ABSTRACT

Introduction: The efficacy of the bond between the restorative materials and the pulp capping materials has an important role in the success of vital pulp therapy.

Aims and Objective: This study was conducted to investigate and compare the shear bond strength (SBS) of Biodentine, Endocem mineral trioxide aggregate (MTA), and TheraCal LC to resin composite using a universal adhesive at their respective initial setting time and to identify the specific modes of failure using a stereomicroscope at ×25 magnification.

Materials and Methods: A total of 60 acrylic blocks were prepared. Each of the blocks were prepared 20 mm high and 12 mm diameter and had a central hole of 2 mm depth and 5 mm diameter. These acrylic blocks were divided into three groups of 20 samples each based on the liner that is condensed into prepared central hole as Group A – Biodentine, Group B – Endocem MTA, and Group C – TheraCal LC. Composite was then bonded to each sample using universal adhesive. SBS analysis was performed at a cross-head speed of 1 mm/min.

Observation and Results: Mean SBS of Group B (Endocem MTA) with composite was found to be significantly lower than that of Group A (Biodentine) with composite which was further significantly lower than that of Group C (TheraCal LC) with composite.

Conclusion: This present study concludes that the bond strength of resin-based light-curable TheraCal LC showed clinically acceptable and highest SBS values when used with a universal silane containing adhesive.

Keywords: Adhesive, Biodentine, Endocem mineral trioxide aggregate, Filtek Z-350 XT, TheraCal LC

INTRODUCTION

The main objective of restorative dentistry is to restore and preserve dental health through proper restorative treatment so as to protect the pulp and restore its function.¹

Pulp capping biomaterials are placed as a protective layer on the floor of deep cavities after removal of deep carious lesions or after traumatic exposure. These biomaterials should have certain properties such as biocompatibility, biointeractivity (i.e., release of biologically relevant ions), and bioactivity (apatite forming ability) to help pulp cell activity and the formation of new reparative dentin.²

Calcium hydroxide-based materials were used before but they have disadvantages such as nonadherence to dentin, dissolution over time, and tunnel defects.³⁴ To overcome these drawbacks, calcium silicate-based liner materials have
been introduced which are biologically well tolerated by the pulp tissue.\[^{2,4}\]

A restoration is placed immediately after vital pulp therapy to create and maintain an effective coronal seal, thus protecting against bacterial attack.\[^{7,8}\] The success of these laminate restorations depends on the bond strength of liner to the dentin and also on the quality of the bond between liner and overlying composite.\[^{9}\]

Mineral trioxide aggregate (MTA), the first calcium silicate-based liner introduced in 1993 has two main disadvantages which are prolonged setting time and high solubility during setting.\[^{10,11}\] Some studies regarding its bond strength to composite show that the highest bond strength is obtained at longer time intervals (2.45 h, 24 h, and 3 days).\[^{12,13}\]

Materials based on tricalcium silicate with a reduced setting time have thus been introduced. Biodentine\[^{TM}\] (Septodont, Saint-Maur-des-Fossés, Créteil, France) has a clinically acceptable setting time (12 min) and physical properties.\[^{14}\] It is known to preserve pulp vitality, help in pulp healing, and provide a natural substitute for dentin through bioactive stimulation of the dentino-pulpal complex.\[^{15}\]

Another calcium silicate-based material called Endocem MTA (Maruchi, Wonju, Korea), derived from Pozzolan cement, has been introduced.\[^{16}\] A pozzolan is a siliceous or silico-aluminous material which in finely divided form and in the presence of moisture, chemically reacts with calcium hydroxide to form compounds having adhesive qualities.\[^{17}\] The advantages of Endocem MTA are its fast setting time (4 min), biocompatibility and osteogenecity comparable with ProRoot MTA.\[^{18}\]

TheraCal LC\[^{TM}\] (Bisco Inc, Schaumburg, IL, USA), a new light-cureable resin-modified calcium silicate-based liner has shorter initial setting time (20 s), improved biological properties and can be easily placed.\[^{19}\]

Curing shrinkage of overlying composite can cause stresses in the bond between liner and composite.\[^{20}\] It has been noted that bond strength of approximately 17 MPa may be required to obtain gap-free restoration margins.\[^{21}\] The few studies that have been conducted to compare the bonding ability of newly introduced calcium silicate-based liners to resin composite with different types of adhesive systems (i.e., total-etch and self-etch) have shown quite variable results.\[^{22-25}\]

Recently, a new single bottle universal or multimode adhesive with silanes (Single Bond Universal\[^{TM}\], 3M ESPE, St. Paul, MN, USA) has been introduced which is a single adhesive and can be used in self-etch or total-etch or selective etch mode.\[^{19}\]

This in vitro study was planned to evaluate and compare the shear bond strength (SBS) of resin composite bonded to three different calcium silicate-based liners: Biodentine, Endocem MTA, and TheraCal LC using a universal adhesive with the help of universal testing machine. The study also aims to identify the specific modes of failure using a stereomicroscope (Swift Stereo SM80, Tokyo, Japan) at a magnification of \(\times 25\).

**MATERIALS AND METHODS**

**Sample size estimation**

The sample size estimation was done using GPower software (version 3.0). The sample size was estimated for \(F\) test and ANOVA: Fixed effects, Omnibus one way was chosen.

A minimum total sample size of 60 was found to be sufficient for an alpha of 0.05, power of 80%, 0.42 as effect size (anticipating a large effect size among three study groups regarding SBS).

**Sample preparation**

A total of 60 cylindrical acrylic blocks measuring (20 mm height and 12 mm diameter) with a hole in the center of the acrylic block (2 mm in depth and 5 mm in diameter) were prepared.\[^{23,24}\]

Specimens were randomly divided into three groups of 20 samples each based on the calcium silicate liner to be used. In the groups A, B, and C, the holes were filled with Biodentine, Endocem MTA, and TheraCal LC, respectively (Table 1).

After the respective liner material was set (setting time of liners as per the manufacturer’s instructions are: Biodentine – 12 min, Endocem MTA – 4 min and TheraCal LC – 20 s), a universal dental adhesive (Single Bond Universal\[^{TM}\]) was applied in a self-etch mode over the liner surface with a bristle brush. It was rubbed for 20 s followed by gentle air drying for approximately 5 s and light-cured for 10 s.\[^{9}\]

The polyethylene tube (2.5 mm diameter, 2 mm height) was placed over the liner surface and filled with the resin composite (Filtek Z-350 XT, 3M ESPE, St. Paul USA) and light-cured with a light-emitting diode curing unit (LY-A180 delivering 1200–2000 mW/cm\(^2\), 420–480 nm) for 20 s.\[^{9,23,24}\]

The polyethylene tubes were removed with a sharp knife after the completion of the resin composite build-up.\[^{9,23,24}\]
All the samples were stored at 37°C in water for 24 h.\[9\]

The measurement of shear bond strength

The specimens were attached to the universal testing machine (Saumya Machineries Private Limited, Ahmedabad, India). A chisel with knife-edge was gently held against the resin composite–respective calcium silicate liner interface and was loaded at a cross-head speed of 1.0 mm/min until bond failure occurred.\[9,23\]

The load at failure was recorded in Newtons (N). SBS was calculated and expressed in Megapascals (MPa) by dividing the peak load at failure to the specimen surface area according to the formula:\[23,24\]

\[
\text{SBS (MPa)} = \frac{\text{Load (N)}}{\text{Area} \pi r^2 (\text{mm}^2)}.
\]

Fracture analysis

The fractured test specimens were examined under a stereomicroscope (Swift Stereo SM80, Tokyo, Japan) at a magnification of × 25 to analyze the nature of failure:\[9,23,24\]

- Cohesive failure – failure with in liner or resin composite
- Adhesive failure – failure within the bonding interface
- Mixed failure – when two modes of failure occur simultaneously (i.e., cohesive failure and adhesive failure).

The data were then collected, tabulated, and subjected to statistical analysis using Statistical Package for Social Sciences (SPSS) version 21 (IBM, Chicago, US).

RESULTS

The mean SBS values and standard deviations are shown in Table 2 and were analyzed using ANOVA test; the post hoc Tukey’s test was used for intergroup comparison. Mean SBS was found to be 6.25 ± 0.4 of Group A (Biodentine) specimens, 4.21 ± 0.27 of Group B (Endocem MTA) specimens, and 16.35 ± 1.19 of Group C (TheraCal LC) specimens [Table 2]. Group C showed the highest SBS value followed by Group A which was further followed by Group B [Table 3]. There was statistically significant difference in the bond strength values among the groups. The observed modes of failure were cohesive in the respective pulp capping materials all the three groups [Figure 1a-c].

DISCUSSION

Bond strength of pulp capping materials to restorative materials is important for the success of restorations and can be measured by bond strength testing.\[26,27\]

This study was conducted to evaluate the SBS of the newly introduced calcium silicate-based liners (Biodentine, Endocem MTA, and TheraCal LC) to resin composite using universal adhesive containing silane in self-etch mode using macro-SBS test.

The universal adhesive is methacryloyloxydecyl dihydrogen phosphate-based (Single Bond UniversalTM, 3M ESPE) with silanes and can be used in self-etch, total-etch or selective etch mode. It shows chemical bonding to calcium ions, and aluminium and zirconium oxides.\[28,29\] The bifunctional silane molecule bonds chemically to silica-containing materials and has methacrylate that allows chemical bonding with resinous substrate.\[30\]

In our study, we used the macro-SBS test which is the most commonly used test to assess bond strength. It is a simple and
quick and popular method. No further specimen processing is required after the bonding procedure. This test is commonly used to screen new adhesives on their bonding effectiveness.\cite{31}

In the present study, mean SBS was found to be 6.25 MPa of Group A (Biodentine) specimens, 4.21 MPa of Group B (Endocem MTA) specimens, and 16.35 MPa of Group C (TheraCal LC) specimens. On intergroup comparison, the difference in the SBS values was found to be statistically significant between all the tested calcium silicate-based liners. In our study, only Group C fulfills the criterion of desirable SBS value (approximately 17 MPa).

The failure mode in all the groups in all the samples was of cohesive type in the respective pulp capping materials. Cohesive failures reflect the inner cohesion power of the material rather than the real interfacial bond strength of the adhesive resin and the material. Cohesive failures may be due to the low internal resistance of the material or the bond strength being greater than the material's internal resistance.\cite{32} In our study, the composite was placed over Biodentine, Endocem MTA, and TheraCal LC after their initial setting time (12 min, 4 min, and 20 s, respectively) as recommended by the manufacturer. The stresses produced by the curing of overlying composite could cause stresses in the pulp capping material.\cite{20}

TheraCal LC exhibited significantly higher bond strength values as compared to Biodentine and Endocem MTA. This could be due to the fact that the harmful effects of the contraction stresses were very less in TheraCal LC as it being a resin-based light-curable liner may achieve sufficient initial bulk strength on curing.\cite{9} While curing contraction of the overlying composite may have caused stresses in immature matrix of Biodentine and Endocem MTA thus leading to further lowering of bulk strength and cohesive failures.\cite{20}

The higher bond strength for TheraCal LC may also be due to the presence of dimethacrylate monomer that promotes chemical adhesion between the TheraCal LC and bonding adhesive.\cite{22,33} The resin bonding agent intermixes with both composite and calcium silicate-based liners by true chemical bonding to create a strong interface. Moreover, a strong chemical bonding among adhesive and Ca, Al, Zr, and silicon ions of calcium silicate-based liners was expected.\cite{9}

The results of our study are in accordance to the study done by Deepa et al.\cite{9} which showed similar results and concluded that bond strength of TheraCal LC (18.24 MPa) to composite resin using universal adhesive (in a self-etch mode) was significantly higher than that of Biodentine and all the samples in TheraCal LC group showed cohesive failures.

The mean bond strength value of Biodentine with composite (6.25MPa) did not fulfill the criterion of desirable bond strength. Hashem et al.\cite{20} stated that for adequate maturation of the material, composite should be placed after 2 weeks. According to Bachoo et al.,\cite{15} Biodentine is a porous material that needs at least 2 weeks' time for crystallization of hydrated calcium silicate gel to get bulk strength enough to withstand the polymerization stresses. They reported that the initial setting time of Biodentine takes approximately 12 min but the full maturation of the material takes 2 weeks. Consequently, the setting reaction of Biodentine may affect the bond strength between Biodentine and restorative materials. Thus in our study, placing composite after the initial setting time could have resulted in lower SBS values of Biodentine.

These results are in accordance to the study done by Deepa et al.,\cite{9} in which Biodentine showed mean SBS of 5.56 MPa to composite resin using universal adhesive (in a self-etch mode). All the samples in the Biodentine group showed cohesive failure.

In a study done by Sakr et al.\cite{34} Biodentine showed SBS of 9.05 MPa when used with self-etch adhesive system. These values were higher than the value obtained in our study though these values did not fall in the desirable range. It could be because of the bonding of composite to calcium silicate-based material after waiting period of 24 h. It could also be because of the difference in methodology (micro-SBS test) and the difference in the adhesive systems used.

Odabaş et al.\cite{35} (2013) conducted a study to measure the SBS of Biodentine with different time intervals (12 min and 24 h). They found that Biodentine showed the highest SBS at 24-h period (19.55 MPa) with self-etch adhesive systems.

Table 2: Shear bond strength (MPa) of samples of three study groups

| Group              | n  | Minimum-maximum   | Mean±SD       |
|--------------------|----|-------------------|---------------|
| Group A (Biodentine)| 20 | 5.54-6.95         | 6.25±0.40     |
| Group B (Endocem MTA)| 20 | 3.76-4.66         | 4.21±0.27     |
| Group C (TheraCal LC)| 20 | 15.09-18.40       | 16.35±1.19    |

SD: Standard deviation

Table 3: Intergroup comparison of shear bond strength between study groups using Tukey's test

| Group B                  | Group C (TheraCal LC) | S: Statistically significant |
|--------------------------|-----------------------|----------------------------|
| Group A (Biodentine)     | <0.0001 (S)           |                            |
| Group B (Endocem MTA)    | 0.0001 (S)            |                            |

The resin bonding agent intermixes with both composite and calcium silicate-based liners by true chemical bonding to create a strong interface. Moreover, a strong chemical bonding among adhesive and Ca, Al, Zr, and silicon ions of calcium silicate-based liners was expected.\cite{9}
The lowest value was obtained at 12 min (9.12 MPa), which is in accordance to the findings of our study.

Aksoy and Ünal[36] conducted a study to compare the SBS of Biodentine using universal adhesive (Single Bond Universal) in self-etch and total-etch mode at different time intervals (12 min, 24 h, 48 h, 72 h, and 96 h) and lower bond strength values were found in the 12 min group as compared to other time periods. According to them, before applying restorative material on Biodentine, a waiting period of at least 24 h can be useful to obtain high SBS values. Furthermore, no significant differences were observed between the self-etch and total-etch mode of universal adhesive. Although the SBS values were still not in the desirable range as found in our study.

In our study, Endocem MTA showed the lowest mean bond strength value (4.21 MPa) which was quite insufficient. This finding matches with the findings of a study by Shin et al.,[16] in which SBS of Endocem MTA to resin composite was evaluated using various adhesive systems and the values were obtained in the range of 3.36 MPa to 4.83 MPa. Endocem MTA did not show any significant difference in the bond strength when used with different adhesive systems. According to them, Endocem MTA comprises small particles of pozzolan cement to increase the surface contact with the mixing water and provide rapid setting time. The beneficial effects of pozzolan addition, such as compressive strength performance and durability, are mostly due to the pozzolanic reaction in which calcium hydroxide is consumed to produce additional calcium silicate hydrate and calcium aluminate hydrate reaction products. These pozzolanic reaction products fill in pores and result in a refining of the pore size distribution or pore structure, which results in a lowered permeability of the binder which could be the reason for the lowest bond strength values between Endocem MTA and composite. Endocem MTA failed to fulfill the criterion of desirable SBS after its initial setting time (4 min).

**CONCLUSION**

Within the limitations of this study, it can be concluded that:

1. TheraCal LC showed maximum SBS when bonded with composite after their initial setting time. It fulfilled the criterion of desirable bond strength.

2. Biodentine and Endocem MTA showed significantly lower SBS values than TheraCal LC when bonded to composite after their initial setting time. Both failed to achieve the desirable SBS.

3. Cohesive failure, within the pulp capping materials, was noticed among all the samples in all the three groups. This shows that the interfacial bond strength between the universal adhesive and the pulp capping agents was higher than the inner strength of the pulp capping materials

4. Low SBS s with cohesive mode of failure in Biodentine and Endocem MTA indicate the low early bulk strengths of these materials.

TheraCal LC can be a material of choice as a liner under composite for single sitting restorative procedure, whereas Biodentine and Endocem MTA due to their low SBS values are not recommended as liners under composite for a single sitting restorative procedure.

Further studies are recommended to evaluate the SBS of Biodentine and Endocem MTA to resin composite with universal adhesive at various longer time intervals.

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**Conflicts of interest**
There are no conflicts of interest.

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