Shear bond strength of a novel light cured calcium silicate based-cement to resin composite using different adhesive systems

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INTRODUCTION

In recent years, considerable attention has been directed towards vital pulp therapy (VPT) in dentistry¹. Vital pulp therapy aims to preserve the vitality and health of the dental pulp following carious exposure or traumatic injury². In permanent adult teeth, several procedures have been described in order to preserve pulp vitality which include ‘controlled’ excavation of deep caries aiming to avoid pulp exposure followed by using biomaterials as a protective layer (indirect pulp capping)³, the use of biomaterials as a dressing over the exposed pulp tissue in direct pulp capping, or using the same biomaterials in pulpotomy procedures which involve partial amputation of the dental pulp⁴,⁵,⁶.

Biomaterials that are advocated for use in VPT procedures should possess certain characteristics such as biocompatibility, the ability to maintain pulp vitality, adherence to dentine and the restorative material, and also to resist forces during restoration placement and function⁷. Mineral trioxide aggregate has been used for this purpose due to its numerous advantages such as biocompatibility⁸,⁹, low solubility⁹, prevention of bacterial leakage¹⁰ and its ability to release calcium hydroxide¹¹. However, it has certain shortcomings such as difficulty in handling and a long setting time which might lead to leakage, and loss of marginal adaptation¹². Furthermore, since MTA requires moisture for complete setting, it is advocated to place a wet cotton pellet over MTA after its application¹³, this will necessitate additional appointments until the permanent restorative material can be used to restore the treated tooth.

In order to overcome the reported shortcomings of MTA, a recently introduced material, TheraCal LC (Bisco, Schaumburg, IL, USA), has been developed. This new material is marketed as a light-curable resin-modified calcium silicate based MTA designed for direct and indirect pulp capping procedures. TheraCal LC contains approximately 45% Portland Cement (PC), 10% radiopaque component (bismuth oxide), 5% hydrophilic thickening agent (fumed silica), and approximately 40% resin, by weight¹⁴. This new material has been reported to have a shorter setting time, lower solubility and higher flowability compared with conventional MTA¹⁵.

Glass ionomer cements have been used as liners, particularly under resin composites, because of their ability to chemically bond to enamel and dentin in addition to their ability to release fluoride¹⁶. They are also used as liners in indirect pulp capping procedures with similar clinical efficacy to contemporary pulp capping materials¹⁷.

The bond strength of restorative materials to pulp capping agents has been an issue of concern, since placement of a restoration following VPT procedures is crucial to ensure their success¹⁷. In spite of the massive advances in adhesive technology over the past decades, the bonded interface to tooth structure remains the weak point of an adhesive restoration¹⁸. The most commonly used test to assess bond strength is the macro-shear test which has gained popularity as a simple and quick method. Moreover, it is a very popular test that is commonly used to screen new adhesive formulations on their bonding effectiveness¹⁹. Standardization of the test protocol has been attempted by preparing specific jigs such as the SDI rig (SDI, Bayswater, Victoria, Australia) (Fig. 1a)²⁰, which is used to aid in proper adaptation and stabilization of the restorative material to the bonded substrate.

The shear bond strength (SBS) of TheraCal LC to resin composite was evaluated in comparison to Mineral trioxide aggregate (ProRoot MTA) and conventional glass ionomer cement (GIC) using two adhesive systems. A hole was prepared in 90 acrylic blocks (6 mm diameter, 2 mm deep) then filled with TheraCal LC, MTA or Fuji IX (n=30/group). Each group was bonded with either an etch and rinse or 1-step self-etch adhesive. Filtek Z250 composite was bonded to each capping material. Bond strength was tested in a universal testing machine, and data were analyzed using 2-way ANOVA and Duncan’s Multiple range test (p<0.05). TheraCal LC showed the highest SBS (p<0.001), while SBS of TheraCal LC and Fuji IX did not differ between either adhesive (p>0.05). TheraCal LC is the preferred choice in pulp capping procedures when using resin composite restorations.

Keywords: TheraCal LC, MTA, Shear bond strength, Resin composite
Several studies investigated SBS of restorative materials including resin composite and GICs to various pulp capping materials using several types of adhesive systems, however information regarding the bond strength between resin composite and TheraCal LC remains limited. Therefore the aim of the present study was to compare SBS of TheraCal LC to resin composite with that of MTA and a conventional GIC using two bonding systems (i.e. etch and rinse system and 1-step self-etch adhesive system). The null hypothesis tested was that SBS of TheraCal LC to resin composite is superior to that of MTA regardless of the adhesive system used.

MATERIALS AND METHODS

The materials used in the current study, their composition, mode of application and manufacturer details are shown in Table 1.

Specimen preparation
A total of 90 acrylic blocks (2 cm high×2 cm wide) containing a central hole 6 mm diameter and 2 mm deep were prepared using cold cure acrylic resin material (Metacryl, Metrodent, West Yorkshire, UK). Thirty blocks were filled with either TheraCal LC, MTA, or Fuji IX. TheraCal LC was inserted into each block (n=30) in two increments of 1 mm using a syringe-tip, then the cement’s surface was flattened using a metal spatula then polymerized with a light emitting diode light curing unit for 20 s (LED.B, Guilin Woodpecker Medical Instrument, Guilin, Guangxi, China) with an intensity of 1,200-mW cm². ProRoot MTA, the powder and liquid were mixed according to the manufacturer’s instructions, and placed in the acrylic blocks using a spatula and pressed flat with a glass slab. The MTA samples were covered with a damp cotton pellet and stored in an incubator at 100% humidity and 37° C for 48 h to allow complete setting.

For the GIC, Fuji IX capsules were mixed according to manufacturer instructions, then extruded into the acrylic blocks. The GIC surface of each sample was flattened using a metal spatula. TheraCal LC and Fuji IX samples were stored at 37° C in 100% humidity for 24 h prior to the bonding procedure.

Restorative material placement
TheraCal LC, MTA and GIC groups were divided into two subgroups (n=15) according to the adhesive system used to bond the resin composite (Table 1). Following application of the adhesive system, each acrylic specimen was placed into a shear bond apparatus (SDI Shear Bond Strength Rig, SDI Limited, Bayswater) (Fig. 1a) to ensure stability of the sample during the bonding procedure of resin composite to the capping materials. A cylindrical stainless steel mold with an internal diameter of 3.5 mm and height of 5.00 mm was placed centrally over the bonding site of each capping material in the acrylic specimen. The mold was filled with resin composite in two layers of 2 mm each and each layer was light-cured for 30 s. After polymerization, each bonded assembly was removed from the jig then placed into a container filled with distilled water for 24 h at 100% relative humidity and 37° C before SBS testing.

Shear bond strength test
Shear bond strength was measured by mounting the specimens in a universal testing machine (Computer control electromechanical universal testing machine model WDW-20, Jinan Chenda Testing Machine Manufacturing, China) and subjecting them to shearing force at a crosshead speed of 1.0 mm/min using a 0.5 mm wide chisel (Fig. 1b). Shear bond strength was calculated and expressed in MPa by dividing the peak load at failure to the specimen surface area according to the formula: SBS (MPa)=Load (N)/Area πr² (mm²).

Fracture analysis
Following the SBS test, the fractured surfaces were examined under a stereomicroscope (Stereomicroscope model XTX-W5C, Proway Optics and Electronics, Ningbo, China) at 20× magnification. The failure mode was classified as follows: (1) adhesive failure, failure at the capping material/resin composite interface, (2) cohesive failure, failure within the capping material or resin composite, and (3) mixed failure, a combination of...
Table 1 Materials used in the study, composition, mode of application and manufacturer information

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Mode/Steps of application</th>
</tr>
</thead>
</table>
| TheraCal™ LC®           | BISCO Dental Products, Schaumburg, IL, USA | AeroSil 8.0%, biocompatible hydrophilic resin 42.5% (Bis-GMA 20%, 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% | 1. Apply in incremental layers. (Layer is not to exceed 1 mm in depth)  
2. Light cure each increment for 20 s. |
| ProRoot® Mineral Trioxide Aggregates | Dentsply Tulsa Dental, OK, USA | Tricalcium silicate, bismuth oxide, dicalcium silicate, tricalcium aluminate, calcium sulfate dehydrate or gypsum | Mix powder/liquid ratio: 1/3. |
| GC Fuji™ IX GP®         | GC, Tokyo, Japan                   | Powder: Alumino-fluorosilicate glass. Liquid: Polyacrylic acid, polybasic carboxylic acid, water. | 1. Shake the capsule then push the plunger.  
2. Mix for 10 s in amalgamator.  
3. Load the capsule into applier and apply. |
| Adper™ Single bond 2    | 3M ESPE, St. Paul, IL, USA         | Scotchbond™ Etchant: 37% H₃PO₄, Adhesive: dimethacrylates, HEMA, colloidal silica, polyalkenoid acid copolymer, ethanol, water, photoinitiator | 1. Apply 35% phosphoric acid etchant 15 s.  
2. Rinse and dry with paper points.  
3. Apply 2 coats of adhesive.  
5. Light-polymerize 10 s. |
| Xeno® V®                | Dentsply Tulsa Dental              | Bifunctional acrylate, acidic acrylate, functionalized phosphoric acid ester, water, tertiary butanol, initiator, stabilizer. | 1. Dry surface.  
2. Apply adhesive.  
3. Agitate for 20s.  
5. Light-polymerize 10 s. |
| Filtek™ Z250 Universal Restorative System | 3M ESPE                        | Zirconia/silica filler, UDMA, Bis-GMA and Bis-EMA resins. | Light cure for 30 s. |

adhesive and cohesive failure.

**Statistical analysis**
Statistical analysis was done using commercially available software (SPSS 10.0, SPSS, Chicago, IL, USA). Means and standard deviation of the SBS of resin composite to each pulp capping material using the bonding systems was calculated. Two-way ANOVA was used to determine the effect of the adhesive systems, the materials and the cumulative effect of these two factors on SBS. *Post hoc* comparisons were carried out using the Duncan’s multiple range test (*p*<0.05). The assumption of normality was established using Leven’s test of homogeneity.

**RESULTS**
Analysis of data with two-way ANOVA demonstrated a statistically significant effect of the capping material as a factor on SBS to resin composite (*p*<0.05); however, there was no significant influence of the adhesive system on the SBS (*p*=0.886). Comparison of means, standard deviations and statistical analysis of the SBS among the tested groups is presented in Table 2. The TheraCal LC groups displayed the highest SBS values to resin composite using both adhesive systems compared with MTA and Fuji IX (*p*<0.05). The SBS of MTA to composite was the lowest among all tested groups, with the lowest value reported using the 1-step self-etch adhesive system. There was no significant difference in SBS of TheraCal LC to resin composite between the two adhesive systems; similarly, GIC had comparable values of SBS regardless of the adhesive system used.

A summary of fracture patterns is given in Table 3. Representative stereomicroscopic photo images of the failure modes are shown in Fig. 2. In the TheraCal LC group equal numbers of mixed and cohesive in composite failure modes were observed when using the etch and rinse adhesive system, failure modes in the 1-step self-etch adhesive system group were mostly mixed (Fig. 2b). In the MTA group, failure was mostly adhesive particularly for the 1-step self-etch adhesive system (Fig. 2c). GIC displayed similar fracture patterns for both adhesive systems with the samples failing mostly cohesively within the GIC (Fig. 2d).
Table 2  Mean values and standard deviation (SD) of SBS (MPa) of tested material to resin composite using two adhesive systems

<table>
<thead>
<tr>
<th>Capping Material</th>
<th>Bonding System</th>
<th>Mean (SD)</th>
<th>Mean (SD)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Etch and rinse system (Group 1)</td>
<td></td>
<td>TheraCal LC</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>MTA</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Fuji IX</td>
</tr>
</tbody>
</table>

\textsuperscript{a,b,c,d} Means within the table with different superscripts differ significantly (\(p<0.05\))

\*\(p\)-Value of ANOVA test of these means was <0.001

Table 3  Fracture modes of the tested materials after the shear bond test

<table>
<thead>
<tr>
<th>Mode of failure</th>
<th>Bonding system</th>
<th>Etch and rinse system</th>
<th>1-step self-etch system</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>TheraCal LC</td>
<td>Fuji IX</td>
</tr>
<tr>
<td>Adhesive</td>
<td></td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>Mixed</td>
<td></td>
<td>7</td>
<td>0</td>
</tr>
<tr>
<td>Cohesive in composite</td>
<td></td>
<td>7</td>
<td>0</td>
</tr>
<tr>
<td>Cohesive in capping material</td>
<td></td>
<td>1</td>
<td>6</td>
</tr>
</tbody>
</table>

DISCUSSION

Over the years, MTA has been increasingly used in various clinical procedures in general dentistry such as furcation repair, root resorption and pulp capping procedures in primary and permanent teeth\textsuperscript{24}. In recent years new materials such as the light cured MTA-like material (TheraCal LC) have been produced to overcome the difficulties in handling as well as the long setting time of conventional MTA\textsuperscript{14}. In addition to biocompatibility and bioactivity of pulp capping materials, their bond strength to restorative materials is important for the quality of fillings and success of restorations since good bonding produces an adhesive joint that is capable of spreading stress relatively evenly over the region of the lining-filling interface\textsuperscript{17} which will aid tolerating stresses created by polymerization shrinkage of overlying resin composite restorations.

The null hypothesis postulated in the current study was accepted. Regardless of the adhesive system used in the current study, TheraCal LC produced the highest SBS to resin composite with the self-etch adhesive showing higher bond strengths than the etch and rinse system, although this was not statistically significant. Similar trends were observed with the GIC while MTA displayed the opposite result.

Cantekin\textsuperscript{25} investigated the bond strength of TheraCal LC and ProRoot MTA to resin composite using...
a 2-step etch and rinse adhesive, and also a Silorane-based composite. The highest SBS values were in the TheraCal LC group when bonded to the resin composite (19.3 MPa). In the current study similar trends were observed with the TheraCal LC group despite a lower SBS value (12.84 MPa). The difference in SBS may be related to the different adhesive system and resin composite material used which have been reported as significant factors that may influence bond strength\(^{39}\). The higher evaporation pressure of acetone present in 2-step etch and rinse system used by Cantekin compared with ethanol solvent present in Single Bond 2 adhesive may have resulted in greater monomer infiltration and thus creating higher bond strengths\(^{30}\). In addition, the bond area in the current study (3.5 mm diameter) was greater than that used by Cantekin (2 mm diameter). It is well established that the larger the bonded area will produce lower SBS values\(^{27}\), due to the increased probability of the presence of critical sized defects\(^{39}\).

The higher SBS value of TheraCal LC compared with MTA and Fuji IX may be due in part to the resin content in TheraCal LC; which provides a chemical bond with the composite. Moreover, TheraCal LC contains a hydrophilic resin monomer that makes it an excellent adhesion promoter enhancing bond strength\(^{26,29,30}\). On the other hand, the lack of resin content in MTA and conventional GIC indicates that their bond to resin composite is purely micromechanical\(^{31}\).

Several studies evaluated SBS of resin composite to MTA compared with several pulp capping biomaterials bonded using different adhesive systems\(^{20,21,23,32-34}\) with variable results. Tunc \textit{et al.}\(^{23}\) evaluated the bond strength of White MTA (WMTA) to a resin composite and a compomer using an etch and rinse system (Adapter\textsuperscript{TM} Single Bond, 3M ESPE) and a 1-step self-etch adhesive system (Prompt L-Pop, 3M Dental Products, St Paul, MN, USA) after a 48 h storage of the MTA. The reported SBS values were higher than in the current study for both adhesive systems (13.22 and 10.73 MPa vs. 7.50 and 4.61 MPa respectively) similar to the findings by Atabek \textit{et al.}\(^{31}\). The difference in the reported values may be due to the smaller bonded area used by Tunc \textit{et al.}\(^{23}\) and different 1-step self-etch adhesive used. The reported values showed significantly higher SBS using etch and rinse system which is in agreement with the current study for MTA.

Altunsoy \textit{et al.}\(^{32}\) studied the SBS of MTA (Angelus), Biodentine and Calcium Enriched Mixture (CEM) to a flowable composite using a 1-step self-etch system and reported lower values compared to the current study (2.01 MPa vs. 4.61 MPa), in spite of the long setting time allowed for MTA (72 h vs. 48 h). Therefore, other factors such as polishing of MTA surface, type of restorative materials and bonding surface area may have affected the outcomes.

It has been previously shown that longer storage times of MTA enables it to reach its optimal physical properties which resulted in higher strength values and lower solubility\(^{35}\). Oskooei \textit{et al.}\(^{17}\) evaluated the SBS of MTA to Filtek Z250 using Single bond etch and rinse system. The results showed lower SBS compared with the current study (4.65 MPa vs. 7.50 MPa) which can be attributed to the shorter storage period allowed for MTA setting (24 h vs. 48 h in the current study), since it has been shown that acid etching procedures affected the hardness and strength of ProRoot MTA after mixing and may lead to displacement of MTA in a cavity and therefore affect its sealability\(^{35,36}\).

In the current study, SBS of the conventional GIC to resin composite was lower than TheraCal LC but significantly higher than MTA. Several factors such as the flow (viscosity) of GIC may be related to its bond strength to resin composite since it affects its adaptability to the surface; low adaptability may lead to partial defects on the restoration margin and small voids which may affect bond strength values to composite\(^{17}\). In addition, the lack of chemical bond between conventional GICs and composite in addition to the low cohesive strength of GICs lead to bond strengths that are likely to be lower\(^{38}\). The lower bond strength to Fuji IX in comparison to TheraCal LC may be attributed to the difference in cohesive strength of these materials. Moreover, during GIC placement, air bubbles may occur within the material which could lead to generation of stress concentration points\(^{39}\). This can also imply that in the GIC subgroups, the bond strength to composite is higher than the cohesive strengths of the capping materials.

A number of previous studies have reported that optimal bond strength to resin composite was achieved using an etch and rinse system since 35% phosphoric acid effectively removes the smear layer which in turn creates a clean surface for better micromechanical retention with composite, in addition to its ability to produce deeper and more retentive microporosities in enamel and dentin\(^{22,23,31,33,36}\). The lesser performance of self-etch adhesives has been attributed to: 1) combining the acidic hydrophilic monomer and hydrophobic monomers into a single step may interfere with the adhesive polymerization; 2) the intrinsically low strength of the adhesive and 3) the inferior degree of monomer polymerization due to the solvent/oxygen inhibition effect during light polymerization of these adhesives\(^{39}\). A number of studies reported that higher bond strength values to composite were achieved using 1-step self-etch adhesive systems\(^{22,40,41}\), which was attributed to a high pH value and better wettability from solvents\(^{22}\), and to the presence of 5% fillers content in the 1-step self-etch system which caused a decreased shrinkage stress leading to higher bond strength to composite\(^{41}\). This is in agreement with the results of the current study for TheraCal LC and GIC. Furthermore, it has been suggested that aggressive etching of GIC using phosphoric acid weakens the cement surface by preferential dissolution of the filler particles\(^{42}\). The zone of weakened cement is therefore more likely to fail cohesively when debonded from resin, possibly resulting in lower bond strengths\(^{40}\).

MTA exhibited higher bond strength to composite when using the etch and rinse system which is in
agreement with previous studies\textsuperscript{30,32,33}. The surface of MTA samples etched with phosphoric acid observed in previous studies\textsuperscript{26,41} showed a crystalline structure that is eroded and cracked following phosphoric acid treatment. This may have contributed to the higher bond strength compared with the weaker acid treatment from the self-etch adhesive system. Furthermore, the water content of MTA could have interfered with the polymerization of the self-etch adhesives\textsuperscript{30} thereby resulting in reduced MTA-adhesive bond strength values and therefore, more adhesive failures of Xeno V+to MTA were found.

To accomplish a successful restorative procedure when two different materials are used, there should be an appropriate bond between the two materials\textsuperscript{43}. Generally, the bond is acceptable when fracture occurs inside each material rather than in the bonded interface (i.e. cohesive rather than adhesive)\textsuperscript{44}. Failure mode analysis showed a greater number of samples exhibited mixed and cohesive failures within the capping material in the TheraCal LC and GIC groups respectively compared with MTA which mainly showed adhesive failure modes which may indicate a stronger bond in the former groups. The higher rate of cohesive failure in tested samples suggests a higher bond strength which has been reported by previous researchers.

It has been previously established that exposure of MTA to a low pH environment may influence its physical and chemical properties. In this context, the acid-etch procedure using phosphoric acid reduces the surface micro-hardness of MTA and most likely weakens its structure\textsuperscript{36}, and this can be the explanation of the greater frequency of cohesive failure in MTA for specimens bonded with etch-and-rinse systems\textsuperscript{35}.

The 1-step self-etch adhesive system has pH of 1.3 which is considered intermediary strong compared with strong phosphoric acid (pH=0.1–0.4)\textsuperscript{30}. Since Xeno V\textsuperscript{+} has lower acidity its use was associated with lower number of cohesive failures in MTA as it does not weaken the MTA structure. Moreover, the self-etch adhesive systems do not etch the surface to the same depth as phosphoric acid.

Within the limitation of this laboratory study it was concluded that TheraCal LC produced the highest SBS to resin composite compared with MTA and Fuji IX. Moreover, its SBS was not affected by type of adhesive system used. It would be clinically beneficial for patients and dentists alike if the final adhesive restoration can be placed over pulp capping material during the same visit, decreasing time and cost. Therefore, the simple application and high bond strength capacity of TheraCal LC to resin composite may support its use and consideration as the material of choice in direct pulp capping. In addition, when placing composite materials over MTA, it might be advisable to use an etch and rinse system prior to composite resin bonding.

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