

# The Effect of Various Mixing and Placement Techniques on the Compressive Strength of Mineral Trioxide Aggregate

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## Abstract

**Introduction:** The aim of this study was to evaluate the effect of various mixing techniques including mechanical and manual mixing as well as the effect of ultrasonic agitation during placement on the compressive strength of mineral trioxide aggregate (MTA). **Methods:** Tooth-colored ProRoot MTA (Dentsply Maillefer, Ballaigues, Switzerland) and white MTA Angelus (Angelus Soluções Odontológicas, Londrina, Brazil) were used. One gram of each powder was mixed with a 0.34-g aliquot of distilled water. Specimens were mixed either by mechanical mixing of capsules for 30 seconds at 4,500 rpm or by a saturation technique and the application of a condensation pressure of 3.22 MPa for 1 minute. Half of the specimens were placed in stainless steel molds and agitated using indirect ultrasonic activation. All specimens were subjected to compressive strength testing after 4 days. **Results:** The compressive strength values of ProRoot MTA were significantly greater than those of MTA Angelus ( $P < .05$ ). The highest compressive strength values were recorded from ProRoot MTA samples that were mixed mechanically and placed using ultrasonic activation (mean = 101.71 MPa), whereas the lowest values were recorded for MTA Angelus samples that were mixed manually and placed without ultrasonic activation (mean = 53.47 MPa). Ultrasonically agitated groups had higher compressive strength values ( $P < .001$ ). The specimens mixed mechanically had higher compressive strength values than those mixed manually ( $P < .05$ ). **Conclusions:** The compressive strength values of ProRoot MTA were significantly greater than those of MTA Angelus. Mechanical mixing enhanced the compressive strength of the material. Regardless of the mixing techniques applied, ultrasonic agitation improved the compressive strength of the material. (*J Endod* 2013;39:111–114)

## Key Words

Compressive strength, manual mixing, mechanical mixing, mineral trioxide aggregate, ultrasonic agitation

Mineral trioxide aggregate (MTA) is a hydraulic cement consisting of fine hydrophilic particles that gradually harden in a moist environment (1, 2). The compressive strength of hydraulic cements is considered as an indicator of the progress of the hydration reaction and a reflection of the setting process (3). Correct proportioning and mixing are essential to ensure that cements attain their optimum physical properties such as consistency, compressive strength, and film thickness (4). Encapsulation along with mechanical mixing can standardize the mixing technique and times (5) and produce consistent mixtures with optimum handling characteristics and physical properties (4).

Few studies have examined variations of mixing and placement techniques and their effects on the properties of MTA-like materials. In a laboratory study that evaluated the sealing ability and retention characteristics of MTA, Hachmeister et al (6) reported that placement technique was more important than the material itself. When comparing manual and mechanical mixing, Nekoofar et al (7) revealed that the application of ultrasonic energy to MTA produced a significantly higher surface microhardness value. Shahi et al (8) compared ultrasonication, trituration, and conventional mixing and concluded that different mixing methods had no significant effect on the push-out bond strength of white MTA.

Little information is available on the effect of various mixing and placement techniques on the compressive strength of MTA and MTA-like materials. Thus, the purpose of this study was to evaluate the effect of various mixing techniques including mechanical and manual mixing as well as the effect of ultrasonic agitation during placement on the compressive strength of MTA. It was hypothesized that mechanical mixing followed by the application of ultrasonic agitation would result in higher compressive strength values.

## Materials and Methods

The parameter investigated was compressive strength, and the materials investigated were tooth-colored ProRoot MTA (Dentsply Maillefer, Ballaigues, Switzerland) and white MTA Angelus (Angelus Soluções Odontológicas, Londrina, Brazil).

## Sample Preparation

One gram of each powder was mixed with a 0.34-g aliquot of distilled water using manual mixing or mechanical mixing with either conventional placement or ultrasonic agitation. Thus, there were 8 groups in total; each containing 10 specimens. ProRoot MTA was used in groups 1 to 4, and MTA Angelus was used in groups 5 to 8. The groups consisted of the following:

- Groups 1 and 5: Mixed mechanically and placed with ultrasonic agitation
- Groups 2 and 6: Mixed mechanically and placed without ultrasonic agitation

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3. Groups 3 and 7: Mixed manually and placed with ultrasonic agitation
4. Groups 4 and 8: Mixed manually and placed without ultrasonic agitation

Stainless steel 2-part split molds with internal dimensions of  $6.0 \pm 0.1$  mm high and a  $4.0 \pm 0.1$  mm diameter were produced (Medical Physics and Clinical Engineering, Cardiff and Vale UHB, Cardiff, UK). The internal surfaces of the molds were coated using polytetrafluoroethylene dry-film lubricant (Rocol, Leeds, UK).

### Mechanical Mixing

Mechanical mixing of MTA was performed by mixing of 1 g MTA powder with 0.34 g distilled water in a plastic mixing capsule containing a plastic pestle at 4,500 revolutions/min for 30 seconds using an amalgamator (Promix TM; Dentsply Caulk, York, PA) (7). The mixture was loaded into the molds with minimum pressure.

### Manual Mixing

An aliquot of 0.34 g distilled water was added to 1 g MTA powder until it was saturated. The mixture was transferred into the mold with minimum pressure. The material was then subjected to 3.22 MPa vertical pressure for 1 minute using a custom-made device (Medical Physics and Clinical Engineering, Cardiff and Vale UHB).

### Ultrasonic Agitation

Half of the specimens in the mechanical mixing groups and half of the specimens in the manual mixing groups were selected randomly. Indirect ultrasonication was applied by placing an endodontic plunger in the center of the material avoiding contact with the walls or floor of the mold and a CPR-2D tip (Obtura Spartan, Fenton, MO) placed in contact with the plunger. The ultrasonic device (Suprasson P5; Satelec, Merignac, France) was then activated for 30 seconds at scale 5. The excess material was removed. A wet cotton pellet was placed on the exposed surface of all specimens, and a damp paper towel was placed under the molds and incubated at 37°C and fully saturated humidity for 4 days.

### Compressive Strength

The compressive strength of specimens was determined according to the method recommended by the British Standards Institution (9) using a Lloyd LR MK1 machine (Lloyd Instruments, Fareham, UK). The load was applied at a speed of 1 mm/min along their long axis. The load at fracture of each specimen was noted, and its compressive strength was calculated in megapascals (MPa) according to the following formula:

$$CS(\sigma) = \frac{4P}{\pi d^2},$$

where CS is the compressive strength,  $P$  is the maximum force applied in newtons, and  $d$  is the mean diameter of the specimen in millimeters.

**TABLE 1.** The Minimum and Maximum Values, Means, and Standard Deviations of the Compressive Strength of the Groups

| MTA type | Mixing/placement technique | Mean       | SD     | Minimum | Maximum |        |
|----------|----------------------------|------------|--------|---------|---------|--------|
| G1       | ProRoot                    | MM + US    | 101.71 | 18.64   | 81.90   | 129.41 |
| G2       | ProRoot                    | MM         | 90.85  | 25.25   | 50.69   | 125.22 |
| G3       | ProRoot                    | Man M + US | 90.78  | 33.60   | 36.88   | 147.26 |
| G4       | ProRoot                    | Man M      | 90.77  | 27.21   | 58.86   | 143.57 |
| G5       | Angelus                    | MM + US    | 81.36  | 24.94   | 50.97   | 124.03 |
| G6       | Angelus                    | MM         | 74.14  | 28.43   | 30.61   | 117.86 |
| G7       | Angelus                    | Man M + US | 54.96  | 17.47   | 32.55   | 81.10  |
| G8       | Angelus                    | Man M      | 53.47  | 22.31   | 24.75   | 89.64  |

Man M, manual mixing; MM, mechanical mixing; US, ultrasonication.

### Statistical Analysis

Multivariate analysis of variance and Tukey honestly significant difference tests were performed to compare the mean values for compressive strength using a significance level of  $P < .05$ .

### Results

The minimum and maximum values, means, and standard deviations of the compressive strength of the groups are shown in Table 1. Overall, the compressive strength values of ProRoot MTA (mean =  $93.38 \pm 26.27$  MPa) were significantly greater than those of MTA Angelus (mean =  $65.06 \pm 25.54$  MPa,  $P < .05$ ). The highest compressive strength values were recorded for ProRoot MTA samples that were mechanically mixed and placed with ultrasonic activation (mean = 101.71 MPa), whereas the lowest values were recorded for MTA Angelus samples that were mixed manually and placed without ultrasonic activation (mean = 53.47 MPa).

Regardless of the MTA type used and the placement method applied, the samples mixed mechanically had higher compressive strength values than those mixed manually ( $P < .05$ ). Also, the groups that were mixed mechanically and placed with ultrasonic agitation had higher compressive strength values than the groups that were mixed manually and placed with ultrasonication ( $P < .05$ ). Regardless of the use of ultrasonics, mechanically mixed MTA Angelus samples had significantly higher compressive strength values compared with those mixed manually ( $P \leq .001$ ). Also, ultrasonically agitated ProRoot MTA had higher compressive strength values than ultrasonically agitated MTA Angelus ( $P \leq .001$ ).

Further statistical analysis revealed a significant difference between the compressive strength values of the mechanically mixed and ultrasonicated ProRoot MTA group and the manually mixed and ultrasonicated MTA Angelus group ( $P < .05$ ). A significant difference was also found between manually mixed MTA Angelus specimens and all ProRoot MTA groups ( $P < .05$ ).

### Discussion

The effect of manual mixing, mechanical mixing of encapsulated MTA, and ultrasonic agitation on the compressive strength of 2 different brands of MTA was evaluated in the present study. The results revealed that mechanically mixed MTA had higher compressive strength values than those mixed manually, and the compressive strength values of ProRoot MTA were significantly greater than those of MTA Angelus.

ProRoot MTA samples that were mechanically mixed and placed with ultrasonic activation had the highest compressive strength values, whereas the lowest values were recorded for MTA Angelus samples that were mixed manually and placed without ultrasonic activation. Also, the groups that were mixed mechanically and placed with ultrasonic agitation had higher compressive strength values than the groups that were mixed manually and placed with ultrasonication. Ultrasonic agitation enhanced the compressive strength of the material regardless of the

mixing techniques applied. Ultrasonically agitated ProRoot MTA had higher compressive strength values than ultrasonically agitated MTA Angelus.

Compressive strength is one of the indicators of the setting and strength of a material (1, 10). Although mechanical tests are unable to reflect the clinical situation, they can help detect and compare the effect of various mixing techniques on different cement types. Mechanical properties of cements might change and interfere with their clinical behavior if mixed inaccurately (11). Mitchell and Douglas (12) found that hand-mixed cements were the weakest because of the air entrapment, whereas encapsulated cements that were mixed and centrifuged before extrusion were the strongest. To produce cements with optimum properties, uniform proportioning and mixing methods must be established. Nekoofar et al (13) drew attention to an inconsistency in the amounts of water in the ampoules of ProRoot MTA packages. Fridland and Rosado (14) found that physical properties of MTA changed when mixed with different water-to-powder proportions. Thus, in order to overcome the lack of consistency in the amount of water supplied, a laboratory digital scale was used.

Most of the studies evaluating the physical properties of ProRoot MTA (15–17) used hand mixing. Thus, the pressure applied during condensation was an uncontrolled variable. In a study examining the effect of compaction pressure on some physical properties of MTA, Nekoofar et al (18) recommended, for research purposes, that a controlled compaction pressure of 3.22 MPa in sample preparation was necessary. In this study, saturation followed by the application of 3.22 MPa compaction pressure was applied to the manually mixed groups to standardize the amount of pressure.

The compressive strength of MTA is affected by the type of MTA (17), mixing liquid (19, 20), condensation pressure (18), acid-etching procedures (10), and mixing techniques (7). Even though the setting time for MTA was measured as 4 hours (1), the compressive strength and push-out strength of MTA reach their maximum several days after mixing (1, 21). The compressive strength of MTA samples that were kept in a humid situation for 4 days was greater than that of 4-hour samples (10). Kayahan et al (10) suggested that restoration with acid-etch composite after MTA placement should be postponed for at least 96 hours. To decrease the chance of MTA displacement, MTA must be allowed to set untouched for 72 hours or longer (22). In the present study, MTA samples were kept for 4 days in moisture to allow optimum setting in accordance with Nekoofar et al (20), Namazikhah et al (23), and Shokouhinejad et al (24). Further long-term studies are needed to evaluate the effect of various mixing and placement techniques on the compressive strength of MTA.

Torabinejad et al (1) measured the compressive strength of gray MTA as 40.0 MPa. Holt et al (19) and Watts et al (25) showed that white MTA had higher compressive strength values than those of gray MTA. The present results revealed that tooth-colored ProRoot MTA had a mean compressive strength value of  $93.38 \pm 26.27$  MPa, which is in accordance with the findings of Nekoofar et al (18) and Watts et al (25), which were  $71.36 \pm 24.81$  MPa and  $81.8 \pm 25.48$  MPa, respectively.

Although the constituents of ProRoot MTA and MTA Angelus are similar (26, 27), the lower compressive strength values of MTA Angelus could be explained by a difference in particle size. MTA Angelus particles had relatively low circularity and wide size distribution and were less homogeneous than ProRoot MTA (28). The present results revealed that the compressive strength of MTA Angelus was greater after mechanical mixing.

Encapsulation along with mechanical mixing techniques can produce standardized and consistent MTA slurries (7, 8). The results of the present study showed that mechanical mixing enhanced the

compressive strength of MTA compared with manual mixing and placement. This could be explained by the concept that mechanical mixing created a less grainy mixture with fewer unhydrated particles resulting in better water diffusion. Conversely, manual mixing and placement were associated with inadequate hydration by limiting the microchannel formation inside the material and compromising the ingress of water to hydrate the material (7). Future studies are required to determine the effect of these techniques on hydration characteristics and other physical properties of MTA.

## Conclusions

The compressive strength values of ProRoot MTA were significantly greater than those of MTA Angelus. Mechanical mixing of encapsulated MTA resulted in higher compressive strength values than those mixed manually. Regardless of the mixing techniques applied, ultrasonic agitation improved the compressive strength of the material.

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