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Push-out bond strength of a nano-modified mineral trioxide aggregate

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Abstract – *Introduction*: To analyze the push-out bond strength of Angelus WMTA (Angelus Dental Products), a nano-modification of WMTA (Kamal Asgar Research Center) and Bioaggregate (Innovative Bioceramix). *Methods*: Sixty 2-mm-thick root sections were prepared from 60 single-rooted human teeth. The dentin disks were randomly divided into three groups (n = 20) and filled with Angelus WMTA, Nano-WMTA, or Bioaggregate, respectively. Push-out bond strength values of the specimens were measured by a universal testing machine and examined under scanning electron microscope at $\times$ 40 magnification to determine the nature of the bond failure. The data were analyzed with a Kruskal–Wallis test. *Results*: The greatest mean for push-out bond strength (138.48 ± 11.43 MPa) was observed for the nano-modification of WMTA. The values decreased to 110.73 ± 11.19 and 25.64 ± 5.27 MPa for Angelus WMTA and Bioaggregate, respectively. There were significant differences between the groups ($P < 0.001$). Inspection of the samples revealed the bond failure to be predominantly adhesive type except for the nano-modification group, as some samples also exhibited cohesive failures. *Conclusions*: It is concluded that the force needed for the displacement of the nano-modification of WMTA (NWMTA) was significantly higher than for Angelus WMTA and Bioaggregate.

Key words: push-out bond strengths; nano; bioaggregate; mineral trioxide aggregate

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An ideal endodontic cement should provide an impervious apical seal, be dimensionally stable, radiopaque, nonresorbable, biocompatible, and be well integrated by the root canal dentin tissues (1). Calcium hydraulic cements such as mineral trioxide aggregate (MTA) exhibit several properties of an ideal root-end filling material and has rapidly gained popularity since its introduction in the market. But this type of cement has some drawbacks including poor handling (2), low acidic resistance (3), and high setting time (4). Recently, two cements were introduced in the market claiming to improve one or some of the MTA drawbacks.

The first one, Bioaggregate (Innovative Bioceramix, Vancouver, BC, Canada), is a novel root-end filling cement primarily composed of calcium silicate, calcium hydroxide, and hydroxyapatite (5). De-Deus et al. (6) found that Bioaggregate cement revealed similar biocompatibility to that seen for MTA when cultured with primary human mesenchymal cells. Most of the constituents of Bioaggregate are similar to those in white MTA, differing mostly by being free of aluminum compounds. It is composed of tricalcium silicate (C_3S), dicalcium silicate (C_2S), calcium phosphate monobasic, and amorphous silicon dioxide with the addition of tantalum pentoxide, instead of bismuth oxide in MTA, for radiopacity (5).

The second one is a nano-modification of WMTA (Kamal Asgar Research Centre, US patent #13/211.880). It is a new root-end filling cement that has similar composition to WMTA, but with very low particle size and high specific surface area of powder which may produce a faster and better hydration process. A previous investigation illustrated similar components of NWMTA and WMTA, but the former was more resistance to an acidic environment and set 10 times faster than WMTA (7).

However, there are no reports on the potential integrity of these kinds of cements with the root canal dentin and push-out bond strength values. The aim of this study was to evaluate the push-out bond strength of WMTA, Bioaggregate, and the nano-modification of WMTA.

Materials and methods

Sixty extracted single-rooted human teeth were used in this study. The crowns of all the teeth were removed. To obtain 2-mm-thick root sections, the middle-thirds of the roots were sectioned transversally using a water-cooled low-speed Isomet diamond saw (Buehler, Lake Bluff, NY, USA). In each section, the canal space was enlarged with a spherical diamond bur and two complete passes of #5 Gates-Glidden bur to obtain 1.3-mm-diameter standardized cavities as performed by previous investigations (8, 9), then sections were immersed in 17% EDTA for 3 min, followed by immersion in 1% sodium hypochlorite for the same period of time. The sections were then washed in distilled water and dried immediately. Afterward, root sections were randomly divided into three groups ($n = 20$), and the cavities were filled with conventional technique and manufacturer's recommendations as follows:

Group 1: WMTA Angelus (Angelus Dental Industry Products, Londrina, Brazil).

Group 2: Nano-modification of WMTA (Kamal Asgar Research Centre, US patent #13/211.880).

Group 3: Bioaggregate (Innovative Bioceramix, Vancouver, BC, Canada).

In Table 1, ingredients of WMTA, Nano-WMTA, and Bioaggregate are described.

The sections were wrapped in pieces of gauze soaked in synthetic tissue fluid (STF), which was prepared as follows:1.7 g of KH_2 PO_4, 11.8 g of Na_2HPO_4, 80.0 g of NaCl, and 2.0 g of KCl in 10 l of H_2O (pH 7.4) according to Saghiri et al. study (8). The specimens were then kept at 37°C for 72 h.

The push-out bond strengths were measured using a Zwick/Roell Z050 universal testing machine (Ulm, Germany). The samples were placed on a metal slab with a central hole to allow the free motion of the plunger. The compressive load was applied to the plug by exerting a downward pressure on the surface of MTA using a 1.00-mm-diameter cylindrical stainless steel plunger at a crosshead speed of 1 mm min^{-1}. The plunger had a clearance of approximately 0.2 mm from the margin of the dentinal wall to ensure contact with MTA only. The maximum load applied to plug at the time of dislodgement was recorded in Newton. To calculate the bond strength in mega pascal (MPa), the recorded value was divided by the adhesion surface area of root canal filling calculated as following:

$$\text{Debond stress (MPa)} = \frac{\text{Debonding force (N)}}{\text{Surface Area (mm}^2\text{)}}$$

The slices were then examined under a scanning electron microscope at ×40 magnification to determine the nature of the bond failure. Each sample was categorized into 1 of the three failure modes: adhesive failures at the cement and dentin interface, cohesive failure within the cement, or mixed failure. The data were analyzed with a Kruskal–Wallis test.

Results

Results of the Kruskal–Wallis test revealed that the mean push-out bond strength values of white MTA, Nano-WMTA, and Bioaggregate were 110.73 ± 11.19, 138.48 ± 11.43, and 25.64 ± 5.27 MPa, respectively. The push-out bond strength values of Nano-WMTA were higher, with statistically significant differences between the groups ($P < 0.001$) (Fig. 1).

Inspection of the samples under a scanning electron microscope at ×40 magnification revealed that the bond failure was adhesive type in the majority of the WMTA and Bioaggregate specimens; however, some of the Nano-WMTA exhibited cohesive failure patterns (Fig. 2).

Discussion

An ideal cement plug must be able to remain in place and integrate with the root canal wall under dislodging

Table 1. The ingredients of WMTA, Nano-WMTA, and Bioaggregate (10, 11, 22)

Cement ingredients					
Nano-WMTA		WMTA		Bioaggregate	
Ingredient	Molecular formula	Ingredient	Molecular formula	Ingredient	Molecular formula
Di-sodium hydrogen phosphate	Na_2HPO_4	Tri-calcium aluminate	$Ca_3Al_2O_6$	Tri-calcium silicate	Ca_3SiO_5
Bismuth oxide	Bi_2O_3	Bismuth oxide	Bi_2O_3	Di-calcium silicate	Ca_2SiO_4
Tri-calcium silicate	Ca_3SiO_5	Tri-calcium silicate	Ca_3SiO_5	Hydroxyapatite	$Ca_{10}(PO_4)6(OH)_2$
Di-calcium silicate	Ca_2SiO_4	Di-calcium silicate	Ca_2SiO_4	Calcium silicate oxide	$Ca_3(SiO_4)O$
Calcium sulfate	$CaSO_4$	Bismuth oxide	Bi_2O_3	Tantalum oxide	Ti_2O_5
Gypsum	$CaSO_4.2H_2O$	Gypsum	$CaSO_4.2H_2O$	Calcium silicate oxide	$Ca_3(SiO_4)O)$
Strontium carbonate	$SrCO_3$			Calcium phosphate silicate	(alpha=$Ca_2SiO_{40}.05Ca_3(PO_4)2$)
Zeolite	$M_2/nO.A_{12}O_3.xSiO_2.yH_2O$				
Tri-calcium aluminate	$Ca_3Al_2O_6$				

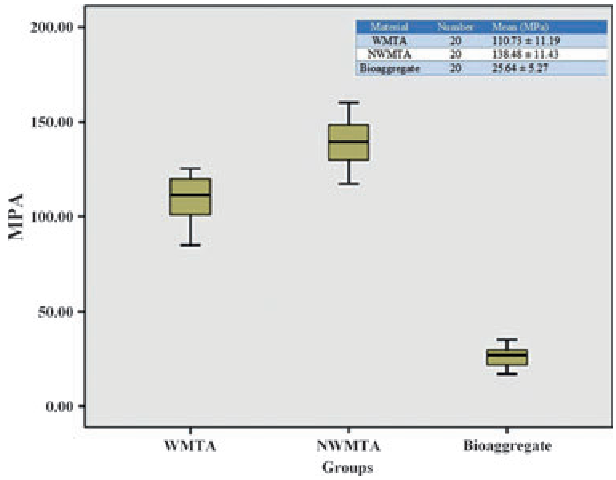

Fig. 1. Means of push-out bond strength values of three types of cements.

forces such as mechanical stresses as a result of tooth function or operative procedures (12). Therefore, the push-out bond strength of perforation repair materials, root-end fillings, and materials used for apical barrier formation is an important factor in clinical practice. To assess the integrity of a material with its surrounding root canal dentin, the push-out bond test has been shown to be efficient, practical, and reliable (8, 13–15). A previous investigation confirmed the value of adhesion and/or integrity between MTA and dentin (16).

In present study, Bioaggregate samples in comparison with WMTA and Nano-WMTA showed lower push-out bond strength. Hashem et al. have indicated that Bioaggregate was more resistant to dislodgement forces than WMTA. However, these authors mentioned that initially dislodgement resistance of Bioaggregate was higher than WMTA while after 34 days, WMTA showed significantly higher bond strength than BA (17). With respect to this result, the manufacturer indicates that during the hydration phase, C-S-H and calcium hydroxide (CH) are formed, while in the presence of amorphous silicon dioxide, the CH byproduct reacts further to form C-S-H. This reaction results in lower amount of CH in Bioaggregate after hydration (18). Another investigation mentioned that the reduction in the amount of CH can play an important role in the weakness of the mixed cement (19). In addition, WMTA and Nano-modified WMTA because of possessing tricalcium aluminate (C_3 A) can show better hydration process as this issue was discussed in previously performed investigations. These authors mentioned that tricalcium aluminate in Portland cement can facilitate the formation of desired silicate phase. This element (C_3 A) can react strongly with water and creates Ca_2AlO_3 (OH) $\cdot nH_2O$, which is addressed as 'flash set' procedure and it is able to enhance the strength of mixed cement relatively (20, 21). Park et al. (22) confirmed that Bioaggregate did not contain any aluminum compound, while other investigations also (23, 24) indicated that tricalcium aluminate plays an important role in the strength of calcium silicate cement such as WMTA and without this ingredient, the strength of the cement may be jeopardized or even hinder the setting time.

The results of Nano-WMTA group specimens indicated that the push-out bond strength of this cement is significantly higher than WMTA. Table 1 describes the differences of ingredients of these two cements, which can explain this finding. Nano-WMTA since introduction has been acclaimed to modify the size of the constituents of WMTA and to increase the surface area of the powder. This modification plus uniform distribution of strontium were shown to be effective in decreasing the setting time and increasing the microhardness even at low pH values (7). Besides these changes, Nano-WMTA in comparison with WMTA contains other particles such as Zeolite according to Table 1. Zeolite is a crystalline hydrated aluminosilicate of alkaline metals and metals of alkaline soils (Ca, K, Na, Mg) so-called klinoptilolite. This substance has shown to be a stabilizer with anticorrosive action against sulfate, which is regarded as the main reason for sulfate attack occurred during the reaction of Portland cement (25). These findings may clarify the push-out bond strength differences detected between Nano-WMTA and WMTA group samples.

Teeth in current study stored in 0.5% chloramine-T at 4°C for up to 15 days before use owing to prevent any cross contamination and also match the methodology with previous studies (8, 9, 13) to make better comparison of results. In addition, the bond failures

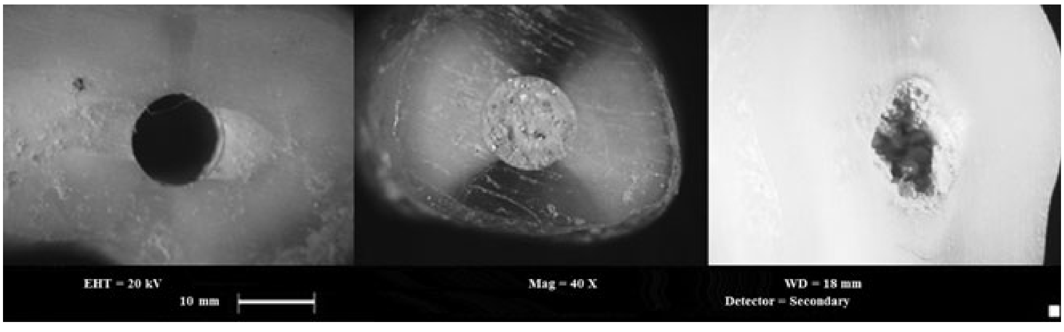

Fig. 2. Mode of failures. (Left) Adhesive failure; note the clean canal wall. (Center) Cohesive failure within cement. (C) Mixed failure; note cement remnants inside the canal.

observed in WMTA and Bioaggregate experimental groups were at the MTA–dentin interface (adhesive type). This finding is in accordance with the results of Vanderweele et al. (9), Saghiri et al. (8), and Shahi et al. studies (15) that revealed MTA–dentin bond failures were predominantly adhesive type. Furthermore, Shokouhinejad et al. (13) showed that the mode of failure after push-out testing of WMTA exposed to pH values of 7.4, 6.4, 5.4, or 4.4 was mainly adhesive type.

In this study, the adhesive type of bond failure for both WMTA and Bioaggregate cements might be attributed to first, the short storage time before testing the push-out bond strength, which was 3 days in the present study, and 4 and 7 days in the studies performed by Shokouhinejad et al. (13) and Vanderweele et al. (9), respectively. Second, the particle size of these cements may have prevented its penetration into dentinal tubules producing adhesive failures (26). In contrast, the type of bond failure of Nano-WMTA samples in some cases was indicated to be cohesive. This difference with the other two groups might be explained by the particle size of Nano-WMTA. Smaller particle size and uniform distribution of constituents can create better interlocking bonds with dentin, which finally leads to cohesive failure mode inside the cement structure (7).

Conclusion

1 Both WMTA and the Nano-WMTA have statistically higher push-out bond strengths than the Bioaggregate cement because of their different compositions.

2 Nano-WMTA showed better resistance against dislodgement forces made in this study in comparison with WMTA. According to this finding, authors may suggest Nano-WMTA as a substitute material for WMTA especially in cases that applied cement undergoes heavy forces such as occlusal forces when cement is used for furcation perforations or direct pulp capping.

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Conflict of interest

The authors affirm that they have no financial affiliation or involvement with any commercial organization with direct financial interest in the subject or materials discussed in this manuscript and deny any conflicts of interest related to this study. M Ali Saghiri and Mehrdad Lotfi hold a US patent for this new endodontic cement.

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