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Comparison of Mechanical and Physical Properties of White and Gray Mineral Trioxide Aggregate Useable in Dentistry

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Abstract

Objectives: Currently, different materials are used to obdurate tooth root canals. An ideal root canal filling material should have some specific properties, including biocompatibility, dimensional stability and insolubility.

Materials and Methods: One of the methods to evaluate these materials is to carry out solubility and microhardness tests. To this end, 20 mineral trioxide aggregate (MTA) samples were prepared for solubility test and 60 samples for microhardness test. MTA (Angelus, Brazil) was prepared based on manufacturer's instructions by mixing the powder and catalyst at a ratio of 1:1 and was placed in Teflon molds. The samples were stored in a room with 95% relative humidity at 37°C for 390 minutes (3 times longer than the setting time). The solubility of the materials was tested by immersing the samples in distilled water and measuring weight loss at 24-hour and 7-day intervals. The microhardness was determined using Vickers test before and after immersion in Ringer's solution at different intervals and the results were compared.

Results: The results showed that solubility of white MTA after 1 day was similar to that of gray MTA; however, after 7 days, its solubility increased. There were no significant differences in microhardness on day 1 and on the subsequent days between white and gray MTA samples ($P<0.05$).

Conclusions: There was no significant difference in the solubility of white and gray MTA samples at 24-hour interval; however, at 7-day interval, the solubility of white MTA (WMTA) was significantly higher than that of grey MTA (GMTA) (with the solubility of both materials being <3%) In addition, there was no significant difference in microhardness of white and gray MTA between the first day and other intervals ($P<0.05$).

Keywords: MTA, Microhardness, Solubility, Ringer's solution.

Introduction

Mineral trioxide aggregate (MTA) is widely used as a biomaterial and is an ideal bioactive agent for the obturation of the root canal system in dentistry. It is a mixture of Portland cement and bismuth oxide (1).

Microanalysis of MTA has shown that its chief ion is calcium. MTA yields hydroxyapatite when it is mixed with interstitial fluid, which is the main component of bone and has proper compatibility with the body tissues. MTA has various applications in the repair of the root and bone in endodontics. Two commercial types of MTA are available, referred to as white and gray ProRoot MTA (2).

Asgary et al showed that the main differences in the ingredients between gray and white MTA were differences in aluminum oxide (Al2O3), magnesium oxide (MgO) and in particular ferrous oxide (FeO). The amounts of Al2O3, MgO and FeO in gray MTA were reported to be approximately 122%, 130% and 100% higher than those in white MTA, respectively (3).

One of the physical properties of root filling materials is their solubility; a material with very low solubility is an ideal material (4-6).

Destruction and disintegration of these materials result in gap formation between the material and the root canal wall, giving rise to an increase in bacterial microleakage over time.

Another important property of such materials is their microhardness which indicates strength and resistance of the material to deformation. This parameter is affected by different factors such as yield strength, tensile strength, elastic modulus, the moisture of the environment and crystalline structure stability of the materials (7,8).

When the MTA powder is mixed with water, a special structure of micro-canals is formed. The majority of these structures are eliminated during the setting reaction and finally, the set cement will consist of a series of pores and micro-canals. Penetration of water results in better hydration of the powder and optimal formation of the

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crystalline structure of the material as a crisscross pattern and a set of needle-shaped scalloped structures (9).

Differences in how the surface crystals of MTA are formed and their content might explain the differences in cellular response to this material (10).

Fridland and Rosado reported that the mixture of MTA loses its consistency in the presence of copious amounts of fluid; in addition, the long setting time results in the initial loosening of MTA in the presence of fluids (11).

In addition, Sheikhrezaie et al evaluated and compared the microhardness of two types of white and gray MTA after contamination with blood. The white and gray MTA samples (W and G, respectively) were placed in 3 groups as follows: no contamination (NC), contamination of material surface with blood (BC) and mixture of material with blood (MB). The samples in the NC group were prepared according to manufacturer's instructions. In the BC group, after preparation the samples according to manufacturer's instructions, their surface was placed in contact with blood for 4 days. In the MB group, the powder was mixed with blood instead of distilled water. Evaluation of the effect of contamination with blood on the materials properties showed significant differences between the NC, BC and M groups and both white and gray MTA ($P<0.001$). The differences between white and gray MTA samples in the NC groups ($P<0.001$) and the WBC and GBC groups were significant ($P=0.043$) (12).

Regarding solubility, Faria-Júnior et al evaluated the antimicrobial activity, pH and solubility of 7 different sealer types at 2- and 7-day intervals. At 2-day interval, the highest solubility was related to Active GP, Sealapex, MTA-S and MTA-F sealers; at 7-day interval, it was recorded with MTA-S and MTA-F sealers. The results showed that Sealapex and MTA-F sealers were associated with the least bacterial counts and highest solubility and pH; in this context, higher solubility and pH might explain the antimicrobial effects of these materials (13). In addition, in a study by Vitti et al, MTA Fillapex sealer exhibited less solubility compared to AH Plus sealer. The flow of MTA Fillapex was consistent with ISO 6876:2001 guidelines (<3%). The solubility of sealers results in the release of calcium components of the sealer, possibly irritating the periapical tissues (14).

In addition, in a study by Amoroso et al and Álvaro et al, MTA Sealapex exhibited higher solubility compared to AH Plus sealer (15).

Finally, in a recent study, evaluation of the properties of different sealers, including MTA, showed that Vickers microhardness for Biodentine (62.35 ± 11.55 HV) was significantly higher than that of MTA (26.93 ± 4.66 HV). The solubility of both sealers was less than the ISO 6875 values. The solubility of Biodentine was significantly higher than that of MTA. The radiopacity of Biodentine was significantly higher than that of MTA and the setting time of Biodentine was significantly lower than that of MTA (16).

Only a limited number of studies are available on the comparison of the properties of white and gray MTA. The aim of the present study was to compare the solubility and microhardness of gray and white MTA.

Materials and Methods

In the present study, the solubility and microhardness of white and gray MTA were evaluated as two important physical and mechanical properties. To this end, MTA manufactured by Angelus (Brazil) was used.

In order to evaluate solubility, 10 samples were prepared in each white and gray MTA group, i.e. a total of 20 samples. The powder and catalyst of the materials were mixed, according to manufacturer's instructions, at a ratio of 1:1 using a metallic spatula on a glass slab, measuring 26×76 mm with a thickness of 1–3 mm, which was covered with a layer of cellophane. The resultant mixtures were placed within Teflon molds in which a nylon thread had been placed for suspending the sample. The molds were then placed in closed containers and a wet cotton pellet was placed in each mold. The complexes consisting of the glass slab, Teflon mold and cellophane layer were stored in a room with 95% relative humidity at 37°C for 390 minutes, which is 3 times longer than the setting time of the material based on the manufacturer's brochure. Then the samples were retrieved from the Teflon molds and weighed with a weighing machine with an accuracy of 0.0001 g. Next, the samples were stored at 23 ± 2°C and a relative humidity of 50 ± 5% in distilled water as a solvent. The weight loss of samples was determined at 24-hour and 7-day intervals using an accurate weighing machine, according to ADA/ANSI specification #57.

In addition, in order to evaluate microhardness, a total of 60 samples were prepared (30 samples for white MTA and 30 samples for gray MTA). The powder and liquid were mixed at a ratio of 1:1 according to manufacturer's instructions and placed within cylindrical plastic molds, measuring 15 mm in diameter and 5 mm in thickness, followed by condensation under a pressure of 3.22 MPa for 1 minute. The samples were stored for 48 hours at room temperature in an open space. Then the samples were divided into two groups as follows: control and Ringer solution. First, the microhardness of all the samples was determined by Vickers test machine. Then the samples were immersed in 250 mL of Ringer's solution (0.86 g of sodium chloride, 0.030 g of potassium chloride, 0.0033 g of calcium chloride, 2H2O) to simulate the oral cavity conditions, and were transferred into an incubator at 37 ± 0.2°C. The samples were retrieved from the incubator at 24-hour, 72-hour and 7-day intervals and kept at room temperature for 48 hours in open space. The surfaces of all the samples in both groups were thoroughly polished and made smooth with abrasive paper. The samples underwent a microhardness test in a Vickers test machine (UHL VMTH AUTO, Germany), which was calibrated using a metallic sample. The microhardness was determined at

3 points on each sample with a 200-Newton force, using a pyramid-shaped indenter with a square base at a 136° angle (Figure 1).

Statistical Analysis

Data were analyzed using SPSS version 20.0.

Results

Solubility Test

The results of independent t-test showed that the mean initial weight of the sample was 0.2219 ± 0.0445 g. The solubility of white and gray MTA after 24 hours of immersion in distilled water was 0.16% and 0.19%, respectively, with no significant difference between these two materials ($P > 0.05$). However, a solubility rate of <3% has been recommended for these two materials based on ISO 6876:2001 and ADA/ANSI specifications. Furthermore, the solubility of white and gray MTA 7 days after immersion in distilled water was 16.32% and 11.33%, respectively, with a significant difference ($P < 0.05$); however, a solubility rate of <3% has been recommended for these two materials based on ISO 6876:2001 and ADA/ANSI specifications (Figure 2 and Table 1).

Microhardness Test

The results of independent t-test showed that the microhardness values of white and gray MTA on the first day were 53.7 and 43.6, respectively, with a significantly higher microhardness of white MTA (WMTA) compared to grey MTA (GMTA) ($P < 0.05$).

At 24-hour interval, the mean microhardness values of white and gray MTA were 45.7 and 45.6, respectively, with no significant difference between the two groups ($P < 0.05$).

At 72-hour interval, the mean microhardness values of white and gray MTA were 51.9 and 47.6, respectively, with a significantly higher microhardness of WMTA compared to GMTA ($P < 0.05$).

At 7-day interval, the mean microhardness values of white and gray MTA were 51.7 and 48, respectively, with a significantly higher microhardness of WMTA compared to GMTA ($P < 0.05$) (Figure 3 and Table 2).

According to Table 3, there was no significant difference in microhardness of white MTA between the first day and other intervals ($P < 0.05$). In addition, there was no significant difference in microhardness of gray MTA between the first day and other intervals ($P < 0.05$).

Discussion

Vitti et al reported that the solubility of MTA sealer was in the standard range specified by ISO 6776:2001 (<3%) (14). In addition, Faria-Júnior et al showed that the solubility of the 2 different types of MTA was different (13). However, Shahi et al reported that the microleakage of white and gray MTA was the same (17). A higher microleakage might be explained by a higher solubility; the results of these two studies are consistent with those of the present study. The major differences between white and gray MTA are

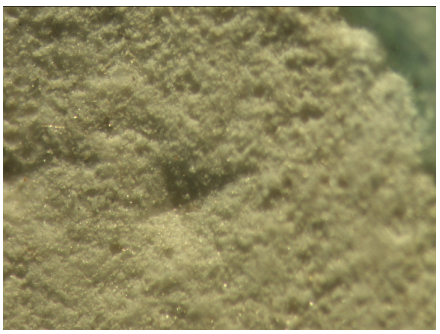

Figure 1. The Effect of the Indenter on a Gray MTA Sample Under a Microscope.

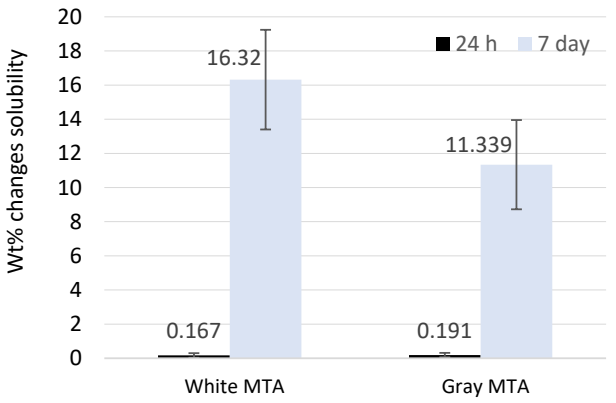

Figure 2. Comparison of the Solubility of White and Gray MTA in Terms of Weight Changes.

Table 1. Comparison of the Solubility of White and Gray MTA in Terms of Weight Changes

Time Interval	MTA	Initial Weight	The Weight Difference	The Weight Difference Percentage (Initial)	Standard Value* (a Minimum of 3% in 24 h)	*P* Value
		Mean ± SD	Mean ± SD			
24 h	White	0.22156 ± 0.04458	0.00034 ± 0.000237	0.1666 ± 0.1325	Less than the standard	0.681
	Gray	0.21347 ± 0.0479	.00041 ± 0.00027	0.1905 ± 0.1238	Less than the standard	
7 days	White	0.1863± 0.0421	.03519 ± 0.00396	16.3255± 2.9195	Less than the standard	0.001
	Gray	0.1896± 0.0435	0.02387 ± 0.00685	11.3394± 2.6137	Less than the standard	

P value of independent *t* test.
*Standard value based on ISO 6876:2001 and ADA/ANSI specifications.

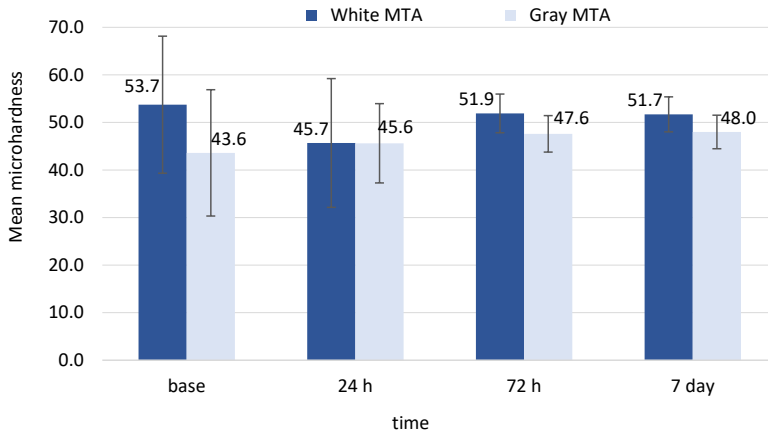

Figure 3. Comparison of Microhardness of White and Gray MTA at 4-Time Intervals.

the lower contents of aluminum, magnesium and ferrous oxides in white MTA. White MTA has smaller particles compared to gray MTA; therefore, a decrease in grit size during abrasion of the surface results in an increase in its mechanical strength, which is evident in the case of white MTA, and might explain its higher microhardness. Sheikhrezaie et al showed a significant difference between white and gray MTA regarding microhardness, which is consistent with the results of the present study. Surface analysis of white MTA and gray MTA showed that gray MTA crystals are 8 times larger than those of white MTA (12).

Table 2. Comparison of Microhardness of White and Gray MTA at 4-Time Intervals

		Mean	Independent *T* Test		
			T Value	*df*	*P* Value
Day 1	White MTA	53.73± 14.41	-2.19	18	0.032
	Gray MTA	43.6 ± 13.28			
24 hours	White MTA	45.68 ± 13.53	0.014	18	0.989
	Gray MTA	45.61 ± 8.33			
72 hours	White MTA	51.9 ± 4.06	2.43	18	0.026
	Gray MTA	47.6 ± 3.8			
7 days	White MTA	51.7 ± 3.6	2.29	18	0.034
	Gray MTA	48 ± 3.52			

Table 3. Two-by-Two Comparison of the Intervals in Terms of Microhardness of White and Gray MTA

	Interval 1	Interval 2	Mean Difference (I-J)	*P* Value
White MTA	Day 1	24 hours	8.05	0.09
	Day 1	72 hours	1.83	0.69
	Day 1	7 days	2.03	0.66
	24 hours	72 hours	-6.21	0.18
	24 hours	7 days	-6.01	0.20
	72 hours	7 days	0.20	0.97
Gray MTA	Day 1	24 hours	-2.014	0.59
	Day 1	72 hours	-3.998	0.29
	Day 1	7 days	-4.398	0.24
	24 hours	72 hours	-1.984	0.59
	24 hours	7 days	-2.384	0.52
	72 hours	7 days	-0.4	0.91

Kaup et al reported that the microhardness of MTA was 26.93 ± 4.66HV (18), which is different from the results of the present study. However, they did not report which MTA they tested (18). Matt et al showed no significant difference in the hardness between these two materials (19). In addition, to avoid mistakes and incorrect interpretation during the comparison of the results of different studies, attention should be paid to the duration of the study and the evaluation intervals because the solubility of sealers might change during the study period and exhibit increases or decreases. Absorption of water by the sealer over time, too, might compensate the decrease in weight due to solubility. In such cases, determination of the type of materials released by atomic absorption spectrophotometry can determine the true solubility of the material. In the present study, microhardness of white MTA was significantly higher than that of gray MTA at 4 evaluation intervals (the first day and 24-hour, 72-hour and 7-day intervals after preparation and immersion in a solution). During these 7 days, the microhardness of white MTA decreased from 53.7 on the first day to 45.7 at 24-hour interval, reaching 51.7 on the 7th day. The microhardness of gray MTA, too, increased from 43.6 on the first day to 48.0 (on the seventh day). Microleakage of a material shows the setting reaction of the material and indicates its strength and general resistance against deformation and stability of its crystalline structure. During the evaluation of microhardness, factors affecting microhardness should be taken into account. Microhardness of MTA might be affected by environmental pH, the thickness of the material, the force applied, the amount of air trapped within the material and moisture. Contact with acidic materials has a deleterious effect on the hardness of GMTA and WMTA. MTA has cuboid and needle-shaped crystals during hydration. The needle-shaped crystals grow between the cuboid crystals and are absent in an acidic environment; therefore, a decrease in the hardness might be attributed to the absence of needle-shaped crystals. Low moisture, low pH and high pressure might exert negative effects on the hardness of MTA (20).

Conclusions

It was concluded from the results of the present study that there was no significant difference in the solubility of white and gray MTA at 24-hour interval; however, at 7-day interval, the solubility of WMTA was significantly higher than that of GMTA (with the solubility of both materials being <3%). In addition, there was no significant difference in microhardness of white and gray MTA between the first day and other intervals ($P < 0.05$).

Conflict of Interests

None to be declared.

Ethical Issues

There were no ethical issues since the study was carried out in vitro.

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