ORIGINAL RESEARCH

Comparative Evaluation of Shear Bond Strength of Tricalcium Silicate-based Materials to Composite Resin with Two Different Adhesive Systems: An *In Vitro* Study

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Abstract

Background: Establishing a strong bond between the pulp capping agent and the restorative material is crucial to the success of the procedure. Without this bond, there is a risk of bacterial infiltration into the pulp, leading to treatment failure. In the past, calcium hydroxide was commonly used for such treatments, but it faced challenges, including poor adhesion to dentin, dissolution over time, and the development of multiple tunnel defects. Mineral trioxide aggregate (MTA), introduced to dentistry in 1993, offered an alternative but came with drawbacks like challenging handling and extended setting times. However, in recent times, several new calcium silicate-based materials have emerged to address MTA's limitations. Two notable examples are Biodentine and MTA Plus. Biodentine, for instance, exhibits excellent sealing ability, while MTA Plus distinguishes itself with a finer particle size compared to traditional MTA. These innovative materials offer promising solutions to enhance the efficacy of pulp capping procedures.

Aim: Therefore, in this research, we conducted a comparative analysis of the shear bond strength (SBS) between composite resin and three materials—MTA, MTA Plus, and Biodentine. We examined the effects of applying two distinct adhesive systems in order to evaluate their influence on the bond strength.

Materials and methods: A total of 60 acrylic blocks were evenly distributed into three groups, each containing 20 blocks—group I received Biodentine, group II was assigned MTA, and group III received MTA Plus. The respective test materials were compacted into the holes within the blocks. Following this, the samples were incubated for a period of 72 hours. Subsequently, the samples were divided into two subgroups, each consisting of 10 blocks—the self-etch and the total-etch subgroup. The SBS values were then carefully measured for analysis.

Result: The SBS of the Biodentine group demonstrated a significantly higher value when compared to the other groups. It's worth noting that when the self-etch adhesive system was employed, the SBS of all the groups experienced a significant reduction.

Conclusion: Biodentine cement proves to be an effective choice for pulp capping procedures, regardless of the specific adhesive system employed. Notably, the total-etch adhesive system consistently yields higher bond strength when compared to the self-etch adhesive system.

Keywords: Biodentine, Mineral trioxide aggregate, Mineral trioxide aggregate Plus, Self-etch adhesive, Shear bond strength, Total-etch adhesive.

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Introduction

To effectively treat deep caries, therapeutic approaches should aim to maintain pulp vitality, facilitating the formation of a dentin bridge.[1] In the absence of a proper seal between the restorative material and the pulp capping agent, bacteria can infiltrate the pulp, ultimately resulting in the failure of the pulp capping procedure.[2]

Calcium hydroxide has traditionally served as the gold standard in vital pulp therapy as it has been observed to promote the formation of reparative dentin while also possessing additional antibacterial properties.[3] Nevertheless, calcium hydroxide, despite its beneficial properties, is not without its limitations. Drawbacks such as inducing pulp cell inflammation, necrosis, and the potential for defects in dentin bridges, along with gradual dissolution over time, have contributed to treatment failures.[4]

Recently, two novel tricalcium silicate-based materials have emerged in the field—Biodentine by Septodont in Saint-Maur-des-Fossés, France and Mineral trioxide aggregate (MTA) Plus by Prevest DenPro in Jammu city, India. Notably, MTA Plus sets itself apart with finer particle size, with approximately 50% of its particles being finer than 1 µm. Additionally, it boasts improved resistance to washout, enhancing its overall performance and reliability compared to other commercially available MTA versions.[5] MTA has been observed to be sensitive to local environmental variations, which can have an adverse impact on its setting and hydration processes.[6] MTA Plus exhibits the ability to create a calcium reservoir and establish a more alkaline environment when compared to ProRoot MTA. This difference can be attributed to the finer calcium silicate powder used in MTA Plus, which may account for its higher levels of ion release, water sorption, porosity, and solubility in comparison to ProRoot MTA.[7]

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Biodentine, a calcium silicate-based dental cement produced by Septodent in Saint-Maur-des-Fossés, Cedex, France is increasingly being employed as a pulp capping material in dental procedures. Biodentine offers several advantages, including excellent marginal adaptation, enhanced physical properties, a shorter setting time (just 10 minutes), and superior bioactivity when compared to MTA.[8,9] Furthermore, Biodentine demonstrates enhanced antibacterial activity in comparison to MTA, and it also exhibits low cytotoxicity, making it a favorable choice for various dental applications.[10]

Resin composites are commonly employed in restorative dentistry, but they cannot be applied directly over freshly mixed MTA due to the adverse effects of phosphoric acid. However, there have been claims that resin composites can be successfully placed over set Biodentine® after a brief waiting period of just 12 minutes. This approach holds the potential to enable a single-visit treatment modality, streamlining the restoration process for patients.[11] The objective of the current study was to assess the bond strength between composite resin and three distinct tricalcium silicate-based materials, employing two distinct bonding systems: a total-etch approach and a self-etch technique.

Materials and Methods

This *in vitro* study was carried out within the Department of Pedodontics & Preventive Dentistry, following the acquisition of ethical clearance. The research focused on assessing the shear bond strength (SBS) of tricalcium silicate-based materials to the composite resin while utilizing two distinct adhesive systems.

Armamentarium Profile

Instruments

- Agate spatula.
- Teflon-coated instrument.
- Polyvinyl chloride (PVC) pipe 6 mm diameter.
- Polyvinyl chloride (PVC) pipe 3 mm diameter.
- Bard Parker (BP) handle with blade.
- Applicator tip.
- Glass slab.

Materials

Tricalcium silicate-based materials:

- ProRoot MTA (Dentsply Tulsa Dental, Tulsa, Oklahoma).
- MTA Plus (Prevest DenPro, Jammu, India).

Biodentine (Septodont: Saint-Maur-des-Fossés, France) (Table 1).
Restorative material:

- Nano-hybrid composite (Kerr Herculite Précis).

Bonding agent:

- OptiBond™ All-In-One (Kerr Corporation, United States of America).
- OptiBond™ Solo Plus (Kerr Corporation, United States of America).

Etchant:

- Scotchbond™ Multipurpose Etchant (3M ESPE).

Equipment

- Light-cure unit.
- Micromotor with straight handpiece.
- Instron 10 kN, Taiwan.

Table 1: Composition of MTA and Biodentine used in this study

VPT material	*Composition*	*Manufacturer*
ProRoot MTA	Dicalcium silicate Tricalcium silicate Tetracalcium aluminate ferrite Bismuth oxide	Dentsply Tulsa Dental, Tulsa, Oklahoma
Biodentine	Dicalcium silicate Tricalcium silicate Tetracalcium aluminate ferrite Zirconium oxide Calcium chloride	Septodent, Saint-Maur-des-Fossés, Cedex, France
MTA Plus	Dicalcium silicate Tricalcium silicate Tetracalcium aluminate ferrite Bismuth oxide	Prevest DenPro, Jammu, India

VPT, vital pulp therapy

Methodology

The study involved 60 cylindrical acrylic blocks, each of which had a hole measuring 3 mm in diameter and 1.5 mm in height prepared within them. Subsequently, the samples were evenly divided among the following groups:

- Group I: (MTA + total-etch)—composite resin bonded to MTA using a total-etch adhesive.
- Group II: (Biodentine + total-etch)—composite resin bonded to Biodentine using total-etch adhesive.
- Group III: (MTA Plus + total-etch)—composite resin bonded to MTA Plus using total-etch adhesive.
- Group IV: (MTA + self-etch)—composite resin bonded to MTA using self-etch adhesive.
- Group V: (Biodentine + self-etch)—composite resin bonded to Biodentine using total etch adhesive.
- Group VI: (MTA Plus + self-etch)—composite resin bonded to MTA Plus using self-etch adhesive.

Shear bond strength (SBS) values were quantified using a universal testing machine, and the obtained values were subjected to statistical analysis for further evaluation.

Sample Preparation

A PVC pipe with a 6 mm internal diameter was divided into 60 individual pieces, each measuring 10 mm in length. Each of these cut pieces was subsequently filled with acrylic resin and allowed to polymerize. After the polymerization process, the PVC pipe was carefully cut away, leaving behind acrylic blocks. These acrylic blocks were then prepared by creating a hole with a 3 mm diameter and a depth of 1.5 mm using a bur.

All acrylic blocks were equally divided into three groups ($n = 20$) and the following test materials were used.

Biodentine (Septodont, Saint-Maur-des-Fossés, France).

Mineral trioxide aggregate (MTA) (Dentsply Tulsa Dental, Tulsa, Oklahoma).

Mineral trioxide aggregate (MTA) Plus (Prevest DenPro, Jammu, India).

Biodentine liquid, sourced from a single-dose container, was discharged into a capsule containing the powder component. Subsequently, the contents were mixed for 30 seconds at a rotational speed of 4000–4200 rpm.

Mineral trioxide aggregate (MTA) was manually mixed with sterile water, following the manufacturer's instructions, at a powder-to-liquid ratio of 3:1.

Mineral trioxide aggregate (MTA) Plus was manually mixed with a salt-free water-soluble polymer gel, following the manufacturer's instructions.

The tested materials (MTA, MTA Plus, and Biodentine) were carefully placed incrementally into the prepared hole within the acrylic cylinder and condensed until they reached the level of the outer surface of the cylinder. Subsequently, the samples of each material group were individually wrapped in a dampened gauge, then transferred to an incubator and kept at 37°C with 100% humidity for a duration of 72 hours.

Following this incubation period, the samples within each group were further divided into two subgroups, with each subgroup containing 10 samples.

Group I: (MTA + total-etch).

Group II: (Biodentine + total-etch).

Group III: (MTA Plus + total-etch).

Group IV: (MTA + self-etch).

Group V: (Biodentine + self-etch).

Group VI: (MTA Plus + self-etch).

In the total-etch groups, Scotchbond™ Multipurpose Etchant (3M ESPE) was applied to the samples for a duration of 30 seconds. Subsequently, the samples were rinsed with normal saline and then air-dried. Following this, the OptiBond™ Solo Plus (Kerr Corporation, United States of America) bonding agent was applied using a micro applicator and cured for 60 seconds.

For the self-etch groups, OptiBond™ All-In-One (Kerr Corporation, United States of America) self-etch bonding agent was applied to the samples using a micro applicator. The bonding agent was then cured for 30 seconds.

A Teflon mold with an internal diameter of 3 mm and a height of 2 mm was positioned over the testing material. This mold was subsequently filled with composite resin, specifically the Kerr Herculite Precis monohybrid composite. The resin was then cured for 20 seconds from the top and 40 seconds from the lateral sides using appropriate curing equipment. This standardized method was consistently applied to all the samples. Once the curing process was completed, the mold was carefully removed from the cured composite.

The SBS values were determined using a universal testing machine, specifically the Instron 10 kN from Taiwan.

Results

The summarized data was effectively presented using tables and graphs shown in Table 2 and Figure 1. Prior to analysis, the data was assessed for normal distribution using the Shapiro–Wilk *W* test, (Table 3 and Fig. 2) and the results indicated that the data followed a normal distribution (p-value greater than 0.05). As a result, parametric tests, specifically the one-way analysis of variance (ANOVA), were employed for the analysis. The level of statistical significance was established at a p-value < 0.05.

Following a positive outcome from the N-ANOVA test (where the p-value was less than the chosen significance level), *post hoc* Tukey's test was employed for the pairwise comparison of

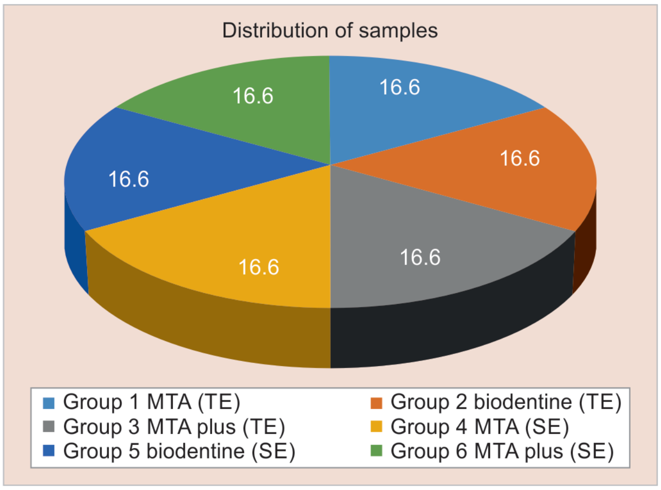


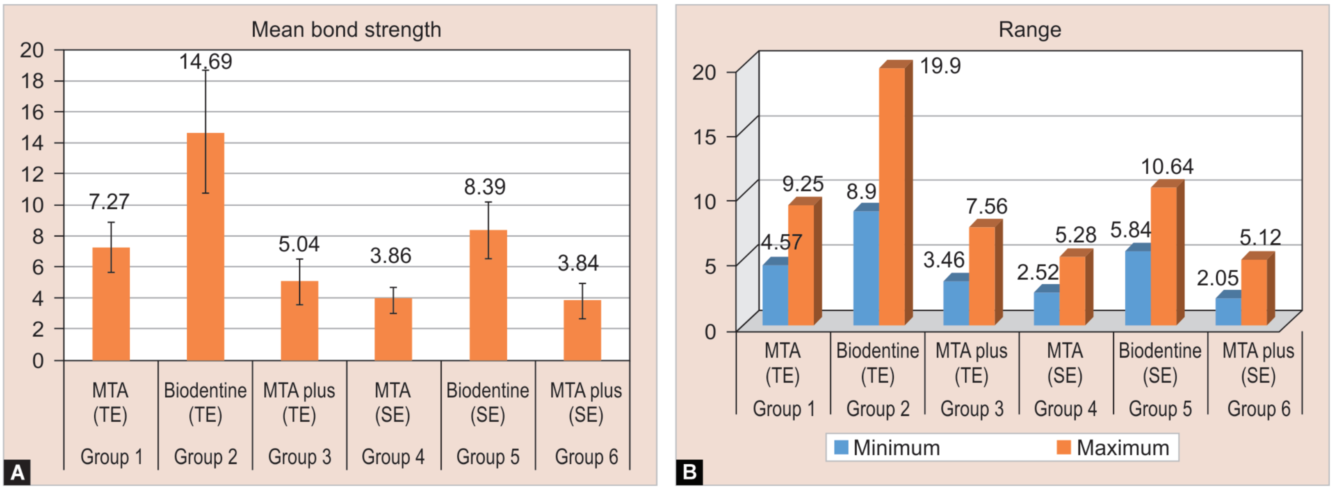
Fig. 1: Distribution of samples

Table 2: Distribution of samples

			N	%
Group I	MTA	Total-etch	10	16.6
Group II	Biodentine	Total-etch	10	16.6
Group III	MTA Plus	Total-etch	10	16.6
Group IV	MTA	Self-etch	10	16.6
Group V	Biodentine	Self-etch	10	16.6
Group VI	MTA Plus	Self-etch	10	16.6
Total			60	100

Table 3: Descriptives of the SBS among six groups

			Mean	*SD*	*Minimum*	*Maximum*
Group I	MTA	Total-etch	7.27	1.64	4.57	9.25
Group II	Biodentine	Total-etch	14.69	3.95	8.90	19.90
Group III	MTA Plus	Total-etch	5.04	1.50	3.46	7.56
Group IV	MTA	Self-etch	3.86	0.88	2.52	5.28
Group V	Biodentine	Self-etch	8.39	1.85	5.84	10.64
Group VI	MTA Plus	Self-etch	3.84	1.10	2.05	5.12



Figs 2A and B: (A) Mean ± standard deviation (SD) of the SBS among six groups; (B) Range of the SBS among six groups

subgroups. This approach allowed for a comprehensive analysis of the significant differences among the subgroups.

Descriptive Data

The comparison of SBS between the total-etch and self-etch groups was conducted using a one-way ANOVA test (Tables 4 and 5).

For the total-etch groups, the analysis revealed statistical significance, with the highest SBS observed in group II. *Post hoc* comparisons showed significant differences between group II and both group I and group III (Tables 4 and 5).

Similarly, in the self-etch groups, the ANOVA test indicated statistical significance, with the maximum SBS observed in group V. *Post hoc* comparisons demonstrated significant differences between group V and both group IV and group VI (Tables 4 and 5).

These findings highlight the variations in SBS among the different groups, indicating the effectiveness of specific bonding techniques in each category.

Discussion

Indeed, MTA comes with some notable limitations. These include its extended setting time, potential for tooth discoloration, and relatively low compression and flexural strength, which are inferior to that of dentin. These factors collectively restrict its applicability to low-stress-bearing areas within dentistry.[9] In recent years, there has been a notable introduction of various materials, such as Biodentine and MTA Plus, with the primary objective of addressing and overcoming some of the disadvantages associated with MTA. These innovative materials aim to provide improved properties and performance in vital pulp therapy and restorative dentistry procedures.[12,13]

Mineral trioxide aggregate (MTA) Plus is a cost-effective dental material with a finer powder consistency. It shares a composition similar to tooth-colored ProRoot MTA and is designed for a wide range of endodontic applications. These applications include pulp capping, cavity lining, pulpotomy, root-end filling, repair of perforations, management of root resorption, apexification procedures, and comprehensive obturation in endodontics. Its versatility makes it a valuable addition to the armamentarium of dental professionals.[13]

Mineral trioxide aggregate (MTA) Plus is primarily composed of tricalcium silicate, bismuth oxide, dicalcium silicate, tricalcium aluminate, and calcium sulfate dehydrate (gypsum). These components constitute the main constituent phases of MTA. It's worth noting that the reaction by-products produced by MTA Plus are similar to those reported for ProRoot MTA, indicating a parallel composition and behavior between the two materials.[14]

The setting time of MTA Plus was found to be influenced by environmental conditions, as noted in previous research. Additionally, MTA Plus exhibited enhanced reactivity and a prolonged ability to release calcium, leading to an increase in local pH to alkaline levels when compared to ProRoot MTA. Another significant advantage of MTA Plus over ProRoot MTA is its cost-effectiveness. Taking into account these advantages, including improved setting time, cost-efficiency, and enhanced handling properties, MTA Plus presents itself as a convenient and viable alternative to conventional MTA in various dental applications.[7]

Biodentine® is comprised of tricalcium silicate, with additional components including calcium carbonate as a filler, zirconium oxide for radiopacity, and a water-based liquid component that includes calcium chloride, serving as a water-reducing agent. The inclusion of calcium chloride aids in achieving shorter clinical and final setting times, while also accelerating early strength development.

Compared to MTA, Biodentine® boasts enhanced sealing ability, higher compressive strengths, a significantly shorter setting time of just 10 minutes, improved biocompatibility, and superior bioactivity and biomineralization properties. These advantages position Biodentine® as a promising alternative to MTA in various dental applications.[8,9]

The research conducted by Kayahan et al. yielded noteworthy findings. They discovered that the SBS of Biodentine was notably higher than that of both ProRoot MTA and MTA Plus. Furthermore, when the study groups were compared based on the application of self-etch and total-etch procedures, the highest SBS was consistently achieved with the total-etch adhesive systems.

These results align with prior studies, which have consistently shown that total-etch adhesives tend to exhibit higher bond strengths compared to self-etch adhesives.[1,15–17] Tunc et al.'s research findings suggested that etch-and-rinse adhesives have the potential to enhance the SBS of composites to MTA.[18] In the current study, the SBS values for ProRoot MTA between the two-step etch-and-rinse adhesive and the self-etching primer system were found to be comparable, with values of 7.26 and 3.9 MPa, respectively. Importantly, this difference was not statistically

Table 4: Comparison of the SBS among total and self-etch groups

			Mean	*SD*	*p-value*	*Post hoc*
Group I	MTA	Total-etch	7.27	1.64	0.000*, significant, 2 > 1, 3	
Group II	Biodentine	Total-etch	14.69	3.95		
Group III	MTA Plus	Total-etch	5.04	1.50		
Group IV	MTA	Self-etch	3.86	0.88	0.000*, significant 5 > 4, 6	
Group V	Biodentine	Self-etch	8.39	1.85		
Group VI	MTA Plus	Self-etch	3.84	1.10		

*One-way ANOVA test; *significance of relationship at $p < 0.05$

Table 5: Comparison of the SBS among total and self-etch groups

	I	*J*	*Mean difference (I–J)*	*Significance*	*Sum of squares*	*df*	*Mean square*	*F*	*Significance*
Total-etch	MTA	Biodentine	−7.42200*	**0.00***					
		MTA Plus	2.23	0.16					
	Biodentine	MTA	7.42200*	**0.00***					
		MTA Plus	9.64700*	**0.00***					
	MTA Plus	MTA	−2.23	0.16					
		Biodentine	−9.64700*	**0.00***					
Self-etch	MTA	Biodentine	−4.53300*	**0.00***					
		MTA Plus	0.02	1.00					
	Biodentine	MTA	4.53300*	**0.00***					
		MTA Plus	4.55000*	**0.00***					
	MTA Plus	MTA	−0.02	1.00					
		Biodentine	−4.55000*	**0.00***					
Total-etch		Between groups			510.338	2	255.169	37.321	0.000*
		Within groups			184.602	27	6.837		
		Total			694.940	29			
Self-etch		Between groups			137.503	2	68.751	38.270	0.000*
		Within groups			48.505	27	1.796		
		Total			186.008	29			

*Significance of relationship at $p < 0.05$

significant. These results are in line with the findings of a study conducted by Tunç et al.,[18] reinforcing the idea that both etch-and-rinse adhesives and self-etching primer systems can yield similar SBS values when used with ProRoot MTA[19] where the SBS of composite to MTA was comparable using etch and rinse and self-etch bonding systems. Similarly, a study by Atabek et al.,[20] concluded that a two-step total-etch adhesive system exhibited a significantly higher SBS to MTA than the one-step self-etch adhesive. In a study conducted by Yelamali S et al.,[21] they investigated the impact of an acid-etch procedure on the bond between composite resin and MTA. The study findings revealed that the acid-etch procedure significantly enhanced the wettability of the MTA surface and, consequently, improved the bond strength between MTA and composite resin. As a result, the authors concluded that the acid-etch procedure plays a crucial role in achieving a stronger bond between MTA and composite resin, emphasizing its importance in dental procedures involving these materials.

In the present study, the SBS values for the total-etch system and self-etch primer system when used with MTA Plus (group III) were found to be 4.7 and 3.5 MPa, respectively. These values were comparatively lower than those obtained with MTA (group II). This difference in bond strength could be attributed to the composition of MTA Plus, which consists of 80% Portland cement and 20% bismuth oxide, whereas ProRoot MTA contains 75% Portland cement, 5% calcium, and 20% bismuth oxide.

It's worth noting that MTA Plus exhibits slightly higher pH levels and calcium ion release compared to ProRoot MTA. These variations in composition and properties can indeed influence the bond strength between composite resin and these different materials.[21]

In this study, the research outcomes revealed that the highest bond strength values were achieved between composite resin and Biodentine when employing total-etch adhesive systems, whereas lower SBS was observed with self-etch adhesives, consistent with findings from another study.[22] Specifically, our findings showed that the composite resin bonded with Biodentine exhibited the highest SBS, measuring 14.7 MPa for the total-etch adhesive and 8.38 MPa for the self-etch adhesive.

In summary, this study's conclusion underscored that etch-and-rinse type adhesives consistently produced higher bond strength compared to self-etch adhesives in the context of bonding composite resin to Biodentine.

Conclusion

Biodentine and MTA Plus have both established themselves as biocompatible materials suitable for vital pulp therapy. Notably, Biodentine exhibits higher bond strength when coupled with composite resin, surpassing that of both ProRoot MTA and MTA Plus, regardless of the adhesive system used. These findings suggest that Biodentine may be a preferred option due to its superior bonding capabilities in various dental applications. Moreover, the study emphasizes that total-etch adhesive systems consistently provide higher bond strength values compared to self-etch adhesives, underscoring the importance of adhesive selection in achieving optimal results in dental procedures.

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