Received: 15th October 2015 Accepted: 18th January 2016 Conflict of Interest: None

Source of Support: Nil

Original Research

Doi: 10.2047/jioh-08-03-06

Effects of Different Acid Etching Times on the Compressive Strength of Three Calcium Silicate-based Endodontic Materials

Sara A Alsubait

Contributor:
Assistant Professor, Department of Restorative Dental Science, College of Dentistry, King Saud University, Riyadh, Saudi Arabia.

Correspondence:
Dr. Alsubait SA. Department of Restorative Dental Science, College of Dentistry, King Saud University, PO Box 85676, Riyadh 11612, Saudi Arabia. Email: s_alsubait@yahoo.com

How to cite the article:
Alsubait SA. Effects of different acid etching times on the compressive strength of three calcium silicate-based endodontic materials. J Int Oral Health 2016;8(3):328-331.

Abstract:

Background: Calcium silicate cements (CSCs), if used for pulp capping or furcation repair, are covered with a restoration. The role of different time intervals of the conditioning process used for final restorations for new CSCs has not been studied. The aim of this study was to evaluate the effects of acid etching on the compressive strengths of biodentine (BD), endosequence bioceramic root repair material-fast set putty (ERRM-FSP), and ProRoot mineral trioxide aggregate (PMTA) at 24 h or 7 days.

Materials and Methods: A total of 144 specimens were prepared (48 per material). For each material, 12 specimens were subjected to acid etching at 24 h or 7 days. Compressive strengths were evaluated in accordance with ISO 9917-1:2007 recommendations. Specimens were crushed along their long axis using a universal testing machine. The maximum load required to fracture each specimen was recorded in megapascals. Compressive strengths were compared with the unetched control groups.

Results: For all materials, the compressive strengths were significantly higher at day 7 than at 24 h. There were no statistically significant differences between the control and etched groups at 24 h or 7 days for the materials tested. Overall, the compressive strength was significantly lower for ERRM-FSP than for PMTA and BD.

Conclusion: The acid-etching procedure did not affect the compressive strength of BD, ERRM-FSP, or PMTA. However, the postponement of the acid-etching procedure until 7 days after CSC placement is suggested to reduce the possibility of displacement.

Key Words: Acid etch, biodentine, endosequence, ProRoot mineral trioxide aggregate, root repair material

Introduction

In vital pulp therapy, a dental material is placed directly over the pulp to maintain the pulp's vitality and function. An ideal pulp capping material should be biocompatible and radiopaque; exhibit antibacterial activity; and possess the tight sealing ability, and the capacity to promote the formation of reparative dentin. Historically, calcium hydroxide ($Ca(OH)_2$) has been the material of choice for vital pulp therapy.[1] Recently, calcium silicate-based cements (CSCs) have been recommended as potential pulp-capping agents.

Relative to $Ca(OH)_2$, mineral trioxide aggregate (MTA), the first CSC, exhibits greater biocompatibility and produces more predictable hard tissue barrier formation.[2] Furthermore, clinical and radiographic successes have been achieved when MTA has been used for direct pulp capping[3] or pulpotomies.[4] However, MTA has certain limitations such as handling difficulties,[5] a long setting time,[6] and the potential for tooth discoloration.[7] Moreover, acid-etching procedures affect the compressive strength of MTA.[8] Several CSCs have been introduced into the market to overcome the unfavorable properties of MTA including biodentine (BD) (Septodont, Saint Maur des Fossés, France) and endosequence bioceramic root repair material (ERRM) (Brasseler USA, Savannah, GA).

BD is a dentine substitute that can be used on crowns and roots. This material consists of a powder and a liquid. The powder is mixed with the liquid in a capsule in an amalgamator for 30 s. According to the manufacturer, the setting time for BD is 12 min. This material not only is biocompatible[9] and radiopaque[10] but also has excellent color stability[11] and a push-out bond strength similar to that of MTA.[12] Moreover, the acid-etching procedure does not affect BD's compressive strength.[13] Studies have demonstrated that BD can induce reparative dentine formation.[14-16] Therefore, BD has been recommended for pulp capping.[14]

ERRM is a newer premixed CSC that sets in the presence of water. It is available in a syringeable paste and condensable putty. Prior research has revealed that ERRM is a biocompatible material[17] with good sealing ability.[18] Relative to MTA, ERRM has similar antibacterial and antifungal activities against *Enterococcus faecalis* and *Candida albicans*, respectively.[19,20] Recently, ERRM-fast set putty (FSP) has become available on the market. The setting time for ERRM-FSP is 20 min. According to the manufacturer, this FSP exhibits the same characteristics as the paste and the putty but exhibits a new fast-setting chemistry and improved syringe delivery.

In vital pulp therapy, the pulp-capping agent is typically covered with a restorative material such as composite resin. Clinically, phosphoric acid is applied before the insertion of the composite to improve the marginal seal and the resin bond to the tooth. To the best of our knowledge, the effects

of different time intervals during the conditioning process used for final tooth restoration on the physical properties of the aforementioned CSCs has not been examined. Therefore, the present study was conducted to evaluate the effects of acid-etching procedures on the compressive strengths of BD, ERRM-FSP, and ProRoot MTA (PMTA) at 24 h or 7 days after mixing. The null hypothesis was that the use of different time intervals after the acid-etching procedure would not affect the compressive strengths of these cements.

Materials and Methods

The materials investigated in the present study were BD, ERRM-FSP, and PMTA (Dentsply, Tulsa, OK, USA). BD and PMTA were mixed according to the manufacturers' instructions. ERRM-FSP was provided premixed. A single operator prepared all the experimental specimens.

Compressive strength

Using an amalgam plugger, each material was incrementally inserted into 2-part split stainless steel molds with an internal diameter of 4.0 mm and a length of 6.0 mm that had been placed on a glass slab. A total of 144 specimens were prepared (48 specimens per group). Wet gauze was placed on top of and under the molds. The specimens were then stored at 37°C.

After 24 h, 24 samples of each material were randomly selected and removed from the molds. The surfaces of each specimen were polished with 600-grit fine-grain sandpaper (Buehler, Lake Bluff, IL, USA) for 20 s to produce smooth surfaces. This polishing process was performed with minimal hand pressure and irrigation with water for debris removal. The samples were then thoroughly dried.

The 24 h specimens for each material were randomly divided into 2 groups with 12 specimens in each group. Samples in the first group were subjected to the acid-etching procedure, whereas samples in the second group were used as controls. For acid etching, 37% phosphoric acid (N-Etch, Ivoclar, Vivadent, NY, USA) was applied to a specimen's top surface for 15 s; the specimen was then rinsed with distilled water for 15 s and thoroughly dried with a stream of air.

Compressive strengths of the 24 h specimens were determined using the method recommended by ISO 9917-1:2007. Specimens were mounted vertically and crushed along their long axis at 1 mm/min using a universal testing machine (Instron 5965, ITW, MA, USA). The maximum load required to fracture each specimen was recorded in megapascals. A blinded operator recorded compressive strength readings. The same steps were repeated for the remaining 72 specimens at 7 days after mixing.

Statistical analysis

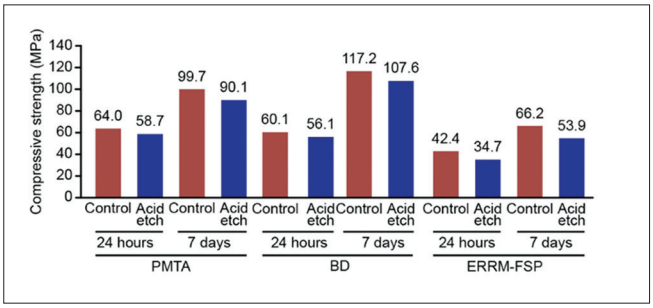
The Shapiro–Wilk test revealed a normal distribution. Independent *t*-tests were used to statistically compare the mean compressive strengths of the etched groups at 24 h and 7 days for each material. This analytical approach was also used to compare the means of the etched and control groups. The mean compressive strengths of different cements were compared using ANOVA followed by Scheffe's *post-hoc* test. A significance threshold of 0.05 was used. Statistical analysis was performed using SPSS statistical software (version 16; SPSS Inc., IL, USA).

Results

The study results are summarized in Graph 1. Compressive strengths for the etched groups were significantly higher at day 7 than at 24 h for PMTA ($P = 0.01$), BD ($p = 0.00$), and ERRM-FSP ($p = 0.01$). There were no statistically significant differences between the control and etched groups at 24 h or 7 days (Table 1). Overall, the compressive strength was significantly lower for ERRM-FSP than for PMTA and BD at the two examined time points ($p = 0.00$). There was no significant difference between PMTA and BD with respect to compressive strength at 24 h ($p = 0.6$). However, at day 7, BD exhibited significantly greater compressive strength than PMTA ($p = 0.04$).

Discussion

The evaluation of a simple mechanical parameter such as compressive strength allows for physical property to be



Graph 1: The mean compressive strengths of control and acid-etched samples for each material (in megapascals) at 24 h and 7 days.

Table 1: Means and SD for compressive strengths (in megapascals) for controls and for samples etched at 24 h and 7 days.

Time interval	Material	Group	Mean	SD	*P* value
24 h	PMTA	Control	64.0	13.0	0.23
		Acid etch	58.7	6.9	
	BD	Control	60.1	7.9	0.41
		Acid etch	56.1	14.7	
	ERRM-FSP	Control	42.4	8.3	0.08
		Acid etch	34.7	11.6	
7 days	PMTA	Control	99.7	13.8	0.41
		Acid etch	90.1	36.9	
	BD	Control	117.2	22.9	0.4
		Acid etch	107.6	30.7	
	ERRM-FSP	Control	66.2	9.6	0.06
		Acid etch	53.9	17.8	

SD: Standard deviation, PMTA: ProRoot mineral trioxide aggregate, BD: Biodentine, ERRM-FSP: Endosequence bioceramic root repair material-fast set putty

correlated with clinical performance.[21] In vital pulp therapy, the applied cement must remain in place despite experiencing dislodging forces resulting from operative procedures. The appropriate time for restoration after the placement of new CSCs has not previously been evaluated. Therefore, in the present study, the effects of 37% phosphoric acid, which is typically applied prior to placing composite restorations, on the compressive strengths of BD and ERRM-FSP were evaluated at 24 h and 7 days after mixing.

Two different time intervals were selected for examination in this study; in particular, 24 h was selected to allow the cements to set. However, the postponement of restorative procedures for at least 96 h after mixing MTA[8] is recommended; therefore, a second-time interval was also assessed.

No prior study has evaluated the effects of different time intervals before acid etching on the compressive strength of BD. Our results demonstrated that significantly greater compressive strength was achieved if acid etching was performed at 7 days after mixing than at 24 h after mixing. This phenomenon might be attributable to the hydration reaction, which is critical to the setting of CSCs.[8] Furthermore, a comparison of the compressive strengths of etched BD and PMTA (the gold standard) revealed that BD was superior for etching performed at day 7; these results are in accordance with the findings obtained by Kayahan *et al.* in 2013.[13] The advantages of BD relative to PMTA include mechanical mixing, which avoids inconsistencies within the material, and improved handling characteristics. Furthermore, the acid-etching procedure did not affect the compressive strength of BD. Therefore, BD could be a suitable replacement for PMTA, particularly in the context of vital pulp therapy. However, our findings indicate that postponing the acid-etching and restoration procedures until 7 days after BD placement is recommended to reduce the possibility of displacement.

ERRM-FSP has the advantage of being available in a premixed syringe that is convenient to use. Our results revealed that specimens etched at 7 days had significantly higher compressive strengths than specimens etched at 24 h. Furthermore, compressive strengths were significantly lower for ERRM-FSP than for PMTA in the control and test groups; this phenomenon might be caused by differences in the compositions of ERRM-FSP and PMTA. Tricalcium aluminate improves the strength of CSCs.[22] However, ERRM-FSP is an aluminum-free material; as a result, relative to PMTA, ERRM-FSP may form fewer ettringite crystals (hydrated calcium-aluminum-sulfate), which play an important role in interlocking the cubic crystals of endodontic materials.[23] Furthermore, the compressive strength is an indirect measure of a CSC's hydration reaction.[8,24] The accelerator in ERRM-FSP might interfere with the cement's hydration reaction and thereby reduce its compressive strength.

A recent study has indicated that ERRM and PMTA have similar compressive strengths.[25] Discrepancies between the findings of this prior study and the current investigation could relate to differences in incubation settings and specimen dimensions. In accordance with ISO 9917-1:2007 recommendations, molds 6 mm in length and 4 mm in diameter were used in the present study.

The *in vivo* environment cannot be replicated using the *in vitro* method employed to assess compressive strengths in the current study. However, the study results might provide information that can aid clinicians in selecting the best CSC, particularly in cases involving vital pulp therapy or the repair of furcation perforations. Future studies are required to determine how different time intervals before the acid-etching procedure affect other physical properties of BD and ERRM-FSP before an appropriate interval can be confidently used in clinical settings.

Conclusion

In conclusion, the acid-etching procedure did not affect the compressive strength of BD, ERRM-FSP, or PMTA. However, higher compressive strengths were achieved for BD, ERRM-FSP, and PMTA by acid etching at 7 days than by etching at 24 h.

Acknowledgments

This work is registered with the College of Dentistry Research Center (FR0239) of King Saud University.

References

1. Stanley HR. Pulp capping: Conserving the dental pulp – Can it be done? Is it worth it? Oral Surg Oral Med Oral Pathol 1989;68(5):628-39.
2. Nair PN, Duncan HF, Pitt Ford TR, Luder HU. Histological, ultrastructural and quantitative investigations on the response of healthy human pulps to experimental capping with mineral trioxide aggregate: A randomized controlled trial. Int Endod J 2008;41(2):128-50.
3. Bogen G, Kim JS, Bakland LK. Direct pulp capping with mineral trioxide aggregate: An observational study. J Am Dent Assoc 2008;139(3):305-15.
4. Qudeimat MA, Barrieshi-Nusair KM, Owais AI. Calcium hydroxide vs mineral trioxide aggregates for partial pulpotomy of permanent molars with deep caries. Eur Arch Paediatr Dent 2007;8(2):99-104.
5. Ber BS, Hatton JF, Stewart GP. Chemical modification of proroot mta to improve handling characteristics and decrease setting time. J Endod 2007;33(10):1231-4.
6. Islam I, Chng HK, Yap AU. Comparison of the physical and mechanical properties of MTA and portland cement. J Endod 2006;32(3):193-7.
7. Belobrov I, Parashos P. Treatment of tooth discoloration after the use of white mineral trioxide aggregate. J Endod 2011;37(7):1017-20.
8. Kayahan MB, Nekoofar MH, Kazandag M, Canpolat C, Malkondu O, Kaptan F, *et al.* Effect of acid-etching

procedure on selected physical properties of mineral trioxide aggregate. Int Endod J 2009;42(11):1004-14.
9. Corral Nuñez CM, Bosomworth HJ, Field C, Whitworth JM, Valentine RA. Biodentine and mineral trioxide aggregate induce similar cellular responses in a fibroblast cell line. J Endod 2014;40(3):406-11.
10. Grech L, Mallia B, Camilleri J. Investigation of the physical properties of tricalcium silicate cement-based root-end filling materials. Dent Mater 2013;29(2):e20-8.
11. Vallés M, Mercadé M, Duran-Sindreu F, Bourdelande JL, Roig M. Influence of light and oxygen on the color stability of five calcium silicate-based materials. J Endod 2013;39(4):525-8.
12. Alsubait SA, Hashem Q, AlHargan N, AlMohimeed K, Alkahtani A. Comparative evaluation of push-out bond strength of ProRoot MTA, bioaggregate and biodentine. J Contemp Dent Pract 2014;15(3):336-40.
13. Kayahan MB, Nekoofar MH, McCann A, Sunay H, Kaptan RF, Meraji N, *et al.* Effect of acid etching procedures on the compressive strength of 4 calcium silicate-based endodontic cements. J Endod 2013;39(12):1646-8.
14. Shayegan A, Jurysta C, Atash R, Petein M, Abbeele AV. Biodentine used as a pulp-capping agent in primary pig teeth. Pediatr Dent 2012;34(7):e202-8.
15. Tran XV, Gorin C, Willig C, Baroukh B, Pellat B, Decup F, *et al.* Effect of a calcium-silicate-based restorative cement on pulp repair. J Dent Res 2012;91(12):1166-71.
16. Nowicka A, Wilk G, Lipski M, Kolecki J, Buczkowska-Radlinska J. Tomographic evaluation of reparative dentin formation after direct pulp capping with $Ca(OH)_2$, MTA, biodentine, and dentin bonding system in human teeth. J Endod 2015;41(8):1234-40.
17. Alanezi AZ, Jiang J, Safavi KE, Spangberg LS, Zhu Q. Cytotoxicity evaluation of endosequence root repair material. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2010;109(3):e122-5.
18. Nair U, Ghattas S, Saber M, Natera M, Walker C, Pileggi R. A comparative evaluation of the sealing ability of 2 root-end filling materials: An *in vitro* leakage study using *Enterococcus faecalis*. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2011;112(2):e74-7.
19. Lovato KF, Sedgley CM. Antibacterial activity of endosequence root repair material and proroot MTA against clinical isolates of *Enterococcus faecalis*. J Endod 2011;37(11):1542-6.
20. Alsalleeh F, Chung N, Stephenson L. Antifungal activity of endosequence root repair material and mineral trioxide aggregate. J Endod 2014;40(11):1815-9.
21. Mitsuhashi A, Hanaoka K, Teranaka T. Fracture toughness of resin-modified glass ionomer restorative materials: Effect of powder/liquid ratio and powder particle size reduction on fracture toughness. Dent Mater 2003;19(8):747-57.
22. Liu WN, Chang J, Zhu YQ, Zhang M. Effect of tricalcium aluminate on the properties of tricalcium silicate-tricalcium aluminate mixtures: Setting time, mechanical strength and biocompatibility. Int Endod J 2011;44(1):41-50.
23. Wang Z, Ma J, Shen Y, Haapasalo M. Acidic pH weakens the microhardness and microstructure of three tricalcium silicate materials. Int Endod J 2015;48(4):323-32.
24. Bentz DP. Three-dimensional computer simulation of portland cement hydration and microstructure development. J Am Ceram Soc 1997;80(1):3-21.
25. Walsh RM, Woodmansey KF, Glickman GN, He J. Evaluation of compressive strength of hydraulic silicate-based root-end filling materials. J Endod 2014;40(7):969-72.