Evaluation of shear bond strength of two resin-based composites and glass ionomer cement to pure tricalcium silicate-based cement (Biodentine®)

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ABSTRACT

Objectives: Tricalcium silicate is the major constituent phase in mineral trioxide aggregate (MTA). It is thus postulated that pure tricalcium silicate can replace the Portland cement component of MTA. The aim of this study was to evaluate bond strength of methacrylate-based (MB) composites, silorane-based (SB) composites, and glass ionomer cement (GIC) to Biodentine® and mineral trioxide aggregate (MTA). Material and Methods: Acrylic blocks (n=90, 2 mm high, 5 mm diameter central hole) were prepared. In 45 of the samples, the holes were fully filled with Biodentine® and in the other 45 samples, the holes were fully filled with MTA. The Biodentine® and the MTA samples were randomly divided into 3 subgroups of 15 specimens each: Group-1: MB composite; Group-2: SB composite; and Group-3: GIC. For the shear bond strength (SBS) test, each block was secured in a universal testing machine. Results: The highest (17.7±6.2 MPa) and the lowest (5.8±3.2 MPa) bond strength values were recorded for the MB composite-Biodentine® and the GIC-MTA, respectively. Although the MB composite showed significantly higher bond strength to Biodentine (17.7±6.2) than it did to MTA (8.9±5.7) (p<0.001), the SB composite (SB and MTA=7.4±3.3; SB and Biodentine®=8.0±3,6) and GIC (GIC and MTA=5.8±3.2; GIC and Biodentine=6.7±2.6) showed similar bond strength performance with MTA compared with Biodentine (p=0.73 and p=0.38, respectively). Conclusions: The new pure tricalcium-based pulp capping, repair, and endodontic material showed higher shear bond scores compared to MTA when used with the MB composite.

Keywords: Silicate cement. Shear strength. Silorane resin.

INTRODUCTION

Calcium silicate-based dental material, known as mineral trioxide aggregate (MTA), was first described in the dental scientific literature in 1993[19], and received US Food and Drug Administration approval for endodontic use in 1998[21]. Over the last decade, MTA has gained great popularity, especially in endodontic and pediatric dentistry, because it possesses improved physical and regenerative characteristics: it supports cementum regrowth[3], has low solubility after setting[11], can set in a wet environment, facilitates the control of bleeding,

provides a strong barrier for bacterial leakage, and can induce mineralized tissue formation[8]. These advanced properties mean that MTA is one of the most successfully used materials in clinical procedures, especially endodontic and surgical applications, including direct pulp capping, apexification, regenerative endodontic treatment (apexogenesis), pulpotomy, and the sealing of perforations. It can also be used as a root-end filling material after root-end resection[12,13,20,24].

MTA is primarily composed of tricalcium and dicalcium silicate and is not only used in the treatment of mature or immature permanent teeth,

but also in primary teeth treatment. Moreover, it has recently been proposed as an alternative agent to formocresol for pulpotomy treatment in primary molars[1,14,15,23]. Several studies have investigated the success of MTA compared to formocresol, as well as other treatments such as ferric sulfate and calcium hydroxide, and it has exhibited a significantly improved clinical performance independent of the time until exfoliation of the primary molar[1,10,14,15,23]. Despite its unique combination of favorable properties, MTA has some critical shortcomings, namely the prolonged setting time, the high solubility during the setting time, the potential for discoloration, and difficultly in handling[4].

Several new calcium silicate-based capping materials have recently been developed in response to the growing demand to overcome the disadvantages of MTA. Biodentine® (Septodont, Saint Maur des Fosses, France), the new high-purity, calcium silicate-based dental cement, was designed as a dentin substitute for resin composite restorations, pulp capping, and endodontic repair material. Biodentine® contains tricalcium silicate, calcium carbonate (filler), zirconium oxide (radiopacifier), and a water-based liquid composed

of calcium chloride as a water-reducing agent for shorter initial and final setting times, as it also accelerates the rate of early strength development. Biodentine® has improved sealing ability, higher compressive strengths, shorter setting time (10 minutes), greater biocompatibility, bioactivity and biomineralization properties than MTA[17,18]. Moreover, Biodentine® shows improved antibacterial properties compared to MTA, as well as a low cytotoxic effect[22].

Resin composites and glass ionomer cements (GICs) are very popular in restorative dentistry because of their esthetic qualities. They cannot be placed directly over freshly mixed MTA because they can affect its setting, and the etching and rinsing of unset MTA can dislodge the material. However, it has been claimed that the setting time of Biodentine® is 12 minutes, so the hypothesis is that resin composites and GICs can be layered over set Biodentine® after 12 minutes, which might enable single-visit procedures.

However, the strength with which restorative materials bond to Biodentine® is unclear. In this study, we evaluated the bond strength of methacrylate-based (MB) composites, silorane-

Material	Manufacturer	Method/steps for Application
Tricalcium-Silicate Cement	Biodentine® (Septodont, Saint Maur des Fosses, France)	Mixing premeasured unit dose capsules in a high-speed amalgamator for 30 s.
MTA	ProRoot MTA (Dentsply Tulsa Dental,USA)	Mix powder and liquid in a 1:3 ratio.
Methacrylate-based composite	Aelite All Purpose Body (Bisco Inc, Schamburg, IL, USA)	Light polymerize for 20 s.
One-Step Plus (2-step total-etch adhesive system)	All-Bond 2 (Bisco Inc, Schamburg, IL, USA)	1. Apply 37.5% phosphoric acid etchant for 15 s. 2. Rinse for 10 s. 3. Dry for 10 s. 4. Mix liquids A and B for 5 s. 5. Apply adhesive for 5 s. 6. Gentle air stream. 7.Light polymerize for 10 s.
Silorane-based composite	Filtek Silorane (3M ESPE, St. Paul, MN, USA)	Light polymerize for 20 s.
Silorane Bond (2-step self-etching primer)	Filtek Silorane Bond (3M ESPE, St. Paul, MN, USA)	1. Dry surface. 2. Apply primer. 3. Gentle air stream for 10 s. 4. Light polymerize for 10 s. 5. Apply adhesive. 6. Gentle air stream. 7. Light polymerize for 10 s.
Conventional glass ionomer cement	GC Fuji IX (GC, Tokyo, Japan)	Mix powder and liquid in a 1:3 ratio.

Figure 1- Manufacturer and application details of the materials used in the study

based (SB) composites, and GIC when used with Biodentine® and MTA.

MATERIAL AND METHODS

The materials used in this study included tricalcium silicate-based cement (Biodentine®, Septodont, Saint Maur des Fosses, France), white MTA (ProRoot MTA, Dentsply Tulsa Dental, Tulsa, OK, USA), MB composite resin (AELITE, Bisco, Inc., Schaumburg, IL, USA), SB composite resin (Filtek™ Silorane, 3M ESPE, St. Paul, MN, USA), and conventional GIC (GC Fuji IX, GC, Tokyo, Japan). The application steps for Biodentine® and resin composites, as well as the powder-to-liquid ratios of MTA and GICs recommended by the manufacturers, are listed in Figure 1.

Specimen preparation

Acrylic blocks (n=90) with 2 mm high and 5 mm diameter central holes were prepared. In 45 of the samples, the holes were fully filled with Biodentine®. In the other 45 samples, the holes were fully filled with MTA.

Biodentine® and MTA specimens were stored at 37°C in 100% humidity for respectively 15 minutes and 96 hours for setting.

Placement of restorative materials

After the settings, both the Biodentine® and the MTA samples were randomly divided into 3 subgroups of 15 specimens each: Group 1: Methacrylate-based (MB) composite; Group 2: Silorane-based (SB) composite; and Group 3: Glass-ionomer cement (GIC). In Groups 1 and 2,

the corresponding adhesive system was applied over the Biodentine® and MTA samples according to the manufacturers' instructions, as displayed in Figure 1. Each resin composite or GIC specimen was placed at the center of the Biodentine® and the MTA surface by placing the packing materials into cylindrically shaped plastic tubes with internal diameter of 2 mm and height of 2 mm. The composite specimens were cured with a light-emitting diode light cure (VALO LED, Ultradent, South Jordan, USA) with an intensity of 1,200 mW/cm^2 for 20 seconds. The GIC specimens were allowed to set for 10 minutes within the plastic tubes. After the polymerization or setting process, the plastic tubes were removed carefully and the specimens were stored at 37°C in 100% humidity for 24 hours. All the samples were prepared and tested by a single investigator (K.C.).

Shear bond strength (SBS) test

For the SBS test, each block was secured in a universal testing machine (Instron, AGS-1000kGW; Shimadzu Corp., Chiroda-Ku, Tokyo, Japan). A chisel-edge plunger was mounted onto the movable crosshead of the testing machine and positioned so that the leading edge was aimed at the Biodentine® or the MTA base/adhesive interface (Figure 2). The force required to remove the restorative material was measured in Newtons (N) (1 MPa=1 N/mm^2), and the SBS was then calculated by dividing the peak load values by the restorative material base area (3.14 mm^2).

Statistical analysis

All calculations were processed using the Statistical Package for the Social Sciences (SPSS) statistical software, version 16 (SPSS Inc., Chicago, IL, USA). The Kolmogorov-Smirnov test was used to evaluate the normality of the data distribution. The means, minimums, maximums, and standard deviations were also calculated. The mean bond strengths of the groups were compared using one-way analysis of variance (ANOVA) and Tukey's *post-hoc* test (honestly significant difference) was used for a two-by-two comparison (p<0.05).

RESULTS

Table 1 shows the descriptive statistics of shear bond strength for each group. The ANOVA that compared the experimental groups revealed the presence of significant differences among the groups (p<0.001). The highest (17.7 MPa) and the lowest (5.3 MPa) bond strength values were recorded for the MB composite-Biodentine® and the GIC-MTA, respectively.

Although the MB composite showed significantly higher bond strength to Biodentine® than it did to MTA (p<0.001), the SB composite and GIC showed
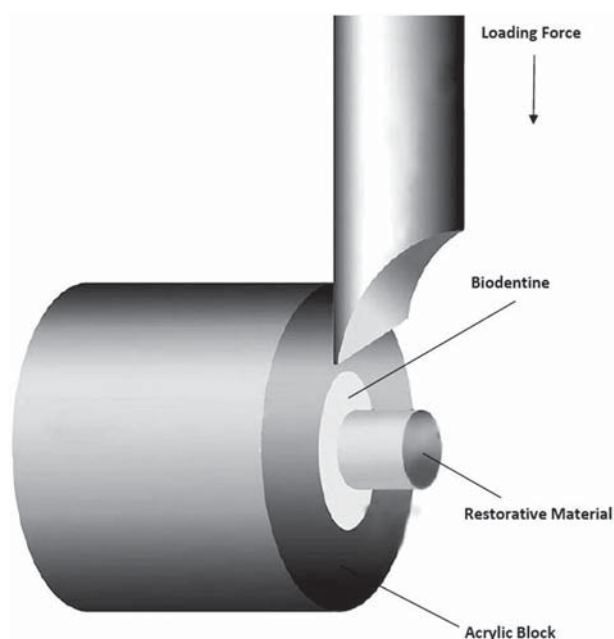

Figure 2- Schematic illustration of the shear bond strength test set-up

Table 1- Shear bond strength values of restorative materials to Biodentine® and conventional mineral trioxide aggregate (MTA)

Restorative materials	Capping Materials				p
	N	Mean	N	Mean	
MB composite	15	17.7±6.2	15	8.9± 5.7	<0.001*
SB composite	15	8.0±3.6	15	7.4±3.3	0.73
GIC	15	6.7±2.6	15	5.8±3.2	0.35
Total	45	10.8±6.6	45	7.2±4.4	0.003*

*There was a statistically significant difference

similar bond strength performance with MTA compared with Biodentine® (p=0.73 and p=0.38, respectively). The *post-hoc* test indicated that the MB composite showed significantly higher bond strength to Biodentine® and MTA (p<0.001) than the other restorative materials. The SB composite and the GIC specimen, however, demonstrated a similar SBS (p=0.17).

DISCUSSION

We designed the present study to evaluate and compare the shear bond strength (SBS) of MB composites, SB composites, and conventional GIC materials when used with Biodentine® and MTA. Several studies have reported obtaining optimal SBS with total etch adhesive systems[5,6,16] when comparing the SBS of various adhesive systems used with MTA for different time intervals, as phosphoric acid has been shown to produce deeper and more retentive micro porosity than even the strongest self-etching adhesive. It has also been demonstrated that restorative procedures should be postponed for at least 72 to 96 hours[7,16,25] after mixing MTA to allow the material to achieve its optimum physical properties. The purpose of the present study was not to assess the waiting times with regard to the SBS of restorative materials when used with MTA, since this has already been evaluated in numerous studies, as previously mentioned. Therefore, the MTA and Biodentine® subjects remained untouched for 96 hours and 15 minutes, respectively, after application, and we applied the MB composite with a total etch adhesive system before the placement of the composite resin restorations.

The strength of the bond between the restorative material and the enamel/dentin, as well as between the restorative material and the cavity liner, is one of the most critical factors for quality dental filling treatment. A bond strength ranging from 17 MPa to 20 MPa may be required to sufficiently resist contraction forces and produce gap-free restoration margins[2,5,9]. Our findings demonstrated that the MB

composite with Biodentine® showed the highest bond strength, and only this group reached an optimal shear bond, with a mean 17.7 MPa SBS value (Table 1). Biodentine® significantly increased the SBS value for the MS composite compared to MTA, and it reached the optimal SBS value. With regard to the MB composite, the difference in the SBS value for Biodentine® and MTA was statistically significant (p<0.001). The results showed that although the SBS values of the SB composite and GIC that were used with Biodentine® (8.0 MPa and 6.7 MPa, respectively) were higher than their SBS when bonded to MTA (7.4 MPa and 5.3 MPa, respectively), the differences between these subgroups were not statistically significant (p<0.05). Several studies have evaluated the effects of the bond strength of MB composite resins and GIC when bonding to MTA, and have indicated that MB composite resins showed low SBS values, ranging from 5.06 MPa to 13.45 MPa, 4 to 48 hours after mixing the MTA, and high SBS values, ranging from 14.44 MPa to 18.25 MPa, 72 to 96 hours after mixing the MTA. SBS values of bonding of GIC to MTA were reported as 8.85 MPa and 9.16 MPa at 45 minutes and 72 hours, respectively, after mixing the MTA[26].

However, a resin system based on the ring-opening polymerization of silorane molecules containing both hydrophobic siloxane and low shrinkage oxirane, rather than the free radical polymerization of dimethacrylate (DMA) monomers of a methacrylate-based composite, has recently been developed. The ring mechanism of the silorane composite allows a reduction in polymerization shrinkage and, according to some studies, siloranes also decrease polymerization stress[29,30]. However, the shear bond of silorane-based materials when bonded to Biodentine® or MTA is unclear. The mean SBS value of the SB composite and the conventional GIC used in the present study did not fall within the clinically acceptable range. Although the bond strength of the silorane composite (7.7 MPa) was greater than the bond strength of conventional GIC (6.0 MPa), the difference was not statistically

significant (p<0.05). However, the SB composite has several advantages with regard to polymerization shrinkage and stress; these were lower than when the MB composite was used (p<0.001).

On the basis of the methodology, the limitations of this study were (i) we based the SBS evaluation on only one setting time, and the adhesive systems have different setting times, and we did not consider alternative bonding systems, and (ii) the study was designed as an *in vitro* evaluation. Future studies should investigate the effects of different bonding protocols on the surface and structural characteristics of Biodentine® to provide an insight into the adhesive mechanisms of the different bonding systems and Biodentine® under strict laboratory conditions and in larger cohorts.

CONCLUSIONS

Within the limitations of this study, the new pure tricalcium-based pulp capping, repair and endodontic material showed clinically acceptable and higher shear bond scores compared to MTA when used with the MB composite. However, the SB composite and GIC bonded to Biodentine® and MTA at a fairly low strength.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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