Original Article

The Effect of Operator-induced Variability on the Physical Properties of ProRoot MTA

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Abstract

Aims: The aim of this study was to compare the influence of operators on the microhardness and compressive strength of mineral trioxide aggregate (MTA). **Materials and Methods:** Forty dental specialists were asked to prepare a series of MTA samples. The tested material was ProRoot MTA (DentsplyMaillefer, Switzerland). Each participant prepared one sample to a consistency they considered acceptable for use in practice (improvised group) and another one according to the manufacturer's recommended water-to-powder (WP) ratio (pre-weighed group). The samples were incubated at 37°C and 95% humidity for 4 days. Parameters evaluated in this study were microhardness and compressive strength. **Results:** Operators mixed MTA samples with varying WP ratios. However, there was no significant difference between the microhardness and compressive strength values of MTA samples between the improvised, the pre-weighed and the control groups. MTA was mixed in a thicker consistency than the manufacturers recommended ratio (0.33) by 62.5% of the operators. **Conclusion:** According to the results of this study, even though the WP ratios that were utilized in the clinical setting vary, microhardness and compressive strength values of MTA was not significantly affected.

Keywords: *Compressive strength, microhardness, MTA, surface microhardness, water-to-powder ratio*

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Introduction

Over the past two decades, mineral trioxide aggregate (MTA) has become one of the most widely studied endodontic materials. During the development of MTA, several strength testing procedures including push-out,[1] compressive,[2,3] three-point flexure,[4,5] and shear bond[6] have been reported broadly in the dental literature. The developments have led the manufacturers to employ two different presentational forms of MTA, namely, a hand-mixing form where the operator dispenses powder and liquid components using a scoop and a dropper bottle, or a mechanical-mixing form where the operator triturates a capsule with a predetermined amount of powder and liquid proportions.

The original MTA product (ProRoot MTA, DentsplyMaillefer, Ballaigues, Switzerland) is sold in single-use sachets of various weights; whereas other MTA products are marketed in bottles or Eppendorf-tubes that allow operators to use a small amount of powder and recap the rest of the material in its original bottle for future use. Although clinicians pay a lot of attention in selecting the material to use for specific applications, they disregard the mixing procedure. Variations due to mixing are more pronounced for hand-mixed water/powder mixtures, even when the proportioning aid was used.[7] The volume of powder dispensed by a scoop solely depends on the operator's decision, therefore, a range of mixing ratios will unavoidably occur in practice.[8] In such cases, the optimum ratio recommended by the manufacturer is rarely achieved. Unfortunately, the physical, chemical, and biomechanical

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properties of MTA are known to be dependent on the mixing regime applied.[2,9,10]

Billington *et al*.[11] and Fleming *et al*.[8] reported that a single operator could regularly achieve consistent mixes when manipulating dental cement, but disparities between different operators were significant. Although MTA is a widely-used material, the issue as to whether this bioactive endodontic cement has a wide therapeutic range or not was addressed. The therapeutic range is the concentration of a drug at which the patient will experience the anticipated clinical outcome with a minimum of undesirable or adverse effects.[12] If the cement has low therapeutic range, it would react very sensitive even to low grade mixing errors.[7] Therefore, the material would need special clinical care during mixing. Low-grade variations of mixing ratio are considered as 10–20% and high-grade variations are considered as 40–60% changes caused by the inaccuracy of proportioning aids.[7] There are few investigations[10,13] concerning the effect of not-recommended mixing ratios on the mechanical properties of ProRoot MTA.

The aim of the current study was to investigate the effect of incongruent mixing ratios routinely encountered clinically by different operators on selected properties of ProRoot MTA. The first hypothesis investigated was that the compressive strength and surface microhardness of MTA would be more susceptible to operator-induced variability than those prepared with a predetermined amount of MTA. In addition, it was hypothesized that the preparation time of MTA would significantly increase when mixed with ratios employed by different operators compared with that of pre-weighed MTA.

Materials and Methods

Specimen preparation

The present study followed the methodology of Fleming *et al*.[8] Oral and written informed consent was taken from all study participants and ethics committee approval was obtained prior to commencing the study (Ethics Committee of Marmara University Health Sciences Institute approval no: 2016-111). Forty dental practitioners, specialized in endodontology (n = 24), pediatric dentistry (n = 7,) or maxillofacial surgery (n = 9) with a minimum of 3-year experience as a specialist, were asked to prepare a series of MTA samples of a standard luting consistency that they considered acceptable for use in practice.

In the improvised group, the operators added ProRoot MTA powder with a LOT number of 0000123922 from a packed sachet of 500 mg to 80 mL of distilled water on a glass slab. The materials were conditioned at room temperature 1 h prior to use. The MTA samples were prepared by each dental practitioner and the weight of the unused MTA powder was determined and the WP mixing ratio calculated. The mixtures that the operator did not consider acceptable for use in their practice were discarded.

In the pre-weighed group, the operators added 240 mg of ProRoot MTA powder to 80 mL of distilled water to verify if the mixing regime, irrespective of the mixing ratio, had any effect on the results.

A control group (n = 10) was prepared by mixing 80 mL of liquid and 240 g of ProRoot MTA powder by a single operator.

The prepared MTA slurry was then transferred by each operator to propylene molds with internal dimensions of 6.0 ± 0.1 mm high and 4.0 ± 0.1 mm diameter. 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. The specimens were incubated at 37°C and fully saturated humidity for 4 days.

Microhardness testing

After 4 days, the specimens were removed from the molds. Specimens containing visible defects were discarded from the study. The specimens were wet polished at room temperature using a silicon carbide-based sandpapers of 1000, 1200, 1500, and 2000-grit particle size (The MetaServ 250, Buehler, Germany) for microhardness testing.

The Vickers microhardness test of each specimen was performed using a Micro-Vickers Hardness Tester Model 401MVD (WolpertWilson, Wolpert Wilson Instruments, Aachen, Germany) and a square-based pyramid-shaped diamond indenter with a full load of 100 g load with a dwell time of 10 s which formed a quadrangular depression with two equal orthogonal diagonals in the polished surface of the cement. Three indentations were made on each specimen and an average was calculated. The Vickers microhardness value was displayed on the digital read-out of the microhardness tester.

Compressive strength testing

For compressive strength testing, the load was applied at a speed of 1 mm/min along the long axis of each specimen. The load at fracture of each specimen was noted, and its compressive strength was calculated in megapascals (MPa) according to the following formula:

$$CS = 4\,P/\pi D^2$$

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.

Preparation time

The preparation time of MTA for both pre-weighed group and the improvised group were determined with a digital stopwatch. The stopwatch was initiated once the operator started mixing the material, and stopped when the operator no longer mixed the material.

Statistical analysis

After the Shapiro-Wilk normality test was applied, the Kruskal-Wallis test was used to reveal whether there are significant differences between groups.

Results

The WP ratio of the specimens in the improvised group varied from 0.20 to 0.46 [Figure 1]. Of all operators, 62.5% mixed MTA in a thicker consistency (>0.33 ± 0.03) than the manufacturers recommended ratio (0.33). The operators also prepared a specimen using 80 mL of liquid and 240 g of MTA powder, which was the manufacturer's recommended ratio.

The mean microhardness values of the improvised group and the pre-weighed group were 67.79 ± 6.64 HV and 64.91 ± 5.4 HV, respectively. The control group samples recorded a mean microhardness value of 64.32 ± 4.04 HV. There was no statistically significant difference between the control and the experimental groups in terms of microhardness values ($P > 0.05$).

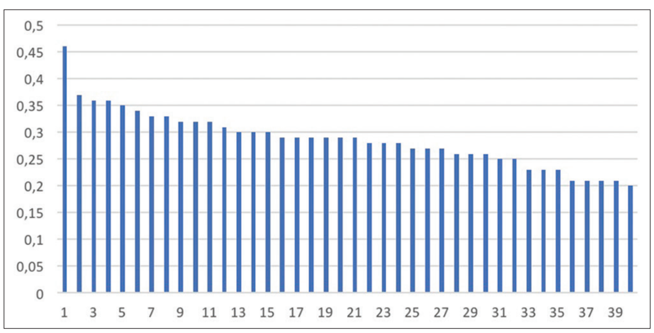

Figure 1: The water-to-powder ratio of MTA that was utilized in the improvised group

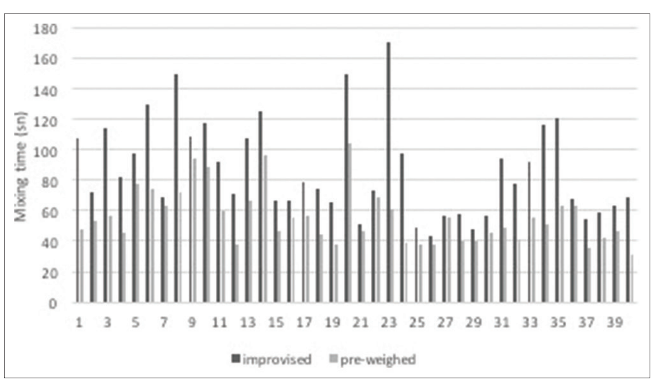

Figure 2: The mixing time of MTA for the improvised and pre-weighed groups

The mean compressive strength values of improvised and pre-weighed groups were 60.64 ± 16.83 MPa and 64.62 ± 18.49 MPa, respectively. For the control group; a mean compressive strength value of 62.58 ± 14.28 MPa was recorded. There were no statistically significant differences were found between the groups in terms of compressive strength values ($P > 0.05$).

Mixing time of MTA for each experimental group was also recorded [Figure 2]. The longest mixing time was recorded for the improvised group (171 s) whereas the shortest mixing time was 31 s in the pre-weighed group. The average mixing times for the improvised and pre-weighed groups were 86.4 and 55.6, respectively. For all the participants, the mixing time of MTA was shortened when a pre-weighed amount was given ($P < 0.05$).

Discussion

In clinical practice, clinicians frequently estimate the amounts of water and powder chairside, thus deviating from the manufacturer's recommendations.[14] Our main objective was to see if this variation from the recommended guidelines could have an effect on the microhardness or the compressive strength of the material. There were no statistically significant differences between the experimental and control groups in terms of compressive strength and surface microhardness.

It has been suggested that simple mechanical tests cannot reflect the clinical situation.[7] However, they can be indicators for setting reaction and the strength of cement on various mixing ratios.[15] Strength is the amount of stress that is necessary to fracture a material. MTA strength can be an important factor, in certain applications where MTA would be subjected to occlusal loading.[16] Therefore, these tests can provide an insight as to whether these cement would need special clinical care during mixing procedure or not.

Fridland and Rosado[13] stated that beyond 0.33 ratio, the material was too liquid to manage and a 0.26 ratio was reported as the minimum that allowed a mix of putty-like consistency to be manipulated. However, the dental specialists who participated in our study, with a minimum of 3 years' clinical experience, mixed and manipulated MTA with WP ratios as high as 0.46 and as low as 0.20. These ratios reflect the consistency of MTA that they found suitable for use in their practice. To simulate a more accurate clinical setting, each operator was asked to prepare as much sample as they desired until they considered the mixture acceptable for clinical use. The mixtures that the operators were not satisfied with were discarded.

The operators who participated in our study, mixed MTA with either up to 39% less or more powder. Behr *et al*.[7] reported that in the case of WP systems, operators tend to prepare the mix higher liquid content. A more fluid cement that was mixed with a 0.50 WP ratio increased the washout[17] and reduced the pushout bond strength of MTA Angelus.[18] An 0.40 WP ratio of the mixture also reduced the compressive strength of ProRoot MTA, MTA Angelus,[10] and calcium-enriched mixture (CEM).[19] However, 25 out of 40 (62.5%) participants of our study mixed the material in a thicker consistency than the recommended WP ratio. Only one participant mixed MTA with a WP ratio exceeding 0.40 (0.46).

Shahravan *et al*.[20] evaluated the histologic pulp reaction to various mixing ratios of MTA as a pulp capping material in healthy human teeth, and reported that 0.28, 0.33, and 0.40 WP ratios of MTA had no significant differences on the histologic outcome of direct pulp capping on healthy pulps. For a microleakage study, Peliccioni *et al*.[21] filled the roots only with powder without any previous hydration. They reported that the absence of water addition did not affect the *in vitro* sealing ability of ProRoot MTA. Hawley *et al*.[14] also mixed MTA with varying WP ratios of 0.26, 0.28, 0.30, and 0.35. They found no significant differences between differing WP ratios in terms of expansion of MTA. Our results revealed that deviating from the manufacturer's recommended WP mixing ratio in a clinical setting would have a negligible influence on the compressive strength or surface microhardness of MTA. MTA is likely to have a wide therapeutic range. The tested mechanical properties of MTA did not significantly change in case of inaccuracy of mixing ratios up to 39%. Operator-induced variability is expected to be tolerated. Therefore, the first hypothesis was rejected.

Mixing time was shortened when the operators were given a pre-weighed amount of MTA. The shortest mixing time was recorded for the pre-weighed group (31 s). The manufacturer recommends the operators to mix the material with the liquid for about 1 min to ensure all the powder particles are hydrated.[22] The average mixing time for the pre-weighed group was 55.62 s. However, when the participants were asked to prepare MTA to a consistency that they desired acceptable for clinical use, the material was mixed for 86.45 s on average. There was no correlation between the mixing time and the tested physical properties of MTA. However, it is recommended to examine the effect of mixing time on the overall performance of MTA.

Conclusion

Deviating from the manufacturer's recommended WP mixing ratio in a clinical setting would have a negligible influence on the compressive strength or surface microhardness of ProRoot MTA. Mixing time was reduced when the operators were given a pre-weighed amount of MTA. Future studies are needed to assess if the varying WP ratios that are utilized in the clinical setting would affect other properties of MTA or different bioactive endodontic types of cement.

Declaration of Patient Consent

The authors certify that they have obtained all appropriate participation consent forms. In the form, the participant(s) has/have given his/her/their consent for his/her/their images and other clinical information to be reported in the journal. The participants understand that their names and initials will not be published and due efforts will be made to conceal their identity, but anonymity cannot be guaranteed.

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Conflicts of interest

There are no conflicts of interest.

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