

Effect of Various Mixing and Placement Techniques on the Flexural Strength and Porosity of Mineral Trioxide Aggregate

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Abstract

Introduction: The aim of this study was to evaluate the effect of mechanical and manual mixing as well as the effect of ultrasonic agitation during placement on the flexural strength and porosity of mineral trioxide aggregate (MTA). **Methods:** White ProRoot MTA and white MTA Angelus 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 4500 rpm or by a saturation technique and application of a condensation pressure of 3.22 MPa for 1 minute. The mixed slurries of all materials were loaded into 2 × 2 × 25 mm molds for testing flexural strength and 3 × 4 mm molds for evaluation of porosity. Half of the specimens were placed in the stainless steel molds by using indirect ultrasonic activation. All specimens were incubated for 4 days. Micro-computed tomography was used to determine the porosity of each specimen, and a 3-point bending test was used to evaluate flexural strength. Tukey honestly significant difference and independent *t* tests were carried out to compare the means at a significance level of *P* < .05. **Results:** Irrespective of mixing and placement techniques applied, the flexural strength values of ProRoot MTA were significantly greater than those of MTA Angelus (*P* < .05). A medium negative correlation was found between flexural strength values and total porosity percentage. **Conclusions:** Although mechanical mixing of encapsulated cements was quicker and provided more consistent mixes, this technique along with ultrasonic agitation was not associated with a significant advantage in terms of flexural strength and total porosity over manual mixing. (*J Endod* 2014;40:441–445)

Key Words

Flexural strength, mechanical mixing, micro computed tomography, MTA, ultrasonic agitation

The manipulation of dental cements frequently involves the mixing of powder and liquid components, with the relative proportions being assessed by eye or with the aid of operator-dependent measuring systems in an uncontrolled situation (1). Mixing variations are likely to occur even when proportioning aids are used (2). For example, hand mixing has been reported to introduce operator-induced variability because of the inaccurate dispensation of the powder and liquid constituents (3). The volume of powder dispensed by using a scoop or the amount of liquid dispensed with a dropper bottle is dependent on the manner in which the scoop is filled or the drop is dispensed (4). The powder-to-liquid mixing ratios used in clinical practice vary when scoop and dropper bottle systems are not used, and the constituents are mixed to the operators' desired consistency (5). Therefore, the optimum ratio recommended by the manufacturer is not always used in clinical practice (6).

It is desirable to establish a homogeneous proportioning and mixing technique for cements to ensure they acquire their optimum properties (4). Capsulation yields consistent mixes by enabling the powder/liquid ratio and mixing regimen to be standardized so that the functional properties of the plastic cement mass will not be susceptible to clinically induced variability (4, 7). Mechanical mixing might also reduce air spaces between adjacent particles, resulting in a more thorough wetting of the powder particles and leading to an improvement in the setting reaction and thus physical properties of the resultant cement (8).

Mineral trioxide aggregate (MTA), a type of hydraulic cement that can set in the presence of water, is a widely used material in endodontics (9). For MTA, mechanical mixing has been shown to enhance the compressive strength of the material (10).

Apart from the mixing regimen applied, Hachmeister et al (11) reported that the delivery system might be more important than the material itself. Matt et al (12) reported that apical barriers placed with ultrasonic activation demonstrated fewer voids than barriers placed without ultrasonic energy. In contrast, Aminoshariae et al (13) reported that hand condensation resulted in better adaptation of MTA than ultrasonic activation. Basturk et al (10) reported that ultrasonic agitation enhanced the compressive strength of hydraulic cements.

Sensitivity to clinical techniques might also interfere with the clinical behavior and optimum performance of materials (2, 14). It has been reported that investigations of simple mechanical tests allow a correlation of mechanical properties with clinical performance (15) and can advise clinicians which cements need special care or entail particular risks during the mixing and placement processes (2).

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To date, there is little information about the effects of various mixing and placement techniques on the flexural strength and porosity of MTA. The purpose of this study was to evaluate the effect of manual and mechanical mixing techniques as well as the effect of ultrasonic agitation during placement on the flexural strength and porosity of MTA. It was hypothesized that mechanical mixing followed by the application of ultrasonic agitation would result in lower porosity and higher flexural strength values.

Materials and Methods

The parameters investigated were flexural strength and porosity; the materials investigated were tooth-colored (white) ProRoot MTA (Dentsply Maillefer, Ballaigues, Switzerland) and white MTA Angelus (Angelus Soluções Odontológicas, Londrina, Brazil).

Eight groups were prepared by manual mixing or mechanical mixing with either conventional placement or ultrasonic agitation. ProRoot MTA was used in groups 1–4, and MTA Angelus was used in groups 5–8. 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; groups 3 and 7, mixed manually and placed with ultrasonic agitation; and groups 4 and 8, mixed manually and placed without ultrasonic agitation.

Sample Preparation

The instruments and the test materials were conditioned at 23°C ± 1°C in the laboratory for 1 hour before use.

Split molds with internal dimensions of 2 × 2 × 25 mm were designed and machined from stainless steel to accommodate beam specimens (Medical Physics and Clinical Engineering, Cardiff and Vale UHB, Cardiff, UK). The molds were open on the upper surface. The internal surfaces of the molds were coated by using 2 layers of polytetrafluoroethylene dry-film lubricant (Rocol, Leeds, UK).

Mechanical Mixing

An empty amalgam capsule was cleaned in accordance with British Standard Institution (16), and 1 g MTA powder and 0.34 g distilled water were added. A plastic rod-like pestle was added to the capsule to facilitate mixing (17). The capsules were sealed and mixed for 30 seconds at 4500 rpm by using an amalgamator (Promix TM; Dentsply Caulk, York, PA). The mixture was loaded into the molds with minimum pressure, and the excess material was removed.

Manual Mixing

An aliquot of 0.34 g distilled water was added to 1 g MTA powder until it was absorbed. The mixture was transferred into the molds with internal dimensions of 6.0 ± 0.1 mm height and 4.0 ± 0.1 mm diameter, with minimum pressure by using the tip of a dental spatula. The material was then subjected to 3.22 MPa vertical pressure for 1 minute by using a custom-made MTA-compression device (Medical Physics and Clinical Engineering, Cardiff and Vale UHB) to standardize the placement technique (18). The material was then transferred into the flexural strength molds by using minimum pressure.

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 a CPR-2D tip (Obtura Spartan, Fenton, MO) in contact with the outer surfaces of the mold, avoiding contact with the material inside the mold. The ultrasonic device

(Suprasson P5; Satelec, Merignac, France) was then activated for 30 seconds at scale 5.

Extruded material was removed, a wet cotton pellet was placed on the exposed surface of each specimen, and the molds were transferred to a plastic container that was sealed and stored at a temperature of 37°C and 95% humidity for 4 days.

Flexural Strength

Ten samples were prepared for testing flexural strength. The specimens were carefully removed from the split mold after 4 days. Specimens with visible cracks or voids were discarded; flexural strength was measured by using a 3-point bend test (Lloyd Instruments, Farnham, UK).

The mean maximum load (N), flexural strength (MPa), and flexural modulus (MPa) values of the specimens were measured by using a 3-point bending test with a 20.0-mm span distance between the lower rollers and a 0.5 mm/min crosshead speed. The load-deflection curves were obtained by means of Nexygen Mt v4.5 PC software (Lloyd Instruments Ltd).

Porosity

Four cylindrical samples with dimensions of 3.0 ± 0.1 mm height and 4.0 ± 0.1 mm diameter were prepared from each group. Samples were mixed by using either mechanical mixing of capsules in an amalgamator for 30 seconds at 4500 rpm or manual spatulation and the application of pressure of 3.22 MPa for 1 minute. Half of the materials were subjected to ultrasonic energy, avoiding contact with the inner walls or floor of the molds. The ultrasonic tip was indirectly activated for 30 seconds at power scale 5. Specimens were then incubated at 37°C in fully saturated humidity for 4 days.

Samples with visible voids or cracks were discarded and replaced. The samples were examined by using the high-resolution micro-computed tomography system SkyScan 1072 (SkyScan, Aartselaar, Belgium). The x-ray tube was operated at 50 kV and 200 μA by using 1-mm aluminum filter. The system was controlled by a PC workstation running under Microsoft Windows 95 (Microsoft Corp, Redmond, WA). The scanning of the specimens was achieved with a rotational angle of 180° around the vertical axis, a rotational step of 0.90°, and a 3.1-second exposure time.

By using NRecon software (Skyscan), images obtained from the scan were reconstructed to show 2-dimensional slices of the inner structure of the MTA samples. Three-dimensional reconstruction, volumetric analysis, and the measurement of volume of porosity were analyzed by using the CTan and CTVol software (SkyScan).

Statistical Analysis

The mean values for flexural strength and porosity were compared by using Tukey honestly significant difference and independent *t* tests (SPSS 9.0; SPSS Inc, Chicago, IL) at a significance level of *P* < .05.

Results

Flexural Strength

The results are shown in Table 1. The flexural strength values of ProRoot MTA were significantly greater than those of MTA Angelus (*P* < .05).

ProRoot MTA samples that were mixed manually and placed with ultrasonic agitation (mean, 11.27 MPa) had the highest flexural strength values, whereas MTA Angelus samples that were mixed mechanically and placed with ultrasonic agitation (mean, 8.73 MPa) had the lowest values.

TABLE 1. Minimum and Maximum Values, Means, and Standard Deviations of Flexural Strength and Porosity

Group	MTA type	Mixing/placement technique	Flexural strength (MPa)			Porosity (%)		
			Mean ± SD	Minimum	Maximum	Mean ± SD	Minimum	Maximum
1	ProRoot	MM + US	10.50 ± 1.82	8.63	12.90	1.81 ± 1.25	0.90	3.60
2	ProRoot	MM	9.99 ± 1.36	8.24	12.02	1.29 ± 1.34	0.41	3.28
3	ProRoot	ManM + US	11.27 ± 1.71	9.15	12.80	1.11 ± 0.46	0.60	1.49
4	ProRoot	ManM	10.36 ± 2.14	7.83	12.75	1.58 ± 1.62	0.52	3.97
5	Angelus	MM + US	8.73 ± 2.11	6.45	12.67	1.85 ± 1.37	0.86	3.40
6	Angelus	MM	8.91 ± 1.99	6.44	11.88	1.11 ± 0.33	0.84	1.57
7	Angelus	ManM + US	8.96 ± 1.45	6.87	10.09	1.44 ± 0.28	1.12	1.63
8	Angelus	ManM	9.52 ± 2.12	7.52	12.78	1.48 ± 0.42	1.07	1.89

ManM, manual mixing; MM, mechanical mixing; SD, standard deviation; US, ultrasonication.

Regardless of the MTA type used, no significant difference was found between the mixing techniques ($P < .05$).

Regardless of the mixing technique, a significant difference was found between ultrasonically agitated ProRoot MTA and ultrasonically agitated MTA Angelus ($P < .05$). ProRoot MTA had higher flexural strength values than those of MTA Angelus.

Porosity

A summary of the results of total porosity percentage (the minimum and maximum values, means, and standard deviations) of the groups is shown in Table 1. There was no statistically significant difference between the percentages of total porosity of any of the groups ($P > .05$).

A negative medium correlation was found between the flexural strength and total porosity values ($r = 26%$). Manually mixed ProRoot MTA group had the highest flexural strength values and the lowest total porosity values (mean, 1.107 ± 0.46), whereas the mechanically mixed and ultrasonically agitated MTA Angelus group had the lowest flexural strength values and the highest total porosity values (mean, 1.85 ± 1.37) (Fig. 1).

Figure 1 shows the 3-dimensional reconstruction of an MTA specimen, revealing the total porosity inside the material.

Discussion

The effect of mechanical mixing of encapsulated MTA, manual mixing, and ultrasonic agitation on the flexural strength and porosity of ProRoot MTA and MTA Angelus was evaluated in the present study. The results revealed that the flexural strength values of ProRoot MTA were significantly greater than those of MTA Angelus, with a negative correlation between flexural strength values and total porosity percentage.

Behr et al (2) reported that the mechanical properties of dental cements might change in a manner that could interfere with their clinical behavior. Kleverlaan et al (14) reported that powder/liquid ratio, temperature, porosity, and diffusion might change the mechanical properties of dental cements. Thus, the variables related to mixing and placement are key factors that influence the performance of dental materials.

Changes in the hydration process might interfere with the biological, chemical, and physical properties of MTA-like materials (19). Hardened MTA is affected by the quantity of water used during mixing, the mixing procedure itself, pressure used for compaction, environmental humidity, and temperature (20, 21). Although moisture is necessary for MTA to set, excess moisture is contraindicated (22). Thus, correct proportioning is essential when preparing an MTA mixture. In most clinical and laboratory studies on the mechanical

and physical properties of MTA, ampules from MTA packages were used. Nekoofer et al (23) drew attention to an inconsistency in the amount of water in the packages of ProRoot MTA (Dentsply International Inc). Considering the inconsistency in these ampules, too much or too little liquid might have been used, causing variations in hydration. Even though a high liquid-to-powder ratio increases the release of calcium hydroxide, the amount of water incorporated in the MTA slurry must be limited because of the serious management problem encountered when transporting or compacting the material (21). To standardize the amount of water that was used in this study, each gram of MTA powder was mixed with 0.34 g distilled water (17).

One limitation inherent in this study was that the thickness of MTA specimens for the flexural strength test was 2 mm. This thickness might not be relevant in some clinical applications. Despite this limitation, the aim of the investigation was to compare the flexural strength and porosity of 2 different brands of MTA as well as to provide insight into the effect of various mixing and placement techniques on its flexural strength.

It has been reported that investigations of simple mechanical parameters such as compressive or flexural strength allow a correlation of mechanical properties with clinical performance (15) and can advise clinicians which cements need special care or entail particular risks during the mixing and placement process (2). Basturk et al (10) reported 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.

Flexural strength may reflect clinical function better than compressive strength, because the site where MTA is placed may be exposed to occlusal loading, for example in furcation perforation repairs, especially before the placement of a permanent restoration (22). Walker et al (22) reported that when amalgam is condensed directly against an MTA repair, it is essential that its strength be higher than the stress associated with amalgam condensation (6–9 MPa) to prevent fracture of the MTA.

Regardless of the mixing or placement technique applied, the mean flexural strength values of ProRoot MTA (10.51 ± 1.66 MPa) and MTA Angelus (9.02 ± 1.84 MPa) were higher than the stress associated with amalgam condensation. Walker et al (22) evaluated the effect of setting time and hydration on the flexural strength of MTA by allowing the specimens to set for either 24 or 72 hours with exposure to moisture on either 1 or 2 sides. For one-sided moisture/72-hour specimens, they reported that the flexural strength of white ProRoot MTA was 11.16 ± 0.96 MPa, which is comparable to the findings of this study.

Aggarwal et al (24) evaluated the effect of distilled water, sodium hypochlorite (5.25%), chlorhexidine gluconate (2%), EDTA solution (17%), and BioPure MTAD on the flexural strength of tooth-colored

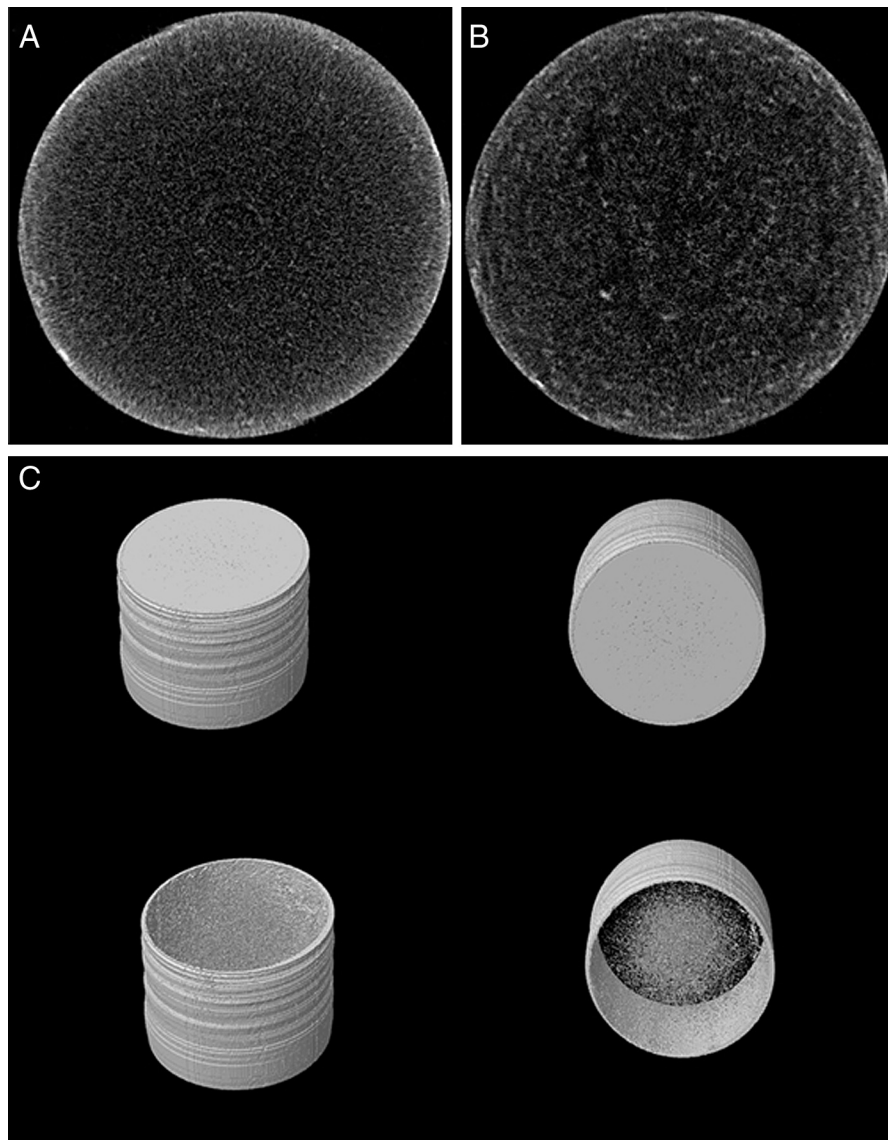


Figure 1. (A) A 2-dimensional image of ProRoot MTA mixed manually and placed ultrasonically, which had the highest flexural strength and lowest porosity values. (B) MTA Angelus mixed mechanically and placed ultrasonically, which had the lowest flexural strength and highest porosity values. (C) Three-dimensional micro-computed tomography images of an MTA specimen showing internal porosity.

ProRoot MTA and reported that the maximum mean flexural strength values (15.714 ± 0.663 MPa) were recorded for the specimens mixed with and exposed to distilled water. Their results are relatively higher in comparison to the present study; however, they allowed the MTA samples to set for 7 days and did not mention whether the specimens were moistened on one side or both. Camilleri (25) investigated the flexural strengths of various hydraulic cements in conjunction with an admixture and demonstrated that the flexural strength values increased with time. This might be why the findings of Aggarwal et al (24) differ from the results of the present study.

The main component of MTA is Portland cement (20), and when Portland cement is mixed with water, it forms a structure of micropores, capillary channels, and loosely trapped water (21). Part of the water in the mix will be used for the chemical reactions involved in the setting process, and another part will be trapped in the pores and capillaries (19). Even though the presence of porosity may be advantageous for the MTA hydration process because these connected pores might provide networks for the water to diffuse into the material (26), the

fact that there was a negative medium correlation between the porosity and flexural strength values might be explained by the porosity causing the material to be weaker.

Various techniques and devices have been used for porosity evaluation, including mercury porosimetry (27), nitrogen perfusion (28), scanning electron microscopy (29), capillary flow porometry (30), and the Archimedes principle method (21). However, all these techniques have drawbacks. Micro-computed tomography is a nondestructive, 3-dimensional imaging technique that can be used as an alternative means of determining both porosity and pore size distribution (31). Even the individual closed pores can be visualized, and the true internal morphology of granules can be revealed (31).

In the present study, micro-computed tomography was used to evaluate the porosity percentage of the specimens. In a study comparing the efficacy of amalgam, Fuji-Plus, Geristore, and ProRoot MTA as intra-orifice barriers, micro-computed tomography was used by Zakizadeh et al (32). They reported that MTA was significantly less porous compared with Fuji-Plus and Geristore. This present study is considered

to be one of the first to use micro-computed tomography to measure percentage of volume of total porosity in MTA specimens.

Even though the difference was not statistically significant, ultrasonicated groups had higher porosity percentage ($1.57\% \pm 0.91\%$) than non-ultrasonicated groups ($1.37\% \pm 0.98\%$). In a laboratory study, Aminoshariae et al (13) reported that ultrasonication caused more voids than hand condensation and concluded that the manufacturers' recommended powder-to-liquid ratio for MTA may not be ideal for ultrasonic placement and might be the reason for voids that resulted with this technique.

The same trend was also observed for mechanically mixed groups showing higher porosity values (1.49 ± 1.05) than those mixed manually (1.42 ± 0.85). By using zinc phosphate cement, Fleming et al (4) reported that mechanical mixing of encapsulated cement resulted in air entrapment in the cement mix, which manifested itself as porosity. Even though different cement types might show different porosity levels, air entrapment caused by the mechanical mixing motion might be the reason for the higher porosity values than manual mixing.

To date, no study has evaluated the flexural strength of MTA Angelus. According to the findings of the present study, ProRoot MTA had higher flexural strength values than MTA Angelus. This might be due to the differences in particle shape and size. ProRoot MTA contains fewer numbers of large particles than MTA Angelus (33), and in that respect ProRoot MTA is more homogeneous than MTA Angelus (34). MTA Angelus particles have wide size distribution compared with ProRoot MTA (33). Although there was no significant difference between the total porosity percentages of the MTA brands used in this study, the lower flexural strength values of MTA Angelus might be explained by its less homogeneous structure.

Conclusion

Regardless of the mixing and placement techniques applied, ProRoot MTA groups had higher flexural strength values than those of MTA Angelus. A medium negative correlation was found between flexural strength values and total porosity percentage. Although mechanical mixing of encapsulated cements is quicker and provides more consistent mixes, this technique combined with ultrasonic agitation conferred no significant advantage in terms of flexural strength and total porosity over manual mixing.

Acknowledgments

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

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