

Dentin bond strength of resin-modified light-curable calcium-silicate-based material

Raissa Dias Fares,¹ Nancy Kudsi Carvalho,¹ Marina Carvalho Prado,¹ Erick M. Souza,² Plinio Mendes Senna,¹ Emmanuel João Nogueira Leal da Silva¹

¹Department of Endodontics, School of Dentistry, Grande Rio University (UNIGRANRIO), Rio de Janeiro, RJ, Brazil

²Department of Endodontics, Maranhão Federal University (UFMA), São Luiz, MA, Brazil

• Conflicts of interest: none declared.

ABSTRACT

Objective: this study aimed to compare the dentin bond strength of a resin-modified light-curable calcium-silicate-based material (TheraCal LC[®]; Bisco, Schaumburg, IL, USA) with White MTA (WMTA[®]; Angelus, Londrina, PR, Brazil). **Materials and Methods:** sixteen human maxillary incisors and canines were selected and three 1-mm-discs were obtained from the middle third of each root. On the coronal surface of each disc, two 1.2-mm-wide-holes were drilled through the dentin. Then, artificial holes were filled with one of the tested materials: WMTA[®] and TheraCal LC[®]. The filled dental slices were stored in a phosphate-buffered saline (PBS) solution (pH 7.2) for 7 days at 37°C. After that, push-out assessment was performed with a 1.0-mm-plunger-tip. Load was applied at a crosshead speed of 0.5 mm/min until sealer displacement. The results were expressed in MPa. Mann-Whitney U test was applied to rank materials regarding dentin push-out bond strength. Significance level was set at $\alpha = 5\%$.

Results: All specimens showed measurable results and no premature failure occurred. TheraCal LC[®] demonstrated superior push-out bond strength values to dentin when compared to WMTA[®] ($P < 0.0001$). **Conclusions:** there is advantage of TheraCal LC[®] over WMTA[®] as regards to the push-out bond strength and, therefore it may be taken as a promising and innovative reparative material.

Keywords: endodontics. root canal obturation. root canal therapy.

Introduction

During pulp-capping procedures, covering materials are applied over directly or indirectly exposed pulp, acting as a barrier for the protection of the dental-pulp complex and preservation of the pulp vitality.¹⁻⁴ Calcium-silicate-based materials such as mineral trioxide aggregate (MTA) are commonly used for pulp-capping despite the main drawbacks related to difficult handling, extended setting time and tooth discoloration.^{5,6} As MTA does fulfill all requirements for a pulp-capping material, calcium-silicate-based materials with different compositions have been designed to overcome these limitations, exhibiting improved physicochemical properties.⁷ In this sense, TheraCal LC[®] (Bisco, Schaumburg, IL, USA) is a resin-modified light-curable calcium-silicate filled material that is designed for pulp-capping and protective liner under base materials (e.g., composites and cements).

According to the manufacturer, TheraCal LC[®] is a singular stable and durable calcium-silicate based material that can be used as a liner or base due to its hydrophilic resin formulation. This material is composed of tricalcium-silicate particles in a hydrophilic monomer, which provides calcium release and low solubility properties that offers major advantages in direct pulp-capping therapies.⁸⁻¹¹ Even though the heat generated by light-cured pulp capping materials represents a matter of concern¹², TheraCal LC[®] demonstrated similar ability to MTA regarding the induction of differentiation

and mineralization of human dental pulp cells.¹³ In addition to that, when TheraCal LC[®] was applied in primate pulps, dentin bridges and mild inflammation in a level acceptable for pulp capping were obtained.¹⁴ Moreover, in response to lactic or hydrochloric acid, pH values of TheraCal LC[®] increase significantly (final pH 9.2-11.3).¹⁵

In contrast to MTA, TheraCal LC[®] presents easy handling resulting from thixotropic properties (becoming less viscous when subjected to stress) and the light-cured set, which permits immediate placement and condensation of the restorative material (<http://www.bisco.com/>).¹⁶ In this context, the use of TheraCal LC[®] seems to be clinically beneficial for patients and practitioners because it permits the final adhesive restoration over pulp-capping material during the same appointment.³

Despite the fact that TheraCal LC[®] has a new interesting approach, the current level of available evidence lacks relevant aspects of TheraCal LC[®], such as the bond strength to dentin that is yet not evaluated. Because TheraCal LC[®] is meant to direct or indirect pulp-capping its overall stability in contact to dentin is an important parameter of its overall performance. Thereby, this study aimed to compare the dentin bond strength of this resin-modified light-curable calcium-silicate pulp protectant/liner with White MTA (WMTA[®]; Angelus, Londrina, PR, Brazil). The following null hypothesis was tested: there is no difference in the push-out performance of both materials evaluated (TheraCal LC[®] and WMTA[®]).

Materials and Methods

Sample Size Calculation

From a prior study,¹⁷ an effect size of 1.39 was added to power $\beta = 95\%$ and $\alpha = 5\%$ and input into a T test family between two independent means (G*power 3.1 for Macintosh). As a result, a total size of 30 slices samples (15 per group) was required for the reliable identification of differences between the groups evaluated.

Specimens Preparation

This study was approved by the local Ethics Committee. Sixteen maxillary canines extracted for periodontal causes were selected. All specimens were previously cleaned – through the removal of hard deposits and soft tissues using curettes and immersing specimens in 5.25% sodium hypochlorite (NaOCl) during 10 minutes – and stored in 0.1% thymol solution at 5 °C. Following this, the coronal and apical root segments were removed from each tooth by using a low-speed saw (ISOMET; Buehler Ltd, Lake Buff, IL) with a diamond disk (Ø 125 mm x 0.35 mm x 12,7 mm; Buehler Ltd), preserving solely the middle third. Under constant water irrigation, 3 horizontal cross-sections (1 ± 0.1 mm thick) were prepared in the remaining middle segment (Figure 1A). Then, the final thickness of each slice was confirmed with the use of a high accuracy digital caliper (Avenger Products, North Plains, OR). Forty-eight root slices were produced following this protocol.

Preparation of Canal-like Holes for the Push-out Assay

Slices were drilled by using a 1.2-mm-cylindrical-carbide-bur, under continuous water irrigation. Two canal-like holes were obtained parallel to the root canal in each root slice by the aid of a vertical drill stand (Dremel Workstation 220, Mount Prospect, IL) in such a way as to standardize the drilling perpendicular to the surface, determining a minimum distance of 1-mm between the holes drilled, the external cementum, and the main root canal walls (Figure 1B). After examining specimens as regards to the presence of dentinal cracks, adequate perpendicular drilling and 1-mm minimum thickness, eighteen slices were excluded from the

study. Afterwards, a final total sample of thirty slices were immersed in a recipient with saline solution and ultrasonic irrigation for 15 minutes.

After irrigation, holes were dried with absorbent paper, and the slices were fixed on a glass plate to stabilize the samples and favor the adequate insertion of the materials. Following this, each hole was randomly signaled, in order to identify the material, and holes were filled with WMTA® or TheraCal LC® (Figure 1C). The creation of bubbles was avoided by the gentle vibration whilst placing materials. Both TheraCal LC® and WMTA® were mixed according to the manufacturers' instructions (Table 1). In a sterilize glass slab, WMTA® was mixed for 30 seconds with distilled water. When the mixture was homogeneous and with a consistency similar to wet sand, the cement was placed in the selected hole with the aid of an MTA carrier delivery system. Then, it was compacted to allow its adaptation into the holes' walls. TheraCal LC® was directly applied into the selected hole with precise placement and condensation. Following this, it was photopolymerized for 20 seconds. Lastly, before the push-out evaluation, specimens were stored in contact with phosphate-buffered saline (PBS) solution (pH 7.2) at 37°C for 7 days.

Push-out Assessment

A universal testing machine (Instron, Canton, Massachusetts, USA) was used to measure the values of push-out groups. Loading was performed with a plunger tip of 1.0-mm-diameter that was positioned over only one of the tested materials for each analysis. The load was constantly applied in a coronal-apical direction with a speed of 0.5 mm/min, until filling-material dislodgment occurred. During the push-out test, a real-time software program (specific for the universal testing machine) was used to plot a load (N) x displacement (mm). The median value of push-out bond strength was calculated to obtain the MPa data according to the following formula^{18,19}:

The adhesion area of endodontic sealers was defined by the cylinder area method, where $\pi = \text{constant } 3.14$; $r = \text{hole radius filled with root sealers (0.6 mm)}$; and $h = \text{thickness of dentin slice (1.0 mm)}$ ^{18,19}:

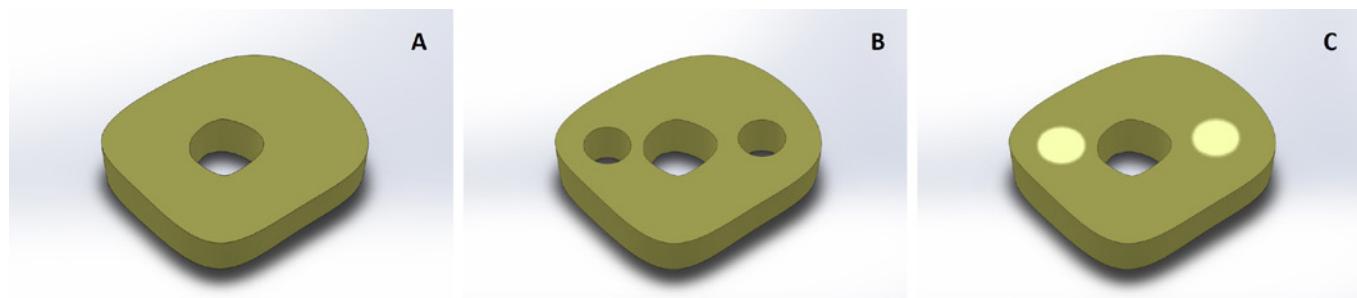


Figure 1. Schematic representation of sample preparation: (A) horizontal cross-sections; (B) canal-like holes prepared in the cross-sections; (C) holes filled with WMTA® or TheraCal LC®.

Table 1. Composition, manufactures and batch numbers of the tested materials.

Materials	Manufactures	Composition
White MTA®	Angelus, Londrina, PR, Brazil	Tricalcium silicate, dicalcium silicate, tricalcium aluminate, calcium oxide, bismuth oxide.
TheraCal LC®	Bisco, Schaumburg, IL, USA	Portland cement, BIS-GMA, barium zirconate powder.

Data Presentation and Analysis

Overall, data did not adhere to a Gaussian Curve (Shapiro-Wilk test, $P < 0.05$). Accordingly, Mann-Whitney U test was applied to rank materials regarding dentinal push-out bond strength ($P < 0.05$). IBM SPSS Inc. for Windows (17.0) was used as statistical package for analysis.

Results

All specimens showed measurable results and no premature failure occurred. A significant difference was observed between materials, in which TheraCal LC® demonstrated higher push-out bond strength values to dentin when compared to WMTA® ($P < 0.0001$). Table 2 discloses the central tendency data of the study.

Table 2. Mean and standard deviation ($\pm SD$), median and interquartile range (IQR) and 95% confidence interval (CI) of the push-out values displayed by the reparative materials, WMTA® and TheraCal LC®.

	Mean ($\pm SD$)	Median (IQR)	95% CI
WMTA®	1.04 (± 0.74)A	0.79 (0.80)	0.67 – 1.40
TheraCal LC®	5.01 (± 1.60)B	4.57 (3.28)	4.22 – 5.81

Discussion

MTA outperformance during pulp-capping procedures has been well documented in endodontic literature.^{15,20} For this reason, WMTA® was selected as the reference material for comparison in the present study. However, even though MTA is the most traditional calcium-silicate material, TheraCal LC®, which is a tricalcium-silicate material modified by photopolymerizable resin, presented better push-out performance. Therefore, the tested null hypothesis was rejected.

Both WMTA® and TheraCal LC® are calcium-silicate based materials, presenting the ability to form an apatite layer when in contact with phosphate-containing physiological fluids, such as PBS.^{21,22} This interaction with the storage medium is known as bioactivity, that positively influences the bond strength performance because of the production of crystalline deposits along the materials-

dentin interface.^{4,23-26} Nevertheless, TheraCal LC® physical-mechanical characteristics and material set are dependent on the application of light-cure during 20 seconds.¹⁰ At the same time, the hydrophilic resin present in TheraCal LC® composition permits water absorption in some levels, which is associated with the initiation of the hydration reaction of the Portland cement.¹⁰

As a result of this light-cured set in association with thixotropic properties, TheraCal LC® demonstrates superior handling characteristics. Consequently, the application of this material is simple and allows precise immediate placement and adequate condensation, even in deep cavity preparations. This peculiarity is very likely related to a strong adhesion. In addition to that, MTA underscores inferior flowability¹⁰ and a water-based chemistry that may led to poorest bonding findings when compared to a resin-based material with dimethacrylate monomers. Accordingly, it seems that the addition of polymerizable methacrylate hydrophilic-monomers in the formula of calcium-silicate-based materials significantly increases push-out bond strength values.

In line with the present finding of great bond strength to dentin, previous reports concluded that TheraCal LC® produces significantly higher microshear bond strength to resin composite when compared to MTA, Fuji IX and Biobond.^{3,6,27,28} On the contrary, a weak bonding between TheraCal LC® and glass ionomer has been reported.⁸ In this context, it has been advised that, after the use of TheraCal LC®, the application of composite resins using a total-etch technique followed by self-etch primer should be performed.^{6,27}

Nevertheless, despite presenting promising properties in several studies^{3,8,11,14,20,25-30}, it should be pointed that TheraCal LC® has recently demonstrated inconsistent results regarding biological³¹ and clinical performance³². These results may have occurred due to differences in methodology and points to the need for new TheraCal LC® studies.

Push-out bond strength represents a relevant physical aspect, as it is crucial for the support and integrity of the endodontic materials conditioned in the dental element. In cases of pulp-capping, in which the preservation of the vital condition of pulp tissues is intended, suitable push-out results are widely desired. Pulp-capping materials should ideally remain bonded to the dentin after conditioned, maintaining its allocation even under the action of mechanical forces from operative procedures, tooth function or dental traumas.^{7,14,28} The current selection protocol of teeth, as well as preparation of slices, lead to greater homogeneity and standardization of the specimens, yielding reduction of the biases' possibilities and greater validation of the results. The use of equivalent dentin substrate for both materials evaluated and the standard format of the holes to which they were conditioned allowed more reliable baseline for push-out assay.^{26,33,34}

Conclusion

In conclusion, TheraCal LC® demonstrated a significant advantage over WMTA® as regards to push-out bond strength. Thus, it may be taken as a promising and innovative pulp-capping material for the treatment of compromised vital pulp. The development of resin-modified light-curable calcium-silicate-based materials

seems to be relevant for the improvement of dentin bond strength performance.

Acknowledgements

This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

References

1. Hilton TJ. Keys to clinical success with pulp capping: a review of the literature. *Oper Dent* 2009;34:615-625.
2. Hilton TJ, Ferracane JL, Mancl L. Comparison of CaOH with MTA for direct pulp capping: a PBRN randomized clinical trial. *J Dent Res* 2013;92:16S-22S.
3. Karadas M, Cantequin K, Gümüş H, Ateş SM, Duymuş ZY. Evaluation of the bond strength of different adhesive agents to a resin-modified calcium silicate material (TheraCal LC). *Scanning* 2016;38:403-411.
4. Mente J, Geletneky B, Ohle M, Koch MJ, Friedrich Ding PG, Wolff D *et al.* Mineral trioxide aggregate or calcium hydroxide direct pulp capping: an analysis of the clinical treatment outcome. *J Endod* 2010;36:806-813.
5. Arias-Moliz MT, Farrugia C, Lung CYK, Wismayer PS, Camilleri J. Antimicrobial and biological activity of leachate from light curable pulp capping materials. *J Dent* 2017;64:45-51.
6. Meraji N, Camilleri J. Bonding over Dentin Replacement Materials. *J Endod* 2017;43:1343-1349.
7. Parirokh M, Torabinejad M. Mineral trioxide aggregate: a comprehensive literature review—part III: clinical applications, drawbacks, and mechanism of action. *J Endod* 2010;36:400-413.
8. Buonavoglia A, Lauritano D, Perrone D, Ardito F, Troiano G, Dioguardi M, *et al.* Evaluation of chemical-physical properties and cytocompatibility of TheraCal LC. *J Biol Regul Homeost Agents* 2017;31:1-9.
9. Camilleri J, Laurent P, About I. Hydration of Biodentine, Theracal LC, and a prototype tricalcium silicate-based dentin replacement material after pulp capping in entire tooth cultures. *J Endod* 2014;40:1846-1854.
10. Gandolfi MG, Siboni F, Prati C. Chemical-physical properties of TheraCal, a novel light-curable MTA-like material for pulp capping. *Int Endod J* 2012;45:571-579.
11. Hinata G, Yoshioka K, Han L, Edanami N, Yoshioka N, Okiji T. Bioactivity and biomineralization ability of calcium silicate-based pulp-capping materials after subcutaneous implantation. *Int Endod J* 2017;50:e40-51.
12. Parirokh M, Torabinejad M, Dummer PMH. Mineral trioxide aggregate and other bioactive endodontic cements: an updated overview - part I: vital pulp therapy. *Int Endod J* 2018;51:177-205.
13. Lee BN, Lee BG, Chang HS, Hwang YC, Hwang IN, Oh WM. Effects of a novel light-curable material on odontoblastic differentiation of human dental pulp cells. *Int Endod J* 2017;50:464-471.
14. Parisay I, Ghoddusi J, Forghani M. A review on vital pulp therapy in primary teeth. *Iran Endod J* 2015;10:6-15.
15. TahaÖzyürek, Demiryürek EÖ. Comparison of the antimicrobial activity of direct pulp-capping materials: Mineral trioxide aggregate-Angelus and Biodentine. *J Conserv Dent* 2016;19:569-572.
16. TheraCal LC; Available at: <http://www.bisco.com/>. Accessed June 20, 2017.
17. Oliveira DS, Cardoso ML, Queiroz TF, Silva EJ, Souza EM, De-Deus G. Suboptimal push-out bond strengths of calcium silicate-based sealers. *Int Endod J* 2016;49:796-801.
18. Sen BH, Piskin B, Baran N. The effect of tubular penetration of root canal sealers on dye microlleakage. *Int Endod J* 1996;29:23-28.
19. Skidmore LJ, Berzins DW, Bahcall JK. An in vitro comparison of the intraradicular dentin bond strength of Resilon and gutta-percha. *J Endod* 2006;32:963-966.
20. Parirokh M, Asgary S, Eghbal MJ, Stowe S, Eslami B, Eskandarizade A, *et al.* A comparative study of White and grey mineral trioxide aggregate as pulp capping agents in dog's teeth. *Dent Traumatol* 2005;21:150-154.
21. Gandolfi MG, Taddei P, Tinti A, De Stefano Dorigo E, Rossi PL, Prati C. Kinetics of apatite formation on a calcium-silicate cement for root-end filling during ageing in physiological-like phosphate solutions. *Clin Oral Investig* 2010;14:659-668.
22. Gandolfi MG, Taddei P, Tinti A, Prati C. Apatite-forming ability (bioactivity) of ProRoot MTA. *Int Endod J* 2010;43:917-929.
23. Camilleri J, Sorrentino F, Damidot D. Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, biodentine and MTA Angelus. *Dent Mater* 2013;29:580-593.
24. Sauro S, Osorio R, Watson TF, Toledoano M. Therapeutic effects of novel resin bonding systems containing bioactive glasses on mineral-depleted areas within the bonded-dentine interface. *J Mater Sci Mater Med* 2012;23:1521-1532.
25. Cannon M, Gerodias N, Viera A, Percinoto C, Jurado R. Primate pulpal healing after exposure and TheraCal application. *J Clin Pediatr Dent* 2014;38:333-337.
26. Carvalho NK, Prado MC, Senna PM, Neves AA, Souza EM, Fidel SR *et al.* Do smear-layer removal agents affect the push-out bond strength of calcium silicate-based endodontic sealers? *Int Endod J* 2017;50:612-619.
27. Cengiz E, Ulusoy N. Microshear Bond Strength of Tri-Calcium Silicate-based Cements to Different Restorative Materials. *J Adhes Dent* 2016;18:231-237.
28. Deepa VL, Dhamaraju B, Bollu IP, Balaji TS. Shear bond strength evaluation of resin composite bonded to three different liners: TheraCal LC, Biodentine, and resin-modified glass ionomer cement using universal adhesive: An in vitro study. *J Conserv Dent* 2016;19:166-170.
29. Fuss M, Wicht MJ, Attin T, Derman SHM, Noack MJ. Protective Buffering Capacity of Restorative Dental Materials In Vitro. *J Adhes Dent* 2017;19:177-183.
30. Giraud T, Rufas P, Chmilewsky F, Rombouts C, Dejou J, Jeanneau C *et al.* Complement Activation by Pulp Capping Materials Plays a Significant Role in Both Inflammatory and Pulp Stem Cells' Recruitment. *J Endod* 2017;43:1104-1110.
31. Jeanneau C, Laurent P, Rombouts C, Giraud T, About I. Light-cured Tricalcium Silicate Toxicity to the Dental Pulp. *J Endod* 2017;43:2074-2080.
32. Bakhtiar H, Nekoofar MH, Aminishakib P, Abedi F, Naghi Moosavi F, Esnaashari E, *et al.* Human Pulp Responses to Partial Pulpotomy Treatment with TheraCal as Compared with Biodentine and ProRoot MTA: A Clinical Trial. *J Endod* 2017;43:1786-1791.
33. Silva EJ, Carvalho NK, Prado MC, Zanon M, Senna PM, Souza EM *et al.* Push-out Bond Strength of Injectable Pozzolan-based Root Canal Sealer. *J Endod* 2016;42:1656-1659.
34. Haghgo R, Ahmadvand M. Evaluation of pulpal response of deciduous teeth after direct pulp capping with bioactive glass and mineral trioxide aggregate. *Contemp Clin Dent* 2016;7:332-335.

Mini Curriculum and Author's Contribution

1. Raissa Dias Fares - DDS. Contribution: Bibliographical research, experimental procedures, manuscript writing. ORCID: 0000-0003-1264-2943
2. Nancy Kudsi Carvalho - DDS; PhD. Contribution: experimental procedures. ORCID: 0000-0003-3893-5330
3. Marina Carvalho Prado - DDS; MSc. Contribution: Experimental procedures. ORCID: 0000-0002-7116-0402
4. Erick M. Souza - DDS; MSc. Contribution: Experimental procedures, image editing. ORCID: 0000-0003-2074-0834
5. Plinio Mendes Senna - DDS; PhD. Contribution: Manuscript review, work supervisor. ORCID: 0000-0003-0743-5376
6. Emmanuel João Nogueira Leal da Silva - DDS; PhD. Contribution: Manuscript review, statistical analysis, work supervisor and paper submission. ORCID: 0000-0002-6445-8243



Submitted: 08/05/2020 / Accepted for publication: 08/13/2020

Corresponding author

Emmanuel João Nogueira Leal Silva

E-mail: nogueiraemmanuel@hotmail.com

