



# Compressive Strength of Mineral Trioxide Aggregate with and without Disodium Hydrogen Phosphate at Different Mixing Ratios

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## ABSTRACT

**Introduction:** Mineral Trioxide Aggregate (MTA) is a substance with favorable physical-mechanical properties. Disodium hydrogen phosphate (DHP) is sometimes added to MTA to reduce its setting time. Therefore, this study was conducted to evaluate the effect of various ratios of liquid to powder of white MTA (WMTA) and addition of DHP on its compressive strength. **Methods and Materials:** One hundred and twenty samples were prepared with a two-piece stainless steel mold with a height of 6 mm and a diameter of 4 mm in order to evaluate the compressive strength where WMTA was used in 60 samples and DHP in white MTA composition (DHPWMTA) was used in other 60 samples. The compressive strength of WMTA and DHPWMTA was measured in various ratios of liquid to powder including 50, 60 and 70% and at 24 h and 21 days ( $n=10$ ). Univariate Analysis of Variance test with SPSS 16 software were used to determine the difference between groups. The level of significance was set at 0.05. **Results:** The maximum and minimum compressive strength of WMTA groups were  $63.25 \pm 1.96$  (50% ratio and 21 days) and  $37.79 \pm 1.28$  (70% ratio and 24 h), respectively. The maximum and minimum compressive strength of DHPWMTA groups were  $63.96 \pm 1.40$  (60% ratio and 21 days) and  $37.37 \pm 1.62$  (70% ratio and 24 h), respectively. The effect of each of factors (type of material, powder to liquid ratio and time) alone were significant on the compressive strength ( $P < 0.05$ ). However, the interactive effect of three factors (type of material, powder to liquid ratio and time) were not statistically significant on compressive strength ( $P > 0.05$ ). **Conclusion:** Adding 2.5 wt% of DHP to white MTA increased samples compressive strength. Compressive strength in liquid to powder ratios of 50 and 60% compare to 70% and at 21 days compared to 24 h was high.

**Keywords:** Compressive Strength; Disodium Hydrogen Phosphate; Mineral Trioxide Aggregate

## Introduction

Mineral Trioxide Aggregate (MTA) is a calcium silicate based biomaterial and is widely used in endodontics due to its favorable properties, including sealing ability, setting capacity in the presence of blood/moisture, biocompatibility, and the ability to induce regeneration in the pulp and periapical tissues [1-3]. According to studies, setting time of MTA is more than 2 h [4]. Various materials like chlorhexidine, NaOCl gel and  $\text{CaCl}_2$

have been added to the MTA to reduce this time but these materials have also reduced the compressive strength of MTA [5]. Compressive strength and an increase in the strength of a given biomaterial over time is an indicator of setting reaction and stability of the material [6]. On the other hand, compressive strength is one of the most important factors in dental cavity filling materials especially when teeth are under occlusal forces such as direct pulp capping, vital pulp therapy, closure of lateral perforation of the roots and orifice plug barrier [7-15].

It was shown in a study that disodium hydrogen phosphate (DHP) solution reduces setting time of MTA as an accelerator and maintains its pH at the same level [16, 17]. Microleakage studies show that sealing power in MTA mixed with DHP is similar to MTA [18]. The study of tissue reaction in subcutaneous connective tissue of rat also showed that adding 2.5 wt% DHP to white MTA increases the biocompatibility of white MTA [19, 20]. Based on Ames test, Samiei *et al.* [1] concluded that MTA with disodium hydrogen phosphate was biocompatible in relation to mutagenicity.

In MTA, hydration of the powder leads to the formation of colloidal gel and its hardening leads to the formation of hard tissue. The hardened MTA properties depend on the size of particles, ratio of powder to liquid, temperature and humidity of used area and amount of air entrapped in the mixture [21, 22]. Special structures of micropores and capillary canals are formed in which water is constituted when Portland cement is mixed with water. Porosity of solution increases when water ratio increases. The amount of water content in the mixture will be a decisive factor in the material's properties including compressive strength [21]. According to a research carried out in valid scientific sources, there has been no study until now evaluating the effect of different liquid to powder ratios of MTA and the effect of adding DHP on compressive strength of MTA; therefore, this study was designed to evaluate the effect of changes in various ratios of liquid to powder of white MTA and addition of DHP on its compressive strength.

## Materials and Methods

One hundred and twenty samples (10 samples for each group) were used for measurement of compressive strength. The studied groups were as follows: 1 g mixture of White MTA (Angelus, Londrina, PR, Brazil)+0.50 mL of liquid, 1 g mixture of MTA+0.60 mL of liquid, 1 g mixture of MTA+0.70 mL of

liquid, 1 g mixture of 2.5 wt% of White MTA (with DHP)+0.50 mL of liquid, 1 g mixture of 2.5 wt% of White MTA (with DHP)+0.60 mL of liquid and 1 g mixture of 2.5 wt% of White MTA (with DHP)+0.70 mL of liquid.

The groups consisted of 6 main groups, with half of each group used to measure compressive strength after 24 h and another half to measure compressive strength after 21 d (12 groups with 10 samples in each).

Tested substances, molds, spatula and glass slides were kept at room temperature for 24 h before mixing. The compressive strength of samples was also calculated according to the British standard for dental glass ionomer cements BS6039: 1981 (British Standard Institution) [23] where there are only changes in the size of molds in such a way that two-piece stainless steel molds with holes and with a height of 6 mm and a diameter of 4 mm were provided [24, 25]. In each group, the material was compressed into the holes after mixing with the condenser and transferred to a 37-degree incubator with 100% moisture content within 3 min after the start of mixing. Then samples were removed from the molds and were checked for void and chipped edge and defective items were set aside. Subsequently, half of the samples from each group were measured after 24 h and the other half were measured after 21 days by the Hounsfield apparatus (Hounsfield test equipment, Model H5KS, UK).

The maximum force required to break each sample was measured and recorded and compressive strength was expressed in MPa as follows:  $C=4P/\pi D^2$  where P is the maximum force applied in Newton and D is the mean sample size in millimeters [26].

## Statistical analyses

Univariate Analysis of Variance test was used to determine the difference between groups. The level of significance was set at 0.05.

**Table 1.** Descriptive statistics of compressive strength of samples based on the type of substance, liquid to powder ratios and time of assessment

Material	Ratio	Time	Mean (SD)	95% Confidence Interval	
				Lower Bound	Upper Bound
WMTA	50%	24 h	42.15 (1.50)	41.16	43.14
		21 d	63.25 (1.96)	62.26	64.23
	60%	24 h	41.33 (2.09)	40.35	42.32
		21 d	59.51 (1.50)	58.52	60.49
	70%	24 h	37.79 (1.28)	36.80	38.78
		21 d	57.27 (1.26)	56.28	58.25
DHPWMTA	50%	24 h	41.44 (1.77)	40.45	42.43
		21 d	62.39 (1.39)	61.40	63.38
	60%	24 h	43.72 (1.78)	42.73	44.71
		21 d	63.96 (1.40)	62.97	64.95
	70%	24 h	37.37 (1.62)	36.38	38.35
		21 d	59.78 (1.03)	58.79	60.77

## Results

The results of the descriptive statistics based on the type of substance, liquid to powder ratios and times of assessment are presented in Table 1. The maximum and minimum compressive strength of WMTA groups were  $63.25 \pm 1.96$  (50% ratio and 21 d) and  $37.79 \pm 1.28$  (70% ratio and 24 h), respectively. The maximum and minimum compressive strength of DHPWMTA groups were  $63.96 \pm 1.40$  (60% ratio and 21 d) and  $37.37 \pm 1.62$  (70% ratio and 24 h), respectively.

Moreover, the results showed that the effect of each of factors (type of material, powder to liquid ratio and time) alone on the compressive strength were significant ( $P < 0.05$ ); however, the interactive effect of three factors (type of material, powder to liquid ratio and time) were not statistically significant on compressive strength ( $P > 0.05$ ). The compressive strength of the DHP mixture to white MTA (DHPWMTA) group was significantly higher than the WMTA ( $P < 0.05$ ). Also, compressive strength after 21 d was significantly greater than 24 h ( $P < 0.05$ ). Based on the LSD post hoc test, the powder to liquid ratio (50% and 60%) were not significantly different ( $P > 0.05$ ), but their difference were statistically significant with the powder to liquid ratio of 70% ( $P < 0.05$ ).

## Discussion

Based on the results of this study, it was found that adding 2.5 wt% of DHPWMTA significantly increase the level of strength. Also, results showed a significant difference between groups in terms of powder/liquid ratio. According to the findings of this study, the groups of 50 and 60% did not have a significant difference, but the group of 70% had a significant difference between the two groups of 50 and 60%. Also, analysis of the results showed that compressive strength was different for 24 h and 21 d and this difference is statistically significant.

MTA is a hydraulic cement composed mainly of dicalcium silicate and tricalcium silicate which produces calcium silicate hydrate gel (CSH) and calcium hydroxide [27] during the hydration process. Setting and strength of the hydraulic cement depend on the formulation of CSH gel and ettringite (hydrous calcium aluminium sulfate mineral) in the germination sites of calcium hydroxide crystals [28].

In addition to the fact that diverse clinical applications of MTA such as the repair of furcation perforation and pulp capping make it necessary for MTA to have sufficient compressive strength, compressive strength is also an indicator for hydration and setting [21].

The aim of the present study was to mix MTA with liquid to powder ratios of 3 to 1, 4 to 1 and 5 to 1; but according to a pilot study, more fluid is required for mixing and consequently, ratios were changed proportional to the desired consistency. The reason for this can be due to type of MTA used in this study (Angelus MTA) which contains 80% Portland cement and 20% bismuth oxide and has no gypsum sulfate and has smaller particle size.

According to ISO 6876 [23], use of a stainless steel mold or a material that is not affected by the cement is recommended for the determination of compressive strength. Various methods have been used to create cylindrical samples of MTA for determination of compressive strength.

One-piece cylindrical molds of PTFE (Polytetrafluoroethylene) were used in the study by Nokofar *et al.* [29]. One-piece plastic molds were used in a study by Kagon *et al.* [5], and one-piece polycarbonate molds were used in another study by Nekofer *et al.* [30]. Two-piece plastic molds were used in a study by Holt *et al.* [31] and two-piece stainless steels were used in the study of Kayahan *et al.* [32]. Given that the MTA is expanded during hydration, Holt *et al.* [31] reported that it is not possible to remove samples from the mold without applying too much force which can lead to damage of samples before testing compressive strength. Therefore, they proposed to use two-piece molds so that less force is used to remove samples. Two-piece stainless steel molds were also used in the present study. Various methods have been used in studies for placing and compressing MTA within the molds. In a study by Nekofer *et al.* [24], an ultrasonic device was used for 30 sec with a moderate strength (5 out of 10) in such a way that the tip of ultrasonic was moved in the MTA mixture without having contact with the walls of the mold. However, Aminoshariae *et al.* [33] performed comparisons between manual placement and ultrasonic placement of MTA in the polyethylene tubes. In that study, samples which were manually compressed with plugger had better matching with tube walls and also showed less voids compared to ultrasonic method. In the present study, MTA was compressed inside molds manually using plugger.

This result confirmed the results of a study by Torabinejad *et al.* [2]. It was determined based on another study which evaluated the effect of adding accelerator where shear strength of MTA with DSP increased as time passes and this increase is significant in the periods studied which is in accordance with the current study [9]. Of course, it should be noted that 2.5 wt% of white MTA with DHP was used in the present study, but percentages of 5, 10 and 15 were used in the study by Huang *et al.* [9].

Prasad *et al.* [26] also evaluated the effect of adding different modifiers on the physical properties of MTA in the 1, 3, and 7-day intervals and concluded that firstly, the compressive strength of white MTA with 15% DHP increased over time and secondly, despite the low compressive strength compared to the control group (white MTA with water), this decrease is not significant.

The reason for this increase with time can be attributed to the MTA compound; as indicated, MTA consists primarily of dicalcium silicate and tricalcium silicate and bismuth oxide. Given that the hydration rate of dicalcium silicate is slower than tricalcium silicate [34], compressive strength of MTA increases over time and reaches its maximum by several days after mixing it.

## Conclusion

This *in vitro* study showed that adding 2.5 wt% of DHP mixture to white MTA increased WMTA's compressive strength and this difference is significant in the ratio of liquid to powder. DHPWMTA's compressive strength in liquid to powder ratios of 50 and 60% is similar to WMTA.

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