# Shear Bond Strength of a Resin Composite to Six Pulp Capping Materials Used in Primary Teeth

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***Aim:*** *The purpose of this in vitro study was to assess the shear bond strength (SBS) and bond failure types of a resin-composite to six pulp-capping materials used in primary teeth.* ***Study design:*** *Eight-disc specimens from each pulp-capping material (6 groups) to bond to Filtek™ Z350 XT Flowable using a standard PVC tube (2X2mm). All groups were prepared according to the instruction of the manufacturer. The SBS was measured with a crosshead speed of 0.5 mm/min using a universal testing machine. Failure mode evaluation was completed using Digital Microscope by two independent examiners.* ***Results:*** *Urbical LC® showed the highest SBS (Mean±SD) followed by ProRoot® MTA and TheraCal LC® (35.422±2.910, 22.114±2.515, and 21.175±1.983) respectively. ANOVA showed significant differences between all groups (P=0.0001). Urbical LC® and Photac™ Fil QuickAplicap™ were statistically significant different from all other pulp-capping materials groups. ProRoot® MTA was statistically significant different than Biodentine® (P=0.0001) and Photac™ Fil (P=0.0001). The total number of bond failure was recorded for cohesive B failure/cohesive in the pulp-capping material (14) and adhesive failure (14).* ***Conclusion:*** *Most of the tested pulp-capping materials bonded to Filtek™ Z350 XT demonstrated clinically acceptable and high SBS. Urbical LC showed the highest SBS while Biodentine® showed the lowest SBS.*

***Keywords:*** *Shear Bond Strength, Pulp Capping Materials, Adhesives, Biodentine, TheraCal LC, ProRoot MTA*

## INTRODUCTION

Pulp capping materials are commonly used in vital deep carious teeth, to avoid pulp exposure and be able to heal from the carious insult.[1] When the remaining dentin thickness is lesser than 1.5mm pulp capping is placed to avoid microleakage which has been linked with secondary caries and bacterial contamination.[1] The restorative material placed over capping materials should seal completely the involved dentin from the oral environment.[2] This concept is backed up by the currently used criteria within adhesive dentistry in that tooth preparation for resin composite restorations should protect the maximum amount of tooth structure possible.[1]

A focus on vital pulp therapy and its objective of maintaining dental pulp health with deep caries and after carious pulp exposures and traumatic injuries has been raised recently.[1,2] The choice of pulp therapy in vital primary teeth with deep carious lesions should be based on a biological approach for caries-affected dentin removal, pulp exposures (if any), reported adverse effects (if any), clinical expertise, and patient preferences.[2,3] Pulp capping is defined as placement of a medicament directly over the exposed pulp (direct pulp cap), or a cavity liner or sealer is placed over residual caries (indirect pulp cap) in an attempt to maintain pulp vitality and avoid the more extensive treatment dictated by extraction or endodontic therapy.[3] The selection of pulp capping biomaterials drastically impacts the success of vital pulp therapy.[1,2] An ideal pulp capping material should be capable of producing reparative dentin, promote

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substantial antibacterial activity, and have appropriate biocompatibility.[1-3] Biomaterials possessing features such as the ability to counteract forces for the course of restoration placement and function, biocompatibility, and the ability to keep pulp vitality and bond the restorative material and dentin, should be encouraged to use in vital pulp procedures.[1-3]

Offering a choice for reparative dentin formation with antibacterial properties, calcium hydroxide is the most commonly utilized for both indirect and direct pulp capping.[4] Some of the common drawbacks of the utilization of calcium hydroxide include development of the material-pulp interface's necrotic layer, surface inflammation, dissolution over time whereby the cap cannot defend and seal the pulp against microleakage, and possible tunnel flaws within the dentin.[4] Resin-modified glass ionomer cement have been utilized as liners, especially under resin composites because their improved bond strength due to its chemical bonding as well as in releasing fluoride.[5] Mineral trioxide aggregate (MTA) has become an increasingly commonly used substitute for calcium hydroxide.[6,7] The physical and chemical advantages of MTA include the biocompatibility, antibacterial activity, and sealing features which promoted its clinical applications in perforation sealing, pulpotomy procedure, open apexes, pulp capping, root canal filling, and root end filling.[6,7] Recent clinical investigations have concluded that MTA-based materials may be trusted to be applied as pulp capping.[6,7] The drawbacks of MTA include potentially tooth discoloration, it is complex to apply, and it possesses high levels of solubility.[6,7] As a result of the ever-increasing requirement to tackle the drawbacks of MTA, a number of novel calcium silicate-based capping materials have been formulated in recent years, one of them being Biodentine.[8] Biodentine is designed as a dentin alternative to pulp capping, resin composite restorations, and endodontic repair material.[6-8] Biodentine is high-purity calcium silicate-based dental cement composed of calcium carbonate (filler), zirconium oxide (radiopacifier), tricalcium silicate, and a water-based fluid comprising calcium chloride as a water-reducing mean to have shorter final and initial setting times which will additionally accelerate the rate of initial strength conversion [6,8] Biodentine has higher compressive strengths, sealing ability, superior biocompatibility and bioactivity, lesser setting time, lesser cytotoxic effect, and enhanced antibacterial activity when compared to MTA.[8] TheraCal LC is a novel calcium silicate-based, resin-modified material containing barium zirconate, tricalcium silicate particles, and polyethylene-glycol dimethacrylate monomers and indicated for application in indirect and direct pulp capping.[5,9] TheraCal LC release calcium, which stimulate the formation of reparative dentin and apatite.[9-11] In addition, due to its handling features and superior flowability compared to MTA, TheraCal LC possesses good sealing abilities.[9] The material's minimal solubility and elevated physical features allows for instantaneous final restorative material placement after light cure of TheraCal LC.[9-11]

There is a limited research on the information concerning the bond strength of the recently capping materials such as Biodentine, Urbical LC® and TheraCal LC to resin composites, despite having been a great number of previously conducted investigations exploring the bond between varying resin composites and pulp capping materials. Therefore, the objective of this *in vitro* investigation was to determine the SBSs and failure mode of six different pulp capping materials used in primary teeth including Biodentine, Urbical LC® and TheraCal LC to a resin composite restorative material. The null hypothesis was no significant difference between SBSs and failure mode of the tested six pulp capping materials to the used resin composite restorative material.

## MATERIALS AND METHOD

### Specimen Preparation

This investigation protocol was approved by the Research and Ethical Committee of Human Studies at King Saud University, College of Dentistry Research Center. In this study, six pulp-capping materials were used. Table 1 shows a summary of the chemical composition and application procedures of the materials used in this study. At the level of significance $\sigma$ =0.05 with estimated standard deviation = 0.85, power=82, the sample size should be at least 8 in each group. Eight-disc specimens from each pulp-capping material were prepared according to the instructions provided by the manufacturers. Each disc was made using cylindrical metal molds 5-mm diameter x 2-mm thick. The molds were placed onto a glass microscopic slide and the materials were placed in the mold, and then Mylar® strip (Mylar Uni-Strip, Caulk/Dentsply, Milford, DE, USA) and a glass microscopic slide were placed on the top surface of each specimen. The glass slide was pressed until it has a tight contact with the metal mold to flatten the surface. The metal mold has a notch in the bottom surface of each specimen to facilitate identification of the top surface where surface was used for surface treatment and bonding. Where applicable every specimen was light cured (Elipar Highlight, 3M ESPE, St. Paul, MN, USA) on each side according to the instructions of the manufacturer. The glass slide and Mylar® strip were removed. All specimens were prepared at room temperature (approximately 25$^{o}$C). Following preparations, all specimens were stored in containers containing 30 ml of distilled water (pH 6.8) in an incubator/humidifier (GI2 So-Low Cincinnati, OH, USA) at 37°C for 24 hours.

### Preparation for Bonding

Each specimen from the six groups of the pulp-capping material was embedded in cylindrical mold filled with acrylic resin (OrthoJet, Lang Dental MFG. Co., Inc., IL, USA) in preparation for bonding. For all groups (1-6) of the pulp-capping materials, each surface was prepared according to the instruction of the manufacturer to bond to Filtek™ Z350 XT Flowable Restorative (3M ESPE, St. Paul, MN, USA) which is a low-viscosity, visible light-cured, radio-opaque flowable nanocomposite. The 35% phosphoric acid etchant (3M ESPE Scotchbond™ Etchant) was applied for 15 seconds and rinsed for 10 seconds. Two consecutive coats of the Adper Single Bond 2 adhesive were applied followed by application of gently air for five seconds to thin the adhesive and then light cure for 10 seconds. Filtek™ Z350 XT Flowable Restorative shade A1 was inserted into a 2X2mm PVC tube which was placed perpendicularly to surface of each specimen and light cure for 20 seconds.

### Bond Strength Testing

The specimens were stored for a period of 72 hours at a temperature of 37ºC with 100% humidity prior to SBSs testing. The s SBSs in MPa were measured with a crosshead speed of 0.5 mm/min using a universal testing machine (Inströn, model no. 8500, Illinois Tool Works Inc., Norwood, MA, USA).

### Failure Mode Evaluation

Following the SBS test, the fractured surfaces were examined using Digital Microscope KH-7700 (Hirox Europe Ltd., Limonest, France) at x40 magnification by two examiners who were uninformed about the experimental groups. The failure mode was classified as follows: Cohesive A = Cohesive in the restorative material, Cohesive B = Cohesive in the pulp capping material, Adhesive failure = Failure at the capping material/resin composite interface, and Mixed failure = A combination of adhesive and cohesive failure (When two modes of failure occur simultaneously).[4,5]

### Statistical Analysis

Descriptive statistics (Mean, standard deviation, standard error, minimum, and maximum) were used to describe the quantitative outcome variable (SBS). A one-way analysis of variance (ANOVA) was used to compare the mean values of these outcome variables, followed by Tukey's HSD post hoc test for multiple comparisons of mean values. Kappa statistics was calculated to quantify an agreement between the two examiners in assessing the types of failures in each of the treatment groups. All statistical analyses were set with a significance level of $p<0.05$. The statistical analysis was carried out with SPSS version 21.0 (Statistical Package for the Social Sciences, SPSS, Chicago, Illinois, USA).

## RESULTS

The mean, standard deviation, standard error, and range of SBS values in MPa of all groups are presented in Table 2 and Figure 1. Urbical LC® showed the highest SBS (Mean$\pm$SD) followed by ProRoot® MTA and TheraCal LC® (35.422 $\pm$ 2.910, 22.114 $\pm$ 2.515, and 21.175 $\pm$ 1.983) respectively. Whereas Biodentine® showed the lowest SBS 10.554 $\pm$ 1.486. A one-way ANOVA showed significant differences between all groups (P=0.0001). Comparison of the mean using Tukey's HSD post hoc test showed the statistically significant differences between the groups (Table 3). Urbical LC® and Photac™ Fil QuickAplicap™ were statistically significant different from all other pulp-capping materials groups. While ProRoot® MTA was statistically significant different than Biodentine® (P=0.0001) and Photac™ Fil QuickAplicap™ (P=0.0001).

Cohen's kappa coefficient which measured inter-rater agreement of bond failure type was 0.888, which indicated strong level of agreement. Table 4 shows bond failure type and distribution (frequency/%) for different groups. The highest total number of bond failures were recorded for cohesive B failure/cohesive in the pulp-capping material (14) and adhesive failure (14). The total Cohesive A = Cohesive in the restorative material was 11while Mixed failure = A combination of adhesive and cohesive failure was 9.

## DISCUSSION

When it comes to the restoration success, the bond strength between the pulp capping materials and the restorative materials with sufficient adhesive joint is of high importance.[8,12] This sufficient adhesive joint should be able to spread stress fairly enough over the whole bond's area.[8,12] A vital procedure within restorative dentistry is the bonding between resin composites and the pulp capping biomaterials in order to lessen contraction forces to allow the creation of gap-free restoration margins and to have needed suitable bond strength.[5,13,14] Bond strength assessments are currently the most popular approaches in estimating the adhesive features of dental materials.[15,16] The null hypothesis of this study was rejected as there was difference between SBSs and failure mode of the tested

**Table 1: Compositions, manufacturers, and steps of application of the six pulp-capping materials used in this study**

| Pulp-Capping Materials | Manufacturers | Compositions | Steps of Application |
|---|---|---|---|
| ProRoot® MTA (Mineral Trioxide Aggregate) | DENTSPLY, Tulsa, OK, USA | Bismuth oxide, tricalcium silicate, dicalcium silicate, calcium dialuminate, and calcium sulfate dehydrated | Mixed powder/liquid ratio: 1/3 |
| Urbical LC® (Calcium-hydroxide) | Promedica, Neumuenster, Germany | Dimethacrylates, calcium hydroxide, pigments, initiators, silicate fillers | Apply Urbical LC directly above the needed area, and remove any excess, light cure the material for 40 seconds |
| TheraCal LC® ( A light-cured, resin-modified calcium silicate filled liner | Bisco Dental Products, Schaumburg, Illinois, USA | AeroSil 8.0%, biocompatible hydrophilic resin 42.5% (Bis-GMA 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% | Apply in incremental layers (Layer is not to exceed 1 mm in depth). Light cure each increment for 20 s. |
| Biodentine® (Bioactive Dentin Substitute) | Septodont, Lancaster, PA, USA | Tricalcium silicate powder<br>Aqueous calcium chloride solution and excipients | Activate the capsule and place on a mixing device for 30 s. |
| Photac™ Fil QuickAplicap™ (Resin-modified glass-ionomer), | 3M ESPE, St. Paul, MN, USA | Glass powder, surface modified with 2-propenoic acid, 2 methyl-3-(trimethoxysilyl)propyl ester,bulk material | Activate the capsule and then mix it with an amalgamator 10 sec (working time > 2 minute), light cure for 20 sec |
| GC Fuji II LC® Capsule (Resin-modified glass-ionomer) | GC America, IL,USA | Powder: Aluminofluorosilicate glass.<br>Liquid: Polyacrylic acid, tartaric acid, distilled water, camphorquinone,<br>dibutyl hydroxy toluene, and three resin complex (mainly HEMA) | Activate the capsule and then mix it with an amalgamator 10 sec (working time > 2.5minute), light cure for 20 sec |

**Table 2: Mean, standard deviation, standard error, and range of SBS values in MPa of all groups**

| Pulp-Capping Materials | N | Mean | Std. Deviation | Std. Error | Minimum | Maximum |
|---|---|---|---|---|---|---|
| Urbical LC® | 8 | 35.422 | 2.910 | 1.029 | 30.321 | 38.580 |
| ProRoot® MTA | 8 | 22.114 | 2.515 | 0.8892 | 18.888 | 25.932 |
| Biodentine® | 8 | 10.554 | 1.486 | 0.525 | 9.052 | 13.263 |
| TheraCal LC® | 8 | 21.175 | 1.983 | 0.701 | 17.557 | 24.020 |
| GC Fuji II LC® CAPSULE | 8 | 20.727 | 2.901 | 1.026 | 16.767 | 23.955 |
| Photac™ Fil QuickAplicap™ | 8 | 14.686 | 2.250 | 0.795 | 11.032 | 18.104 |

**Table 3: Comparison of the mean between different groups**

| Pulp-Capping Materials | 95% Confidence Interval for Mean | | Pulp-Capping Materials | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Lower Bound | Upper Bound | Urbical LC | ProRoot® MTA | Biodentine | TheraCal LC | GC Fuji II LC® CAPSULE | Photac™ Fil QuickAplicap™ |
| Urbical LC® | 32.990 | 37.855 | 1 | | | | | |
| ProRoot® MTA | 20.011 | 24.217 | 0.0001* | 1 | | | | |
| Biodentine® | 9.312 | 11.796 | 0.0001* | 0.0001* | 1 | | | |
| TheraCal LC® | 19.518 | 22.833 | 0.0001* | 0.969 | 0.0001* | 1 | | |
| GC Fuji II LC® CAPSULE | 18.302 | 23.152 | 0.0001* | 0.854 | 0.0001* | 0.999 | 1 | |
| Photac™ Fil QuickAplicap™ | 12.805 | 16.567 | 0.0001* | 0.0001* | 0.015* | 0.0001* | 0.0001* | 1 |

*Statistically significant

**Table 4: Bond failure type and distribution (frequency/%) for different groups**

| Pulp-Capping Materials | Bond Failure Type (N/%) | | | |
|---|---|---|---|---|
| | Cohesive A* | Cohesive B** | Adhesive*** | Mixed**** |
| Urbical LC® | 0/0 | 7/87.5 | 0/0 | 1/12.5 |
| ProRoot® MTA | 1/12.5 | 4/50 | 3/37.5 | 0/0 |
| Biodentine® | 3/37.5 | 0/0 | 0/0 | 5/62.5 |
| TheraCal LC® | 5/62.5 | 0/0 | 0/0 | 3/37.5 |
| GC Fuji II LC® Capsule | 0/0 | 2/25 | 6/75 | 0/0 |
| Photac™ Fil QuickAplicap™ | 2/25 | 1/12.5 | 5/62.5 | 0/0 |
| Total | 11/22.92 | 14/29.17 | 14/29.17 | 9/18.75 |

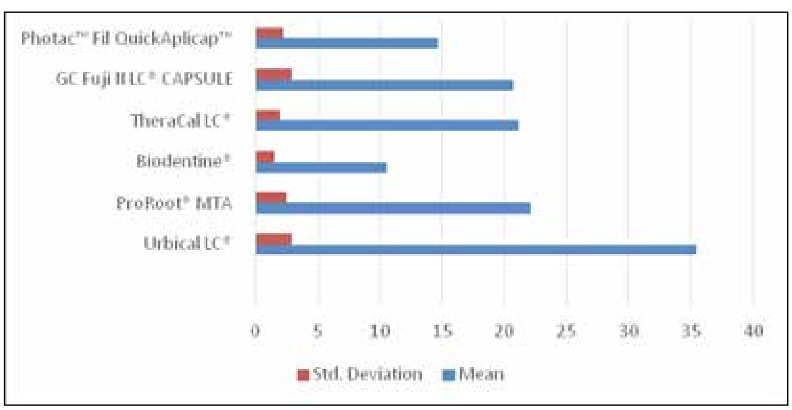
*Cohesive A = Cohesive in the restorative material

**Cohesive B = Cohesive in the pulp capping material

***Adhesive failure = Failure at the capping material/resin composite interface

****Mixed failure = A combination of adhesive and cohesive failure

**Figure 1: Mean and standard deviation of SBS in MPa for all groups**



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six pulp capping materials to the used resin composite restorative material. An essential element of the successful restoration is the assurance that pulpal health is sealed and maintained during the pulp capping process.[3] It has been reported that dentin bond strengths in the range of 17-20 MPa are sufficient to resist the polymerization shrinkage of resin composites and accepted as SBS.[17]

The findings of the current investigation concluded that Urbical LC® demonstrated the highest (35.42 MPa) SBS, whilst Biodentine® demonstrated the lowest SBS (10.55 MPa) among the included pulp capping materials. The notably high SBS of Urbical LC® is considered to be clinically very reliable to resist contraction forces as well as any given margin defects to come afterward as the recorded SBS was 35.42 MPa which fulfils the required reported range of SBSs (17-20 MPa).[17] To the best of our knowledge only one investigation compared SBS of Urbical LC® to the resin composite. Another study compared SBS of Urbical LC (22.359±0.952) to Filtek Z250 XT etched with phosphoric acid[14]. In addition, SBS of calcium enriched mixture cement to a resin composite was reported to be 3.24 MPa and 1.91 MPa with or without acid etching respectively with no significant difference.[18] The SBS of MTA to the resin composite recorded in this study was 22.11 MPa which is similar to another study (18.69 MPa)[13] and considered suitable to resist contraction forces whilst simultaneously ensuring leakage-free restoration.[8,10,11] The results of this study showed that the SBS for GC Fuji II LC (RMGIC) was 20.72 MPa while for Photoac Fil was 14.68 MPa. These results correlate with the conclusions drawn from other studies on MTA.[18-20]

In this study Biodentine demonstrated a SBS of 10.55 MPa as a capping material bonded to the tested resin composite. This result correlates with a previous investigations which reported SBS of 5.67 MPa and 9.34 MPa of Biodentine to a resin composite.[5,13] This low SBS may be due to the setting reaction which takes a duration of roughly 12 minutes, while the complete maturation can take from 2 weeks to a month.[13] As a result of this, Biodentine setting reactions may impact the restorative material and capping bond strength.[13] In this study, the SBS of TheraCal LC to the resin composite was 21.17 MPa which was close to the bond strength reported in a previous investigations (18.25 MPa).[5] Biodentine and TheraCal LC both release silicon and calcium ions into the underlying dentin and when compared to the RMGIC fluoride ions, silica is a superior stimulant for dentin matrix remineralization.[5,21,22] Both capping materials are sufficiently accepted by preserved odontoblast cells, as demonstrated by cytotoxicity studies.[5,21-23] Further, the results from such investigations are related to the application of Biodentine and TheraCal LC as a substitute for RMGIC, MTA, and calcium hydroxide, and as a liner, assuming the bond is suitable to sustain polymerization stresses (17-20 MPa).[5,17,22] Our results demonstrated that Biodentine did not fulfill these necessities for a suitable bond strength with the resin composite while TheraCal did.

When it comes to stimulating reparative dentin formation calcium hydroxide offers pulpal compatibility and ability.[18] However, calcium hydroxide has several undesired features such as they vanish over time, possess insufficient mechanical features, and offer a too-weak protection against microleakage.[4,18,23] Bearing this in mind, light-activated calcium-hydroxide products have been put forward, which have obtained several enhancements when it comes to their physical features.[4,18,23]

In this study, the examination of the failure modes demonstrated that the two most common failure modes among all investigated pulp capping materials were cohesive B failure/cohesive in the pulp-capping material (14) and adhesive failure (14). This result correlates with previously conducted studies concerning MTA with more cohesive failure and Biodentine with more adhesive failure.[13] In contrast, the two main failure types recorded in the present study within Biodentine group were mixed and Cohesive A equivalent to Cohesive in the restorative material types. Whilst the most common type of failure within the ProRoot MTA was cohesive B failure/cohesive in the pulp-capping material. The most noted mode of failure within the RMGIC-based materials was that of adhesive type, whereas the TheraCal LC pulp capping material was Cohesive A = Cohesive in the restorative material types. The mixed and cohesive failures reported for the MTA and Biodentine groups may have been as a result of the small compressive strengths of those materials.[6-8,24]

Taking the best material properties and combining them would always be the objective when aiming to identify the most promising restoration in mind of maintaining pulpal health. One of the limitations of this study was the use of one resin composite and not using more pulp capping materials such as glass-ionomer cement. It would be beneficial if more and different restorative materials and etch and rinse as well as selfetch adhesive systems is tested. Another limitation is measuring bond strength within a short period. It would be beneficial if bond strengths occur after aging the specimens and thermocycling. The results of this investigation should consider the *in vitro* setting of the study, which may not simulate cumulative long-term effect *in vivo*. However, the clinical condition in the mouth is not easy to mimic in the laboratory.[25] On the other hand, in this *in vitro* study, standardization of experimental conditions was advantage and the research does describe a number of positive links between *in vitro* efficacy and clinical efficacy. In addition, the results demonstrated a clear correlation between SBS of the six tested pulp-capping biomaterials to the tested resin composite.

## CONCLUSIONS

Under the experimental conditions and the methodology of this *in vitro* study, the following conclusions can be made:

1. Most of the tested pulp-capping materials bonded to Filtek™ Z350 XT Flowable Restorative demonstrated clinically acceptable and high SBS.
2. Urbical LC showed the highest SBS to Filtek™ Z350 XT Flowable Restorative.
3. Biodentine® showed the lowest SBS to Filtek™ Z350 XT Flowable Restorative which considered unacceptable.
4. ProRoot MTA and TheraCal LC demonstrated SBSs to Filtek™ Z350 XT Flowable Restorative resin composites within the acceptable range.
5. The total number of bond failure was recorded for cohesive B failure/cohesive in the pulp-capping material (14) and adhesive failure (14).

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