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Chemo-mechanical properties of the new bioceramic cement PBS CIMMO® compared to MTA REPAIR HP Angelus® cement

Abstract

Objective: To evaluate the bond strength (BS), pH and solubility (Sol) of a new PBS HP® bioceramic cement compared to MTA REPAIR HP® cement.

Material and methods: Twenty human premolars were selected for BS analysis. Roots were cut into thirds (cervical, medium and apical) and filled with one of the cements (n=10): PC (PBS HP) or MC (MTA REPAIR HP). Half of these samples were subjected to 2,000 thermal cycles. After 48 hours, the BS test was performed in a universal testing machine (0.5mm/min). Cylindrical specimens (1.0mm in diameter and 10mm in height) were made with PC or MC and immersed in distilled water for pH analysis. After 0.3 and 24 hours, the pH was measured. Solubility was evaluated through the mass loss of the samples after 24 hours. Teflon rings of 20mm in diameter and 1.5mm in height were filled with PC or MC and kept at 37ºC in 100% humidity. Descriptive and exploratory analyzes of BS, pH and Sol data were performed ($p<0.05$).

Results: There was no significant difference in BS between the two types of cement. Thermo cycling did not affect the BS ($p>0.05$). MC cement showed significantly higher pH values than the PC at the initial and 3 hours ($p<0.05$). There was no difference between them ($p>0.05$) after 24 hours. There was no significant difference regarding solubility ($p>0.05$).

Conclusion: Bioceramic cements have similar properties, as they did not differ in relation to dentin bond strength, pH after 24 hours and solubility.

Keywords: dental infiltration, endodontics, materials testing, ph, physical properties, solubility

Volume 13 Issue4 - 2022

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Received: December 2, 2022 | **Published:** December 19, 2022

Abbreviations: MTA, Mineral Trioxide Aggregate; PBS HP, Pozzolana Biológico Silva Hight Plasticity; BS, Bond Strength

Introduction

Endodontic repair cements are commonly used to seal the communication between the pulp cavity and the periodontium, such as perforations and root resorption.[1] They are also used in the apical sealing of teeth submitted to paraendodontic surgery.[2] They must be biocompatible, resist the displacement of masticatory forces and have physical, chemical, and biological properties that do not adversely affect the cells involved in the repair process.[3]

Bioceramic cements are bioactive ceramic compounds whose composition includes tricalcium and dicalcium silicates, calcium phosphates, calcium hydroxide, and a radiopacifying agent.[4] They have excellent properties due to the similarity with the biological process of hydroxyapatite formation and, therefore, they can induce regenerative responses.[5] They have as properties: high pH, no resorption, easy handling inside the root canals, increased root resistance, low cytotoxicity, no contraction, and are chemically stable.[6,7] Due to these characteristics and, with the advent of nanotechnology, it became possible to use them as endodontic repair cements.[8]

MTA REPAIR HP ANGELUS® (Angelus, Londrina, PR, Brazil) was marketed at the beginning of 2016 as a new formulation of the White MTA. Launched internationally as a repair cement in the form of a bioceramic material of high plasticity and handling, in which calcium tungstate ($CaWO_4$) has been used as a radiopacifier instead of bismuth oxide to improve the physicochemical and biomechanical properties of material.[9] The liquid state of this product is obtained by adding an organic plasticizer (to gain plasticity and thus increase its handling) in distilled water.[10,11] These bioactive materials are recommended mainly for dental pulp treatments (pulp capping, pulpotomy), apexification, apexogenesis, root end obturation and root canal resorption repair.[12]

Recently, a significant number of bioceramic endodontic repair cements have been introduced to the market[1], including PBS HP CIMMO® cement - Pozzolana Biológico Silva Hight Plasticity (CIMMO, Pouso Alegre, MG, Brazil) having the same base as MTA, but with natural additives responsible for its resistance. It is a biological cement composed of mineral oxides in the form of fine hydrophilic particles, indicated for root perforation (fornix), root perforation by internal resorption, retro-filling, direct pulp protection, apicigenesis and apicification.[13,14]

Despite the technological advances, knowledge of the properties of the cements evaluated in this study is still scarce. The importance of evaluating bond strength (BS), pH and solubility is highlighted, as they are fundamental attributes for the materials used in endodontic repair. These cements, if not well attached to the root dentin, can move during tooth movement, and lose their repair function.[15]

Thermo cycling is a laboratory method of exposing dental materials and teeth to temperature ranges like those occurring in the oral cavity.[16] Through these cycles, thermal stresses can affect the BS between the repair material and the dental structure and cause microleakage.[17]

The evaluation of the pH and solubility of these cements is also extremely necessary. The pH is elevated by the release of calcium and hydroxyl ions and, the alkalinization of the medium can benefit the repair, promote mineralization[18,19] and cause an antibacterial effect. On the other hand, if soluble, they lose their sealing capacity and, consequently, allow bacterial microleakage and tissue fluids.[5] This in vitro study was necessary to evaluate the BS, pH and solubility of bioceramics PBS HP CIMMO® and MTA REPAIR HP ANGELUS® to be used clinically safely and effectively. The null hypotheses tested were that there is no significant difference in BS with or without thermocycling ($H0_1$), in the pH measured at different times (initial, 3 hours and 24 hours) ($H0_2$) and in the solubility ($H0_3$) of the two bioceramic cements evaluated.

Material and methods

This study was approved by the Research Ethics Committee of Faculdade San Leopoldo Mandic (CAAE: 03416818.0.0000.5374).

Push-out test: Selection and preparation of root canal

Twenty mono-reticulated human premolar teeth were selected and preserved in 0.1% thymol solution. The external surfaces of these teeth were cleaned with curettes, gauze and 5.25% sodium hypochlorite. They were washed in water for 24 hours and dried on paper towels. Roots were cross sectioned at the cementitious junction using a double-sided diamond disc (carburundum disc); under constant refrigeration roots size was standardized at approximately 14mm in length.

Root canal was prepared with the NiTi ProDesign Easy Logic rotary file system (Easy dental equipment, Belo Horizonte, Brazil) at the working length (actual tooth length minus 1mm) using #15/05 as the initial instrument and #40/05 for the final instrumentation and to make the apical stop.

During biomechanical preparation, canals were irrigated with 5mL of 5.25% sodium hypochlorite (Asfer, São Paulo, SP, Brazil), using sterile plastic syringes and Navitips irrigation tips (Ultradent, Indaiatuba, SP, Brazil). Irrigating solution was removed with an aspiration cannula. After biomechanical preparation, canals were treated with 3mL of 17% EDTA (Asfer, São Paulo, SP, Brazil) and stirred with a K15 file (Dentsply Maillefer, Germany) for 3 minutes to remove the smear layer. The last irrigation of the canal was carried out with 5mL of sterile 0.9% saline (Eurofarma, São Paulo, SP, Brazil). Canals were dried with 40 paper cones (Dentsply, Germany).

Preparation of samples for push-out test

Roots were fixed in acrylic plates with sticky wax and sectioned perpendicularly along their axes with a high concentration diamond disc (Extec Corp, Enfield, CT, USA) coupled to a precision cutter (Isomet 1000, Buehler Lake Bluff, Illinois, USA). After a first cut made 1 mm from the cervical margin (not used), 6 consecutive cuts were made 2 mm from each other, so that it was possible to obtain 2 slices of each root third: cervical, middle, and apical. With the aid of a digital caliper, in each slice, the smallest radius (apical side of the slice), the largest radius (cervical side) and thickness (height) were measured for later calculation of the joining area. The root slices were kept in individual containers (Eppendorf) with saline solution at 37°C for 24 hours to maintain hydration. After that time, the root dentin was dried with absorbent paper tips.

The 20 roots and their respective slices were randomly divided into 2 groups (n=10), according to the selected endodontic cement, PBS HP cement (CIMMO, Pouso Alegre, MG, Brazil) or MTA REPAIR HP cement (Angelus, Londrina, PR, Brazil). Both are described in Table 1. The justification for the sample size (n=10) was based on previous studies.[20–23] Each slice was positioned on a glass slide placed on a glass plate. The cements were handled in the proportion of 1:1 powder/liquid and inserted into the conduits of the slices with the aid of a flexible metallic spatula, number 24 (SS White Duflex, Rio de Janeiro, RJ, Brazil). After filling, another slide and another plate were placed on the slices and kept in an oven at 37ºC, in the presence of 100% humidity for 48 hours. Excess cement was removed with scalpel blades and 220 and 600 grit sandpapers until the cement / dentin interface in the preparation became visible.

Table 1 Composition, manufacturer and batch of tested cements

Commercial Name	Composition	Manufacturer	Batch
MTA REPAIR HP	Powder: tricalcium silicate, dicalcium silicate, tricalcium aluminate, calcium oxide, calcium tungstate. Liquid: water and plasticizer.	Angelus (Londrina, PR, Brazil)	101555 and 46486
PBS HP	Powder: calcium oxide, calcium carbonate, magnesium oxide, dicalcium silicate, aluminum oxide, sodium oxide, potassium oxide and pozzolan plus additives. Liquid: sterile distilled water.	CIMMO (Pouso Alegre, MG, Brazil)	005 and 007

One slice of each root third of each root (n=10) was randomly selected to be subjected to two thousand thermal cycles, in a thermocycling simulation machine (model MSCT-3e, Elquip, São Carlos, SP, Brazil). Cycles consisted of immersions in temperature from 5ºC to 55ºC, with 30 seconds of immersion in each bath. The slices that were not subjected to thermocycling remained in an oven at 37ºC until the moment of the bond strength test.

Bond strength test (Push-Out)

Samples were placed in a device composed of a base with a central stainless-steel hole attached to the testing machine (model EMIC DL2000; EMIC, São José dos Pinhais, PR, Brazil) to perform the push-out bond strength test. Slice was centered over the hole and a metal rod with an active tip of 1.0 mm in diameter, fixed to the load cell (50KgF per 0.5 mm/min), was then positioned over the center of the bioceramic cement and the push-out test was conducted at a speed of 0.5 mm/min. The force required to move the bioceramic cement was recorded in Kg/F and converted to stress values (in MPa), adopting the following formula:

$$MPa = Kg / Fx9.81 / area\left(mm^2\right)$$

The adhesive area was calculated using the formula on the side of the trunk of the cone, where π=3.1416; R=largest radius of the conduit; r=smaller radius of the conduit; h=specimen thickness.

$$MPa = Kg / Fx9.81 / area\left(mm^2\right)$$

Analysis of pH

To measure the pH, 1mm-inner diameter and 10 mm-length polyethylene tubes were used with only one of its ends open. The other end of the tube was closed by compressing its edge with a heated instrument. Cements were inserted into the tubes with the aid of Paiva pressers until they were filled. To standardize the quantity, the tubes were weighed before and after laying the cement (±0.002g). Ten specimens of each cement used in the test were prepared and, individually, each was immersed in polypropylene tubes (Falcon Tubes) containing 10mL of distilled water, according to previous studies.[24–26]

After that, they were closed and taken to the incubator at 37ºC (NT 705 Incubator Greenhouse, Nova Técnica, São Paulo, Brazil), where they remained for the entire experimental period. Before the immersion of the specimens, the pH was checked. Evaluations were carried out in the periods of 3 and 24 hours.[24–26]

Solubility analysis

Cement specimens (n=10) were made following ANSI/ADA standard guidelines #57 for the determination of solubility.[27] Sample size was based on previous studies.[28,29] Teflon ring molds of 20mm in diameter and 1.5mm in height were filled with cements, proportioned and handled in an environment with a temperature around 25ºC. Each mold was placed on a glass slide covered by a cellophane film. A nylon thread was placed inside the mass of the still soft cements so that, during the immersion of the cement in distilled water, the specimen could be kept suspended, not touching the flask wall during the experimental period. After filling the rings on the glass sheets with cement, another slide, also protected with cellophane, was placed on these rings and a light pressure was applied until they touched the surface of the rings, leaving the cement with a uniform and smooth thickness. The set consisting of the glass slide, teflon mold, nylon thread and cement was taken to an incubator and kept at 37ºC in 100% relative humidity for the period of 1.5 setting time of each material. Then, they were placed in a dehumidifier for 24 hours to remove possible suspended particles. Specimens were weighed on a 3-decimal precision scale (Quimis Aparelhos Científicos Ltda., São Paulo, SP, Brazil). Specimens were immersed in 7.5ml of distilled and deionized water, packed in sealed flasks, taking care that the specimens suspended and immersed in the water did not touch the walls of the container. Flasks were sealed and the system maintained at 37ºC and 100% relative humidity for 24 hours. Specimens were removed from the liquid, rinsed in distilled and deionized water and placed in a dehumidifier for 24 hours. They were weighed to obtain the final mass. Loss of mass was noted as being the solubility of the tested material.

Statistical analysis

Descriptive and exploratory analyzes were performed on shear strength, pH, and solubility data. Resistance data do not meet the assumptions of a parametric analysis and were analyzed by Mann Whitney's nonparametric tests for comparisons between cements. Wilcoxon test was used for comparisons between groups with and without thermocycling and Friedman and Nemenyi for comparisons between thirds. The pH data have an asymmetric distribution and were analyzed by generalized linear models for repeated measures over time. Solubility data were analyzed using the Student's t test. All analyzes were performed in the R[30] program considering the significance level of 5%.

Results

There was no significant difference in the BS (push-out) between the two types of cements (MTA REPAIR HP ANGELUS® and PBS HP CIMMO®) (p>0.05) (Table 2).

Table 2 Shear strength by extrusion (Push-Out) as a function of cement, thermocycling, and thirds

Thermocycling	Third	Cement				p-value
		1MTA		2PBS		
		Mean (standard deviation)	Median (minimum value - maximum value)	Mean (standard deviation)	Median (minimum value - maximum value)	
Without p-value	Cervical	0.187 (0.198)	0.114 (0.007-0.592)	0.284 (0.257)	0.214 (0.068-0.912)	0.2568
	Middle	0.585 (0.796)	0.202 (0.023-2.246)	0.639 (0.783)	0.426 (0.002-2.611)	0.5937
	Apical	0.468 (0.241)	0.514 (0.015-0.792)	1.064 (1.495)	0.427 (0.065-4.163)	0.8798
		0.0821		0.0608		
With p-value	Cervical	0.215 (0.330)	0.082 (0.016-1.082)	0.215 (0.269)	0.142 (0.006-0.926) b	0.5454
	Middle	0.422 (0.599)	0.171 (0.054-2.011)	0.498 (0.693)	0.220 (0.017-2.271) ab	0.8206
	Apical	0.395 (0.478)	0.178 (0.016-1.301)	0.590 (0.488)	0.412 (0.102-1.429) a	0.1509
		0.3012		0.0017		

MTA REPAIR HP®; PBS HP CIMMO®; p (cycling. MTA cervical) =0.6465; p (cycling. MTA médio) =0.6465; p (cycling. MTA apical) =0.6465; ®; p (cycling. PBS cervical) =0.5076; p(cycling. PBS médio) =0.5751; p (cycling. PBS apical) = 0.8785. Distinct vertical letters indicate significant differences (p<0.05)

There was also no significant difference between groups with and without thermocycling (p>0.05). Resistance was significantly higher in the apical than in the cervical third (p<0.05) for PBS HP CIMMO® cement, with thermo cycling. There was no significant difference in resistance between the thirds (p>0.05), for the other groups.

The pH was significantly higher in MTA REPAIR HP ANGELUS® cement than in PBS HP CIMMO® cement (p<0.05) for 0 and 3 hours (Table 3).

There was no significant difference between cements for 24 hours (p>0.05). There was a significant increase in pH over time for both cements (p<0.05).

None of the cements showed solubility below 3% as recommended by ANSI/ADA (p>0.05). There was no significant difference between the two cements in terms of solubility (percentage of mass loss) (p>0.05).

Table 3 pH of cement as a function of time

Time	Cement			
	1MTA		2PBS	
	Mean (standard deviation)	Median (minimum value - maximum value)	Mean (standard deviation)	Median (minimum value - maximum value)
0h	6.30 (0.48) Ac	6.36 (5.56-7.08)	5.63 (0.12) Bc	5.60 (5.46-5.83)
3h	8.71 (1.08) Ab	9.10 (3.94-10.05)	7.87 (0.90) Bb	7.82 (6.74-92.3)
24h	9.46 (0.65) Aa	9.60 (8.33-10.27)	9.72 (0.39) Aa	9.68 (9.17-10.24)

MTA REPAIR HP®; PBS HP CIMMO®; p(cement)=0.0310; p(time)<0.0001; p(interaction)=0.0007. Distinctive letters (uppercase comparing horizontally and lowercase vertically) indicate significant differences (p≤0.05).

Discussion

The bond strength of endodontic repair cement is important to maintain the sealing of the communication area between the pulp cavity and the periodontium. These cements must have adequate strength so that the intracoronary restorative material can be safely condensed without displacement after the endodontic repair of the perforated region.[31,32]

Push-out test is efficient and reliable to assess the bond strength, since a gradually increasing pressure is applied to the material until the displacement occurs.[15,31] It is noteworthy that this study is the first to evaluate the bond strength of a new bioceramic cement PBS HP CIMMO® compared to the classic MTA REPAIR HP ANGELUS® using push-out and simulated aging using thermo cycling.

These results showed that there was no significant difference in the BS between the two types of cement and neither between the groups with and without thermocycling (p>0.05). However, for PBS HP cement, after thermo cycling, the resistance was significantly greater in the apical third than in the cervical one, while for the other groups there was no significant difference in resistance between the thirds (p>0.05). It is suggested that this greater resistance in the apical third occurred due to a hygroscopic expansion of the PBS HP cement after thermo cycling. This expansion is common in bioceramic cements due to the presence of calcium silicate[32] and, in addition, there may also have been a better frictional retention of this cement because the apical third has a smaller duct diameter. These findings must be confirmed in future studies. In addition to the bond strength, pH can also influence endodontic repair. Alkalinity is essential for the fixation reaction of any material permanently sealed in the root canal.[18,31] In this study, the pH of MTA REPAIR HP was higher than that of PBS HP for 0 and 3-hour times, whereas, in the time of 24 hours, there was no significant difference between the cements (p>0.05).

Some studies that evaluated the pH of bioceramic cements,[33–34] including MTA REPAIR HP, corroborate our findings with similar alkalinizing activity after 3 hours. Lower pH values in all periods for cements were found by other authors.[35,36] The pH is affected by the release of calcium and hydroxyl ions and by the alkalization of the medium, a condition that can influence repair, and promote mining.[18–19]

Cement potential for bonding to root dentin and dimensional changes possibly suffered by it are directly related to its solubility.[36] Absence of solubility is a desired characteristic because it avoids spaces that can favor bacterial colonization.[18] According to ANSI/ADA specifications, the loss of mass of each sample is expressed as a percentage of the original mass (solubility) and the ideal value should be lower than 3%. Results of this study showed that there was no significant difference in solubility between the two groups of cements studied (p>0.05), but the loss of mass of each sample, expressed as a percentage, did not present values below 3%. An acceptable explanation for this is that calcium silicate cements contain water in their composition and, therefore, during the final drying of the samples, there is a great loss of mass caused by the evaporation of this free water in the mixture.[32] Therefore, it must be taken into account that the loss of mass occurs not only due to the solubility of the material, but also due to the reactions of adjusting the material and also to the formation of soluble calcium salts and calcium hydroxide during the hydration of the material. In addition, MTA Repair HP ("High Plasticity") contains a plasticizer in the mixing liquid that was included to facilitate the manipulation and insertion of the material in the root cavity and to improve its manipulation. However, it makes the material more soluble.[12]

Despite the limitations of this in vitro study, it can be seen from the characteristics evaluated that both tested cements can be used as endodontic sealers. But other properties must be investigated and clinical trials must be conducted to confirm these results.

Conclusion

Bioceramic repair cements have similar properties, since they did not differ in terms of BS to dentin, pH after 24 hours, and solubility.

Acknowledgments

We would like to express our gratitude to the Department of Restorative Dentistry, São Leopoldo Mandic Institute and Dental Research Center.

Conflicts of interest

The authors have no conflict of interests related to this study.

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