UNIVERSIDADE FEDERAL DE ALFENAS

JAQUELINE MARTINS CRIVELARI

AVALIAÇÃO FÍSICO-QUÍMICA DE UM CIMENTO BIOCERÂMICO REPARADOR CONTENDO CINCO ÓXIDOS

ALFENAS/MG

2025

JAQUELINE MARTINS CRIVELARI

AVALIAÇÃO FÍSICO-QUÍMICA DE UM CIMENTO BIOCERÂMICO REPARADOR CONTENDO CINCO ÓXIDOS

Trabalho de conclusão de curso apresentado como parte dos requisitos para obtenção do grau de Bacharel em Odontologia pela Faculdade de Odontologia da Universidade Federal de Alfenas. Área de concentração: endodontia.
Orientador: Dr. Bruno Martini Guimarães
Coorientadora: Dra. Camila Soares Lopes

ALFENAS/MG

2025

Sistema de Bibliotecas da Universidade Federal de Alfenas
Biblioteca Central

Crivelari, Jaqueline Martins.
Avaliação Físico-Química de um Cimento Biocerâmico Reparador Contendo Cinco Óxidos / Jaqueline Martins Crivelari. - Alfenas, MG, 2025.
44 f. : il. -

Orientador(a): Bruno Martini Guimarães.
Trabalho de Conclusão de Curso (Graduação em Odontologia) - Universidade Federal de Alfenas, Alfenas, MG, 2025.
Bibliografia.

1. Endodontia Regenerativa. 2. Materiais Dentários. 3. Composição Química. 4. Selante do Canal Radicular. I. Guimarães, Bruno Martini, orient. II. Título.

Ficha gerada automaticamente com dados fornecidos pelo autor.

JAQUELINE MARTINS CRIVELARI

AVALIAÇÃO FÍSICO-QUÍMICA DE UM CIMENTO BIOCERÂMICO REPARADOR CONTENDO CINCO ÓXIDOS

O Presidente da banca examinadora abaixo assina a aprovação do Trabalho de Conclusão de Curso apresentado como parte dos requisitos para obtenção do título de Bacharel em Odontologia pela Universidade Federal de Alfenas.
Área de concentração: Endodontia

Aprovada em: 17 de novembro de 2025

Prof. Dr. Bruno Martini Guimarães
Universinade Federal de Alfenas

Assinatura gov.br
Documento assinado digitalmente
BRUNO MARTINI GUIMARAES
Data: 17/11/2025 09:07:07-0300
Verifique em https://validar.iti.gov.br

Prof. Dr. Carlos Augusto de Souza Lima
Universinade Federal de Alfenas

Prof. Dra. Daniela Coelho de Lima
Universinade Federal de Alfenas

Prof. Dra. Tatiany Gabrielle Freire Araújo Guimarães
Universinade Federal de Alfenas

RESUMO

Um dos principais objetivos do tratamento endodôntico é reparar dentes e mantê-los em suas condições funcionais. Para que isso seja possível, muitos cimentos têm sido utilizados, sendo que, por muitos anos, o material de escolha foi o Hidróxido de Cálcio, pois apresenta como propriedades ação antimicrobiana e indução de deposição de tecido mineralizado. Os cimentos biocerâmicos vêm demonstrando maior eficácia terapêutica devido à sua biocompatibilidade. O cimento reparador MTA (agregado trióxido mineral) é um material à base de silicato de cálcio amplamente utilizado em procedimentos reparadores na Odontologia. Porém, por não apresentar todas as caracerísticas de um material reparacor ideal, novos cimentos têm sido desenvolvidos, como o “5MO”, um cimento à base de cinco óxidos minerais. O presente estudo tem como objetivo avaliar as propriedades físico-químicas deste novo cimento, comparando o MTA Branco.

Palavras-chave: Endodontia regenerativa, Materiais Dentários, Composição Química, Selante do Canal Radicular.

ABSTRACT

One of the main goals of endodontic treatment is to repair teeth and keep them in their functional condition. To make this possible, many cements have been used and for many years, the material of choice was Calcium Hydroxide, as it has antimicrobial properties and induction of deposition of mineralized tissue. Bioceramic cements demonstrating greater therapeutic efficacy due to their biocompatibility. The repairing cement MTA (mineral trioxide aggregate) is a material based on calcium silicate widely used in restorative procedures in Dentistry. However, as it does not have all the characteristics of an ideal repairing material, new cements have been developed, such as “5MO”, a cement based on five mineral oxides. The present study aims to evaluate the physicochemical properties of this new cement comparing with the properties of pure silicate and white MTA.

Keywords: Regenerative endodontics, Dental materials, Chemical composition, Root canal sealant.

LISTA DE FIGURAS

LISTA DE TABELAS

LISTA DE ABREVIATURAS E SIGLAS

EDS	Espectrômetro de Energia Dispersiva
EDX	*Energy Dispersive X-Ray*
FTIR	*Fourier Transform - Infrared Spectroscopy*
MEV	Microscopia Eletrônica de Varredura
MTA	*Mineral Trioxide Agregate*
SEM	*Scanning Electron Microscopy*
5MO	*Five Mineral Oxides*

SUMÁRIO

1 INTRODUÇÃO

Os procedimentos reparadores são de grande importância na endodontia (Bancas; Trope, 2004), pois permitem a manutenção dos dentes em condições funcionais (Roig *et al*., 2011). Para isso, materiais bioativos têm sido a escolha em procedimentos pulpares e endodônticos para melhorar os resultados da cicatrização, principalmente reduzindo a probabilidade de extração (Primus; Tay; Niu, 2019). Em caso de exposição pulpar, esse material não irritante é aplicado sobre a polpa com o intuito de desinflamar o tecido pulpar e manter o dente funcional (Barret, 1877).

A busca por um material capaz de induzir a regeneração e reparo de cavidades expostas começou no último século (Al-Nalahwi; Rachi; Hasna, 2021). Durante muito tempo, o hidróxido de cálcio foi o material de escolha e passou por diversos estudos avaliando sua eficácia como material reparador, apresentando resultados satisfatórios nas suas propriedades antimicrobiana e indutora da deposição de tecido mineralizado (Cvek, 1972; Schöder, 1972). Porém, os cimentos biocerâmicos surgiram demonstrando efeitos mais efetivo em tratamentos endodônticos (Hasna *et al*., 2020), devido à sua biocompatibilidade e excelente eficácia terapêutica (Darweesh *et al*., 2020).

O agregado trióxido mineral (MTA) é um material reparador bioativo derivado do cimento de Portland (Torabinejad, 1993), descrito como um pó hidrofílico composto por silicato tricálcico, aluminato tricálcico, óxido tricálcico, óxido de silicato e alguns outros óxidos minerais (Darweesh *et al*., 2020). É indicado para diferentes situações clínicas, tais como apicigênese, apicificação, revascularização pulpar e selamento de perfurações endodônticas (Linswanont; Kulviti; Santiwong, 2018; Linswanont *et al*, 2017). Sua radiopacidade se deve ao óxido de bismuto adicionado à sua estrutura (Torabinejad, 1993). Suas propriedades físicas, químicas e biológicas do MTA têm sido estudadas por décadas, porém, avanços ainda são necessários para que se obtenha uma composição que atenda as características ideais para um material reparador (Santos *et al*., 2005; Gandolf *et al*., 2011; Gandolf *et al*., 2012).

Um cimento endodôntico reparador ideal deve apresentar características físicas de estabilidade dimensional e de cor (Camilleri; Mallia, 2011; Marciano *et al*., 2014; Duque, 2018), radiopacidade (Islam; Chng; Yap, 2006), tempo de presa adequado, ausência de solubilidade em contato com fluídos (Fridland; Rosado, 2003; Cavenago *et al*., 2014), escoamento para um adequado selamento (Duarte *et al*., 2012; Duque

et al., 2018); propriedades químicas e biológicas de alcalinidade, liberação de íons cálcio (Duarte *et al*., 2003), bioatividade (Gandolfi *et al*., 2010), adesão celular (Balto, 2004) e biocompatibilidade (Holland *et al*., 2002; Camilleri *et al*., 2004); e propriedades antimicrobianas (Tanomaru-Filho *et al*., 2007). Muitas das propriedades ideais para um cimento reparador foram demonstradas pelo MTA, porém outras ainda necessitam de aprimoramento (Parirokh; Torabinejad, 2010).

Novos materiais têm sido desenvolvidos com o objetivo de solucionar estes inconvenientes do MTA (Gandolfi *et al*., 2015; Cintra *et al*., 2017; Silva *et al*., 2017). O cimento de cinco óxidos minerais, 5MO (Golden Yatti LLC, Muscat, Oman), também é derivado do cimento Portland e foi desenvolvido com o intuito de tratar complicações endodônticas. Este cimento se mostrou efetivo em casos de capeamento pulpar, apicectomia e selamento de perfurações (Hasna, *et al*., 2022). Na sua composição encontramos o óxido de titânio (TiO2), além dos óxidos de silício, cálcio, alumínio e magnésio. Embora não haja informações disponíveis sobre o efeito direto do óxido de titânio presente no 5MO, várias pesquisas demonstraram que o titânio puro é um material atóxico e apresenta excelente biocompatibilidade (Heravi, *et al*., 2013).

Por ser um material novo, as propriedades físico-químicas do cimento 5MO foram pouco exploradas, mas torna-se mais uma opção de material biocerâmico reparador viável no cotidiano clínico e objeto de estudo para pesquisas.

1.1 OBJETIVO GERAL

O objetivo geral deste estudo será analisar as propriedades físico-químicas dos cimentos obturadores à base de silicato de cálcio, o 5MO (Golden Yatti LLC, Muscat, Oman) que contém cinco óxidos em comparação ao MTA branco (Angelus Indústria de produtos Odontológicos S/A, Londrina, Brasil), considerado padrão ouro.

2 ARTIGO

RESEARCH ARTICLE

Experimental study comparing the physicochemical properties and surface characterization of 5MO and White MTA

Larissa C. Novaes Batista[1], Jaqueline Martins Crivelari[1], João Vitor da Cruz Pegoraro[1], Ana Cristina Padilha Janini[2], Marina A. Marciano[2], Camila Soares Lopes[1], Bruno Martini Guimarães[1]

[1] School of Dentistry, Federal University of Alfenas (Unifal-MG), Alfenas, Minas Gerais, Brazil.

[2] Department of Restorative Dentistry - Endodontics, Piracicaba School of Dentistry, University of Campinas (Unicamp), Piracicaba, São Paulo, Brazil

Corresponding author:

Bruno Martini Guimarães

School of Dentistry, Federal University of Alfenas (Unifal-MG), Gabriel Monteiro da Silva Street, 700 - Zip Code: 37130-000 - Alfenas - Minas Gerais - Brazil.

Phone: 55 14 99181 7474. E-mail: bruno.guimaraes@unifal-mg.edu.br

---

1 Artigo científico elaborado e submetido de acordo com as normas do periódico internacional Journal of Biomedical Materials Research Part B.

ABSTRACT

Repairing endodontic materials play a crucial role in maintaining dental functionality by improving healing outcomes and preventing extractions. Mineral trioxide aggregate (MTA), a calcium silicate-based cement, stands out for its bioactivity and biocompatibility, being considered the gold standard in endodontic repairs. However, new materials have been developed, such as the five mineral oxides cement (5MO). This study aimed to compare the physicochemical properties of 5MO and White MTA, focusing on pH, setting time, solubility, chemical composition, and surface characterization. The pH was analyzed at 37°C at 3, 12, 24, 72 hours, and 7, 14, 21 days, and the setting time test followed ISO 6876-2012 standard. For the solubility test, samples were weighed, stored in distilled and deionized water for 24 hours, dried, and weighed again. Solubility was calculated using the samples' weight loss (%). Surface analysis of the cements was performed by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), and chemical analysis by Fourier-transform infrared spectroscopy (FTIR) (37-100°C). Data were analyzed using Shapiro-Wilk and Student's t-test ($p < 0.05$). Both materials maintained an alkaline pH throughout the study period, essential for antimicrobial activity and tissue mineralization. However, White MTA maintained higher alkalinity at 21 days, indicating potential long-term stability advantages. Setting time analysis revealed that 5MO has a significantly longer setting time compared to White MTA, which may impact clinical handling efficiency. Furthermore, 5MO showed higher solubility after immersion in PBS, potentially compromising material stability and sealing effectiveness over time. Surface characterization using SEM/EDS highlighted distinct particle morphologies and compositions, while FTIR analysis further differentiated chemical bonds and hydration products between the cements. In conclusion, although 5MO exhibits promising alkalinity and elemental composition favorable for tissue regeneration, its longer setting time and higher solubility compared to White MTA may limit its clinical utility.

Keywords: Dental materials; chemical properties; calcium silicate; Regenerative endodontics.

INTRODUCTION

Repair procedures are crucial in endodontics as they help in maintaining teeth in functional conditions[51]. Bioactive materials play a significant role in pulp and repair procedures in order to improve healing outcomes and reduce the need for extraction[45]. Hydraulic cements are particularly notable for their ability to form apatite at the material-dentine interface in the presence of moisture[8]. Calcium silicate-based cements, for example, are commonly used in regenerative endodontic treatment due to their biocompatibility and bioactivity[27]

Mineral Trioxide Aggregate (MTA) is a calcium silicate-based material widely used in repair procedures in endodontics[24], considered the gold standard repair material with promising clinical outcomes[8]. It is primarily composed of tricalcium silicate, dicalcium silicate, and a radiopacifying agent[44]. The performance of MTA is attributed to its bioactivity[8], sealing ability[43], expansion[32], moisture retention, and release of hydroxyl and calcium ions, creating an alkaline pH that is unfavorable for bacterial growth and promotes mineralized barrier formation in adjacent tissues[42]. While MTA (WMTA) has been extensively studied for its physical, chemical, and biological properties over the years, there is ongoing research to further enhance its characteristics as an ideal repair material[53,31,33].

An ideal endodontic repair cement should possess physical characteristics such as dimensional stability, color[11,41,26], radiopacity[37], suitable setting time, resistance to solubility[29,16], flow for proper sealing[25,26], as well as chemical and biological properties including alkalinity, calcium ion release[23], bioactivity[34], cellular adhesion[6], biocompatibility[35,9], and antimicrobial properties[56]. WMTA demonstrates many of these ideal properties, there is still room for improvement[44].

New materials such as the Five Mineral Oxides cement (5MO), derived from Portland cement[1], have shown promise in in vitro studies as a non-cytotoxic material with antimicrobial properties[1,2]. In clinical applications, 5MO has proven effective as a retrofilling material[28] and repair material for perforations[5,3]. Although the physical-chemical properties of 5MO are still being explored, it represents a viable bioceramic repair material option for both clinical practice and research purposes. The literature also does not present works comparing these properties with those of MTA.

The development of biocompatible and bioactive materials in Endodontics signifies a significant advancement with promising prospects for clinical use[24]. However, careful consideration and prior studies are essential in selecting a new endodontic material based on physical-chemical properties, which can influence its biological behavior and clinical prognosis[14]. The aim of this study was to analyze and compare 5MO with WMTA in terms of their physical-chemical properties including pH, setting time, solubility, and surface characterization, with the null hypothesis being that there would be no difference between these two repair cements.

MATERIALS AND METHODS

The materials used in the study were the WMTA (Angelus, Londrina, Paraná, Brazil) and 5MO (GOLDEN YATTI LLC, MUSCAT, OMAN). The preparation of WMTA was done using 0,145g of powder (1 spoon) to one drop of liquid. The cements was mixed in a glass plate using a metallic spatula for 40 seconds to obtain a homogeneous consistency, as recommended by the manufacturer. For 5 MO, was used the ratio 3:1 (powder: water) and mixed for 30 seconds. The root canal sealers were mixed in accordance with the instructions provided by the manufacturers (Table 1).

Table 1- The repair materials evaluated, their respective manufacturers, chemical composition and proportions utilized.

	Manufacturers	Composition	Proportions
5MO	Golden Yatt LLC Registration N° 31211591	Powder: Calcium oxide, silicate oxide, titanium oxide, aluminum oxide, and magnesium oxide. Liquid: distilled water	1g powder / 0.33ml distilled water
WMTA	Angelus Indústria de Produtos Odontológicos S/A. CNPJ 00.257.992/0001-37.	Powder: Tricalcium silicate, dicalcium silicate, tricalcium aluminate, calcium oxide, calcium tungstate Liquid: distilled water.	0.145g powder/ 0.25 ml distilled water

Physicochemical properties

pH

The pH of the cements was determined after 3, 12, 24, 72 hours, 7, 14, 21 days. 10 samples from each group were used, in accordance with other studies found in the literature. The samples were prepared using polyethylene tubes (10 x 1 mm) filled by the materials WMTA and 5MO. Each tube was immersed in plastic containers with 10

ml of distilled water and then stored at 37°C with a relative humidity of 95%. At each period, the tubes were removed from the plastic container and placed in a new one with 10 mL of distilled water. The pH analysis was performed by using the previously calibrated Ultrabasic pH metre (Denver Instrument Company, Arvada, Colorado, USA)[19].

Setting time

Setting time was determined by using microgranulated plaster molds (Durone-IV; Dentsply, Petrópolis, Rio de Janeiro, Brazil) that were previously manufactured (10 x 1mm) and immersed in distilled water for 24 hours before testing. The sample consisted of 6 elements from each group, also following examples from the literature. The plaster molds were filled with the sealers WMTA and 5MO after manipulation. Periodically, a 100 ± 0.5 g Gilmore needle with a diameter of 2 ± 0.1 mm was placed vertically on the surface of the sample to determine the setting time according to ISO 6876:2012. Throughout the analysis, the materials were kept in an oven, and the needle was cleaned between analyses. Setting times were determined as the period between manipulation and the moment when the needle no longer produced marks on the surface of the cements[13.]

Solubility

The sample was also composed of 6 elements from each group, according the literature. Circular molds measuring 1.5 mm in height and 7.75 mm in internal diameter were made[15] and filled with the materials and with a nylon thread included in the mass cement. The samples were stored in an oven at a temperature of 37°C for a period of time three times the setting time of each material. The specimens were weighed on an HM-200 precision balance (A & D Engineering, Inc., Bradford, MA) to obtain their initial mass The specimens were weighed on an HM-200 precision balance (A & D Engineering, Inc., Bradford, MA) to obtain their initial mass and suspended using nylon threads inside plastic containers with lids containing 7 .5 mL phosphate-buffered saline solution (PBS), and kept in an oven at 37°C for 7 days. After these periods, the specimens were removed from the solution and placed in a desiccator. The final mass was measured every 24 hours until the mass stabilized. Mass loss was expressed as a percentage of the original mass.

Surface characterization

Scanning Electron Microscopy/Energy Dispersive X-Ray (SEM/EDX)

For surface characterization, Scanning Electron Microscopy (SEM) analysis coupled to the Energy Dispersive Spectrometer (EDS – Energy Dispersive System) was used. Three samples of each cement (10mm diameter x 1mm thick) were prepared and after setting time at 37°C and 95% humidity, each sample was carbon- coated separately, using double-sided carbon conductive tape. These samples were placed on a platinum SEM plate and the assembly was placed in the hood of the Emitech K450 metallizer for 1 minute and 30 seconds under a current of 50 mA. In this step, a thin layer of conductive element, in this case carbon, was deposited on the surface so that the electrons could be conducted. Then, the samples were taken to the Scanning Electron Microscope (SEM), which had an energy dispersive spectrometer (EDS) attached for semi-quantitative analysis. The beam energy was maintained at 20 kV and the beam current reached the samples with values of 6000 pA, as needed to adjust the brightness/contrast and quality (counts) of the EDS analyzes[54]. A magnification of 1000 times was performed.

FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR spectra were obtained using potassium bromide (KBr) pellets with 0.05 g of each material and 0.1 g of KBr. The mixture was immediately transferred to a 13 mm diameter pressing die, where it was placed under vacuum in a 10-ton RIIK ring pressing machine for 1 min. Spectra were obtained using a calibrated FT-IR spectrometer (Vertex 70v; Bruker, Billerica, MA, USA) operating in normal transmittance mode, spectral resolution of 4 cm À1 and range of 400 to 4000 cm À1 under vacuum. FTIR spectral data does not require signal processing[47]. The sample number was 3 elements for each group of cements.

Statistical analysis

All data was analyzed with GraphPad Prism 9.02 (GraphPad Software, Inc., CA, USA) statistical program ($\alpha = 0.05$). The normal distribution of these data was confirmed using the Shapiro-Wilk normality test and the physicochemical properties were submitted to Student's t test.

RESULTS

In all time periods, both WMTA and 5MO promoted an alkaline pH. At 21 days, WMTA was significantly more alkaline than 5MO ($p=0.004$), demonstrating greater stability over time; in other periods, there was no significant difference between the cements ($p>0.05$).

Acording to table 2, both repair materials exhibited mass loss after immersion in PBS. WMTA had lower mass loss than 5MO ($p = 0.0107$). Regarding setting time values, there was no difference between the cements, with 5MO demonstrating the longest setting time ($p < 0.0001$).

Table 2 – pH, solubility and setting time values (mean and standard deviation) of W MTA and 5MO.

	pH values							**Solubillity** (% mass loss)	**Seeting Time** (minutes)
	3 hours	12 hours	24 hours	72 hours	7 days	14 days	21 days		
W MTA	10.96±0.44[a]	10.73±0.49[a]	10.46±0.43[a]	10.54±0.57[a]	9.6±0.54[a]	8.85±0.75[a]	9.50 ±0.8[a]	4.04±1.23[b]	51.02±3.94[b]
5MO	10.56±0.67[a]	10.60±0.25[a]	10.53±0.39[a]	10.45±0.58[a]	9.2±0.67[a]	8.75±0.59[a]	8.6±0.3[b]	6.02±1.46[a]	70.76±2.93[a]

Different letters on the same column represent a significant difference between **materials** ($p < 0.05$)

In the analysis of cements using Energy Dispersive Spectroscopy (EDS), the Figure 1 (A) shows the spectrum for 5MO sample, showing that this material is primarily composed of carbon, oxygen, calcium, and silicon, along with smaller amounts of strontium, barium, aluminum, titanium, bismuth, sulfur, magnesium, and iron. In contrast, in Figure 1 (B), the spectrum for WMTA shows that this cement is predominantly composed of oxygen, carbon, calcium, and tungsten, but also contains smaller quantities of silicon, strontium, and aluminum. The presence of magnesium, sulfur, titanium, iron, barium, and bismuth in 5MO, as well as the absence of tungsten, makes the composition of this cement different from WMTA. In the Table 3, which represents the chemical composition of both materials, it is possible to observe that similarity occurs only in the presence of carbon, oxygen, aluminum, silicon, calcium, and strontium in both cements.

Table 3 – Chemical Composition of 5MO and W MTA according to EDX

Elemento	%5MO	%WMTA
C	50,13	19,87
O	36,87	39,79
Mg	0,09	
Al	0,74	0,93
Si	3,93	35
S	0,19	
Ca	5,14	20,67
Ti	0,24	
Fe	0,06	
Sr	1,49	2,27
Ba	0,92	
Bi	0,19	
W		13,14

Image captured by Scanning Electron Microscopy (SEM) observed in Figure 2 (A) reveal that 5MO exhibits an irregular surface with small, lighter-colored particles prominently visible. In contrast, WMTA in Figure 2 (B) shows a surface composed of particles of various sizes, more elongated, with white spots among a grayish coloration.

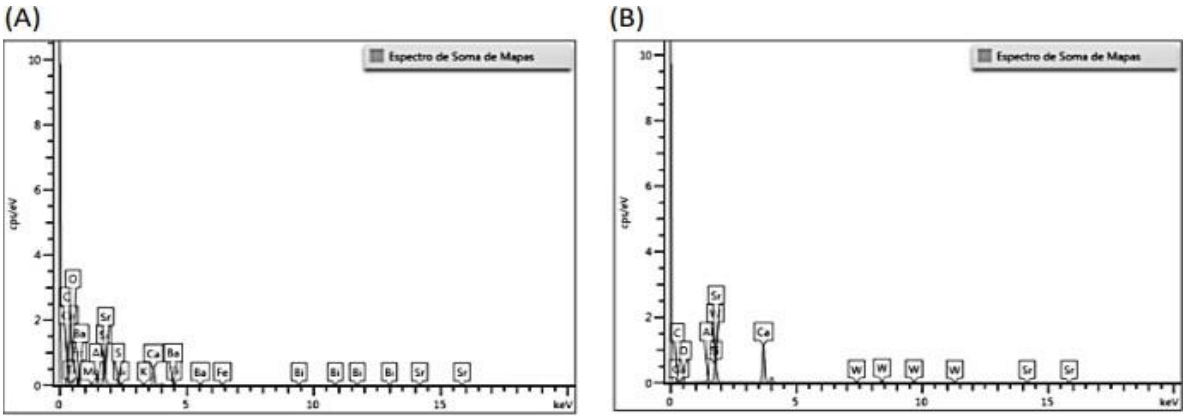

Figure 1: EDS Spectrum. (A) 5MO Sample; (B) WMTA Sample

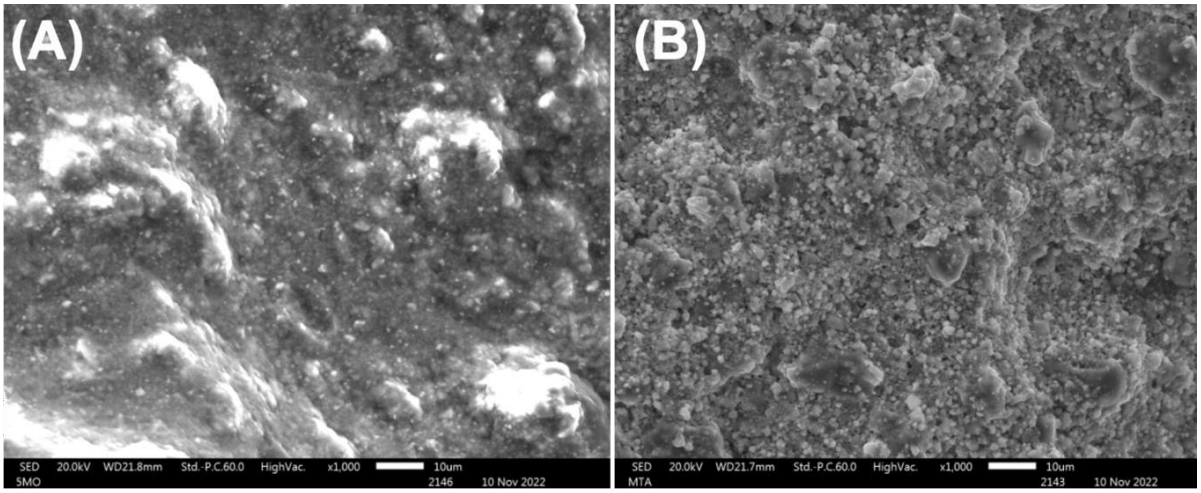

Figure 2: Scanning electron microscope photomicrograph at 1000x magnification. (A) 5MO sample; (B) WMTA sample.

The figure 3 displays FTIR spectra for WMTA and 5MO. An absorption band at 1400 cm-1 related to the C-O group was observed. The band at 3200 cm-1 corresponds to the C-H group, which is prominent in hydrated materials. Furthermore, a significant peak ranging from 2750 to 3250 cm-1, attributed to the O-H group present

in the cement composition, was noted. Bands at 750 and 1150 cm-1 were associated. The 5MO graph presents the same peaks as that of MTA but with more noise, which may be associated with the impurity of the formulation.

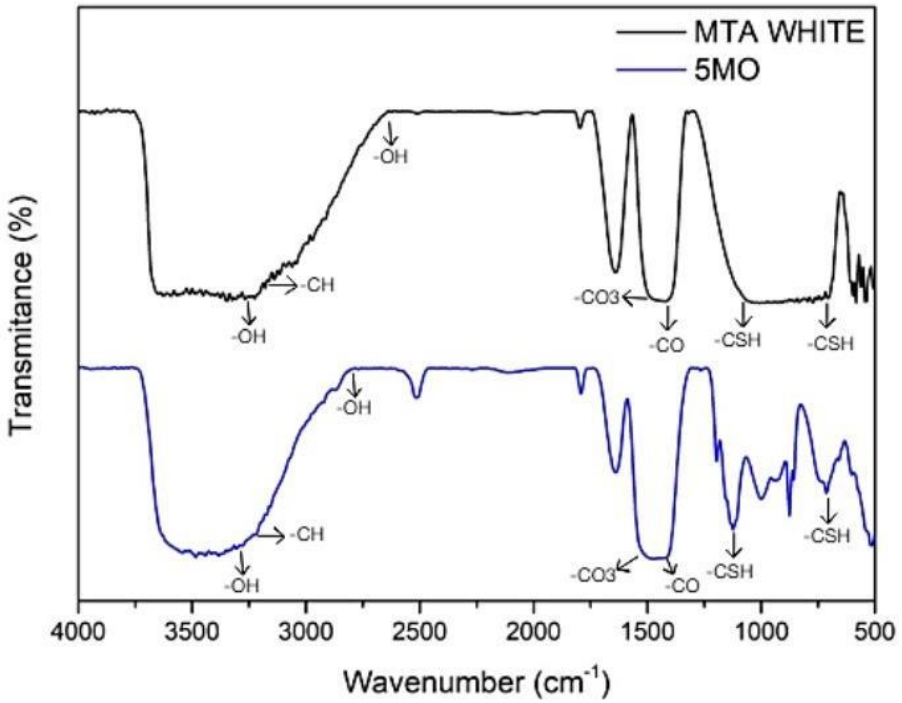

Figure 3: FTIR spectra of 5MO and WMTA.

DISCUSSION

The current study evaluated the physicochemical, morphological structure, and chemical composition properties of 5MO compared to WMTA. Significant differences were found in the physicochemical properties of the materials, leading to the rejection of the null hypothesis. 5MO exhibited a longer setting time and higher solubility, but lower alkalinity after 21 days.

The release of hydroxyl and calcium ions results in the alkaline pH of the medium. The composition, proportion of silicates, and particle sizes have a significant influence on the hydration process of the material and the release of calcium hydroxide. During the hydration of calcium silicate and calcium oxide[12], the synthesis of calcium hydroxide occurs, with a continuous release of calcium ions and hydroxyl ions[27].WMTA contains calcium silicates and calcium oxide in its composition, while 5MO cement only contains oxides in its formulation. This could explain the significant decrease in alkalinity of the medium after 21 days compared to MTA.

Throughout all periods, both materials maintained an alkaline pH in the medium, which plays a significant role in the antibacterial activity and osteogenic potential of the materials[27]. This alkaline environment creates favorable conditions for tissue regeneration and infection prevention, crucial characteristics in endodontics[13]. WMTA initially raised the pH of the medium, which then varied between 10.96 and 8.85, consistent with findings from previous studies[13,19].

In this study, 5MO demonstrated a longer setting time and greater mass loss compared to other related studies on setting time and solubility of calcium silicate-based endodontic materials[49]. The significant mass loss observed in the 5MO material can be attributed to the presence of calcium oxide in its composition, which contributes to its high solubility[58,7] similar to materials containing calcium hydroxide[58].

Both repair materials exhibited mass loss after being immersed in PBS, with a solubility exceeding the recommended 3% according to ISO 6876:2012 standards. Another study also reported mass loss for MTA White[13]. Materials with extended setting times are more prone to dissolution during endodontic procedures[20] and those with high solubility may lead to inadequate sealing and the presence of gaps in the filling[16], potentially causing reinfection and compromising the prognosis.

The liquid utilized for immersion can interact with the material, potentially impacting the results obtained in solubility tests[52]. The cements utilized in this study are hydrophilic and rely on humidity to set[27]. Consequently, the solubility methodology outlined by Carvalho-Júnior[15] was adopted, with PBS selected as the immersion medium as it closely mimics physiological conditions[57.]

Chemical analysis and characterization of different materials were conducted using a combination of SEM/EDX and FTIR analyses. In this study, scanning electron micrographs of WMTA revealed a more uniform and homogeneous surface compared to 5MO. While there are no existing studies in the literature comparing these specific materials, other studies employing similar methodologies have noted that WMTA exhibits multiple aggregates of large, round particles with an elongated shape[17,40]. The size and distribution of particles in the composition of endodontic materials can significantly impact their characteristics[40]; specifically, larger particles tend to have excellent hydration capability[12]. At 5MO the particles showed more evident this could be related to the high solubility and setting time of this material observed in the present study.

Rocha et al.[50] analyzed the FTIR spectra of MTA Angelus and identified absorption bands at 660, 896, and 1410 cm^{-1} associated with C-O. In the present study, absorption bands at 1400 cm^{-1} were observed for "WMTA" and "5MO". Abu Zeid et al.[4] demonstrated bands at 2800-2900 cm^{-1} related to the C-H group, which were also evident at 3200 cm^{-1} in both materials analyzed in this study. Following material setting, the formation of CO3 resulted in peaks at 1440 and 1472 cm^{-1}, as observed at 1600 cm^{-1} in this study. Bands at 449, 524, and 996 cm^{-1} indicated the presence of hydrated calcium silicate in WMTA which were detected at 750 and 1150 cm^{-1} in the sample examined in this study. Furthermore, Mahmoud et al.[40] identified a broad peak between 3000-3600 cm^{-1} attributed to the water O-H group in fully set materials for MTA Angelus, suggesting the formation of hydrated phases such as calcium hydroxide and hydrated calcium silicate as reaction products. In this study, significant peaks of this O-H group were observed at 2750 and 3250 cm^{-1}

Analyzing aspects of Scanning Electron Microscopy (SEM) coupled with Energy Dispersive Spectrometer (EDS), Camilleri et al.[10] found that "WMTA" is composed of silicon, calcium, and oxygen, with the latter two being more concentrated. Hasna[1] demonstrated in their study the presence of titanium, sulfur, potassium, and the

absence of tungsten in "5MO". Furthermore, according to Sarzeda et al.[54], calcium and oxygen elements form calcium oxide, which subsequently transforms into calcium hydroxide, enabling these repair materials to effectively induce remineralization. The chemical composition data from previous studies are also observed in this work, where White MTA has 39.79% oxygen and 20.67% calcium, consistent with the aforementioned authors. Additionally, comparing White MTA and 5MO cements, it was found that both contain carbon, oxygen, aluminum, silicon, calcium, and strontium. However, it was noted that WMTA also contains tungsten, probably related to its radiopacifier, while 5MO contains titanium, iron, barium, and bismuth. It was also observed that White MTA has a high percentage of calcium in its composition. In 5MO, calcium is present in smaller quantities, with carbon standing out at 50.13% in its composition.

5MO exhibited an alkaline pH and the presence of calcium and oxygen ions, which are crucial for antimicrobial activity and for inducing the formation of mineralized tissue during repair[1]. However, in the current study, this material displayed higher mass loss and setting time compared to WMTA indicating potential drawbacks that could hinder its clinical utility. Surface characterization and FTIR tests show that 5MO appears to lack purity in its formulation, which may lead to the aforementioned disadvantages.

CONCLUSION

In conclusion, this study comprehensively evaluated and compared the physicochemical characteristics of 5MO and WMTA as potential endodontic repair materials. Substantial variations were observed between the two cements in several parameters, contradicting the initial hypothesis. Although 5MO presents favorable properties such as high pH and satisfactory repair elements, the lack of purity in its formulation contradicts the advances that MTA has undergone.

REFERENCES

1. Roig M, Espona J, Mercadé M., Duran-Sindreu F. Horizontal root fracture treated with MTA, a case report with a 10-year follow-up. *Dental Traumatoly*. 2011; **27**(6): 460-3. doi: 10.1111/j.1600-9657.2011.01018.x
2. Primus C, Tay FR, Niu LN. Bioactive Tri/dicalcium Silicate Cements for Treatment of Pulpal and Periapical Tissues. *Acta Biomater*. 2019; **96**: 35-54. doi: 10.1016/j.actbio.2019.05.050
3. Camilleri J, Atmeh A, Li X, Meschi N. Present status and future directions: Hydraulic materials for endodontic use. *Int Endod J*. 2022; **55**(S3): 710 – 77. doi: 10.1111/iej.13709
4. Estrela C, et al. Mechanism of action of Bioactive Endodontic Materials. *Braz Dent J*. 2023; **34**(1):1-11. doi: 10.1590/0103-6440202305278
5. Duarte MAH, et al. Tricalcium silicate-based cements: properties and modifications. *Braz Oral Res*. 2018; **32**(suppl 1): e70. doi: 10.1590/1807- 3107bor-2018.vol32.0070
6. Parirokh M, Torabinejad M. Mineral Trioxide Aggregate: A Comprehensive Literature Review-Part I: Chemical, Physical, and Antibacterial Properties. *J Endod.* 2010; **36**: 16 - 27. doi: 10.1016/j.joen.2009.09.006
7. Medeiros PL, et al. Sealing ability of MTA, CPM, and MBPc as root-end filling materials: a bacterial leakage study. *J Appl Oral Sci*. 2016; **24**: 148 - 52. doi: 10.1590%2F1678-775720130103
8. Gandolfi MG, et al. Setting time and expansion in different soaking media of experimental accelerated calcium-silicate cements and ProRoot MTA. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod*. 2009; **108**: e39- 45. doi: 10.1016/j.tripleo.2009.07.039
9. Marciano MA, et al. Physical and Chemical Properties and Subcutaneous Implantation of Mineral Trioxide Aggregate Mixed with Propylene Glycol. *J Endod*. 2016; **42**(3): 474-9. doi: 10.1016/j.joen.2015.10.014
10. Santos AD, et al. Physico-chemical properties of MTA and a novel experimental cement. *Int Endod J*. 2005; **38**(7): 443-7. doi: 10.1111/j.1365- 2591.2005.00963.x
11. Gandolfi MG, et al. Development of the foremost light-curable calcium-silicate MTA cement as root-end in oral surgery. Chemical-physical properties,

bioactivity and biological behavior. *Dental Materials*. 2011; **27**(7): e134-57. doi: 10.3390/ijms232213914

12. Gandolfi MG, Siboni F, Prati C. Chemical-physical properties of TheraCal, a novel light-curable MTA-like material for pulp capping. *International Journal of Endodontics*. 2012; **45**(6): 571-79. doi: 10.1111/j.1365-2591.2012.02013.x
13. Camilleri J, Mallia B. Evaluation of the dimensional changes of mineral trioxide aggregate sealer. *International Journal of Endodontics*. 2011; **44**(5): 416-24. doi: 10.1111/j.1365-2591.2010.01844.x
14. Marciano MA, et al. Assessment of color stability of white MTA Angelus and bismuth oxide in contact with tooth structure. *Journal of Endodontics*. 2014; **40**(8): 1235-40. doi: 10.1016/j.joen.2014.01.044
15. Duque JA, et al. The effect of mixing method on tricalcium silicate-based cement. International *Journal of Endodontics*. 2018; 51(1): 69-78. doi: 10.1111/iej.12774
16. Islam I, Chng HK, Yap AUJ. Comparison of the physical and mechanical properties of MTA and Portland cement. *Journal of Endodontics*. 2006; **32**(3): 193-97. doi: 10.1016/j.joen.2005.10.043
17. Fridland M, Rosado R. Mineral trioxide aggregate (MTA) solubility and porosity with different water-to-powder ratios. *Journal of Endodontics*. 2003; 29(12): 814-17. doi: 10.1097/00004770-200312000-00007
18. Cavenago BC, et al. Influence of powder-to-water ratio on radiopacity, setting time, pH, calcium ion release and a micro-CT volumetric solubility of white mineral trioxide aggregate. *Int Endod J.* 2014; **47**(2):120–126. doi: 10.1111/iej.12120
19. Duarte MAH,et al. Evaluation of the propylene glycol association on some physical and chemical properties of mineral trioxide aggregate. International *Journal of Endodontics*. 2012; **45**(6): 565-70. doi: 10.1111/j.1365- 2591.2012.02012.x
20. Duarte MAH, et al. pH and calcium ion release of 2 root-end filling materials. Oral Surgery, Oral Medicine, Oral Pathology, *Oral Radiology and Endodontology*. 2003; **95**(3): 345-47. doi: 10.1067/moe.2003.12
21. Gandolfi MG, Taddei P, Tinti A, Prati C. Apatite-forming ability (bioactivity) of ProRoot MTA. *International Journal of Endodontics*. 2010; **43**(10): 917-29. doi: 10.1111/j.1365-2591.2010.01768.x

22. Balto HA. Attachment and morphological behavior of human periodontal ligament fibroblasts to mineral trioxide aggregate: a scanning electron microscope study. *Journal of endodontics*. 2004; 30(1): 25-29. doi: 10.1097/00004770-200401000-00005
23. Holland R, et al. Reaction of rat connective tissue to implanted dentin tubes filled with a 30 white mineral trioxide aggregate. *Brazilian Dental Journal*. 2002; **12**(1): 3-8. PMID: 11870957
24. Camilleri J, et al. Biocompatibility of two commercial forms of mineral trioxide aggregate. International *Journal of Endodontics*. 2004; 37(10): 699-704. doi: 10.1111/j.1365-2591.2004.00859.x
25. Tanomaru-Filho M, et al. Radiopacity Evaluation of New Root Canal Filling Materials by Digitalization of Images. *Journal of endodontics*. 2007; **33**: 249-51. doi: 10.1016/j.joen.2006.08.015
26. Gandolfi MG, et al. Calcium silicate and calcium hydroxide materials for pulp capping: biointeractivity, porosity, solubility and bioactivity of current formulations. *Journal of Applied Biomaterials and Functional Materials*. 2015; **13**(1): 43-60. doi: 10.5301/jabfm.5000201
27. Cintra LTA, et al. Cytotoxicity, biocompatibility, and biomineralization of the new high-plasticity mta material. *J Endod*. 2017; **43**(5):774-778. doi: 10.1016/j.joen.2016.12.018
28. Silva EJNL, et al. Push-out Bond Strength of Fast-setting Mineral Trioxide Aggregate and Pozzolan-based Cements: ENDOCEM MTA and ENDOCEM Zr. *J Endod*. 2017; **43**(5):801-804. doi: 10.1016/j.joen.2016.12.007
29. Abu Hasna A, et al. Biological and chemical properties of five mineral oxides and of mineral trioxide aggregate repair high plasticity: an in vitro study. *Sci Rep*. 2022; **12**(1): 14123. doi: 10.1038/s41598-022-17854-0
30. Abu Hasna A, et al. Antimicrobial Action, Genotoxicity, and Morphological Analysis of Three Calcium Silicate-Based Cements. *Biomed Res Int*. 2022; **2022**: 1-7. doi: 10.1155/2022/2155226
31. Flores Orozco EI, et al. Case Report: Interdisciplinary management of a complex odontoma with a periapical involvement of superior anterior teeth. *F1000Res*. 2019; **8**:1531. doi: 10.12688/f1000research.20337.1
32. Al-Nahlawi T., Ala Rachi M., Abu H. A. Endodontic perforation closure by five mineral oxides silicate-based cement with/without collagen sponge

matrix. *International Journal of Dentistry.* 2021; **2021**:8. doi: 10.1155/2021/4683689

33. Abu Hasna A., et al. Apicoectomy of perforated root canal using bioceramic cement and photodynamic therapy. *Journal of Dentistry*. 2020; **2020**:1. doi: 10.1155/2020/6677588
34. Cardinali F, Camilleri J. A critical review of the material properties guiding the clinician's choice of root canal sealers. *Clin Oral Investig* 2023; **27**(8):4147-55. doi: 10.1007/s00784-023-05140-w
35. Coaguila – Llerena H, et al. Physicochemical properties of a bioceramic repair material BioMTA. *Brazilian Dental Journal*. 2020; **31**: 511-515. doi: 10.1590/0103-6440202003163
36. Campi LB, et al. Physicochemical properties, cytotoxicity and bioactivity of a ready-to-use bioceramic repair material. *Brazilian dental jornal*. 2023; **34**: 29-38. doi: 10.1590/0103-6440202304974
37. Carvalho Junior J, et al. Solubility and Dimensional Change after Setting of Roof Canal Sealers: A Proposal for Smaller Dimensions of Test Samples. *Journal of Endodontics*. 2007, 33(9): 1110-16. doi: 10.1016/j.joen.2007.06.004
38. Sarzeda GDR, et al. Análise da composição química dos cimentos MTA Angelus branco, cinza e HP Repair através de Microscopia Eletrônica de Varredura (MEV) acoplada a Espectrômetro de Energia Dispersiva (EDS). *Revista de Odontologia UNESP*. 2019; **48**: e20190093. doi: 10.1590/1807- 2577.09319
39. Qiu L, Hurt AP, Coleman NJ. The Aplication of 29Si NMR Spectroscopy to the Analysis of Calcium Silicate-Based Cement using Biodentine as an Example. *Jounal of Functional Biomaterials*. 2019; **10**(2): 25. doi: 10.3390/jfb10020025
40. Kato G, et al. Ex Vivo Osteogenesis Induced by Calcium Silicate-Based Cement Extracts. *J Funct Biomater*. 2023; **14**(6):314. doi: 10.3390/jfb14060314
41. Camilleri J, Sorrentino F, Damidot D. Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, Biodentine and MTA Angelus. *Dent Mater*. 2013; **29**(5):580-93. doi: 10.1016/j.dental.2013.03.007
42. Queiroz MB, et al. Development and evaluation of reparative tricalcium silicate-ZrO_2 - Biosilicate composites. *J Biomed Mater Res B Appl Biomater*. 2021; **109**(4):468-476. doi: 10.1002/jbm.b.34714

43. Vivan RR, et al. Evaluation of the physical and chemical properties of two commercial and three experimental root-end filling materials. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod*. 2010; **110**(2):250-6. doi: 10.1016/j.tripleo.2010.04.021
44. Bosso-Martelo R, et al. Physicochemical properties of calcium silicate cements associated with microparticulate and nanoparticulate radiopacifiers. *Clin Oral Investig*. 2016; **20**(1):83-90. doi: 10.1007/s00784-015-1483-7
45. Húngaro Duarte MA, et al. Effect of different radiopacifying agents on the physicochemical properties of white Portland cement and white mineral trioxide aggregate. *J Endod*. 2012; **38**:394–397. doi: 10.1016/j.joen.2011.11.005
46. Liu WN, Chang J, Zhu YQ, Zhang M. Effect of tricalcium aluminate on the properties of tricalcium silicate-tricalcium aluminate mixtures: setting time, mechanical strength and biocompatibility. *Int Endod J*. 2011; **44**(1):41-50. doi: 10.1111/j.1365-2591.2010.01793.x
47. Collares FM, et al. Influence of radiopaque fillers on physicochemical properties of a model epoxy resin-based root canal sealer. *J Appl Oral Sci.* 2013; **21**(6):533–539. doi: 10.1590%2F1679-775720130334
48. Rosatto CMP, et al. Physicochemical properties and osteoclastogenesis for three premixed calcium silicate-based sealers post set. *Braz Oral Res*. 2022; **36**:e065. doi: 10.1590/1807-3107BOR-2017.vol31.0061
49. Torres FFE, et al. Effect of immersion in distilled water or phosphate-buffered saline on the solubility, volumetric change and presence of voids within new calcium silicate-based root canal sealers. *Int Endod J*. 2020; **53**(3):385-391. doi: 10.1590/1807-3107bor-2021.vol35.0028
50. Chang SW. Chemical Composition and Porosity Characteristics of Various Calcium Silicate-Based Endodontic Cements. *Bioinorg Chem Appl*. 2018; **2018**:2784632. doi: 10.1155/2018/2784632
51. Mahmoud O, et al. Morphological and Chemical Analysis of Different Types of Calcium Silicate-Based Cements. *International Journal of Dentistry*. 2022; **2022**: 1-16. doi: 10.1155/2022/6480047
52. Rocha AC, et al. Physicochemical analysis of MTA Angelus® and Biodentine® conducted with X ray diffraction, dispersive energy spectrometry, X ray fluorescence, scanning electron microscope and infrared spectroscopy.

Revista Odontológica Mexicana. 2015; **19** (3): e170-176. doi: 10.1016/j.rodmex.2016.02.023

53. Abu Zeid ST, et al. Chemistry and bioactivity of NeoMTA Plus™ versus MTA Angelus® root repair materials. *Journal of Spectroscopy*.2017, **2017**: 1-9. doi: 10.1155/2017/8736428
54. Camilleri J, et al. The constitution of mineral trioxide agreggate. *Dental Material*. 2005; **21**:(4): 297-303. doi: 10.1016/j.dental.2004.05.010

3 CONSIDERAÇÕES FINAIS

O presente trabalho teve como objetivo fazer a análise físico-química de um novo material biocerâmico contendo 5 óxidos (5MO) em comparação ao MTA Branco (Angelus), sendo este considerado padrão ouro.

Com base na análise comparativa entre o MTA Branco e o cimento 5MO, observa-se que ambos os materiais apresentam propriedades físico-químicas distintas, as quais podem influenciar sua efetividade clínica e estabilidade a longo prazo.

O MTA Branco demonstrou maior manutenção da alcalinidade após 21 dias, indicando uma possível superioridade em termos de durabilidade. Ademais, apresentou um tempo de presa significativamente reduzido em relação ao 5MO, característica que pode favorecer o manuseio clínico e otimizar a eficiência dos procedimentos terapêuticos.

Por sua vez, o cimento 5MO revelou uma alcalinidade adequada e uma composição elementar potencialmente benéfica à regeneração tecidual. Contudo, o tempo de presa mais prolongado e a maior solubilidade observada após a imersão em solução de PBS podem representar limitações quanto à sua estabilidade dimensional e capacidade de selamento ao longo do tempo.

.

REFERÊNCIAS

AL-NALAHWI, T.; RACHI M. A.; HASNA, A. A. Endodontic perforation closure by five mineral oxides silicate-based cement with/without collagen sponge matrix. **International Journal of Dentistry**, v. 2021, p. e4683689, 2021.

BALTO, H. A. Attachment and morphological behavior of human periodontal ligament fibroblasts to mineral trioxide aggregate: a scanning electron microscope study. **Journal of endodontics**, v. 30, n. 1, p. 25-29, 2004.

BANCAS, F.; TROPE, M. Revascularization of immature permanent teeth with apical periodontitis**:** new treatment protocol? **Journal of endodontics**, v. 30, n. 4, p. 196-200, 2004.

BARRET, W. C. Pulp Capping. **Amerian Journal od Dental Science**, v. 10, n. 12, p. 552-55, 1877.

CAMILLERI, J. *et al*. Biocompatibility of two commercial forms of mineral trioxide aggregate**. International Journal of Endodontics**, v. 37, nº. 10, p. 699-704, 2004.

CAMILLERI, J.; MALLIA, B. Evaluation of the dimensional changes of mineral trioxide aggregate sealer. **International Journal of Endodontics**, v. 44, n. 5, p. 416-24, 2011.

CAMILLERI, J.; MONTESIN, F. E.; BRADY, K.; SWEENEY, R.; CURTIS, R. V.; FORD, T. R. The constitution of mineral trioxide agreggate. **Dental Materialn**, v. 21, n. 4, p. 297-303, 2005.

CAMPI, L. B. *et al*. Physicochemical properties, cytotoxicity and bioactivity of a ready-to-use bioceramic repair material. **Brazilian dental journal**, v. 34, p. 29-38, 2023.

CAVENAGO, B. C. *et al*. Influence of powder-to-water ratio on radiopacity, setting time, pH, calcium ion release and a micro-CT volumetric solubility of white mineral trioxide aggregate**. International Journal of Endodontics**, v. 47, n. 2, p. 120-26, 2014.

CECI M, B. R.; CHIESA, M,.; COLOMBO M.; POGGIO, C. Biological and chemical-physical properties of root-end filling materials: A comparative study. **Jornal of Conservative Dentistry**, v. 18, n. 2, p. 94-9, 2015.

COAGUILA-LLERENA, H. *et al.* Physicochemical properties of a bioceramic repair material-BioMTA. **Brazilian Dental Journal**, v. 31, p. 511-515, 2020.

CVEK M. Treatment of non-vital permanent incisors with calcium hydroxide**: f**ollow- up of periapical repair and apical closure of immature roots. **Odontol Revy**, v. 23, p. 27-44, 1972.

DARWEESH, F. A.; SHAHAWY, D.; ZAGHLOUL, N. M., MOHAMMAD, A. H. Impact of a new silicate-based capping material on healing potentiality of traumatically exposed healthy dog´s dental pulp. **Journal Egyptian Dental**, v. 2020, p. 1-8, 2020.

DOCIMO, R.; FERRANTE, V. C.; COSTACURTA, M. The physical-mechanical properties and biocompatibility of BiodentineTM: A review. **Journal Osseointegration and Oral Reabilitation**, v. 13, n. 1, p. 47-50, 2021.

DUARTE M. A. H. *et al.* pH and calcium ion release of 2 root-end filling materials. **Oral Surgery, Oral Medicine, Oral Pathology , Oral Radiology and Endodontology**, v. 95, n. 3, p. 345-47, 2003.

DUARTE M. A. H. *et al*. Evaluation of the propylene glycol association on some physical and chemical properties of mineral trioxide aggregate. **International Journal of Endodontics**, v. 45, n. 6, p. 565-70, 2012.

DUARTE, M. A. H., *et al*. Tricalcium silicate-based cements: properties and modifications. **Brazilian oral research**, v. 32, (suppl 1), e70, 2018.

DUQUE J. A. *et* al. The effect of mixing method on tricalcium silicate-based cement. **International Journal of Endodontics**, v. 51, n. 1, p. 69-78, 2018.

FRIDLAND, M.; ROSADO, R. Mineral trioxide aggregate (MTA) solubility and porosity with different water-to-powder ratios. **Journal of Endodontics**, v. 29, n. 12, p. 814-17, 2003.

GANDOLFI, M. G. *et al*. Calcium silicate and calcium hydroxide materials for pulp capping: biointeractivity, porosity, solubility and bioactivity of current formulations. **Journal of Applied Biomaterials and Functional Materials**, v. 13, n. 1, p. 43-60, 2015.

GANDOLFI, M. G. *et al*. Development of the foremost light-curable calcium-silicate MTA cement as root-end in oral surgery**.** Chemical-physical properties, bioactivity and biological behavior. **Dental Materials**, v. 27, n. 7, p. e134-57, 2011.

GANDOLFI, M. G.; SIBONI, F.; PRATI, C. Chemical-physical properties of TheraCal, a novel light-curable MTA-like material for pulp capping**. International Journal of Endodontics**, v. 45, n. 6, p. 571-79, 2012.

GANDOLFI, M. G; TADDEI, P.; TINTI, A.; PRATI, C. Apatite-forming ability (bioactivity) of ProRoot MTA. **International Journal of Endodontics**, v. 43, n. 10, p. 917-29, 2010.

GUIMARÃES, B. M. *et al*. Chemical-physical properties and apatite - forming ability of Mineral Trioxide Aggregate Flow. **Journal of Endodontics**, v. 43, n. 10, p. 1692-96, 2017.

GUIMARÃES, B. M. *et al.* Physicochemical properties of calcium silicate-based formulations MTA Repair HP and MTA Vitalcem. **Journal of Applied Oral Science, revista FOB**, v. 26, e2017115, 2018.

HASNA, A. A.; SANTOS D.P.; OLIVEIRA T. R. G. de; PINTO, A. B. A.; PUCCI, C. R.; LAGE-MARQUES, J. L. Apicoectomy of perforated root canal using bioceramic cement and photodynamic therapy. **International Journal of Dentistry**, v. 2020, p. 1-8, 2020

HERAVI, F. *et al. In* Vitro Cytotoxicity Assessment of an Orthodontic Composite Containing Titaniumdi oxide Nano-particles. **Journal of Dental Research**, v. Dental Clinics, p. e20082118, 2013.

HOLLAND, R. *et al*. Reaction of rat connective tissue to implanted dentin tubes filled with a white mineral trioxide aggregate. **Brazilian Dental Journal**, v. 12, n. 1, p. 3-8, 2002.

ISLAM, I.; CHNG, H. K.; YAP, A. U. J. Comparison of the physical and mechanical properties of MTA and Portland cement. **Journal of Endodontics**, v. 32, n. 3, p. 193-97, 2006.

LINSUWANONT, P.; KULVITI, S.; SANTIWONG, B. Reinforcement of Simulated Immature Permanent Teeth after Mineral Trioxide Aggregate Apexification. **Journal of Endodotics**, v. 44, n. 1, p. 163-67, 2018.

LINSWANONT, P.; WIMONSUTTHIKUL, K.; POTHIMOKE, U.; SANTIWONG, B. Treatment Outcomes of Mineral Trioxide Aggregate Pulpotomy in Vital Permanent Teeth with Carious Pulp Exposure: The Retrospective Study. **Journal of Endodontics**, v. 43, n. 2, p. 225-30, 2017.

MARCIANO, M. A. *et al.* Assessment of color stability of white MTA Angelus and bismuth oxide in contact with tooth structure. **Journal of Endodontics**, v. 40, n. 8, p. 1235-40, 2014.

OCHOA-RODRÍGUEZ, V. M. *et al*. Addition of zirconium oxide to Biodentine increases radiopacity and does not alter its physicochemical and biological properties. **Journal of Applied Oral Science**, v. 27, p. e20180429, 2019.

PARIROCK, M; TORABINEJAD, M. Mineral trioxide aggregate: a comprehensive literature review--Part III: Clinical applications, drawbacks, and mechanism of action. **Journal of Endodontics**, v. 36, n. 3, p. 400-13, 2010.

PRIMUS, C; TAY, F. R.; NIU, L. N. Bioactive Tri/dicalcium Silicate Cements for Treatment of Pulpal and Periapical Tissues. **Acta Biomater**, v. 96, p. 35-54, 2019.

QIU, L.; HURT, A. P.; COLEMAN, N. J. The Aplication of 29Si NMR Spectroscopy to the Analysis of Calcium Silicate-Based Cement using Biodentine as an Example. **Jounal of Functional Biomaterials**, v. 10, n. 2, p. 25, 2019.

ROIG, M.; ESPONA, J.; MERCADÉ, M.; DURAN-SINDREU, F. Horizontal root fracture treated with MTA, a case report with a 10-year follow-up. **Dental Traumatoly**, v. 27, n. 6, p. 460-3, 2011.

SARZEDA, G. D. R. *et al.* Análise da composição química dos cimentos MTA Angelus branco, cinza e HP Repair através de Microscopia Eletrônica de Varredura (MEV) acoplada a Espectrômetro de Energia Dispersiva (EDS). **Revista de Odontologia UNESP**, v. 48, p. e20190093, 2019.

VOGEL, G. L.; CHOW, L. C.; BROWN, W.E. A Microanalytical Procedure for the Determination of Calcium, Phosphate and Fluoride in Enamel Biopsy Samples. **Caries Research**, v. 17, n. 1, p. 23-32, 2009.

ZANJANI, V. A. *et al*. Physiochemical properties of experimental nano-hybrid MTA. **Journal of Medicine and Life**, v. 11 (1), p. 51, 2018.