

Effect of Alkaline Environment on Micro-Hardness of Mineral Trioxide Aggregate with Different Setting Times

Diatri Nari Ratih^{1*}, Sylvia Widhiharsari²

1. Department of Conservative Dentistry, Faculty of Dentistry, Universitas Gadjah Mada, Yogyakarta, Indonesia.

2. Conservative Dentistry Specialist Study Program, Faculty of Dentistry, Universitas Gadjah Mada, Yogyakarta, Indonesia.

Abstract

The purpose of this study was to determine the effect of alkaline environment on micro-hardness of mineral trioxide aggregate (MTA) with different setting times.

Forty-five samples were prepared in Teflon molds. Samples were randomly divided into 3 groups of 15 each. Group 1: immersed in pH 7.4 (a control), Group 2: immersed in pH 9.4, Group 3: immersed in pH 11.4. Each sample was tested using Vickers hardness tester on days 3, 14, and 28. MTA, which immersed in pH of 9.4 for 28 days had the highest micro-hardness (80.22 ± 0.74), while the MTA which immersed in pH of 11.4 for 3 days produced the lowest micro hardness (53.57 ± 1.07).

At conclusion, the alkaline environment influences the micro-hardness of MTA with setting times of 3, 14 and 28 days. pH 9.4 and setting time of 28 days showed higher micro-hardness than pH 11.4 with the setting time of 3 days.

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Introduction

Mineral trioxide aggregate (MTA) has been widely used in dentistry as materials for apexification, retrograde filling, pulp capping, pulpotomy, repairing root perforation and root canal obturation.^{1,2} Apexification is non-surgical treatments by applying a biocompatible material at the open root apex of non-vital tooth.³ Currently MTA is more popular than calcium hydroxide as an apexification material, since these materials have advantages such as biocompatibility, antibacterial, seal-ability, and its capacity to promote hard tissue formation, radiopaque, and has the ability to set although contaminated by humidity.⁴

MTA has been introduced since 1993 as a material with gray color (Grey ProRoot MTA). The newer tooth-colored material, commonly referred to as white MTA (White ProRoot MTA), was developed for its application in esthetically

sensitive areas.⁵ MTA is a mixture of tricalcium aluminate, dicalcium silicate, tricalcium silicate, tetracalcium-aluminoferrite, and bismuth oxide. Previous research demonstrated that MTA is superior to calcium hydroxide as a apexification materials, due to the disadvantages of calcium hydroxide, such as prolonged treatment time (approximately 3 to 20 months), the need for multiple visits and radiographs.⁶ In some cases, prolonged treatment may also cause root resorption occurred due to trauma or root fracture between visits.⁷

Apexification procedures require application of intracanal medication for sterilization of root canal system. Recently, non-setting calcium hydroxide has been a material of choice for intracanal medication. This is because calcium hydroxide has antimicrobial properties as it has a high pH.⁸ The high pH can eliminate or reduce bacterial contamination during treatment. Previous research showed that the application of calcium hydroxide as a root canal sterilization would increase the pH of surrounding dentin tissue in the range between 11.1 to 12.2, while before application dentin tissues have a pH of 6.4 to 7.4.⁹ This alkaline environment is probable to interfere the reaction of MTA and it might lower the physical properties of the MTA, such as micro-hardness.

*Corresponding author:

Diatri Nari Ratih

Department of Conservative Dentistry,
Faculty of Dentistry, Universitas Gadjah Mada,
Yogyakarta, Indonesia.
E-mail: diatri@ugm.ac.id

In clinical condition, micro-hardness of a material is very important as an indicator of the setting process and determine the material strengths, especially during the hydration process of the MTA that seems to be influenced by several conditions, such as alkaline environment.¹⁰ Therefore, this present study was undertaken to determine the effect of alkaline environment (pH 9.4 and 11.4) to the micro-hardness MTA with a different setting (3, 14, and 28 days).

Materials and methods

Forty-five cylindrical specimens (diameter: 5 mm, height: 2 mm) were prepared in Teflon molds. Initially, the powder of MTA (White ProRoot MTA, Densply Tulsa Dental, Johnson City, TN, USA) was mixed with a sterile water with a ratio of 3: 1 for 1 minute to obtain putty-like consistency. MTA then applied to the Teflon molds with micro-apical placement (MAP) (Dentsply Mailefer, Ballaigues, Switzerland) and compress with a condenser. A glass microscope slide with a load weight of 500 g was used to compress MTA in the mold for 30 seconds to ensure consistent and reproducible packing of the samples.

The samples were assigned randomly into 3 groups of 15 each. Each group was immersed in the synthetic tissue fluid with different pH. Group 1: samples were immersed in the synthetic tissue fluid with a pH of 7.4 (as a control), group 2: samples were immersed in the synthetic tissue fluid with a pH of 9.4, and Group 3: samples were immersed in synthetic tissue fluid with a pH of 11.4. During immersion samples were kept in an incubator with a temperature of 37°C. The synthetic tissue fluid was prepared as follows: 1.7 g KH₂PO₄, 11.8 g Na₂HPO₄, 80 g NaCl, and 2 g KCl were dissolved in 10 L of H₂O and buffered in potassium hydroxide at pH values of 9.4 and 11.4.¹¹ The synthetic tissue fluid for immersion were replaced every day.

Each sample was tested using Vickers hardness tester (Buechler Ltd, Lake Bluff, IL, USA) on days 3, 14, and 28. Before testing, each sample was clamped on board measuring machine. Each specimen was placed and clamped on a board measuring machine, therefore the surface to be measured located perpendicularly to the axis of the indenter.

Hardness test was performed using a Vickers diamond indenter and a 50 g load applied for 5 seconds.¹⁰ Three indentations were made on the surface of MTA. One indentation was made in the center and two indentations were made toward the periphery of the specimen. Furthermore, the results of three indentations were averaged to obtain a hardness value of each sample.

Under the microscope, the length of the diagonal of each indentation was directly measured, and Vickers micro-hardness tester automatically processed the data and the hardness value of each sample was obtained (in VHN units). The data then were analyzed using a two-way ANOVA, followed by Tukey's test with 95% level of significance.

Results

The results (Figure 1) showed that MTA, which was immersed in synthetic tissue solution with pH of 11.4 had the lowest micro-hardness than those with pH of 7.4 and 9.4, while the setting time of 28 days produced the highest micro-hardness compared to the setting time of 3 and 14 days. It also revealed that MTA, which was immersed in synthetic tissue solution with pH of 9.4 and setting time of 28 days generated highest micro-hardness than other groups except the control group, whereas pH 11.4 and setting time of 3 days demonstrated the lowest micro-hardness.

A two-way ANOVA followed by Tukey's test exhibited that a significant different occurred in all groups ($p < 0.05$), except for the pH of 7.4 with setting time of 14 days did not differ significantly from the pH of 9.4 with setting time of 3 days ($p > 0.05$).

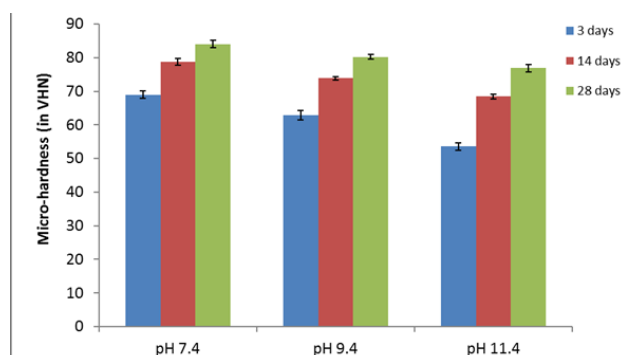


Figure 1. The results of the effect of alkaline environment on the microhardness of MTA in different setting time.

Discussion

Mineral trioxide aggregate has been shown to release soluble fractions, mainly calcium hydroxide, in both the short- and long-term sufficient to maintain the pH of the surrounding environment at a high level (pH. 11-12).⁷ MTA releases calcium ions as a result of hydration of calcium oxide, which is the main component of MTA.⁴ Rahimi et al.¹² recommended that MTA should be allowed to set untouched for 72 hours or longer to decrease the chance of MTA displacement. In the present study, the samples were initially measured for micro-hardness after 3-day of immersion, followed by 14-day and 28-day of immersion.

The micro-hardness of a material is not a measure of a single property. It is influenced substantially by other fundamental properties of the material such as yield strength, tensile strength, modulus of elasticity, and crystal structure stability. Therefore, it can be used as an indicator of the setting process and the overall strength or resistance to deformation when compared with baseline information. It can also reveal the effect of various setting conditions on the overall strength of a material.¹³

The results of the present study showed that either the alkaline environment or setting time affects the micro-hardness MTA. The alkaline environment adversely affects MTA, particularly micro-hardness of MTA, leading to the assumption that alkaline solution interferes with MTA, and may disturb the hydration process of MTA, which in turn lead to decrease the micro-hardness of MTA.¹⁴ Hydration process of MTA occurs rapidly at first, however with increasing time, the hydration process become slower. With increasing pH due to releasing of calcium hydroxide during hydration process of MTA as well as in the presence of an alkaline environment could destroy the gel of calcium silicate hydrate gel during the initial hydration process of the MTA.¹⁵ Furthermore, an alkaline environment influences the solubility of tricalcium aluminate, resulting in adversely influence of ettringite formation (calcium sulfoaluminate). Sulfate will react with excessive sodium resulting in formation of Na_2SO_4 , which then could disrupt the ettringite reaction.^{16,17} This phenomenon would reduce micro-hardness of MTA.

The results showed that regardless of the time setting, an alkaline environment with a pH of

9.4 produced greater micro-hardness than pH 11.4, but lower than pH 7.4 (a control group). This condition occurred probably due to pH 9.4, the amount of cement that is not hydrated decreases and the surface hardness of cement as an indicator setting process increases.¹⁸ Therefore, if the MTA exposed to an alkaline environment with a pH greater than 9.4, a greater adverse effect is likely to occur.

Vivan et al.¹⁵ reported that with increasing concentration of calcium hydroxide may induce the chemical shrinkage of cement and could change the micro-hardness of MTA. In addition, it has been found than an alkaline accelerator influences the early hydration of MTA. Nekoofar et al.¹⁰ revealed that calcium silicate gel breakdown during the hydration of MTA cement in the presence of high concentration of NaOH. Thus, due to that phenomenon, alkaline pH might change the physical properties of MTA.

The principal setting process of MTA is initiated on contact with water when a chemical reaction between water and cement commences, this is essentially a hydration reaction. The hydration reaction is primarily controlled by aluminates that are the first compounds to react with water and cause the flash setting of the cement. This stage is crucial to gain sufficient primary strength within the cement and is usually followed by hydration of the silicate phases. The hydrated calcium silicate phases that comprise 70-80% of the cement contribute the most to the binding power and strength of the material. During this process, fine hydrophilic particles react chemically with water and subsequently harden. Cement hydration connects the original cement particles together resulting in a colloidal gel that develops bonding properties and is responsible for hardening.^{19,20}

MTA setting process is divided into two stages. After being mixed with sterile water, the hydration reaction of tricalcium silicate and dicalcium silicate commences and generates gel containing calcium silicate hydrate, which contains a lot of calcium hydroxide. Calcium hydroxide will then react slowly with other minerals in the MTA powder to form other hydrate compounds. Calcium silicate contributes the greatest strength to the MTA. In addition, calcium silicate contributes to bond to calcium hydroxide crystals, which easily released from the gel. Tricalcium aluminate reacts quickly on hydration process, whereas tetracalcium-

auminoferit reacts more slowly than tricalcium aluminate.^{21,22} A variety of factors may influence setting reaction of MTA including the ratio of powder and liquid, temperature, humidity and pH environments, condensation pressure.²³

MTA setting process will occur slowly if there is no interference from the day 3 to 28, and with increasing time the hydration strength increases.¹⁹ This phenomenon also occurred in this present study that revealed micro-hardness increased with increasing time, and setting time of 28 days to produce the highest micro hardness.

Conclusions

Based on the results of this study it can be concluded that an alkaline environment influences micro-hardness of MTA with setting times of 3, 14 and 28 days. Alkaline environment with pH 9.4 and setting time of 28 days showed higher micro-hardness than pH 11.4 with setting time of 3 days.

In clinical situation, it is recommended that the use of MTA as apexification materials should not be used calcium hydroxide as intracanal medication since it could influence the micro-hardness which ultimately leading to failure of the treatment.

Declaration of Interest

The authors report no conflict of interest.

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