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PHYSICAL PROPERTIES OF A NEW MINERAL TRIOXIDE AGGREGATE  
MATERIAL

A Thesis submitted in partial fulfillment of the requirements for the degree of Master of Science at Virginia Commonwealth University.

by

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## Abstract

### PHYSICAL PROPERTIES OF A NEW MINERAL TRIOXIDE AGGREGATE MATERIAL

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A Thesis submitted in partial fulfillment of the requirements for the degree of Master of Science at Virginia Commonwealth University.

Virginia Commonwealth University, 2004

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The objective of this study was to compare the time to final set and compressive strength of the white mineral trioxide aggregate (MTA) formulation to the original grey MTA. To test compressive strength, each MTA formulation was placed into Teflon split molds for four hours at 37° Celsius (C) and 100% humidity. Compressive strength of both MTA formulations was measured at 24 hours (n=12) and 21 days (n=19) using an Instron Testing Machine. For determination of time to final set, each MTA formulation

(n=6) was placed into a metal mold and maintained at 37° C and 100% humidity while setting. At five-minute time intervals, an indenter needle was lowered onto the surface of the MTA material and allowed to remain in place for five seconds before it was removed from the specimen surface. This process was repeated until the needle failed to make a complete circular indentation in the MTA specimen. Results of a two-way ANOVA indicate that white MTA had a significantly higher compressive strength (mean=32.7 MPa) than grey MTA (mean=25.2 MPa) at 24 hours and no statistically significant differences at 21 days (white mean=38.6 MPa and grey mean=38.0 MPa). Using one-way ANOVA, results indicate that grey MTA had a significantly longer time to final setting time (mean=296 min) compared to white MTA (mean=276 min). Based on this study, the results suggest that white MTA is an effective substitute for grey MTA.

## Introduction

When nonsurgical root canal treatment has been unsuccessful or is contraindicated, surgical root canal treatment is often conducted to save teeth. This procedure routinely consists of root-end exposure of the involved apex, resection of its apical end, a retrofilling preparation and the placement of a root-end filling material. Various materials have been used as root-end filling materials including amalgam, Super-EBA, Intermediate Restorative Material (IRM), glass ionomers and composite resins (1). According to Gartner and Dorn (2), an ideal root-end filling material should prevent leakage of microorganisms into the periradicular tissues, be non-toxic, non-carcinogenic, biocompatible with host tissues, insoluble in tissue fluids and dimensionally stable.

Originally amalgam was the material of choice for root-end fillings, however, there are several disadvantages associated with amalgam including initial marginal leakage, secondary corrosion, moisture sensitivity and concerns over mercury toxicity. While Super-EBA and IRM have addressed some of the disadvantages concerning amalgam, these materials also have potential disadvantages including moisture sensitivity, irritation to vital tissues, solubility and difficulty in handling. While some investigators have shown glass ionomer cements (3, 4, 5) and composite resins (6,7,8) to

provide a better seal than amalgam, the issue of moisture contamination remains a valid concern with these materials. MacNeal and Beatty (9) demonstrated that the seal of two glass ionomers was adversely affected when the root-end cavities were contaminated with moisture at the time of material placement.

Mineral Trioxide Aggregate (MTA) was developed by Torbinejad et al. to address shortcomings of routinely used root-end filling materials. The principle components in MTA cement are tricalcium silicate, tricalcium aluminate, tricalcium oxide and silicate oxide (10). MTA is a powder consisting of hydrophilic particles that set in the presence of moisture (11). Hydration of the powder results in a colloidal gel that solidifies to a hard structure. Characteristics of set MTA depend on the size of the particles, the water-to-powder ratio, temperature and humidity at the application site and the amount of air trapped in the mixture (12).

Several studies of MTA have demonstrated that the material possesses many ideal properties. The sealing ability of MTA in root-end fillings was found to be superior to that of amalgam, IRM and Super-EBA using both dye (12, 13) and bacterial (14) leakage methods. When MTA was used as a root-end filling material in monkeys, results revealed no periradicular inflammation, new bone formation and the growth of cementum directly against the MTA material (15). In addition, MTA has been used as a capping material in mechanically exposed pulps (16), for root-end induction (17), repair of root

perforations (18, 19) and as a barrier during internal bleaching of endodontically treated teeth (20).

In 1995, Torbinejad et al. (11) presented research findings involving the physical and chemical properties of the original grey MTA formulation. The results of that study indicated that grey MTA had mean compressive strengths of 40.0 MPa and 67.3 MPa at 24 hours and 21 days respectively and a mean time to final set of 165 minutes. Recently a new formulation of MTA has been introduced to the dental materials armamentarium under the trade name ProRoot MTA (Tulsa Dentsply, Tulsa, OK). There appears to be little in the literature concerning the physical properties of this new formulation of MTA. The purpose of this study is to compare the time to final set and compressive strength of the new white MTA formulation to the original grey MTA and determine whether or not these two MTA formulations are equivalent with respect to the physical properties tested.

## Materials and Methods

The compressive strength of the two MTA formulations was determined according to a modified method of the American Dental Association (ADA) specification No. 30, section 7.3 (21). The instruments and test materials were conditioned at 23 +/- 1° C in a cabinet one hour prior to testing. A split mold apparatus, consisting of five individual specimen wells each measuring six mm in height and three mm in diameter, was fabricated from Teflon (Custom Design & Fabrication, Richmond, VA.). This split mold assembly was placed onto a 4 x 6 inch glass slab prior to the start of mixing. Each MTA formulation was mixed according to the manufacturer's instructions and placed into the individual specimen wells utilizing an amalgam carrier. The MTA material was condensed into each well using a large amalgam condenser. After removing any excess MTA material from the wells, a 4 x 6 inch glass slab was placed on top of the split mold assembly. Next, the entire assembly was placed into an oven maintained at 37 +/- 1° C and 100% humidity for four hours from the start of mixing. The specimens were then removed from the molds and examined for voids and chipped edges. Defective specimens were discarded. Twelve acceptable specimens were prepared for each MTA formulation to measure compressive strength at 24 hours and 19 specimens were prepared for measuring compressive strength at 21 days. The specimens were kept in

100% relative humidity prior to testing of their compressive strengths utilizing an Instron Testing Machine model TTC (Instron Corp., Canton, Mass. USA). Each MTA specimen was positioned with the flat ends between the plates of the apparatus, cushioned by a thin layer of tin foil, with care taken to ensure that the load was applied to the long axis of each test specimen. The maximum load required to fracture each specimen was recorded and the compressive strength (K) was calculated in Megapascals using the formula

$$K = 4F/\Pi d^2$$

where F is the maximum applied load in Newtons and d is the mean diameter of the specimen in millimeters (21).

The time to final set of the two MTA formulations was determined according to the method recommended by the International Organization for Standardization (ISO) No. 6876, section 7.4 (22). Before mixing, the test materials, mixing spatulas and glass slabs were placed in a cabinet with a maintained temperature of 23 +/- 1° C for 1 hour. Each MTA formulation was mixed according to the manufacturer's instructions and placed into a circular metal mold measuring ten mm in diameter and two mm in height. The assembly, comprising the mold and test material, was maintained at 37° C and relative humidity not less than 95%. At five-minute time intervals, an indenter needle one mm in diameter was lowered onto the surface of the MTA material and allowed to remain in place five seconds before it was removed from the specimen surface. This process was repeated until the needle failed to make a complete circular indentation in the

MTA specimen. The time to final set was recorded as the duration of time that elapsed from the start of mixing to when the indenter needle failed to make a complete circular indentation in the MTA material. This test was repeated six times for each of the MTA formulations.

## Results

The compressive strength results are shown in Table 1 and Figure 1. The compressive strength of the white MTA formulation at 24 hours was compared to the compressive strength of the white MTA at 21 days using a two-way ANOVA. The same was done for the grey MTA formulation. Since there was evidence of an interaction ( $F(1,58) = 2.89$ ,  $p\text{-value} = 0.0942$ ), the difference in compressive strengths between the two MTA formulations may not have been consistent across the two time periods. Therefore, the compressive strength of the formulations was compared separately within each time period. The results indicate that at 24 hours, the two MTA formulations were significantly different ( $F(1,58) = 5.67$ ,  $p\text{-value} = 0.0206$ ) and at 21 days the two formulations were not significantly different ( $F(1,58) < 1$ ,  $p\text{-value} = 0.7945$ ). This pattern is illustrated in Figure 2; the values plotted are shown in Table 2. As shown, the white MTA specimens have a compressive strength 7.5 MPa higher than the grey MTA specimens at 24 hours. However, at 21 days, the specimens differed by only 0.7 MPa.

The time to final set for each of the MTA formulations is shown in Table 3 and Figure 3. A one-way ANOVA was used to compare the time to final set for the two MTA formulations. All comparisons were made using two-tailed tests with  $\alpha = 0.05$  using JMP software (version 5.0.1, SAS Institute, Cary, NC). The results indicated the

grey specimens had significantly longer time to final set compared with the white specimens ( $t = 2.275$ ,  $df = 10$ ,  $p\text{-value} = 0.0462$ ).

Table 1. Average Compressive Strength

<b>Time</b>	<b>Color</b>	<b>Compressive Strength (MPa)</b>				
		<b>n</b>	<b>Mean</b>	<b>SD</b>	<b>Range</b>	
24 hrs	Grey	12	25.2	7.1	17.1	38.5
24 hrs	White	12	32.7	8.5	22.7	47.2
21 days	Grey	19	38.0	8.4	23.4	55.3
21 days	White	19	38.6	7.1	25.4	58.7

Table 2. Results of the Two-Way ANOVA

<b>Time</b>	<b>Color</b>	<b>Compressive Strength (MPa)</b>				
		<b>n</b>	<b>Mean</b>	<b>SE</b>	<b>95%CI</b>	
24 hrs	Grey	12	25.2	2.2	20.7	29.6
24 hrs	White	12	32.7	2.2	28.2	37.2
21 days	Grey	19	38.0	1.8	34.4	41.5
21 days	White	19	38.6	1.8	35.1	42.2

Table 3. Average Time to Final Set

<b>Color</b>	<b>Time To Final Set (Minutes)</b>				
	<b>n</b>	<b>Mean</b>	<b>SD</b>	<b>Range</b>	
Grey	6	296.2	18.9	277	330
White	6	275.7	11.4	255	287

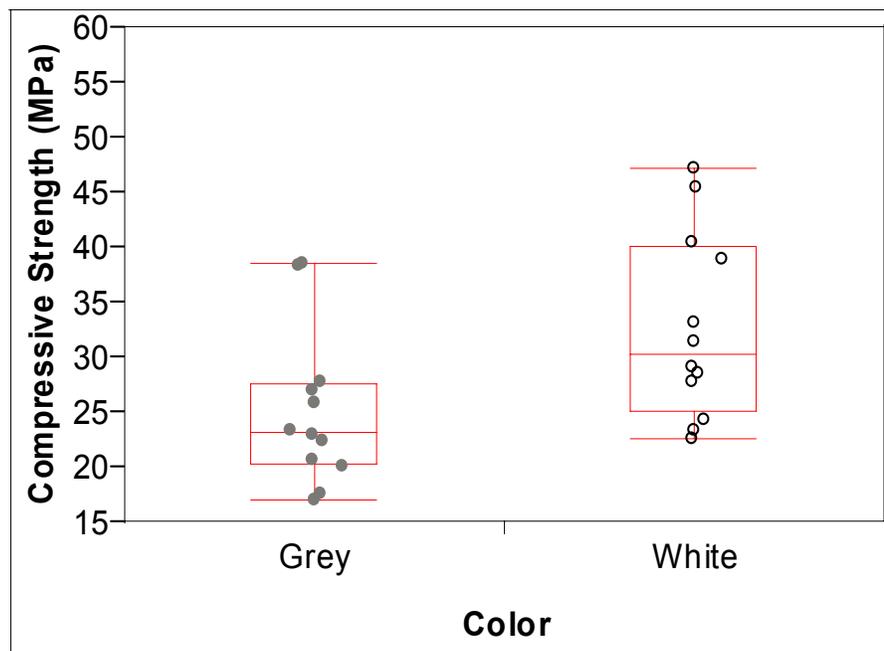


FIG 1. Compressive Strength at 24 Hours

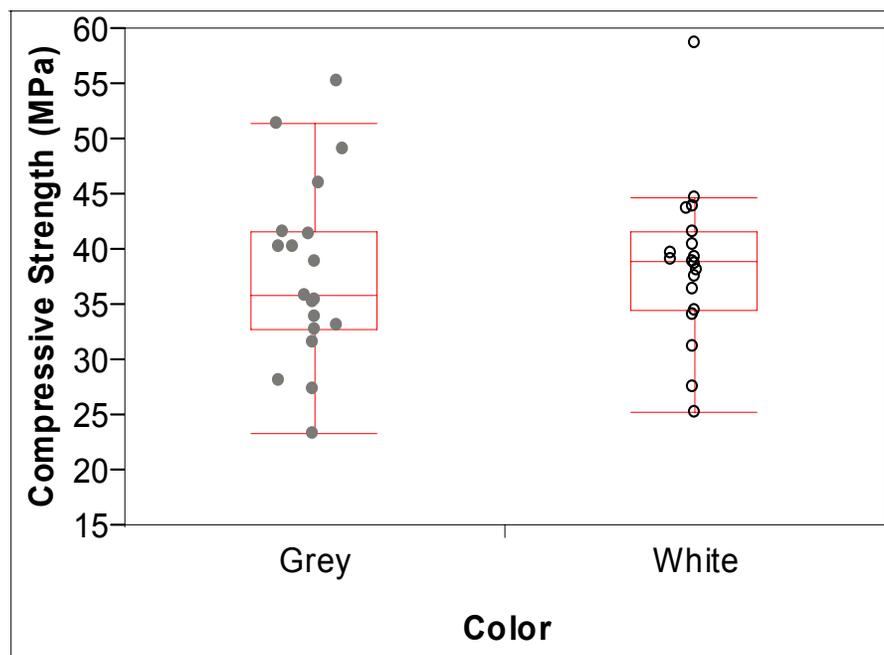


FIG 2. Compressive Strength at 21 Days

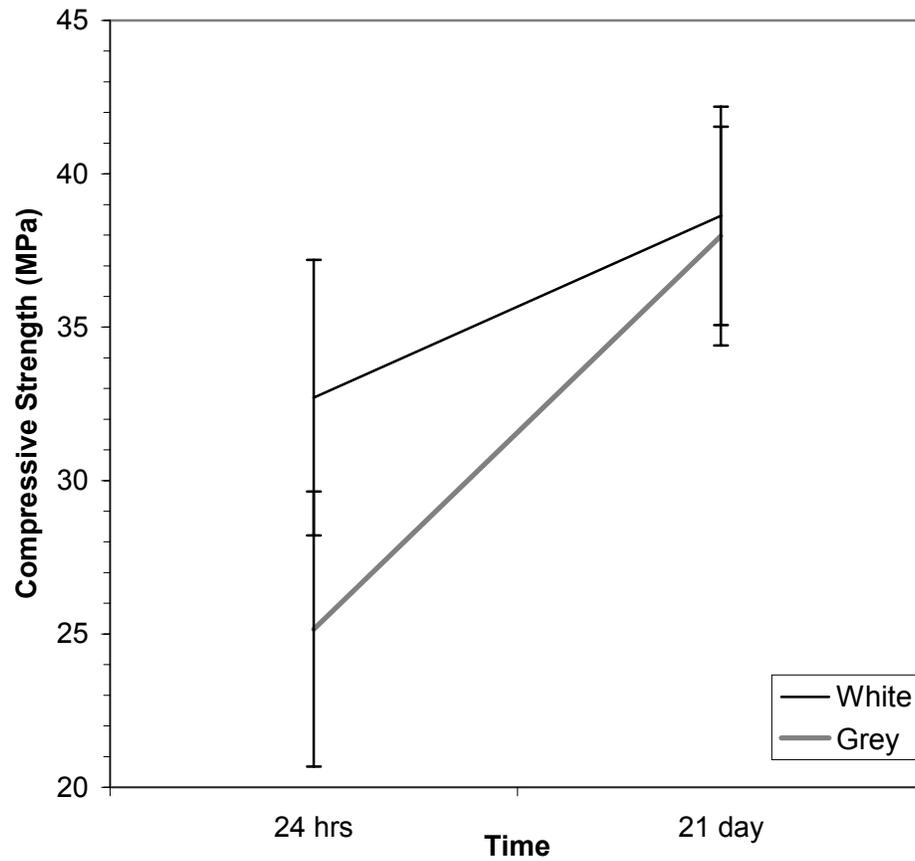


FIG 3. Compressive Strength (MPa) of white and grey MTA measured at 24 hrs and 21 days. Their least square means were measured in Megapascals (MPa) and vertical bars represent 95% CI.

## Discussion

The results of this study demonstrate that at 24 hours the mean compressive strengths of the two MTA formulations (white MTA mean = 32.7 MPa and grey MTA mean = 25.2 MPa) were significantly different while at 21 days the mean compressive strengths (white MTA mean = 38.6 MPa and grey MTA mean = 38.0 MPa) were not significantly different. The results of the time to final set for each MTA formulation indicated the grey MTA specimens had a significantly longer time to final set (grey MTA mean = 296 minutes) compared with the white MTA specimens (white MTA mean = 275 minutes). In their study, Torbinejad et al. (11) reported the mean compressive strength of grey MTA at 24 hours (mean = 40.0 MPa) and at 21 days (mean = 67.3 MPa) respectively. In the same study, Torbinejad et al. reported that the mean time to final set for grey MTA was 165 minutes.

The difference in the compressive strengths and time to final set between the current study and the Torbinejad study may be the result of several factors. First, the specimen mold sizes between the studies differed dramatically. For determining compressive strength, Torbinejad used stainless steel split molds measuring twelve mm in height and six mm in width while the current study used Teflon split molds measuring 6 mm in height and 3 mm in width. The decision to use smaller mold sizes in this study

was done in an effort to reduce material cost and to utilize the MTA material in dimensions more relevant to clinical practice. This difference in the specimen mold sizes between the two studies could have influenced the compressive strength reported in each study.

Secondly, the physical characteristics of MTA are influenced by several factors including the quantity of water used during mixing, the mixing procedure itself, pressure used for compaction, environment humidity and temperature (11). Some of these factors are not easy to control and, therefore, it is difficult to standardize methods used to determine properties of MTA. Any variation in mixing, handling and/or atmospheric environment experienced by the MTA material can strongly influence the overall physical characteristics of the material. More specifically, hand packing the MTA material into the mold apparatus most likely led to the incorporation of internal material voids not observed by the examiner during visual examination of the specimens. However, Aminoshariae et al. (23) recently demonstrated that hand placement of MTA into simulated root canals resulted in significantly fewer voids compared with an ultrasonic placement method. In hindsight, it would have been advantageous to radiograph the MTA specimens prior to compressive strength testing in an effort to identify and discard specimens having obvious internal voids. The presence of such internal voids could have lessened the compressive strength values reported in this study and the Torbinejad study. Again, any variation in the mixing and handling procedures or

environmental factors could influence the overall physical characteristics of the MTA material. Although this study as well as the Torbinejad study attempted to follow the methods prescribed by ADA Specification No. 30 (21) and ISO No. 6876 (22) for determination of the compressive strengths and time to final set, it would be naïve to think that there were no variances within or between the studies with respect to specimen preparation.

Another factor that may have significantly influenced the results of these studies involves the testing apparatus itself. For instance, although both studies utilized the Instron Testing Machine for determination of compressive strength, these testing devices were inevitably calibrated differently, leading to overall variances between the results of the two studies. In addition, the mass and tip diameter of the indenter needle used to determine time to final set was dramatically different between the two studies. The Torbinejad study used an indenter needle with a mass of 100 grams and a needle tip diameter of 2.0 mm. The current study used an indenter needle with a mass of 450 grams and a needle tip diameter of 1.0 mm. These differences in testing equipment for each study undoubtedly influenced the overall reported values for time to final set between the two studies. Nonetheless, the current study reported that the grey MTA formulation took significantly longer to set compared to the white MTA formulation.

Another difference between the current study and the Torbinejad study is the fact that the current study tested both the original grey MTA formulation and the newer white

MTA formulation while the Torbinejad study exclusively investigated the grey MTA formulation. This author decided that investigating both MTA formulations using the same testing parameters and comparing the results would be the most informative.

Based upon the results of this study, white MTA may be considered a reasonable substitute for grey MTA with respect to compressive strength and time to final set. Further research is warranted to determine if the two MTA formulations are equivalent based on other physical and chemical properties including chemical composition, pH and solubility. Results of additional studies may indicate if one of the two MTA formulations is superior to the other based upon chemical and physical properties tested.

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Literature Cited

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