

In-Vitro Comparison of Primary Setting Time and Surface Porosity of Two Types of Mineral Trioxide Aggregate (MTA)

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Abstract

Background and Aim: Use of MTA-based cements has disadvantages like prolonged setting time, and inappropriate application. This study aims to compare two types of MTA in terms of setting time and surface porosity.

Materials and Methods: Two MTA cement groups were investigated, 1: ProRoot MTA and 2: Angelus MTA. Primary Setting Time of 5 samples from each group was evaluated according to ISO 6876: 2001 standard with a 10mm diameter and 1mm thickness stainless steel mold in a 37±1 °C incubator with 95% relative humidity using Gilmore needle system. After final setting, surface porosity of samples was assessed using an electron microscope with 2000 and 5000 magnifications. For data analysis, statistical t-test was used.

Results: According to statistical tests, primary setting times of materials in two groups differed significantly ($P<0.001$) with group1: 38.72 ± 0.61 and group2: 22.46 ± 0.61 minutes, and microscopic investigations revealed better surface properties in ProRoot MTA compared with those in Angelus MTA.

Conclusion: Angelus MTA had shorter primary setting time compared with ProRoot MTA, but ProRoot showed better surface properties and less porosity after final setting.

Key Words: MTA cement , primary setting time , electronic microscope

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Introduction

Initially introduced by Torabinejad in 1993 [1], mineral trioxide aggregate (MTA), is a mixture of Di-Calcium Silicate, Tri-Calcium Silicate, Tri-Calcium Aluminates, and Tetra-Calcium Alumina Ferrite [2]. Added to water, this substance produces a colloidal gel that contains calcium hydroxide, and creates an impermeable barrier after setting [3]. Since the introduction of MTA as root-end filling material, it has found diverse endodontic applications in root restoration and bone regenera-

tion. Advantages of this material include facilitating bone regeneration, cementum, and reproduction of periodontal fibers, ability to make a homogeneous dentin barrier, elicitation of minimal inflammatory response, good biological compatibility, radio-opacity and hydrophilicity [4]. MTA is used in apexogenesis, apexification, direct pulp capping, and repair of perforations, orthograde apical plug, and retrograde root-end fillings [4, 6]. When used as root-end filling, MTA is exposed to interstitial fluid, and calcium ions (in calcium hy-

droxide) combine with phosphate ions (in interstitial fluid) making hydroxyapatite crystals [3].

Like other cements used in dentistry, setting time of MTA cement is clinically important because appropriate mechanical properties are obtained after setting. Thus, materials should have the shortest setting time (as mentioned in standards). Setting time is categorized into primary and final [7]. According to ISO 6876:2001 standard, primary setting time is the time between mixing and the time when Gilmore needle no longer leaves circular impression on the surface of the sample. However, final setting time of a cement may take 24 hours up to a week [8]. Primary setting should not occur too quickly, causing insufficient working time, it should not take too long either that would waste both patient's and the dentist's time [9, 10].

In some treatments, shortness of setting time of dental cements is clinically important. Pulp capping treatment is commonly performed by calcium hydroxide. High pH (12.5) of calcium hydroxide causes secretion of hard dentin barrier [11]. In the oral cavity and under restorations, this material becomes soluble causing dentin bridge porosity [3]. MTA with its high pH (nearly 11) has been introduced as a pulp capping material in recent years. For clinical applications in pulp capping, MTA primary setting time is too long, therefore, more than one session is needed. Because it takes too long to set, it can easily break off due to pressure or during cleaning, even at normal storage temperatures [12] and filling materials cannot be placed on it [1, 13]. Due to its prolonged primary setting time, the dentist should place a moist cotton pellet over MTA for 24 hours before inserting the restorative material. This causes further lengthening of treatment time and cost. Since this cement, in clinical terms, has a shorter and more reasonable primary setting time, it can be used in single-visit pulp capping treatment. Moreover, root canal treatments take less time. Manufacturers have attempted to shorten MTA cement setting time by adding some catalysts. Incorporation of materials like calcium sulfate dihydrate and calcium chloride in powder and tartaric acid in liquid is effective in

accelerating chemical reactions of dental materials [14, 15].

In the brochure for each material, only the final setting time is mentioned, but the primary setting time is not. According to manufacturers, the final setting time is 2 hours and 45 minutes for ProRoot MTA cement (DENTSPLY Company), and 15 minutes for Angelus MTA (A Brazilian product.) In the brochures of these two products (available in Iran) that have been recommended for one session pulp capping, there are no mentions of the primary setting times. Only based on a study by Islam, this time for ProRoot MTA was found to be approximately 38 minutes [10].

Given the unclear primary setting time, this study aims to experimentally find and compare primary setting time of two MTA products (tooth-colored) and assess their surface porosity using scanning electron microscopy (SEM).

Materials and Method

In this experimental study, two types of MTA were used, ProRoot MTA (Dentsply, Tulsa Dental, USA), and Angelus MTA (Angelus, Londrina, Brazil). The entire experimental process was performed according to ISO 6876:2001 standard specified for measurement of primary setting time of water base cements. Of each tooth-colored MTA type, 5 samples were prepared. First, a few sachets of powder and liquid of each MTA with same batch number were prepared for each group. Then, powder and liquid were mixed with a 3: 1 ratio to prepare the mixture of each type of cement. The mixture was placed in a stainless steel cylindrical mold of 10 mm diameter and 1 mm height (based on ISO 6876:2001) [8].

Based on the standards, all equipments used in preparation of samples were washed, dried and sterilized before the experiment. Before the experiment, all equipments and materials, slab, and spatulas were incubated at 23 ± 1 °C for one hour. The mold (Humboldt, USA) and Gilmore needle system were also incubated at 37 ± 1 °C and relative humidity of 95%. After mixing each material in any of the groups, the chronometer (AKAI timer,

China, accurate to 1/100 second) was set at zero according to manufacturer's recommendation, and switched on when the mixing time was over. The molds were overfilled with MTA and pressed down inside the mold by mixing spatula and the plastic instrument. Next, a mixing spatula rinsed with distilled water was used to smooth the surface. For the measurement of primary setting time, Gilmore system with the marker weight of 100 ± 0.5 grams and cylindrical needle tip of 2 mm in diameter and 5 mm length was used. According to the standards, 120 ± 10 seconds after the end of mixing process in the mold, the mixture was incubated at 37 ± 1 °C with relative humidity of 95%. At 30-second intervals (based on ISO6876:2001), Gilmore needle was gently applied to horizontal surface of the material for 5 seconds. Any indentation on the surface would indicate that the material was not set yet. This process was repeated until no indentation could be seen on the surface of the material [10]. At this stage, the time recorded by the chronometer was taken as the primary setting time. This process was performed for all samples in both groups.

In the next stage, the time for final setting (according to the brochure) was allowed to elapse. For SEM evaluation, two samples out of each group were coated with 20 nm gold using Polaron Sputter Coater (Quorum Technologies, Newhaven, UK) and examined under electron microscope (LED 440i, Leo Electron Microscopy LTD, Cambridge, UK, and SEM) with 1000x and 5000x magnification. From the images produced, surface porosity and homogeneity of the two materials were evaluated after final curing. The collected data were analyzed using t-test.

Results

Two tooth-colored MTA types were studied, and results are presented in tables 1 and 2. Kolmogorov Smirnov test showed normal distribution of data for all groups ($P > 0.05$). The mean and standard deviation of primary setting time of two MTA types are shown in table 1. T-test revealed a significant difference in primary setting time between

the two types ($P < 0.001$), with Angelus MTA having the shorter time of the two.

Table 1. The mean and standard deviation of primary setting time according to MTA type (in minutes)

Material type	Number	Mean	Standard deviation
ProRoot MTA	5	38/72	0/61
Angelus MTA	5	22/46	0/61

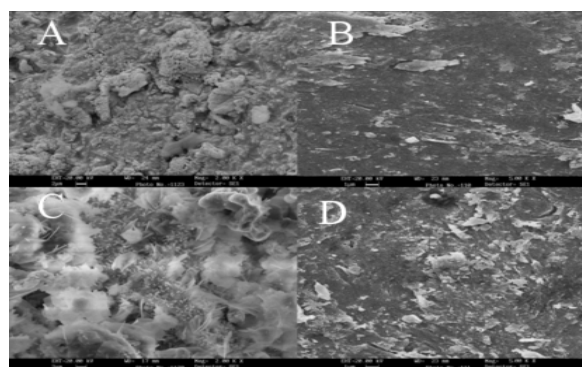
According to the t-test, primary setting time of MTA types was significantly different from the time stated by the manufacturers (table 2).

Table 2. Primary setting time of ProRoot and Angelus MTA types compared with time specified by the manufacturers

MTA	Degree of freedom	Mean difference	P.V
ProRoot (2 hours 45 minutes)	4	-126/28	0/00 0
Angelus (10 minutes)	4	12/5	0/000

Examination of samples under SEM revealed the least surface porosity after final setting was for ProRoot MTA (figure 1).

Figure 1. ProRoot MTA micrograph (A, B), and angelus MTA micrograph (C, D) in 2000x and 5000x magnifications



Discussion

It was shown in this study that the primary setting time of tooth-colored MTA cements was signifi-

cantly different from each other and also, different from the manufacturer specifications. But, considering less surface porosity in ProRoot MTA compared with Angelus MTA, it is possible that ProRoot MTA is more suited for some treatments.

Results of this study appear to be in line with those obtained by Islam et al. in 2006 [10]. Primary setting time obtained for ProRoot MTA concurred with that in Islam study (40 ± 2.94 minutes), indicating compatibility of controlled experimental conditions and precise adherence to the standards in both studies. Both followed ISO6876:2001 standards specific to measurement of primary setting time of water-based cements used in root canal treatment. Angelus MTA is Brazilian cement with unknown properties including primary setting time. Based on the information extracted from the manufacturer's brochure, primary setting time of 10 minutes, and final setting time of 15 minutes were specified. These were significantly different from the results of this study according to the standard conditions. As stated by the manufacturer in the brochure, primary setting time for ProRoot MTA is 2 hours and 45 minutes (similar to the gray type), which is significantly different from that found in this study, and also in the study by Islam et al.

In his study, Torabinejad calculated the primary setting time from the start of mixing, which is in conflict with the standard definition given in the literature. However, the difference in primary setting time of ProRoot MTA between this study and that of Torabinejads' is so vast that it cannot be due to the difference in definition only [1]. There is still a need for research in this area into different materials, and their other properties like compressive strength, and microleakage, and impact of other materials on them. Also, many studies have identified the effect of trace elements (such as Arsenic) on MTA properties, and considering presence of different trace elements in the two cements used in this study, different primary setting times could be well-expected for these cements [16]. Of the two cements, manipulation of ProRoot MTA was easier and the prepared material was more cohesive compared with Angelus MTA. However,

examination of these cements under SEM revealed less surface porosity and more homogeneity in ProRoot MTA compared with Angelus MTA. This characteristic makes ProRoot MTA more suitable cement for some applications like pulp capping. As one of the most suitable features of pulp capping material is creation of a better seal for protection of the pulp [6].

Manufactures of both materials in this study did not document any specific accelerating materials in their product. However, given the results of this study, it may be necessary to conduct chemical analysis of the materials in future researches. As against the ISO 6876:2001 standard that proposes use of dental plaster for the mold, and given the reaction of plaster with the MTA setting and accelerated response that could confound the results, in this study, as in the studies by Torabinejad and Islam et al., the mold was stainless steel (though of the same dimensions as in the standard) [10].

As surface characteristics indicate suitability of material in terms of structure and hydration, a suitable material for pulp capping is a material with least surface porosity that can provide an adequate seal and protect the pulp.

Conclusion

1. Primary setting time was different in two types of MTA and also different from the manufacturers' specification.
2. ProRoot MTA showed longer primary setting time than Angelus MTA.
3. Electron microscopy revealed less surface porosity and more surface homogeneity in ProRoot MTA compared with Angelus MTA.

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