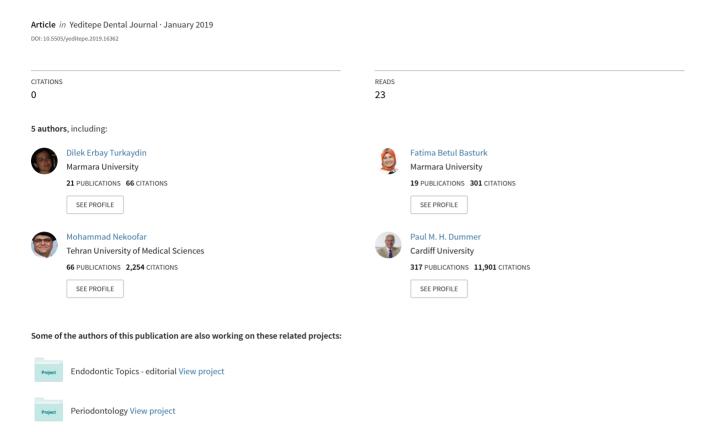
## Does the mixing and placement regime affect the pH of Mineral Trioxide Aggregate?



# Does the mixing and placement regime affect the pH of Mineral Trioxide Aggregate?

### Karıştırma ve yerleştirme teknikleri Mineral Trioksit Agregatının pH değerini etkiler mi?

Assist. Prof. Dilek Türkaydin

Marmara University, Faculty of Dentistry, Department of Endodontics, İstanbul

Orcid ID: 0000-0002-4185-2643

**Assoc. Prof. Fatima Betül Baştürk**Marmara University, Faculty of Dentistry,
Department of Endodontics, İstanbul

Orcid ID: 0000-0002-5494-9209

Prof. Mohammad Hossein Nekoofar

Tehran University of Medical Sciences, Tehran, Iran

Orcid ID: 0000-0003-0530-8621

Prof. Mahir Günday

Marmara University, Faculty of Dentistry, Department of Endodontics, Istanbul

Orcid ID: 0000-0002-2019-8734

**Prof. Paul Dummer** 

Cardiff University, Faculty of Dentistry, UK

Orcid ID: 0000-0002-0726-7467

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#### Corresponding author:

Assist. Prof. Dilek Turkaydin

Marmara University, Başıbüyük 9/3, Maltepe, Istanbul

Phone: 905058021958

E-mail: dilek.turkaydin@marmara.edu.tr

#### **SUMMARY**

**Aim:**The objective of this study was to measure in a laboratory setting the pH of tooth coloured ProRoot MTA and MTA Angelus following various mixing and placement techniques, including mechanical mixing, manual mixing and indirect ultrasonic activation.

Materials and Method: Tooth coloured ProRoot MTA and White MTA Angelus were used. One gram of each powder was mixed with a 0.34 g of distilled water that were allocated to eight experimental groups, each containing three specimens. Four groups were prepared by mechanical mixing of capsules for 30 s at 4500 rpm the other four were mixed manually. Half of the specimens in each group were placed in moulds using indirect ultrasonic activation. pH values were recorded directly from within the freshly mixed material and were analyzed using one-way ANOVA at a 0.05 level of significance.

**Results:** No significant difference in pH was found between the mixing and placement techniques or the materials tested. The highest pH value recorded was in the ProRoot group that was mixed manually and placed ultrasonically (11.64). The Angelus group, which was mixed manually without an ultrasonic agitation, had the lowest pH values (10.42).

**Conclusion:** Mechanical mixing and ultrasonication conferred no significant disadvantage in terms of the initial pH of the material. Since mechanical agitation of encapsulated cements provides more consistent mixes, it might be possible to use this technique combined with ultrasonic agitation as an alternative to manual mixing, both in clinical and in laboratory conditions, in order to achieve standardization of the material so as to enhance its properties.

Keywords: PH, placement, MTA, mixing.

#### ÖZET

**Amaç:** ProRoot MTA ve MTA Angelus'un mekanik ve elle karıştırma teknikleriyle karıştırılıp, indirekt ultrasonik aktivasyon ile yerleştirilmesinin materyalin pH değerleri üzerindeki etkisini in vitro olarak incelemektir.

Gereç ve Yöntem: Beyaz ProRoot MTA ve beyaz MTA-Angelus kullanılmıştır. Bir gramlık toza 0,34 g distile su eklenmiş ve sekiz grup hazırlanmıştır. Grupların yarısı, kapsüllere yerleştirilerek 4500 rpm hızda 30 sn boyunca karıştırılmış, diğer yarısı ise elle karıştırılmıştır. Hazırlanan bu karışımların yarısına indirekt ultrasonik aktivasyon uygulanmıştır. Numunelerin pH değerleri karışım hazırlandıktan hemen sonra ölçülmüş ve değerler one-way ANOVA ile 0,05 anlamlılık düzeyinde değerlendirilmiştir.

**Bulgular:** Test edilen materyaller bazında, karıştırma ve yerleştirme tekniklerinin arasında herhangi bir anlamlı fark bulunamamıştır. En yüksek pH değeri elle karıştırılıp ultrasonik aktivasyon uygulanan ProRoot grubunda (11,64), en düşük pH değeri ise elle karıştırılıp ultrasonik aktivasyon uygulanmayan MTA Angelus grubunda kaydedilmiştir (10,42).

Sonuçlar: Mekanik karıştırma ve ultrasonik aktivasyon, mater-

yalin pH değeri üzerinde herhangi bir olumsuz etki göstermemiştir. Karıştırma ve yerleştirme teknikleri materyalin pH değerleri üzerinde herhangi bir etkiye sebep olmamıştır. Kapsüller içerisindeki simanların mekanik olarak karıştırılmasının daha tutarlı kıvamda karışımlar hazırladığı göz önünde bulundurulursa, bu tekniği ultrasonik aktivasyonla birlikte uygulamak hem klinik hem de laboratuvar koşulları altında standardizasyonun sağlanması açısından bir alternatif olarak düşünülmelidir.

Anahtar kelimeler: pH, karıştırma, MTA, yerleştirme.

#### **INTRODUCTION**

Mineral trioxide aggregate (MTA) is a hydraulic cement that is able to set in the presence of blood or other fluids.¹ In an aqueous environment, it releases calcium hydroxide, which makes the hydrated cement highly alkaline.² In the clinical environment, the manufacturers' recommended water-to-powder ratio is difficult to achieve when the material is mixed manually³ In fact, hand-mixing has been reported to cause inaccuracies during dispensation of the powder and liquid constituents due to the use of scoop and dropper bottle systems.⁴,⁵ In this scenario the volume of powder dispensed is dependent upon the method of filling the scoop whilst dropper bottles frequently dispense uncalibrated volumes of liquid.⁶ These variations are further exacerbated when the constituents are mixed according to the operators' desired consistency.⁵

The pressure applied during condensation of MTA is another uncontrolled variable. In a study examining the effect of compaction pressure on some physical properties of MTA, Nekoofar et al.<sup>7</sup> reported that higher condensation pressures produced lower surface microhardness values. Ultrasonication is an alternative method of placing MTA without applying high condensation pressure with the compressive strength,<sup>8</sup> surface micro-hardness<sup>9</sup> and fill-density<sup>10</sup> of MTA all being enhanced when placed by ultrasonic agitation.

The effect of various mixing and placement techniques on the compressive strength,<sup>8</sup> push-out bond strength,<sup>11</sup> microstructure,<sup>12</sup> hydration,<sup>13</sup> flexural strength and porosity<sup>14</sup> of MTA have been studied. However, information on the effect of mechanical mixing and ultrasonication on the pH of MTA is lacking. Biological properties of MTA are based on its alkaline pH and calcium release. Thus, it is important to note if the mixing and placement regime had any effects on the pH of MTA.

Therefore, the objective of the present study was to measure in a laboratory setting the pH of Tooth coloured Pro-Root MTA and White MTA-Angelus when prepared with various mixing and placement techniques. The null hypothesis is that the mechanical mixing and ultrasonication would not affect the pH of these materials.

#### **MATERIALS AND METHODS**

The parameter investigated was pH, and the materials investigated were Tooth-coloured ProRoot MTA (Dentsply Sirona Endodontics, Ballaigues, Switzerland) and White MTA-Angelus (Angelus Solucoes Odontologicas, Londrina, Brazil).

Eight groups of specimens were prepared. Four groups were prepared with ProRoot MTA while the other four were prepared with MTA-Angelus. Within the groups of each material, two subgroups were mixed manually and two were mixed mechanically with each subgroup being placed conventionally with hand instruments or using ultrasonic agitation. 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

Groups 4 and 8: Mixed manually and placed without ultrasonic agitation

The instruments and test materials were conditioned at 23°C±1°C in the laboratory for 1 h before use. Mechanical mixing was performed by mixing 1 g MTA powder with a 0.34 g aliquot of distilled water in a plastic mixing capsule containing a plastic pestle. It was then mixed at 4500 rpm for 30 s using an amalgamator (Promix TM; Dentsply Caulk, York, PA, USA).15 The mixture was loaded into ring moulds using minimum pressure. In the manual mixing group, 0.34 g of distilled water was added to 1 g of MTA powder until it was absorbed and the slurry was then mixed manually. Half of the specimens in the mechanical mixed groups and half of the specimens in the manually mixed groups were selected randomly to apply indirect ultrasonication. Indirect ultrasonic activation was applied by placing the CPR ultrasonic tip (Spartan, Fenton, MO, USA) in contact with the outer surfaces of the mould. The ultrasonic device (Suprasson P5; Satelec, Merignac, France) was then activated for 30 s at scale 5.

The pH of each sample was measured with a pH meter, (pH 1000 L, pHenomenal®, VWR International, Dublin, Ireland), which was calibrated with buffer solutions at pH 4.0, 7.0 and 11.0. The electrode was inserted into the freshly mixed material and readings were taken immediately after mixing at a temperature of 24°C. Statistical analysis of the pH vales in each subgroup was carried out using one-way ANOVA at 0.05 level of significance.

#### **RESULTS**

The mean pH values of each group are shown in Figure 1.

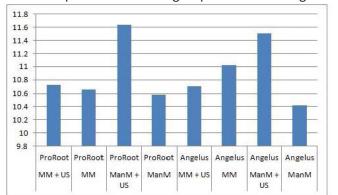


Figure 1. The pH values of all tested groups. MM: Mechanical mixing, ManM: Manual mixing, US: Ultrasonication.

The highest pH value recorded was in the ProRoot group that was mixed manually and placed ultrasonically (11.64). The Angelus group, which was mixed manually without an ultrasonic agitation, had the lowest pH values (10.42). Regardless of the mixing or placement technique, there was no significant difference between the mean pH values of Tooth coloured ProRoot MTA (10.9) and White MTA-Angelus (10.92). Also, there was no statistically significant difference in terms of the mixing and placement techniques applied (p>0.05).

#### **DISCUSSION**

It is known that the hydration of MTA can be influenced by various mixing and placement methods applied.<sup>13</sup> Therefore, the effect of manual mixing, mechanical mixing, and ultrasonic agitation on the pH of ProRoot MTA and MTA-Angelus was evaluated in the present study. There was no statistically significant difference in any of the parameters tested (p>0.05).

The release of hydration by-products from Mineral Trioxide Aggregate, such as calcium hydroxide, may affect the pH of the material.<sup>13</sup> MTA is affected by various factors, including the mixing techniques applied,14,15 the quantity of water used during mixing,8 pressure used for compacting the material<sup>7</sup> and environmental humidity.<sup>16</sup> Fleming et al.5 emphasized that the ideal mixing conditions of the cement components are seldom achieved. In fact, in a study4 in which 40 dental nurses prepared a series of three cement samples which they considered were acceptable for clinical use, the compressive strength of 70% of the samples were below the standard value. In order to overcome these variables, mechanical mixing of encapsulated MTA has been recommended. 9,17 Nowadays, there are several commercially available MTA-like materials with ready-to-be mechanically mixed capsules. 18,19

Torabinejad et al.<sup>20</sup> assessed the pH value of ProRoot MTA with a pH meter using a temperature compensated electrode and found that the initial pH was 10.2. However, it was not clear if the readings were taken from a solution in which the samples were immersed or from the fresh-

ly mixed samples directly. Their initial readings of the pH values for ProRoot MTA coincide with our findings (10.9). In the present study, no significant difference was measured between the pH values of the MTA-Angelus (10.92) and ProRoot MTA (10.9) groups. Duarte et al.<sup>21</sup> evaluated the pH of ProRoot MTA and MTA-Angelus after 3, 24, 72, and 168 hours. By immersing the specimens in distilled water, they found that the mean pH values recorded were the highest during the first three hours then tended to decrease. They reported that the mean pH values and calcium ion release were slightly higher for MTA-Angelus than for ProRoot MTA. However, the difference was not reported as statistically significant.

One limitation inherent to this study is that the moulds used were not in contact with any kind of liquid. In a clinical situation, freshly mixed MTA would be exposed to tissue fluid and/or blood immediately. However, laboratory studies are important in helping to understand the properties of materials. In this study, all pH readings were recorded under standardized conditions. Thus, the results for each group and subgroup are comparable.

Duarte et al.<sup>21</sup> compared the pH of MTA-Angelus with the pH of ProRoot MTA using an immersion method. They found that the mean pH values after 24 h for MTA Angelus and ProRoot MTA were 9.42 and 9.32 respectively. Similarly, de Vasconcelos et al.<sup>22</sup> reported the pH of MTA Angelus as 9.42, which is lower than our findings. It may be that these differences can be attributed to the fact that the authors measured the pH from immersion tubes in water, and not directly from the mass of the material as in the present study. Many studies on the evaluation of the pH of different types of MTA have been undertaken based on the immersion technique.15,21-23 The immersion method has the advantage of researchers being able to record the measurements even after the setting time. However, it does not represent the pH of the material, but rather its ability to alkalize.22 In addition, when freshly mixed MTA becomes immersed in water, the water-to-powder ratio is altered, affecting the properties of the material.<sup>2,8</sup> In this study, in order to compare the mixing and placement methods without altering the water-to-powder ratio, we measured the initial pH by placing the pH-meter into the mixed material. Chng et al.24 and Islam et al.25 also measured the pH of MTA during the setting process directly within the freshly mixed material.

Although several studies<sup>15,21-26</sup> have evaluated the pH of MTA and MTA-like materials, they are not easily comparable because of the different methodologies used and/ or the lack of information in the methodology or in the results. Nevertheless, all of the studies showed that these materials are highly alkaline.

The results of our study allow the null hypothesis to be accepted., that is the various mixing and placement

regimens did not influence significantly the pH of the materials.

#### CONCLUSION

Mechanical mixing and ultrasonication had no significant impact on pH of the ProRoot MTA and MTA Angelus. Since mechanical agitation of encapsulated cements provides more consistent mixes, it might be possible to use this technique combined with ultrasonic agitation as an alternative to manual mixing, both in clinical and in laboratory conditions, in order to achieve standardization of the material so as to enhance its properties.

Conflict of Interest Statement

The authors deny any conflict of interests related to this study.

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