Effect of Acid Etching Procedures on the Compressive Strength of 4 Calcium Silicate–based Endodontic Cements

Mehmet Baybora Kayahan, DDS, PhD,[*‡] *Mohammad Hossein Nekoofar, DDS, MSc, DoIBoE, PhD,*[†‡] *Amy McCann, BDS,*[‡] *Hakkı Sunay, DDS, PhD,*[*] *Rabia Figen Kaptan, DDS, PhD,*[*] *Naghmeh Meraji, DDS, MSc, DoIBoE,*[†] *and Paul M.H. Dummer, BDS, MScD, PhD, DDSc, FDSRCS(Ed)*[‡]

Abstract

Introduction: The purpose of this study was to evaluate the effect of acid etching on the compressive strength of 4 calcium silicate–based cements. **Methods:** One gram of each corresponding powder of ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN), MTA Angelus (Angelus, Londrina, PR, Brazil), and CEM cement (BioniqueDent, Tehran, Iran) and a 0.33-g aliquot of liquid were placed in a plastic mixing capsule that was then mechanically mixed for 30 seconds at 4500 rpm in an amalgamator. For the preparation of Biodentine (Septodont, Saint Maur-des-Fossés, France), the liquid provided was added to the powder within the plastic capsule supplied by the manufacturer and then mechanically mixed for 30 seconds at 4500 rpm using the amalgamator. The resulting slurries were then placed incrementally into 40 cylindrical molds to give a total of 160 specimens that were incubated at 37°C for a week. Twenty specimens of each material were then subjected to the acid etch procedure. The compressive strength of the samples was then calculated in megapascals using a universal testing machine. The results were then subjected to 2-way analysis of variance analysis of variance followed by the Tukey post hoc test. **Results:** The application of acid etch significantly reduced ($P < .0001$) the compressive strength of Angelus MTA and CEM cement; however, it did not reduce the compressive strength of ProRoot MTA or Biodentine. Regardless of the acid etch application, Biodentine showed significantly higher compressive strength values than the other materials ($P < .0001$), whereas CEM cement had the lowest compressive strength values. There was no significant difference between CEM cement and MTA Angelus. The compressive strength of ProRoot MTA was significantly lower ($P < .0001$) than Biodentine but significantly higher ($P < .0001$) than MTA Angelus and CEM cement in both the test and control groups. **Conclusions:** When the application of acid etchants is required, Biodentine and ProRoot MTA seem to be better options than MTA Angelus or CEM cement. *(J Endod 2013;39:1646–1648)*

Key Words

Biodentine, calcium silicate–based cement, CEM cement, compressive strength, MTA Angelus, ProRoot MTA

An ideal root repair material should be able to seal communications between the root canal system and surrounding tissues and be biocompatible, dimensionally stable, radiopaque, and insoluble when in contact with tissue fluids. Similar requirements are necessary for materials used in vital pulp therapies. For many years, various materials such as calcium hydroxide, zinc oxide–eugenol cements, composite resin, and glass-ionomer cements have been used in vital pulp therapies and the repair of root perforations although none of them fulfill all the requirements of an ideal material (1).

Mineral trioxide aggregate (MTA), a type of calcium silicate–based cement, is biocompatible (2) and antibacterial (3) and can set in an aqueous environment; it also provides a good seal against bacteria and fluids (4). It was first developed and introduced as a root repair material and subsequently approved by the Food and Drug Administration for use in direct pulp capping and pulpotomy procedures, particularly in teeth with immature apices (apexogenesis) (5, 6). In addition, conduction of the cementum and bone formation over its surface is another advantage of MTA (7).

Regardless of its distinctive combination of biological and physical characteristics, the unpredictable setting and hardening reaction of commercially available MTA formulations are potential drawbacks (8). Moreover, the adverse effect of an acidic environment (9, 10) and acid etch procedures on MTA (11), which are often used to increase the sealing ability; retention of resin restorations; and bond strength of MTA to composite resins (12) may restrict its use.

Several modified calcium silicate–based cements, such as MTA Angelus (Angelus, Londrina, PR, Brazil), CEM cement (BioniqueDent, Tehran, Iran), and Biodentine (Septodont, Saint Maur-des-Fossés, France), have been marketed with claims that they have improved physical and chemical properties over the original MTA products (13–15). The hydration process of these calcium silicate–based materials (hydraulic cements) results in the formation of calcium hydroxide (16); therefore, their application in vital pulp therapies has been recommended (17, 18). However, information on

From the *Department of Endodontics, Faculty of Dentistry, Yeditepe University, Istanbul, Turkey; †Department of Endodontics, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran; and ‡Endodontology Research Group, School of Dentistry, College of Biomedical and Life Sciences, Cardiff University, Cardiff, United Kingdom.

Address requests for reprints to Dr Mohammad Hossein Nekoofar, School of Dentistry, College of Biomedical and Life Sciences, Cardiff University, Heath Park, Cardiff CF14 4XY, UK. E-mail address: nekoofarmh@cardiff.ac.uk
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the effect of conditioning processes used during tooth restoration, including exposure to phosphoric acid, on the mechanical properties of these materials is limited (11).

In this study, the effects of acid etch procedures on the compressive strength of Biodentine, CEM cement, MTA Angelus, and ProRoot MTA were compared. The null hypothesis was that the acid etch procedure does not reduce the compressive strength of these silicate-based cements.

Materials and Methods

The materials investigated were ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN), MTA Angelus, CEM cement, and Biodentine (Septodont, France). The mixing of ProRoot MTA and MTA Angelus was standardized by placing 1.00 g of each corresponding powder and a 0.33-g aliquot of distilled water in a plastic mixing capsule containing a plastic pestle that was then mechanically mixed for 30 seconds at 4500 rpm in an amalgamator (Silamat; Ivoclar Vivadent AG, Schaan, Liechtenstein) (19).

For the preparation of Biodentine, 5 drops of the liquid provided were added to the powder within the plastic capsule supplied by the manufacturer and then mechanically mixed for 30 seconds at 4500 rpm using the amalgamator. A customized encapsulated CEM cement was also prepared by placing 1.00 g CEM cement powder and 0.33 g liquid inside a plastic mixing capsule containing a plastic pestle, which was then mechanically mixed for 30 seconds at 4500 rpm using an amalgamator.

The resulting slurry of each of the 4 materials was then placed incrementally into 40 silicon, cylindrical molds to give a total of 160 specimens. In accordance with ISO 9917-1:2003, the dimension of each mold was 6 $\pm$ 0.1 mm in length and 4 $\pm$ 0.1 mm in diameter. The placement of the slurries was standardized by applying 1.68 MPa compaction pressure (20) to each specimen followed by agitation with an ultrasonic tip for 30 seconds (CPR-2D; Obtura Spartan, Fenton, MO). All specimens were then incubated in a fully saturated humidity at 37°C.

After 7 days, the specimens were removed from the molds. The upper and lower faces of the cylindrical specimens were then polished using minimum hand pressure and silicon-carbide 1200-grit fine-grain sandpaper (3M, St Paul, MN) to provide a smooth surface; during this process, constant irrigation with water was used to remove surface debris. The samples were then dried thoroughly and their dimensions rechecked with calipers. Twenty specimens of each material were then randomly selected and subjected to the acid etch procedure. This consisted of an application of 37% phosphoric acid on one of the flat surfaces on each cylinder for 15 seconds before being rinsed with distilled water for a further 15 seconds and then dried thoroughly.

The specimens were then mounted vertically and crushed along their long axis at 1 mm/min using a Universal Testing Machine (Lloyd LR MK1 machine; Lloyd Instruments, Fareham, UK), which recorded the failure load in newtons; the compressive strength of the samples was then calculated in megapascals. The results were then subjected to 2-way analysis of variance followed by the Tukey post hoc test using the Statistical Package for the Social Science Version 16 (SPSS Inc, Chicago, IL).

Results

The results are summarized in Figure 1. Regardless of the acid etch application, Biodentine showed significantly higher compressive strength values than the other materials ($P < .0001$), whereas CEM cement had the lowest compressive strength values. There was no significant difference between CEM cement and MTA Angelus. The compressive strength

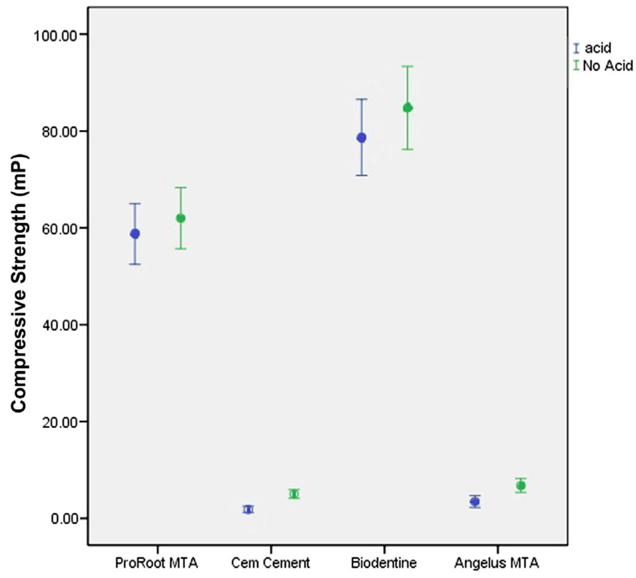

Figure 1. The mean compressive strength and standard deviation of control and test samples for each material (megapascals). 95% confidence interval.

of ProRoot MTA was significantly lower ($P < .0001$) than Biodentine but significantly higher ($P < .0001$) than MTA Angelus and CEM cement in both test and control groups. The application of acid etch significantly reduced ($P < .0001$) the compressive strength of Angelus MTA and CEM cement; however, it did not reduce the compressive strength of ProRoot MTA and Biodentine.

Discussion

In many challenging endodontic procedures, such as pulp treatment of traumatized immature teeth with reversible pulpitis and pulpotomy of primary teeth, the application of MTA as a bioactive silicate cement in contact with exposed pulp tissue may lead to a favorable outcome (21–25). However, the number of high-quality, well-designed, and large scale randomized controlled clinical trials with long-term follow-up are limited (26, 27). Many laboratory and *in vivo* studies have shown the exceptional sealing ability and biological properties of MTA and MTA-like materials, which has resulted in their increased clinical use. Obviously, information about recently developed MTA-like materials such as Biodentine and CEM cement are limited. The aim of this study was to evaluate the effect of acid etching, which is used before the placement of composite restorations for the enhancement of the micromechanical bond and to prevent microleakage, on the compressive strength of 4 calcium silicate–based cements.

Compressive strength is regarded as one of the main physical characteristics of hydraulic cements, which is correlated to its stage of hydration (28, 29). In addition, in vital pulp therapies, the cement may encounter (indirectly) masticatory loads and should have sufficient compressive strength to withstand them. Regardless of the application of acid etch, Biodentine showed a significantly higher compressive strength ($P < .0001$) than the other materials, whereas CEM cement had the lowest compressive strength. Therefore, in clinical applications such as direct pulp capping, the use of Biodentine appears to have advantages, particularly because it can be placed in bulk and has a short setting time.

According to the results of the current study, the compressive strength of MTA Angelus was significantly lower than ProRoot MTA in both the control and test groups. This difference in compressive strength values can be because of differences in their composition. ProRoot MTA consists of 75% Portland cement, 20% bismuth oxide, and 5% calcium sulfate dehydrate; however, MTA Angelus contains 80% Portland cement and 20% bismuth oxide with no addition of calcium sulfate in an attempt to reduce the setting time (14). The formation of ettringite crystals depends on the presence of calcium sulfate dehydrate (30), and because of the lack of this component in MTA Angelus, it may be concluded that this formulation of MTA lacks ettringite crystals. The lack of these crystalline formations was most probably the reason for the lower compressive strength values of MTA Angelus compared with ProRoot MTA; however, further research is required to confirm this hypothesis.

In the current study, regardless of the application of acid etch, CEM cement had the lowest compressive strength. It has been shown that CEM cement contains larger amounts of sulfate and calcium oxide compared with both ProRoot and Angelus MTA (15). High sulfate contents can cause gradual dissolution and decomposition of the products of the hydration process (eg, calcium hydroxide and calcium silicate hydrate gel [31]) that acts as the binding agent of silicate-based cements (32). Consequently, the calcium to silicate ratio declines, resulting in a loss of strength (31). Additionally, calcium sulfate dehydrate increases the setting time of silicate-based cements (33). Therefore, it can be suggested that the higher sulfate content in CEM cement is the reason for its lower compressive strength values.

In the case of acid application, several studies (11, 34, 35) have reported the adverse effect of acidic pH on several physical properties of ProRoot MTA, such as push-out bond strength (36), porosity, microhardness (9), and compressive strength (11), suggesting it would be better to postpone restorative procedures for 96 hours to 1 week. The manufacturer of Biodentine also recommended delaying the placement of the final restoration for at least 1 week to achieve more mature crystalline formations.

According to the results of the current study, acid etching procedures after 7 days did not reduce the compressive strength of ProRoot MTA and Biodentine. However, a significant reduction of the compressive strength was noted in MTA Angelus and CEM cement. For ProRoot MTA, the results are in accordance with Kayahan et al (11), who showed the application of phosphoric acid after 96 hours of storage did not reduce the compressive strength of ProRoot MTA.

Conclusion

After a 1-week interval, an acid etching process had no adverse effect on the compressive strength of Biodentine and ProRoot MTA but significantly decreased this property in MTA Angelus and CEM cement samples. When the application of acid etchants is required, Biodentine and ProRoot MTA seem to be better options than MTA Angelus or CEM cement.

Acknowledgments

The authors deny any conflicts of interest related to this study.

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