Effect of Varying Water-to-Powder Ratios and Ultrasonic Placement on the Compressive Strength of Mineral Trioxide Aggregate

Fatima B. Basturk, DDS, PbD,^{*†} *Mohammad Hossein Nekoofar, DDS, MSc, DoIBoE, PbD,*^{†‡} *Mahir Gunday, DDS, PbD,* * *and Paul M.H. Dummer, BDS, MScD, PbD, DDSc, FDSRCS (Ed)*[†]

Abstract

Introduction: The purpose of this study was to compare the compressive strength of mineral trioxide aggregate (MTA) when mixed with 2 different waterto-powder (WP) proportions using either hand or ultrasonic placement. Methods: Tooth-colored ProRoot MTA (Dentsply Maillefer, Ballaigues, Switzerland) and white MTA Angelus (Angelus Soluçoes Odontologicas, Londrina, Brazil) were investigated. One gram of each MTA powder was mixed with either 0.34 or 0.40 g distilled water. The 4 groups were further divided into 2 groups of 5 specimens for each of the following techniques: conventional (ie, hand placement) and placement using indirect ultrasonic activation for 30 seconds. All specimens were subjected to compressive strength testing after 4 days. The results were statistically analyzed with multivariate analysis of variance and Tukey Honestly Significant Difference tests at a significance level of *P* < .05. **Results:** The mean compressive strength values of ProRoot MTA (84.17 \pm 22.68) were significantly greater than those of MTA Angelus (47.71 ± 14.29) (*P* < .01). Specimens mixed with the 0.34 WP ratio had higher compressive strength values (72.85 ± 25.77) than those mixed with the 0.40 WP ratio (56.69 ± 24.85) (*P* < .05). The highest compressive strength values were recorded for ProRoot MTA specimens that were mixed in the 0.34 WP ratio, and then the samples were placed with ultrasonic activation (mean = 91.35 MPa). The lowest values were recorded for MTA Angelus samples that were mixed in the 0.40 WP ratio, and the specimens were placed without ultrasonic activation (mean = 36.36 MPa). Ultrasonic activation had no significant difference in terms of compressive strength. Conclusions: When using ProRoot MTA and MTA Angelus, higher WP ratios resulted in lower compressive strength values. Ultrasonication had no significant effect on the compressive strength of the material regardless of the WP ratio that was used. Therefore,

adherence to the manufacturer's recommended WP ratio when preparing MTA for use in dental applications is advised. (*J Endod 2015;41:531–534*)

Key Words

Compressive strength, mineral trioxide aggregate, ultrasonic agitation, water-to-powder ratio

M ineral trioxide aggregate (MTA) is a powder that consists of fine hydrophilic particles that harden in contact with water. The physical characteristics of hardened MTA depend on several factors including the water-to-powder ratio (1); the mixing liquid (2, 3); the mixing and placement technique (4–6); the compaction pressure (7); and, lastly, the room temperature (8).

The problems identified with hand mixing in clinical practice were caused mainly by variations in the water-to-powder (WP) ratio resulting from the operator's imprecise mixing techniques when compared with the manufacturer's recommendations (9). The volume of powder is dependent on the operator's decision to accurately fill the scoop (10). In addition, variations in liquid volume arise in response to the positioning of the liquid bottle and the inclusion of air bubbles inside the dispenser (11). Temperature and humidity of the environment as well as the mixing technique and the time spent on mixing are the external factors that may cause iatrogenic variability in the consistency of the cement produced (11). In clinical practice, dental cements are routinely mixed according to the operator's desired consistency, namely by estimation, without the aid of scoop and dropper bottles (10). Therefore, in such instances, the optimum ratio recommended by the manufacturer is not always followed in clinical practice (12).

In an attempt to eliminate operator-induced variability on mixing, encapsulation in which the optimum powder and liquid proportions are predetermined and supplied as capsules was introduced into the market for various hand-mixed cements, such as glass ionomer cements, zinc phosphate luting cements, and calcium silicate cements (10, 13, 14).

Ultrasonication has been reported to enhance the compressive strength (5), surface microhardness (15), and sealing ability (16) of MTA. Yet, in a study evaluating the adaptation of MTA using hand compaction or ultrasonication, Aminoshariae et al (6) concluded that the manufacturer's recommended WP ratio of 1:3 for MTA may not be the most favorable for ultrasonic placement and that it might have caused voids inside the material. When using ultrasonic agitation for the placement of MTA, no significant differences were reported for porosity (4) or push-out bond strength (17) of the material.

From the *Department of Endodontics, Faculty of Dentistry, Marmara University, Istanbul, Turkey; [†]Endodontology Research Group, School of Dentistry, Cardiff University, Cardiff, UK; and [‡]Department of Endodontics, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran.

Address requests for reprints to Dr Mohammad Hossein Nekoofar, School of Dentistry, Cardiff University, Heath Park, Cardiff CF14 4XY, UK. E-mail address: nekoofarmh@cardiff.ac.uk

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According to the manufacturer's instructions for ProRoot MTA (Dentsply Maillefer), 1 g powder supplied in 1 packet should be mixed with a 0.34-g aliquot of distilled water. This mixture results in a waste of the cement because only a small amount of MTA slurry is needed for most cases (18). In an attempt to overcome this waste, the manufacturer of MTA Angelus (Angelus Soluçoes Odontologicas, Londrina, Brazil) developed smaller packets containing 0.14 g MTA powder. Yet, the amount of liquid mixed with the MTA powder is still dependent on the clinician's choice or that of his or her chairside assistant. Thus, clinicians tend to prepare a mixture according to their best estimations rather than the manufacturer's recommended guidelines. This variation in WP ratio could have an effect on the compressive strength of MTA in a clinical setting. The compressive strength of hydraulic cement is related to the proper hydration reaction that occurs between MTA and water (19). Thus, the purpose of this study was to investigate the influence of WP ratio variations on the compressive strength of 2 commercially available mineral trioxide aggregates placed by hand or using ultrasonic instruments. The first null hypothesis was that increasing the water content of MTA slurry above that recommended by the manufacturer would have no impact on the compressive strength of MTA. The second null hypothesis was that ultrasonic placement of MTA would not alter the compressive strength of MTA regardless of the WP ratio applied.

Materials and Methods

Tooth-colored ProRoot MTA (Dentsply Maillefer) and white MTA Angelus (Angelus Soluçoes Odontologicas) were investigated. One gram of each MTA powder was mixed with either 0.34 g or 0.40 g distilled water. The 4 groups were further divided into 2 groups of 5 for each of the 2 placement techniques: conventional (hand) or ultrasonic placement. There were 8 groups in total: groups 1 and 5: ProRoot MTA placed with ultrasonic agitation (n = 5 for each group), groups 2 and 6: ProRoot MTA placed with conventional placement (n = 5for each group), groups 3 and 7: MTA Angelus placed with ultrasonic agitation (n = 5 for each group), and groups 4 and 8: MTA Angelus placed with conventional placement (n = 5 for each group).

The instruments and the test materials were conditioned at $23^{\circ}C \pm 1^{\circ}C$ in the laboratory for 1 hour before use. MTA was mixed and transferred to polytetrafluoroethylene cylindrical molds with internal dimensions of 6 ± 0.1 mm high and a 4 ± 0.1 mm diameter. Half of the materials were subjected to indirect ultrasonic energy. A CPR-1 ultrasonic tip (Dentsply Tulsa Dental) placed in contact with an endodontic plugger was attached to a Suprasson P5 Booster (Satelec, Merignac, France), and the tip was activated for 30 seconds at a power scale of 5. The tip of the endodontic plugger was inserted into and moved throughout the MTA slurry without touching the walls of the molds. The excess material was removed. A damp paper towel was placed on top and bottom of the molds. The specimens were incubated at $37^{\circ}C$ in 100% humidity.

Four days later, the samples were submitted to compressive strength tests using a universal testing machine (Lloyd Instruments, Fareham, UK) at a speed of 1 mm/min along the long axis. The load needed to fracture the specimen was recorded, and its compressive strength was calculated according to the following formula:

Compressive Strength (σ) = $\frac{4P(ultimate \ load)}{\pi d^2(mean \ diameter \ of \ the \ specimen)}$

The tests that were preformed and used to compare the mean values for compressive strength were the multivariate analysis of variance and the Tukey HSD tests along with the IBM Statistical Package of Social Science version 22 (SPSS Inc, Chicago, IL). A significance level of P < .05 was used.

Results

The means and standard deviations of the compressive strength of all groups were shown in Table 1. The mean compressive strength values of ProRoot MTA (84.17 \pm 22.68) were significantly greater than those of MTA Angelus (47.71 \pm 14.29) (P < .01) (Table 2). The highest compressive strength values were recorded for ProRoot MTA specimens that were mixed in a 0.34 WP ratio and placed with ultrasonic activation (mean = 91.35 MPa). The lowest values were recorded for MTA Angelus samples that were mixed in a 0.40 WP ratio and placed without ultrasonic activation (mean = 36.36 MPa). Regardless of the MTA type or the placement method applied, specimens mixed with the 0.34 WP ratio had higher compressive strength values (72.85 \pm 25.77) than those mixed with the 0.40 WP ratio (56.69 \pm 24.85) (P < .05). Compressive strength values of MTA Angelus groups that were mixed with the 0.34 WP ratio had higher compressive strength values than those mixed with the 0.40 WP ratio (P < .01). Also, ultrasonically agitated ProRoot MTA had higher compressive strength values than ultrasonically agitated MTA Angelus (P < .001). Even though the difference was not statistically significant, ultrasonicated groups had higher compressive strength values (68.69 \pm 28.12 MPa) than nonultrasonicated groups $(64.42 \pm 24.78 \text{ MPa}).$

Further analysis revealed a significant difference between the compressive strength values of ProRoot MTA groups that were mixed with the 0.34 WP ratio and the compressive strength values of all MTA Angelus groups regardless of ultrasonication used during the placement of MTA (P < .01).

Discussion

In the present study, the compressive strength of ProRoot MTA and MTA Angelus placed with either hand compaction or ultrasonication was measured. The resultant cement's compressive strength using the manufacturer's WP ratio along with an increased WP ratio was also compared. The results revealed that MTA in the 0.34 WP ratio had higher compressive strength values than those in the 0.40 WP ratio, and the compressive strength values of ProRoot MTA were significantly greater than those of MTA Angelus. Ultrasonic placement had no significant effect on the compressive strength of either formulation of MTA.

MTA becomes a source of calcium hydroxide when it comes in contact with water (20). Also, the expansion of MTA is a water-dependent mechanism attributable to water uptake (21). Therefore, it might be logical to conclude that a high WP ratio might be beneficial. However, the excessive amount of water incorporated in the mix might lead to a management problem when transporting, placing, or compacting the material (1). Fridland and Rosado (1) revealed that a WP ratio

TABLE 1. The Means and Standard Deviations of the Compressive Strength of All Groups

WP ratio		$Mean \pm SD$
0.34	US (+)	$\textbf{91.36} \pm \textbf{22.71}$
	US (–)	$\textbf{88.16} \pm \textbf{20.02}$
0.40	US (+)	$\textbf{77.83} \pm \textbf{32.80}$
	US (–)	71.15 ± 11.23
0.34	US (+)	54.50 ± 14.92
	US (–)	$\textbf{52.53} \pm \textbf{16.10}$
0.40	US (+)	41.45 ± 7.15
	US (–)	$\textbf{36.36} \pm \textbf{7.25}$
	WP ratio 0.34 0.40 0.34 0.40	WP ratio 0.34 US (+) US (-) US (+) 0.40 US (+) US (-) US (+) 0.34 US (+) US (-) 0.40 US (-) US (+) US (-) US (-)

SD, standard deviation; US, ultrasonication; WP, water-to-powder.

TABLE 2.	Compressive S	Strength Value	es according	to the Wat	er-to-Powder
Ratio, Min	eral Trioxide A	Aggregate Typ	e, and Ultras	sonication	Applied

	Compressive strength	
	Mean ± SD	P value
WP		
Normal	$\textbf{72.85} \pm \textbf{25.77}$.030*
High	$\textbf{56.69} \pm \textbf{24.85}$	
MTA		
ProRoot	$\textbf{84.17} \pm \textbf{22.68}$.001 [†]
Angelus	47.71 ± 14.29	
US		
US (+)	$\textbf{68.69} \pm \textbf{28.12}$.566
US (–)	64.42 ± 24.78	

MTA, mineral trioxide aggregate; SD, standard deviation; US, ultrasonication; WP, water-to-powder *P < .05.

 $^{\dagger}P < .01.$

higher than 0.33 was not viscous enough for practical use and a 0.26 WP ratio was the minimum that allowed a mix of putty-like consistency to be manipulated. In a study evaluating the histologic pulp reaction to various WP ratios of white MTA as a pulp capping material in healthy human teeth, Shahravan et al (22) reported that 0.28, 0.33, and 0.40 WP ratios of white MTA had no significant differences on the histologic outcome of direct pulp capping on healthy pulps. Yet, they attributed the increase in the number of mildly inflamed samples to higher WP ratios because of the increased solubility and porosity inside MTA (22). In a study investigating the solubility and porosity of MTA using different WP ratios, it was reported that the porosity of MTA increased as the WP ratios increased (1). The presence of porosity may be advantageous for the MTA hydration process because the connected pores provide networks for the water to diffuse into the material (23). However, Basturk et al (4) reported a negative correlation between the porosity and flexural strength of MTA, which might be explained by the porosity causing the material to be weaker.

MTA has a composition very similar to Portland cement with the addition of bismuth oxide and gypsum (20). Bentz and Aïtcin (24) reported a direct link between the water-to-powder ratio and the spacing between the cement particles and the cement paste—the lower water-to-cement ratio, the stronger the concrete. Strength is considered to be the amount of stress that is necessary to fracture a material. MTA strength can be an important factor, especially in certain applications, such as pulp capping or repair of furcation perforation in which MTA would be subjected to occlusal loading (25).

In the present study, ProRoot MTA had higher strength values than MTA Angelus, which was in agreement with earlier studies (4, 5). MTA Angelus particles have a wide size distribution compared with ProRoot MTA particles (26), and as a result, ProRoot MTA is more homogeneous compared with MTA Angelus. Incongruities in the cement's microstructure might result in larger local water-to-powder distances, which are inversely related to the strength of the material (24). Smaller particles are better able to absorb moisture (27). Therefore, the difference in the compressive strength of ProRoot MTA and MTA Angelus could be attributed to the differences in particle shape and size, which might affect flexural (4) and compressive strength (5). Exposure to atmospheric moisture may also cause an increase in particle size that may adversely affect the properties and subsequent clinical performance of the material. Thus, the single use of each package of material has been recommended for both ProRoot MTA and MTA Angelus (27), and, therefore, in our study the contents inside each package were used only once after the manufactured seal had been broken.

In a study investigating the adaptability of MTA to the walls of simulated root canals using direct ultrasonic activation or hand compaction, Aminoshariae et al (6) reported that hand compaction gave better adaptation to the walls, and it created fewer voids in the set phase than ultrasonic placement. Furthermore, Yeung et al (28) concluded that hand compaction followed by indirect ultrasonic activation for 1 second resulted in a denser MTA fill than that accomplished by hand compaction alone. They speculated that longer activation time might produce voids, which resulted in a lower weight. Yet, in a study evaluating the effect of mechanical and manual mixing as well as the effect of ultrasonic agitation during placement on the compressive strength of MTA, it was reported that a 30-second indirect ultrasonic activation improved the compressive strength of the material (5). The results of the present study revealed that ultrasonic placement resulted in higher compressive strength values (68.69 ± 28.12 MPa) compared with hand compaction $(64.42 \pm 24.78 \text{ MPa})$, with no significant differences between them. The variable results in these studies might be caused by the use of direct or indirect ultrasonic agitation as well as the duration of the activation.

Pelliccioni et al (29) reported that the lack of water addition during the preparation of the cement did not affect the *in vitro* sealing ability of ProRoot MTA. Hawley et al (18) evaluated the effect of varying WP ratios on the setting expansion of MTA, and they reported no significant difference in terms of MTA setting expansion. Even though the results of these studies revealed that differences in the WP ratios did not seem to influence some properties of the material, some positive properties of the final mix may be lost. In a study evaluating solubility and porosity with different WP ratios, Fridland and Rosado (1) concluded that the amount of water used in preparing the MTA mix had a direct effect on its solubility.

The present study showed that higher WP ratios result in lower compressive strength values and that ultrasonication had no significant effect on the compressive strength of the material regardless of the WP ratio applied. Therefore, clinicians should be encouraged to use the recommended WP ratio when preparing MTA cement in clinical practice. Encapsulation of predetermined amounts of water and powder are recommended in order to standardize the MTA mixture before its intramural placement.

Conclusion

In conclusion, we discovered that the higher the WP ratio the lower the compressive strength values of MTA. Ultrasonication had no significant effect on the compressive strength of MTA regardless of the various water-to-powder ratios. Therefore, clinicians should be encouraged to use the recommended WP ratio when preparing an MTA slurry in clinical practice.

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

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