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Comparative evaluation of the effect of carbonic acid as a solvent on the microhardness of mineral trioxide aggregate, Biodentine, and root dentin: An *in vitro* study

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Purpose: Mineral trioxide aggregate (MTA) has been shown a potential endodontic material in several clinical applications. Certain inherent disadvantages of MTA led to the introduction of Biodentine (BD). The retrieval of MTA and BD has been challenging for most clinicians. This study aimed to compare and evaluate the effect of carbonic acid (CA) on the microhardness of MTA, BD, and root dentin (RD).

Methods: Uniform MTA, BD, and RD discs were prepared under standardized conditions following the manufacturer's instructions. The preliminary microhardness of all the samples was tested after 24 h. All samples were soaked in CA for 5 min, and the microhardness was tested. The results were statistically analyzed using paired sample t-test.

Results: BD showed no statistically significant difference, whereas MTA and RD showed statistically significant differences in microhardness before and after exposure to CA.

Conclusion: CA can dissolve set MTA, but cannot dissolve BD. Nevertheless, CA, when used to remove set MTA, shows a detrimental effect on RD within 5 min of its exposure. Therefore, it is not clinically recommended to use CA for the retrieval of set MTA.

Keywords: Biodentine, carbonic acid, microhardness, mineral trioxide aggregate, root dentin.

Introduction

Since its introduction in 1993 by Torabinejad et al., mineral trioxide aggregate (MTA) has been shown potential as an endodontic material in several clinical applications. It can be used in surgical and nonsurgical applications, such as direct pulp capping, perforation repairs, apexification, root-end filling, and root canal filling. The main advantages of this material are its ability to set in the presence of moisture (1) and its cementogenic/dentinogenic properties (2). Despite the superior characteristics of MTA, the inherent disadvantages of the material are its prolonged setting time (3) and its inability to be retrieved from the root canal system (4).

Retrieval is a prerequisite for any root canal filling material so that it is possible to re-treat in case of endodontic failures (5). MTA sets into a hard mass, thus making its retrievability tough, and can pose significant procedural problems during re-treatment (6). The use of ultrasonic

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instruments to remove the hard pastes is effective to a certain extent (7), but complications such as instrument separation and limitations of its usage in curved canals have to be considered. Boutsioukis et al. (4) studied the retrievability of MTA from the root canal using a rotary and ultrasonic instrument and found it irretrievable. Another calcium silicate-based material, Biodentine (BD) (Septodont, Saint-Maur-des-Fossés, France), has been developed to overcome the shortcomings of MTA (8). The presence of a setting accelerator in BD results in a faster setting, thereby improving its handling properties and strength (9). Carbonic acid (CA) can be effectively used as an adjunct to dissolve set white MTA (WMTA) (10). The efficacy of CA in the retrievability of MTA has been well evaluated in the endodontic literature (11,12). However, no such study has been conducted to test the efficacy of CA on the retrievability of BD in the literature so far. Additionally, CA used for MTA retrieval can pose potential damage to the tooth structure, resulting in the danger of weakening the tooth structure, thus reducing the fracture resistance of the tooth. The null hypothesis postulates that CA, when used for retrieval, does not affect the dissolution of MTA, BD, and dentin disc. Therefore, the aim of this *in vitro* study was to compare and evaluate the effect of CA in the microhardness of BD, MTA, and root dentin (RD).

Materials and Methods

Materials used were WMTA (Angelus-WMTA, Brazil), BD (Septodont, Saint Maur des Fosses, France), CA, and freshly extracted teeth. The number of samples assessed for each group was 13, which was adequate for a power of 80%.

Discs, each of 7 mm diameter and 3 mm depth, of WMTA and BD were prepared under standardized conditions using silicon molds. WMTA powder and BD were mixed according to the manufacturer's instruction, a thick creamy consistency, and immediately condensed into molds of 3 mm depth. A moist cotton pellet was kept on the condensed WMTA and BD for 24 h. All the specimens were examined under the optical microscope, and those found to have cracks, defects, or gaps were excluded from the study. Thirteen discs fulfilling these criteria were included in the study.

Single-rooted premolars with relatively similar dimensions and morphology with closed apices extracted for orthodontic reasons were collected from adult patients. Teeth with previous root caries, cracks, curved canals, endodontic treatment, internal resorption, or calcification were excluded. Teeth were thoroughly cleaned from soft tissue or calculus deposition, and then stored in distilled water. Selected 13 teeth were sectioned transversely with the help of a diamond disc in a micromotor straight handpiece using water spray as a coolant to obtain a tooth slice of 5 mm height. All sections were stored in distilled water. Exposing the sectioned surface, teeth were embedded in self-cure acrylic using a custom-made mold to give the specimen the correct shape. Dentin (Group 1), BD (Group 2), and WMTA (Group 3) disc surfaces were serially polished starting from size 400 to 600, 800, and finally 1200 grit abrasive papers. Then, specimens were stored in a closed container to preserve the surface fineness.

Microhardness was measured for each sample at baseline and after application of CA. The baseline microhardness value was measured using Vickers Microhardness Tester (Digital Mitutoyo HM-200 Micro Hardness Testing Machine, USA) on polished cement and dentin surfaces of each sample. Samples were loaded with a diamond indenter point with a weight of 50 g for 5 s to produce a stamp in a homogeneous region of the cement surface. The indentation was made on the dentin surface approximately at 200 μm from the canal–dentin interface for standardization. A square-based pyramid-shaped diamond indenter produced a quadrangular depression with two equal orthogonal diagonals in the polished surface of the cement and dentin. The resulting impression of the two diagonals was observed with an optical microscope, and the average length of the two diagonals was measured by the built-in scaled micrometer and converted into Vickers hardness number (VHN), which was displayed on the digital readout of the microhardness tester.

Soon after recording the baseline readings, all samples were soaked in CA for 5 min. Thereafter, the microhardness of all these samples was measured one more time using the same Vickers Microhardness Tester. The microhardness before and after CA treatment was evaluated and compared.

Results

The results were statistically analyzed using paired sample t-test (Table 1). There was a statistically significant difference between Group 1 (dentin disc) and Group 3 (MTA) before and after the exposure to CA. However, Group 2 (BD) showed no statistically significant difference ($p< 0.05$) (Fig. 1).

Discussion

The null hypothesis of the present study was partially rejected because significant differences were observed in microhardness among Group 1 and Group 3. CA is a component of blood and a weak inorganic acid with a pH of

Table 1. Results of paired sample 't' test before and after carbonic acid treatment

Paired Samples Test								
Before and after carbonic acid treatment	Paired differences					t	df	p-value
	Mean	Std. deviation	Std. error mean	95% Confidence interval of the difference				
				Lower	Upper			
Group 1								
DENTIN	22.577	2.337	.648	21.165	23.989	34.831	12	.000*
Group 2								
BIODENTIN	.346	.588	.163	-.009	.702	2.122	12	.055
Group 3								
MTA	.362	.206	.057	.237	.486	6.318	12	.000*

*Statistically significant difference between the pair.

5.48 (10). The calcium-depleting nature of CA acid may have the potential to decrease the strength of MTA by interrupting the crystallization of calcium silicate hydrate (13). CA produces a significant reduction in hardness of WMTA within 5 min of exposure (10). Therefore, in the present study, 5 min was selected as the time period for CA treatment.

In this study, dissolution of WMTA was assessed by reduction in surface hardness after exposure to acids because clinically, only the material surface will be exposed to solvents and not the entire mass. Fresh CA was used in this study because the availability of carbon dioxide for interaction reduces over a period of time (10). In *in vitro* studies, the carbon dioxide released from CA is not buffered, and therefore its action will be more aggressive compared to *in vivo* conditions where the released carbon dioxide is buffered by fluids in the periapical tissues.

Exposure to CA significantly decreased the surface hardness of WMTA. The probable reason could be that the CA ions act on the calcium silicate hydrate and calcium hydroxide in the WMTA.

The chemical reactions are as follows (14):

$$H_2CO_3 \rightarrow 2H^+ + CO_3^{2-}$$

$$Ca(OH)_2 + H_2CO_3 \rightarrow Ca_2CO_3 \text{ (calcium carbonate)}$$

A value of 2% CA significantly reduced the microhardness of WMTA after exposure for 20 min. This was probably because long-term contact of MTA with CA decomposed the calcium silicate hydrate gel into calcium carbonate, acid-insoluble silica gel, and water. As the reaction of acid and base led to the formation of salt and water, complete carbonation eventually resulted in the decalcification of the calcium silicate gel, which decreased strength (12). The 2% CA significantly reduced the surface microhardness of partially and completely set MTA within 10 min of exposure (10). The results of all these studies were in line with the results of the present study.

Although there was a reduction in the microhardness of the BD disc after CA treatment, it was not statistically significant compared with the microhardness of the BD disc prior to CA treatment. This could be due to the chemical changes occurring only on the surface of the substance rather than within it. BD exhibited different chemical and

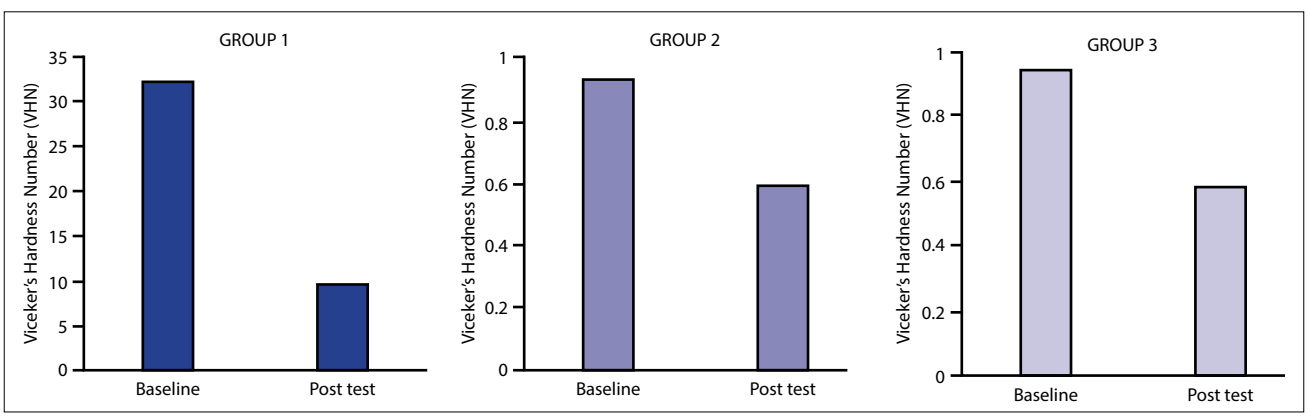

Fig. 2. Comparison of mean baseline values of microhardness and posttreatment microhardness values by Vicker's microhardness test of dentin disc (graph 1), BD (graph 2), and MTA (graph 3), respectively.

mechanical properties when stored in a bicarbonate-rich medium that could be attributed to carbonation. Due to surface carbonation, BD exhibited higher strength at earlier stages; however, this was associated with a limited release of calcium and silicon ions to the surrounding environment (14).

In addition to the consumption of calcium hydroxide, carbonation resulted in the deposition of calcium carbonate on the surface (15). With the precipitation of calcium carbonate on the surface of the cement, the leachability of ions is affected (16), which may reduce the available calcium ions in the solution (15). Based on the current findings, carbonation may affect the levels of ions released by the cement, either directly through the carbonation process that consumes both calcium and hydroxyl ions (17) or indirectly by limiting the leachability of ions such as Ca^{2+} and Si^{4+} (15).

This investigation revealed statistically significant variations in surface microhardness of dentin discs before and after exposure to CA within 5 min. CA released the highest amount of calcium ions from RD within 10 min of its exposure (18). This is attributed to the chelating effect of CA (18), which could also be a reason for decreased surface hardness after CA treatment. Therefore, using CA to dissolve MTA might pose the danger of weakening the tooth structure, thus reducing the fracture resistance of the tooth (10). Changes in the mineral content ratio may alter the original proportion of organic and inorganic components, reducing the microhardness, increasing the permeability and solubility of the root canal dentin, and inhibiting resistance to bacterial ingress, thereby permitting leakage (19).

The limitation of the study is that CA gets buffered in periapical tissues when used clinically, but in the present study freshly prepared unbuffered CA was used. Therefore, the effect of CA ions will be more under *in vitro* situations than when used clinically. This in turn can alter the results of the study. Accordingly, further studies are needed to confirm the *in vivo* effects of the tested solvents on the retrievability of MTA and BD.

Conclusion

CA can dissolve set MTA, but cannot dissolve BD. Nevertheless, CA, when used to remove set MTA, shows a detrimental effect on RD within 5 min of its exposure. Therefore, it is not clinically recommended to use CA for the retrieval of set MTA.

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