Bond Strength of Different Restorative Materials to Light-Curable Mineral Trioxide Aggregate

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*The **aim** of the present study was to evaluate the bond strength of methacrylate-based (MB) composites, silorane-based (SB) composites, and glass-ionomer cement (GIC) in comparison to TheraCal and to compare those findings with the reference pulp capping material (MTA). **Study design:** A total of 90 acrylic blocks were prepared. Each of the blocks were **prepared as 15 mm high and 10 mm diameter and the blocks had a** 2 mm high and a 5 mm diameter central hole. In 45 of the samples, the holes were fully filled with TheraCal and in the other 45 samples, the holes were fully filled with MTA. The TheraCal 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). For the shear bond strength (SBS) test, each block was secured in a universal testing machine. After the SBS test, the fractured surfaces were examined under a stereomicroscope at ×25 magnification. **Results:** The analysis of variance that compared the experimental groups revealed the presence of significant differences among the groups (P < 0.001). The highest (19.3 MPa) and the lowest (3.4 MPa) bond strength value were recorded for the MB composite-TheraCal and the GIC-TheraCal, respectively.There were significant differences in bond strength between the TheraCal and the MTA groups for the MB composite subgroup (P <0.001) and the SB composite subgroup (P <0.05); however, there was no significant difference in bond strength for the GIC subgroup (P >0.05). **Conlusions:** The results from this in vitro study suggest that the new pulp capping material, known as light-curable MTA, showed clinically acceptable and higher shear bond scores compared to MTA when used with the MB composite.*

Key words: MTA, bond strength, composite

INTRODUCTION

In the last decade, calcium silicate based hydraulic cement, known as mineral trioxide aggregate (MTA), has received great attention in the field of dentistry, especially in endodontic and pediatric dentistry, because it can be set in the presence of blood and other biological fluids[1] and it has presented as being biocompatible, so it can be used safely when placed adjacent to pulp and periodontal tissues.[2,3] In addition to its good biological biocompatibility, MTA presents some improved physical and regenerative characteristics; it supports cementum regrowth,[4] it has low solubility after setting,[5] it can be set in a wet environment, it facilities the control of bleeding, it provides a strong barrier for bacterial leakage, and it has the ability to induce mineralized tissue formation.[6]

Despite its unique combination of favorable properties, MTA has some shortcomings. The prolonged setting time and high solubility of MTA during the setting time are two of its primary drawbacks.[7] In addition, MTA is relatively difficult to manipulate after it is mixed with water, as the mixed material has a "grainy" and "sandy" consistency. Moreover, it is recommend that a wet cotton pellet be placed next to MTA following its placement because MTA requires water to initiate and complete the setting reaction.[8] In this respect, other filling materials should be placed adjacent to MTA at the additional appointment. This increases the number of appointments required and delays the placement of the final restoration; consequently, leakage of the temporary restorative material may cause undesirable results, impacting the success of the treatment.

In response to the growing demand to overcome the disadvantages of MTA regarding its long setting time, high solubility, and its difficulty in using it for clinical application, a light-curable MTA has recently been developed. The new light-curable material, a resin-modified calcium silicate-based MTA liner designed with a direct and indirect pulp capping material, has been introduced as TheraCal® (Bisco, Inc., Schamburg, IL, USA). This light-curable MTA contains approximately 45% Portland Cement (PC), 10% radiopaque component (bismuth oxide), 5% hydrophilic thickening agent (fumed silica), and approximately 40% resin, by weight.[9] The incorporation of light-curable resins has been proposed for many

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materials, such as the resin-modified glass-ionomer cements (GIC), to improve their mechanical properties and reduce their setting time. It is equally important to note that this light-curable material has a shorter setting time, lower solubility, higher flowability, and less film thickness compared with MTA.[9] The light-curable MTA is self-sealing, which aids in antimicrobial activity. It initially bonds to dentin helping it resist accidental air-drying removal, making the application advisable in extremely wet and blood-contaminated areas. It has also been hypothesized that due to fact that the final restoration can be completed during the same appointment, the rate of clinical success will be increased.[1]

Because there is no published data on bond strength with regards to light-curable MTA, the aim of the present study was to evaluate the bond strength of methacrylate-based (MB) composites, silorane-based (SB) composites, and glass-ionomer cement (GIC) in comparison to TheraCal and to compare those findings with the reference pulp capping material (MTA).

MATERIALS AND METHOD

The material used in this study included light-curable MTA (TheraCal, Bisco, Inc., Schamburg, IL, USA), conventional white MTA (ProRoot MTA, Dentsply Tulsa Dental, Tulsa, OK, USA), MB composite resin (AELITE, Bisco, Inc., Schamburg, IL, USA), SB composite resin (Filtek™ Silorane, 3M ESPE, St. Paul, MN, USA) and conventional GIC (GC Fuji IX, GC, Tokyo, Japan). The compositions of each material, the powder-to-liquid ratios of the MTA and GICs, as well as the application steps for the light-curable MTA and resin composites that are recommended by the manufacturers, are listed in Table 1.

Specimen Preparation

A total of 90 acrylic blocks were prepared. Each of the blocks were **prepared as 15 mm high and 10 mm diameter and the blocks had a** 2 mm high and a 5 mm diameter central hole. In 45 of the samples, the holes were fully filled with TheraCal and in the other 45 samples, the holes were fully filled with MTA.

Each of the TheraCal specimens was light-cured with an LED cure unit (VALO LED, Ultradent, South Jordan, USA) with an intensity of 1200-mW cm² for 20 sec.

All of the MTA surfaces were wrapped with pieces of wet gauze and the specimens were stored at 37° C in 100% humidity for 96 hours for setting.

Placement of Restorative Materials

After 4 hours, both the TheraCal 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 Group 1 and Group 2, the corresponding adhesive system was applied over the TheraCal and MTA samples, according to the manufacturers' instructions, which are shown in Table 1. Each of the resin composite specimens and the GIC specimen were placed at the center of the MTA and the TheraCal surface by applying packing materials into cylindrically shaped plastic tubes with an internal diameter of 2 mm and a height of 2 mm (Figure 1). The composite specimens were cured with a light emitting diode light cure (VALO LED) with an intensity of 1200 mV/cm² 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 .

Figure- 1. Schematic illustration of the preparation of the samples.
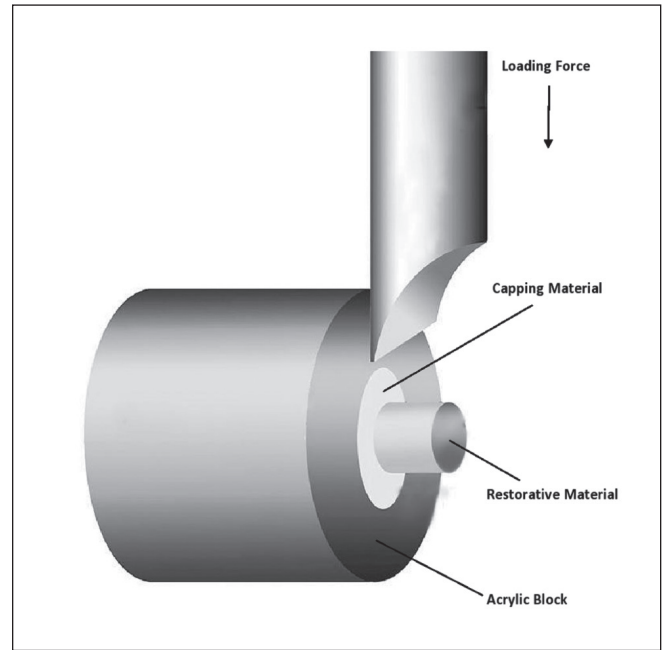
Shear Bond Strength (SBS) Test

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 MTA or the TheraCal base/adhesive interface (Figure 2). The force required to remove the restorative material was measured in Newtons (N) (1 MPa = 1 N/mm²), and the SBS was then calculated by dividing the peak load values by the restorative material base area (3.14 mm²).

Evaluation of Fracture Patterns

After the SBS test, the fractured surfaces were examined under a stereomicroscope (SZ 40, Olympus, Tokyo, Japan) at ×25 magnification. The fractured surfaces were classified as follows: (1) adhesive fracture, failure at the site of the TheraCal/MTA-restorative material, (2) cohesive fracture, failure within the restorative material/base material, and (3) mixed fracture, a combination of adhesive fracture and cohesive fracture.

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 test the normality of the distribution of the data. The means, minimums, maximums, and standard deviations were 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 (significance level, $P < 0.05$).

Table-1. Materials used in this study and their composition, manufacturer and application details.

Material	Manufacturer	Composition	Mode/steps for Application
Light cured MTA	TheraCal (Bisco Inc, Schamburg, IL, USA)	AeroSil 8.0%, biocompatible hydrophilic resin 42.5% (BisGMA 20%, biocompatible resin-FDA 77.25%, modifying agent 2.4%; initiating agent 0.32%, stabilizer for the initiating agent 0.032%), active ingredients in MTA 44.5%, and barium sulfate 5%	Light-polymerize for 20 s.
MTA	ProRoot MTA (Dentsply Tulsa Dental,USA)	Tricalcium silicate, bismuth oxide, dicalcium silicate, tricalcium aluminate, calcium sulfate dehydrate or gypsum	Mix powder/liquid ratio:1/3.
Methacrylate- based composite	Aelite All Purpose Body (Bisco Inc, Schamburg, IL, USA)	Bis-GMA, Bis-EMA, UDMA, TEGDMA, zirconia/sílica, silica, camphoroquinone	Light polymerize for 20 seconds
One-Step Plus (2-step total-etch adhesive system)	All-Bond 2 (Bisco Inc, Schamburg, IL, USA)	Acetone Biphenyl dimethacrylate Hydroxyethyl methacrylate Dental glass	1. Apply 37.5% phosphoric acid etchant for 15 seconds 2. Rinse for 10 seconds 3. Dry for 10 seconds 4. Mix liquids A and B for 5 seconds 5. Apply adhesive for 5 seconds 6. Gentle air stream 7. Light polymerize for 10 seconds
Silorane-based composite	Filtek Silorane (3M ESPE, St. Paul, MN, USA)	Siloxane and oxirane (23% of the composition). The inorganic filler contains fine quartz particles and radiopaque yttrium fluoride (76%).	Light polymerize for 20 seconds
Silorane Bond (2-step self-etching primer)	Filtek Silorane Bond (3M ESPE, St. Paul, MN, USA)	3M ESPE hydrophobic bifunctional monomer, camphor quinine/ a silane-treated silico fillers, stabilizer.	1. Dry surface 2. Apply primer 3. Gentle air stream for 10 seconds 4. Light polymerize for 10 seconds 5. Apply adhesive 6. Gentle air stream 7. Light polymerize for 10 seconds
Conventional glass ionomer cement	GC Fuji IX (GC, Tokyo, Japan)	Powder:Alumino-fluorosilicate glass, Liquid: Polyacrylic acid, polybasic carboxylic acid, water	Mix powder/liquid ratio:1/3.

RESULTS

The descriptive and comparative statistics of shear bond strength for each of the restorative and capping materials are given in Table 2. The analysis of variance that compared the experimental groups revealed the presence of significant differences among the groups (P <0.001). The highest (19.3 MPa) and the lowest (3.4 MPa) bond strength value were recorded for the MB composite-TheraCal and the GIC-TheraCal, respectively.

There were significant differences in bond strength between the TheraCal and the MTA groups for the MB composite subgroup (P <0.001) and the SB composite subgroup (P < 0.05); however, there was no significant difference in bond strength for the GIC subgroup (P >0.05).

Although the MB composite showed significantly higher bond strength to TheraCal than it did to MTA (P <0.001), the SB composite showed significantly higher bond strength performance with MTA than it did with TheraCal (P <0.05).

The post-hoc test indicated that the MB composite showed a significantly higher bond strength to either TheraCal or MTA (P <0.001) than the other restorative materials. The SB composite and the GIC specimen, however, demonstrated a similar shear bond strength (P = 0.56) (Table 3).

Fracture analyses of the tested materials are given in Table 4. Most of the observed fracture modes in the TheraCal groups were adhesive fractures; the **following** was cohesive in the capping materials and mixed and cohesive in the restorative materials. In contrast to the MB composite, the SB composite showed predominantly adhesive failure and adhesive fracture in both the TheraCal and MTA groups.

DISCUSSION

The essential objective of pulp therapy is the successful treatment of a pulpally involved tooth and retaining that tooth in a healthy condition so that it can fulfill its role as a useful component of the primary and young permanent dentition.[10] For successful vital pulp treatment, the pulp capping liner that is used must act as a strong barrier to prevent dental pulp complex **for microbial attacks** and induce the formation of the new dentine bridge or dentine-like bridge between the pulp and the restorative material. In recent years, MTA has become widely used in the practice of pediatric dentistry as a pulpotomy medicament in primary teeth and as a capping material in young permanent teeth because of its beneficial characteristics, such as the fact that it is highly protective for microleakage and it is biocompatible. It also promotes regeneration of the original tissues when it is placed in contact with the dental pulp.[11,12] On the other hand, despite its desirable physical and biological properties, MTA does have some disadvantages, including a long setting time, high solubility, and poor handling characteristics. To overcome all the disadvantages of MTA, light-cured MTA was developed in recent years.[9]

In the literature, several studies have evaluated the chemical-physical characteristics of TheraCal, a light-curable pulp capping material consisting of resin and calcium silicate (Portland Cement), and it has been suggested that light-cured MTA has demonstrated a short setting time and low solubility and that it releases significantly more Ca ions than either MTA or a calcium hydroxide based (Dycal®) liner.[1,9,13,14]

The present study was designed to evaluate and compare the shear bond strength of MB composites, SB composites, and conventional GIC materials when used with TheraCal and MTA. Several studies have reported that optimal shear bond strength was obtained with total etch adhesive systems[15-17] because phosphoric acid has been proven to produce deeper and more retentive microporosities than even the strongest self-etching adhesive; it has also been shown that restorative procedures should be postponed for at least 72 to 96 hours[17-19] after mixing MTA to allow the material to achieve its optimum physical properties. The purpose of this current study was not to assess or compare the effect of different adhesive materials or the waiting time intervals on the shear bond strength of restorative materials when used with either TheraCal or MTA, as this has been evaluated by numerous previous studies mentioned above. Therefore, in the present investigation, all of the MTA subjects were untouched for 96 hours after application and the MB composite was applied with a total etch adhesive system before the placement of the composite resin restorations began.

The bond strength between the restorative material and the enamel/dentine, as well as between the restorative material and the cavity liner, is one of the most critical factors for quality dental filling treatment. It has been estimated that a bond strength ranging from 17 MPa to 20 MPa may be required to sufficiently resist contraction forces in order to produce gap-free restoration margins[15,20,21] Our findings demonstrated that the MB composite with TheraCal showed the highest bond strength and this is the only group that reached an optimal shear bond with a mean 19.3 MPa SBS value (Table 2). The following are the results for the MB composite-MTA, the SB composite-MTA, the GIC-MTA, the SB composite-TheraCal, and **the GIC-TheraCal**, respectively. According to our results, TheraCal significantly increased the SBS value for the MS composite and it reached the optimal SBS value when compared to MTA. For the MB composite, the difference in the SBS value for TheraCal and MTA reached a statistically significant level (*P*<0.001). On the other hand, the positive effect of TheraCal on the shear bond strength was limited in the MS composite; high SBS values were not seen with the SB composite and GIC specimen subgroups. According to our results, the SBS values of the SB composite and GIC bonded to TheraCal (3.6 MPa and 3.4 MPa, respectively) were lower than the SBS of these materials bonded to MTA (7.4 MPa and 5.8 MPa, respectively). Although, there was a significant difference in the SBS of TheraCal and MTA for the SB composite subgroup (*P*<0.05), the differences in the GIC subgroup did not reach a statistically significant level (P>0.05).

The choice between restorative material and adhesive material is an important factor to consider in order to insure a successful restorative treatment. Several studies have evaluated the effects of the bond strength of MB composite resins and GIC to MTA, and they 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 18.25MPa, 72 to 96 hours after mixing the MTA. Yesilyurt et al.[22] measured the SBS of conventional GICs bonded to MTA that had been allowed to set for two different time intervals, and they reported that the SBS values for the 45-minute and 72-hour WMTAs were 8.85 MPa and 9.16 MPa, respectively. On the other hand, a resin system was recently developed that is 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. The ring mechanism of the silorane composite allows a reduction in polymerization shrinkage[23-26] and, according to some studies, siloranes also decrease polymerization stress.[24,27] However, the shear bond of silorane-based materials that are bonded to TheraCal or MTA is unclear. The mean SBS value of the SB composite and the conventional GIC used in this study did not fall within the clinically acceptable range. Although, the bond strength for the silorane composite, at 5.5 MPa, was greater than the bond strength of conventional GIC, at 4.6 MPa, the difference did not reach a statistically significant level (P □0.05) (Table 3). On the other hand, the SB composite has several advantages in relation to polymerization shrinkage and stress; in that regard, it showed a fairly lower performance compared to the MB composite.

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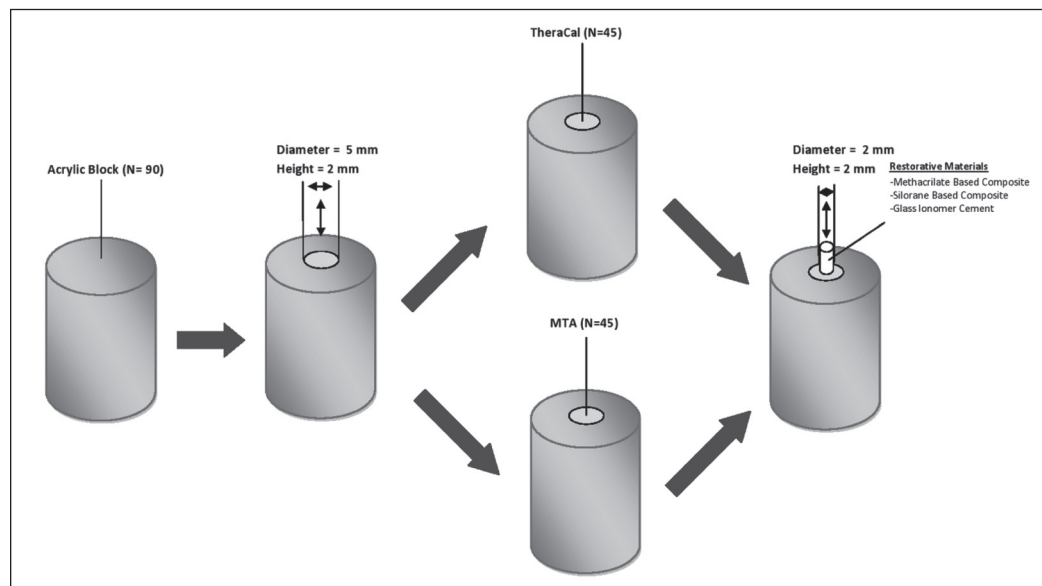

Table-2. Shear bond strength values of restorative materials to Light-curable and conventional MTA.

Restorative materials	Capping Materials								P
	Light-curable *MTA*				Conventional MTA				
	N	Mean	Min	Max	N	Mean	Min	Max	
MB composite	15	19.3±8.4[A]	9,6	39.9	15	8.5±5.3[B]	1.4	21.8	<0.001
SB composite	15	3.6±2.5[C,B]	0.5	10.4	15	7.4±3.3[D,B]	4.2	13.8	0.02
GIC	15	3.4±1.3[E,C]	0.9	6.1	15	5.8±3.2[E,B,C]	1.4	12.4	0.16
Total	**45**	8.8±9.1	0.2	39.9	**45**	7.2±4.1	1.4	21.8	0.31

The different letters mentioned that there was a significant difference according to post-hoc test. The same letters mentioned there was no significant difference.

Table-3. Mean shear bond strength values of restorative materials

Material	N	Mean	SD	95% confidence interval		P
				Lower Bound	Upper Bound	
MB composite	30	13.9	8.8	10.6	17.2	**A**
SB composite	30	5.5	3.5	4.2	6.8	**B**
GIC	30	4.6	2.7	3.5	5.6	**B**
Total	90	8.1	7.1	6.5	9.5	**<0.001**

The different letters mentioned that there was a significant difference according to post-hoc test. The same letters mentioned there was no significant difference.

Table-4. Fracture modes of the tested materials after the shear bond test.

	Light-curable *MTA*			Conventional *MTA*			Total
	MB composite	SB composite	GIC	MB composite	SB composite	GIC	
Adhesive	4	12	8	7	11	7	46
Mix	4	0	3	0	0	3	10
Cohesive in capping material	6	3	3	8	4	4	32
Cohesive in restorative material	3	0	1	0	0	1	5

After SBS testing, it is expedient to determine the site of material failure, and the fractured surfaces were examined under a stereomicroscope. Based on these observations, fracture occurred mainly within the adhesive failure; the **following** was cohesive in the capping materials and mixed and cohesive in the restorative materials. The silorane composite predominantly showed adhesive failure and adhesive fracture in both the TheraCal and MTA groups. On the other hand, while it was observed that a stronger bond existed between the MB composite and TheraCal as well as the MB composite and MTA, a higher percentage of cohesive failures were yielded within these materials (Table 4).

CONCLUSIONS

We found that the MB composite-TheraCal specimens and the GIC-TheraCal specimens demonstrated the highest and the lowest bond strength, respectively. The results from this *in vitro* study suggest that the new pulp capping material, known as light-curable MTA, showed clinically acceptable and higher shear bond scores compared to MTA when used with the MB composite. **Therefore, the simple application and high bond strength capacity with MB composite resin of this pulp capping material might be considered for clinical use in endodontic and pediatric dentistry.** On the other hand, further *in vivo* and *in vitro* studies are required to evaluate its clinical performance.

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