanderweele *et al.* observed that as the time interval between the placement of pulp-capping biomaterial and definitive restoration increased, the likelihood of cohesive failure decreased and the incidence of adhesive failure increased.^[38]

It is desirable for bond failure to occur inside the material rather than at the bonded contact. (Cohesive fracture is preferred over adhesive failure).^[39] A bond is often preferred when the fracture occurs inside the material rather than at the bonded contact (i.e., a cohesive fracture is preferable to adhesive failure).^[40]

In the present study, we observed that the samples with higher bond strength showed cohesive and mixed failure modes under stereomicroscope. The majority of samples in this study displayed cohesive failure inside the pulp-capping agents, indicating a favorable bond between the restorative material and the pulp-capping agent.

CONCLUSION

Within the limitation of this *in vitro* study, it was concluded that

1. Both the time of bonding and type of adhesive affected the shear bond strength of bulk-fill composite to pulp capping agent
2. For MTA highest bond strength was observed with two-step SE group at 15 min
3. CEM exhibited the highest shear bond strength with two-step SE group at 72 h
4. Highest bond strength for Biodentine group was observed with total-etch group at 72 h
5. Fracture mode in the majority of samples of MTA and CEM was cohesive, whereas adhesive fracture was seen in most of the samples of Biodentine.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

REFERENCES

1. Nowicka A, Lipski M, Parafiniuk M, Sporniak-Tutak K, Lichota D, Kosierkiewicz A, *et al.* Response of human dental pulp capped with Biodentine and mineral trioxide aggregate. *J Endod* 2013;39:743-7.

2. Aeinehchi M, Eslami B, Ghanbariha M, Saffar AS. Mineral trioxide aggregate (MTA) and calcium hydroxide as pulp-capping agents in human teeth: A preliminary report. *Int Endod J* 2003;36:225-31.
3. Yesilyurt C, Ceyhanli KT, Kedicli Alp C, Yildirim T, Tasdemir T. *In vitro* bonding effectiveness of new self-adhering flowable composite to calcium silicate-based material. *Dent Mater J* 2014;33:319-24.
4. Altunsoy M, Tanriver M, Ok E, Kucukyilmaz E. Shear bond strength of a self-adhering flowable composite and a flowable base composite to mineral trioxide aggregate, calcium-enriched mixture cement, and Biodentine. *J Endod* 2015;41:1691-5.
5. Camilleri J, Sorrentino F, Damidot D. Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, Biodentine and MTA Angelus. *Dent Mater* 2013;29:580-93.
6. Parameswaran M, Vanaja Madanan K, Kumar Maroli R, Raghunathan D. Efficacy of calcium enriched mixture cement, mineral trioxide aggregate and calcium hydroxide used as direct pulp capping agents in deep carious lesions – A randomised clinical trial. *Eur Endod J* 2023;8:253-61.
7. Utneja S, Nawal RR, Talwar S, Verma M. Current perspectives of bio-ceramic technology in endodontics: Calcium enriched mixture cement – Review of its composition, properties and applications. *Restor Dent Endod* 2015;40:1-13.
8. Asgary S, Eghbal MJ, Parirokh M. Sealing ability of a novel endodontic cement as a root-end filling material. *J Biomed Mater Res A* 2008;87:706-9.
9. Deepa VL, Dhamaraju B, Bollu IP, Balaji TS. Shear bond strength evaluation of resin composite bonded to three different liners: TheraCal LC, Biodentine, and resin-modified glass ionomer cement using universal adhesive: An *in vitro* study. *J Conserv Dent* 2016;19:166-70.
10. Nekoofar MH, Motevasselian F, Mirzaei M, Yassini E, Pouyanfar H, Dummer PM. The micro-shear bond strength of various resinous restorative materials to aged Biodentine. *Iran Endod J* 2018;13:356-61.
11. Kayahan MB, Nekoofar MH, Kazandağ M, Canpolat C, Malkondu O, Kaptan F, *et al.* Effect of acid-etching procedure on selected physical properties of mineral trioxide aggregate. *Int Endod J* 2009;42:1004-14.
12. Atabek D, Sillelioğlu H, Olmez A. Bond strength of adhesive systems to mineral trioxide aggregate with different time intervals. *J Endod* 2012;38:1288-92.
13. Yelamali S, Patil AC. "Evaluation of shear bond strength of a composite resin to white mineral trioxide aggregate with three different bonding systems" – An *in vitro* analysis. *J Clin Exp Dent* 2016;8:e273-7.
14. Bayrak S, Tunç ES, Saroğlu I, Eğilmez T. Shear bond strengths of different adhesive systems to white mineral trioxide aggregate. *Dent Mater J* 2009;28:62-7.
15. Neelakantan P, Grotra D, Subbarao CV, Garcia-Godoy F. The shear bond strength of resin-based composite to white mineral trioxide aggregate. *J Am Dent Assoc* 2012;143:e40-5.
16. Tunç ES, Sönmez IS, Bayrak S, Eğilmez T. The evaluation of bond strength of a composite and a compomer to white mineral trioxide aggregate with two different bonding systems. *J Endod* 2008;34:603-5.
17. Jaberi-Ansari Z, Mahdilou M, Ahmadyar M, Asgary S. Bond strength of composite resin to pulp capping biomaterials after application of three different bonding systems. *J Dent Res Dent Clin Dent Prospects* 2013;7:152-6.
18. Kudva A, Raghunath A, S Nair PM, Shetty HK, D'Costa VF, Jayaprakash K. Comparative evaluation of shear bond strength of a bioactive material to composite resin using three different universal bonding agents: An *in vitro* study. *J Conserv Dent* 2022;25:54-7.
19. Elmi M, Ehsani M, Esmaeili B, Khafri S. Comparison of bond strength of a composite resin with two different adhesive systems and a resin modified glass ionomer to calcium enriched mixture. *J Conserv Dent* 2018;21:369-72.
20. Ajami AA, Bahari M, Hassanpour-Kashani A, Abed-Kahnamoui M, Savadi-Oskoei A, Azadi-Oskoei F. Shear bond strengths of composite resin and giomer to mineral trioxide aggregate at different time intervals. *J Clin Exp Dent* 2017;9:e906-11.
21. Tulumbaci F, Almaz ME, Arıkan V, Mutluay MS. Shear bond strength of different restorative materials to mineral trioxide aggregate and Biodentine. *J Conserv Dent* 2017;20:292-6.
22. Davidson CL, de Gee AJ, Feilzer A. The competition between the composite-dentin bond strength and the polymerization contraction stress. *J Dent Res* 1984;63:1396-9.
23. Salama F, Alahaidib F, Alshamrani M, Allam R. Bond strength of resin modified glass ionomer cement and resin composite to four pulp capping biomaterials. *IOSR J Dent Med Sci* 2017;16:68-75.
24. Sulwińska M, Szczesio A, Bołtacz-Rzepkowska E. Bond strength of a resin composite to MTA at various time intervals and with different adhesive strategies. *Dent Med Probl* 2017;54:155-60.
25. Cantekin K, Avci S. Evaluation of shear bond strength of two resin-based composites and glass ionomer cement to pure tricalcium silicate-based cement (Biodentine®). *J Appl Oral Sci* 2014;22:302-6.
26. Tyagi N, Chaman C, Tyagi SP, Singh UP, Sharma A. The shear bond strength of MTA with three different types of adhesive systems: An *in vitro* study. *J Conserv Dent* 2016;19:130-3.
27. Doozaneh M, Kooheima F, Firouzmandi M, Abbasiyan F. Shear bond strength of self-adhering flowable composite and resin-modified glass ionomer to two pulp capping materials. *Iran Endod J* 2017;12:103-7.
28. Ajami AA, Jafari Navimipour E, Savadi Oskoei S, Abed Kahnamoui M, Lotfi M, Daneshpooy M. Comparison of shear bond strength of resin-modified glass ionomer and composite resin to three pulp capping agents. *J Dent Res Dent Clin Dent Prospects* 2013;7:164-8.
29. Oskoei SS, Kimyai S, Bahari M, Motahari P, Eghbal MJ, Asgary S. Comparison of shear bond strength of calcium-enriched mixture cement and mineral trioxide aggregate to composite resin. *J Contemp Dent Pract* 2011;12:457-62.
30. Bouillaguet S, Gysi P, Wataha JC, Ciucchi B, Cattani M, Godin C, *et al.* Bond strength of composite to dentin using conventional, one-step, and self-etching adhesive systems. *J Dent* 2001;29:55-61.
31. Kaaden C, Powers JM, Friedl KH, Schmalz G. Bond strength of self-etching adhesives to dental hard tissues. *Clin Oral Investig* 2002;6:155-60.
32. Inoue S, Vargas MA, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, *et al.* Microtensile bond strength of eleven contemporary adhesives to enamel. *Am J Dent* 2003;16:329-34.
33. Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, *et al.* Relationship between bond-strength tests and clinical outcomes. *Dent Mater* 2010;26:e100-21.
34. Pashley DH, Tay FR, Breschi L, Tjäderhane L, Carvalho RM, Carrilho M, *et al.* State of the art etch-and-rinse adhesives. *Dent Mater* 2011;27:1-16.
35. Ha HT. The effect of the maturation time of calcium silicate-based cement (Biodentine™) on resin bonding: An *in vitro* study. *Appl Adhes Sci* 2019;7:1-13.
36. Tsujimoto M, Tsujimoto Y, Ookubo A, Shiraishi T, Watanabe I, Yamada S, *et al.* Timing for composite resin placement on mineral trioxide aggregate. *J Endod* 2013;39:1167-70.
37. Bachoo IK, Seymour D, Brunton P. A biocompatible and bioactive replacement for dentine: Is this a reality? The properties and uses of a novel calcium-based cement. *Br Dent J* 2013;214:E5.
38. Vanderweele RA, Schwartz SA, Beeson TJ. Effect of blood contamination on retention characteristics of MTA when mixed with different liquids. *J Endod* 2006;32:421-4.
39. Odabaş ME, Bani M, Tiralı RE. Shear bond strengths of different adhesive systems to Biodentine. *ScientificWorldJournal* 2013;2013:626103.
40. Zarean P, Roozbeh R, Zarean P, Jahromi MZ, Broujeni PM. *In vitro* comparison of shear bond strength of a flowable composite resin and a single-component glass-ionomer to three different pulp-capping agents. *Dent Med Probl* 2019;56:239-44.