UNIVERSIDADE FEDERAL DE ALFENAS

LARISSA CARVALHO NOVAES BATISTA

ESTUDO EXPERIMENTAL COMPARANDO AS PROPRIEDADES FÍSICO-QUÍMICAS E CARACTERIZAÇÃO DE SUPERFÍCIE DO 5MO E DO MTA BRANCO

ALFENAS/MG

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Dissertação apresentada como parte dos requisitos para obtenção do título de Mestre em Ciências Odontológicas pela Faculdade de Odontologia da Faculdade Federal de Alfenas. Área de concentração: Biologia dos tecidos do complexo bucomaxilofacial.
Orientador: Prof. Dr. Bruno Martini Guimarães

ALFENAS/MG

2024

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Batista, Larissa Carvalho Novaes.

Estudo experimental comparando propriedades físico-químicas e caracterização de superfície do 5MO e do MTA Branco / Larissa Carvalho Novaes Batista. - Alfenas, MG, 2024.
55 f. : il. –

Orientador(a): Bruno Martini Guimarães.

Dissertação (Mestrado em Ciências Odontológicas) - Universidade Federal de Alfenas, Alfenas, MG, 2024.
Bibliografia.

1. Materiais dentários. 2. Propriedades químicas. 3. Silicato de cálcio. 4. Endodontia regenerativa. I. Guimarães, Bruno Martini, orient. II. Título.

Ficha gerada automaticamente com dados fornecidos pelo autor.

LARISSA CARVALHO NOVAES BATISTA

AVALIAÇÃO DAS PROPRIEDADES FÍSICO-QUÍMICAS DE UM CIMENTO BIOCERÂMICO REPARADOR QUE CONTÉM 5 ÓXIDOS

O Presidente da banca examinadora abaixo assina a aprovação da Dissertação apresentada como parte dos requisitos para a obtenção do título de Mestre em Ciências Odontológicas pela Universidade Federal de Alfenas. Área de concentração: Odontologia

Aprovada em: 31 de julho de 2024.

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Documento assinado eletronicamente por **Bruno Martini Guimarães**, **Coordenador(a) do Programa de Pós-Graduação em Ciências Odontológicas**, em 31/07/2024, às 16:18, conforme horário oficial de Brasília, com fundamento no art. 6º, § 1º, do Decreto nº 8.539, de 8 de outubro de 2015.

Dedico este trabalho aos meus
filhos Otávio e Pedro.

AGRADECIMENTOS

Nesses anos de mestrado, na maioria das vezes não foi fácil. Dividir o tempo entre jornada dupla de trabalho, estudo e estrada, foi um desafio constante. Por isso, não posso deixar de agradecer a todas as pessoas que fizeram parte desse caminho e me ajudaram a concretizar esse processo.

Primeiramente, agradeço meu maior incentivador, Rodrigo. Meu amor, aquele que compartilhou comigo todos os momentos bons e ruins e que me fez ser forte em momentos em que pensei que não iria conseguir. Sou profundamente grata por você ser meu parceiro de vida e meu porto seguro.

Agradeço aos meus filhos Otávio e Pedro, meus maiores tesouros e motivação diária para meu crescimento profissional. Obrigada por serem pacientes nos momentos de ausência e por nunca deixarem de me apoiar.

Aos meus pais, Claudete e Carlos. Minha fonte de inspiração, minha base. Se não fosse por vocês, não teria chegado aqui hoje. Tenho muito orgulho e alegria de poder seguir os seus passos e hoje poder dizer que sou dentista como meu pai e professora como minha mãe.

Agradeço às minhas queridas tias Lur, Eli e Fram, que me acolheram e mimaram sempre de forma carinhosa e amorosa, com almoços, cafezinhos e caminhadas. Também não posso deixar de agradecer minha prima/irmã Lailah, que sempre me encorajou, foi meu exemplo e uma das responsáveis por eu ter chegado até aqui. À minha irmã, Ine, por estar sempre comigo em todos os momentos da minha vida.

O mestrado me deu um grande presente, uma amiga querida, companheira, dedicada, que não mediu esforços pra me ajudar a concluir essa jornada. Jaque, nossa amizade começou aqui, mas sei que ainda teremos muitas páginas para escrever juntas.

Aos meus queridos amigos e colegas de trabalho, Paulo e Fer por me apoiarem, por me cobrirem nos momentos que precisei ir para Alfenas, por me deixarem desabafar nos momentos de cansaço e também pelas risadas.

Gostaria de expressar minha profunda gratidão ao meu orientador, Bruno Martini Guimarães, pela sua paciência, sabedoria e apoio ao longo desses anos. Sua compreensão e orientação foram fundamentais para o meu crescimento acadêmico e profissional. Agradeço também à Camilinha por todo o suporte e pela confiança

depositada em mim como profissional. Vocês sempre terão um lugar especial em meu coração.

À equipe e aos funcionários da UNIFAL, expresso meu profundo agradecimento pelo ambiente acadêmico acolhedor e pelos recursos valiosos que foram disponibilizados para minha pesquisa. Seu profissionalismo e dedicação foram essenciais e contribuíram de maneira significativa para o desenvolvimento deste estudo.

Agradeço à empresa Angelus e ao amigo Amjad Abu Hasna pelo fornecimento dos cimentos utilizados durante a pesquisa.

O presente trabalho foi realizado, parcialmente, com o apoio da Fundação de Amparo à Pesquisa do Estado de Minas Gerais (FAPEMIG).

O presente trabalho foi realizado com apoio da Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Código de Financiamento 001.

“A experiência é o nome que
damos aos nossos erros.”
(Oscar Wilde, 1854)

RESUMO

Materiais endodônticos reparadores desempenham um papel crucial na manutenção da funcionalidade dentária ao melhorar os resultados de cicatrização e prevenir extrações. O agregado de trióxido mineral (MTA), um cimento à base de silicato de cálcio, destaca-se por sua bioatividade e biocompatibilidade, sendo considerado padrão ouro em reparos endodônticos. Porém, novos materiais têm sido desenvolvidos, como por exemplo, o cimento de cinco óxidos minerais (5MO). Este estudo teve como objetivo comparar as propriedades físico-químicas dos materiais 5MO e MTA Branco, com foco no pH, tempo de presa, solubilidade, composição química e caracterização de superfície. Analisou-se o pH à 37°C em 3, 12, 24, 72 horas, e 7, 14, 21 dias e o tempo de presa seguiu a norma ISO 6876-2012. Para o teste de solubilidade, as amostras foram pesadas, armazenadas em PBS por 24 horas, secas e pesadas novamente. A solubilidade foi calculada usando a perda de peso das amostras (%). A análise de superfície dos cimentos foi realizada por microscopia eletrônica de varredura (MEV) e espectroscopia por dispersão de energia acoplada (EDS) e a química por teste de espectroscopia infravermelha transformada de Fourier (FTIR) (37-100°C). Os dados foram analisados pelo teste de Shapiro-Wilk e t de *Student* ($p < 0.05$). Ambos os materiais mantiveram pH alcalino ao longo do período de estudo, essencial para atividade antimicrobiana e mineralização tecidual. No entanto, o MTA Branco manteve maior alcalinidade aos 21 dias, indicando potenciais vantagens de estabilidade a longo prazo. A análise do tempo de presa revelou que o 5MO possui um tempo de presa significativamente maior em comparação com o MTA Branco, o que pode impactar a eficiência do manuseio clínico. Além disso, o 5MO demonstrou maior solubilidade após imersão em PBS, comprometendo potencialmente a estabilidade do material e a eficácia de vedação ao longo do tempo. A caracterização de superfície utilizando MEV/EDS destacou morfologias e composições de partículas distintas, enquanto a análise FTIR diferenciou ainda mais as ligações químicas e os produtos de hidratação entre os cimentos. Em conclusão, embora o 5MO apresente uma alcalinidade promissora e uma composição elemental favorável à regeneração tecidual, seu tempo de endurecimento mais longo e maior solubilidade em comparação com o MTA Branco podem limitar sua utilidade clínica.

Palavras-chave: Materiais dentários; propriedades químicas; silicato de cálcio; Endodontia regenerativa.

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.

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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 EXPANDIDA

Os procedimentos de reparo são fundamentais na endodontia, pois permitem a preservação dos dentes em condições funcionais (Roig *et al*., 2018). Para atingir esse objetivo, materiais bioativos são selecionados em procedimentos pulpares e de reparo, visando melhorar os desfechos de cicatrização e diminuir a probabilidade de extração (Primus, Tay, & Niu, 2019). No contexto endodôntico, os cimentos hidráulicos são notáveis por sua capacidade de formar apatita na interface material-dentina na presença de umidade. Um exemplo são os cimentos à base de silicato de cálcio, amplamente utilizados devido à sua biocompatibilidade e bioatividade (Estrela *et al.*, 2023).

A busca por um material capaz de induzir a regeneração e reparo de cavidades expostas começou no século passado (Al-Nalahw, Rachi, & Hasna, 2021). Durante muito tempo, o hidróxido de cálcio foi o material de escolha, com vários estudos demonstrando sua eficácia como material reparador, especialmente em relação às suas propriedades antimicrobianas e à indução da deposição de tecido mineralizado (Cvek, 1972). No entanto, cimentos biocerâmicos surgiram posteriormente, mostrando ser mais eficazes em tratamentos endodônticos devido à sua biocompatibilidade superior e excelente eficácia terapêutica (Hasna *et al*., 2020; Darweesh *et al*., 2020).

O agregado de trióxido mineral (MTA) é um material à base de silicato de cálcio amplamente utilizado em reparos endodônticos (Duarte *et al.,* 2018), considerado o padrão ouro pelos promissores desfechos clínicos que proporciona (Camilleri *et al*., 2022). Este cimento biocerâmico, derivado do cimento de Portland (Torabinejad, 1993), possui em sua composição silicato dicálcico e silicato tricálcico como principais componentes. A primeira versão comercialmente disponível foi a cinza, composto basicamente de cimento Portland cinza e como radiopacificador, o óxido de bismuto. A versão branca surgiu em 2004, tendo em sua composição o cimento Portland branco com baixo teor de Ferro (Primus, C.; Tay, F. R.; Niu, L. N., 2019).

O desempenho do MTA é atribuído à sua bioatividade e capacidade de selamento, relacionada à expansão do material, à retenção de umidade e à liberação de íons hidroxila e cálcio, criando um ambiente de pH alcalino que inibe o crescimento bacteriano e facilita a formação de uma barreira mineralizada (Marciano *et al*., 2016). Além disso, é comprovadamente não citotóxico (Campi *et al*., 2023), biocompatível e bioativo (Delfino *et al*., 2021; Queiroz *et al*., 2021).

Embora as propriedades físicas, químicas e biológicas do MTA sejam amplamente estudadas e reconhecidas, ainda é necessário aprimorar sua composição para atender às características ideais de um material de reparo. Um cimento de reparo endodôntico ideal deve possuir características como estabilidade dimensional, cor adequada, radiopacidade, tempo de presa apropriado, baixa solubilidade em contato com fluidos e fluidez suficiente para um selamento eficaz. Além dessas propriedades físicas, é fundamental que o material também apresente características químicas e biológicas, como alcalinidade, liberação de íons de cálcio, bioatividade, adesão celular, biocompatibilidade e propriedades antimicrobianas. O MTA, embora apresente muitas dessas propriedades ideais, ainda tem espaço para melhorias (Santos *et al*., 2005; Gandolf *et al*., 2011; Gandolf *et al*., 2012).

Em resposta a algumas limitações do MTA, novos materiais foram desenvolvidos. Por exemplo, o cimento de cinco óxidos minerais (5MO) (Golden Yatti LLC, Muscat, Oman, 2021), derivado do cimento Portland, foi reconhecido em estudos in vitro por ser não citotóxico e apresentar propriedades antimicrobianas. Clinicamente, o 5MO tem se mostrado eficaz como material retroativo e para o reparo de perfurações (Hasna, *et al*., 2022). No entanto, suas propriedades físico-químicas ainda são pouco exploradas, representando uma área promissora tanto para a prática clínica quanto para a pesquisa. A literatura também não apresenta trabalhos fazendo a comparação dessas propriedades com as do MTA.

Desenvolver materiais biocompatíveis e bioativos é um avanço crucial na endodontia, oferecendo perspectivas promissoras para aplicações clínicas. Ainda assim, a adoção de novos materiais endodônticos deve ser fundamentada em estudos pré-clínicos abrangentes (Duarte *et al.*, 2018). Propriedades como solubilidade, pH, liberação de íons e tempo de presa impactam significativamente o comportamento biológico dos materiais, afetando, assim, o prognóstico clínico (Cardinali; Camilleri, 2023).

Portanto, o objetivo deste estudo foi analisar e comparar o 5MO com o MTA Branco da Angelus (descritores da empresa) em relação às suas propriedades físico-químicas, tais como pH, tempo de presa, solubilidade e caracterização de superfície. A hipótese nula estabelecida foi que não haveria diferença entre esses dois cimentos de reparo.

A busca constante por materiais que induzam a regeneração e reparo com maior eficácia continua a ser um campo ativo de pesquisa dentro da endodontia. Ao

comparar o 5MO e o MTA Branco, espera-se identificar não apenas as diferenças nas suas propriedades, mas também entender melhor como essas características influenciam os desfechos clínicos, proporcionando inovações que possam melhorar práticas endodônticas e, consequentemente, a saúde dental dos pacientes.

1.1 OBJETIVO GERAL

Este estudo teve como objetivo comparar as propriedades físico-químicas dos materiais 5MO e MTA Branco, com foco no pH, tempo de presa, solubilidade, composição química e caracterização de superfície.

2. ARTIGO

RESEARCH ARTICLE

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

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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.

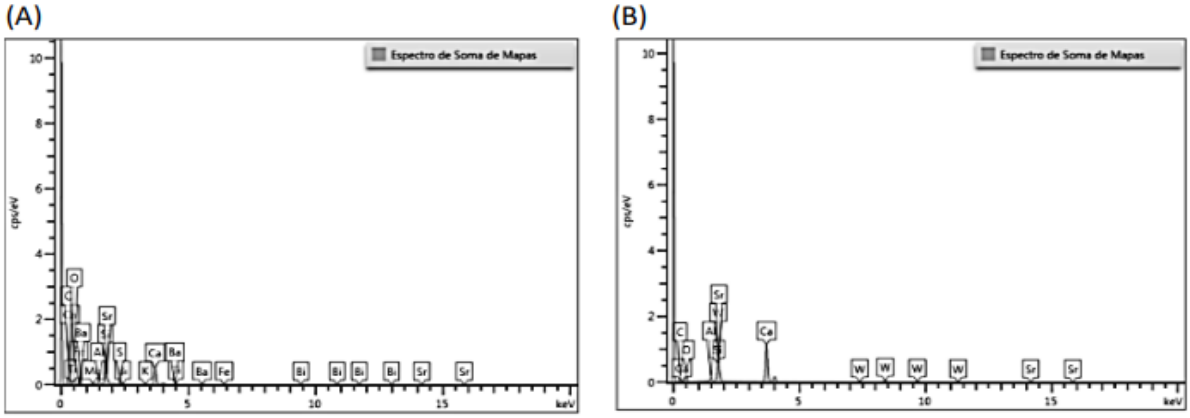

Figure 1: EDS Spectrum. (A) 5MO Sample; (B) WMTA Sample

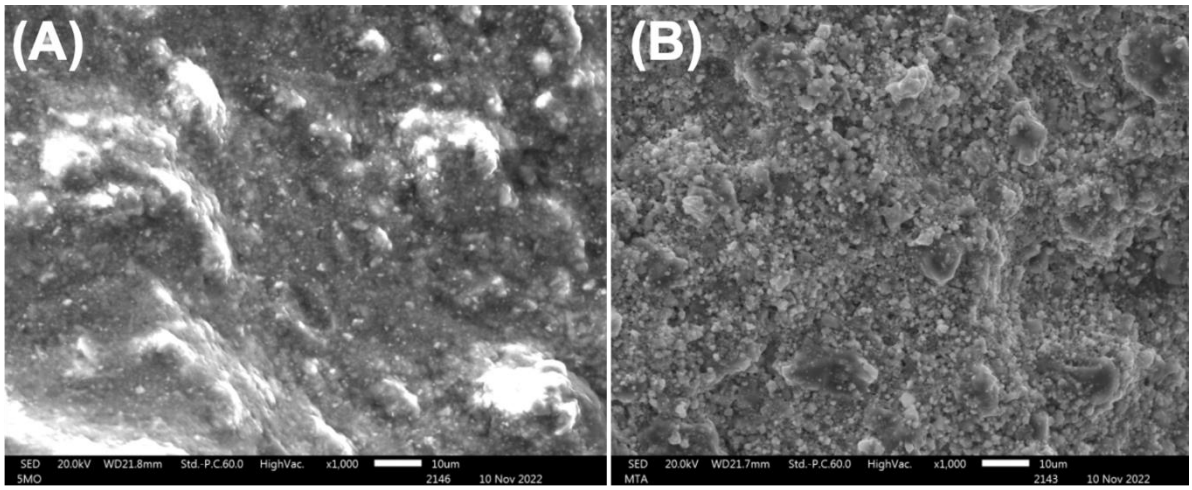

Figure 2: Scanning electron microscope photomicrograph at 1000x magnification. (A) 5MO sample; (B) WMTA sample.

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.

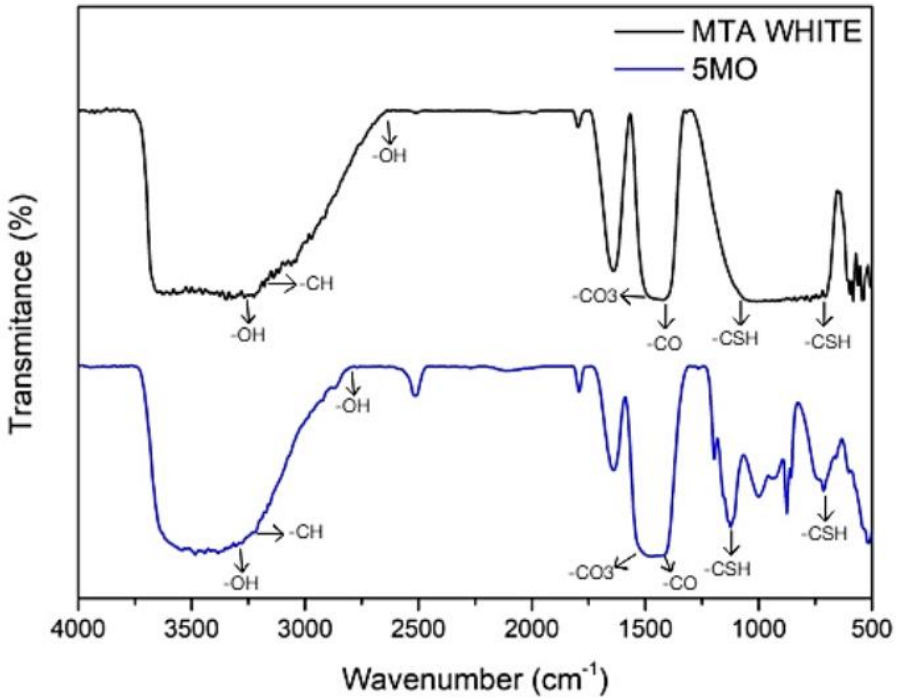

Figure 3: FTIR spectra of 5MO and WMTA.

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.

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3 CONSIDERAÇÕES FINAIS

Com base nos dados obtidos na comparação entre o MTA Branco e o 5MO, é evidente que ambos os materiais possuem propriedades distintas que podem impactar sua efetividade e estabilidade a longo prazo.

O MTA Branco demonstrou manter uma alcalinidade superior após 21 dias, sugerindo potencialmente uma maior durabilidade ao longo do tempo. Além disso, apresentou um tempo de presa significativamente mais curto em comparação com o 5MO, o que pode facilitar o manuseio clínico e a eficiência do tratamento.

Por outro lado, o 5MO exibiu uma alcalinidade promissora e uma composição elemental favorável à regeneração tecidual. No entanto, seu tempo de presa mais prolongado e maior solubilidade após imersão em PBS podem comprometer sua estabilidade e eficácia de selamento em longo prazo.

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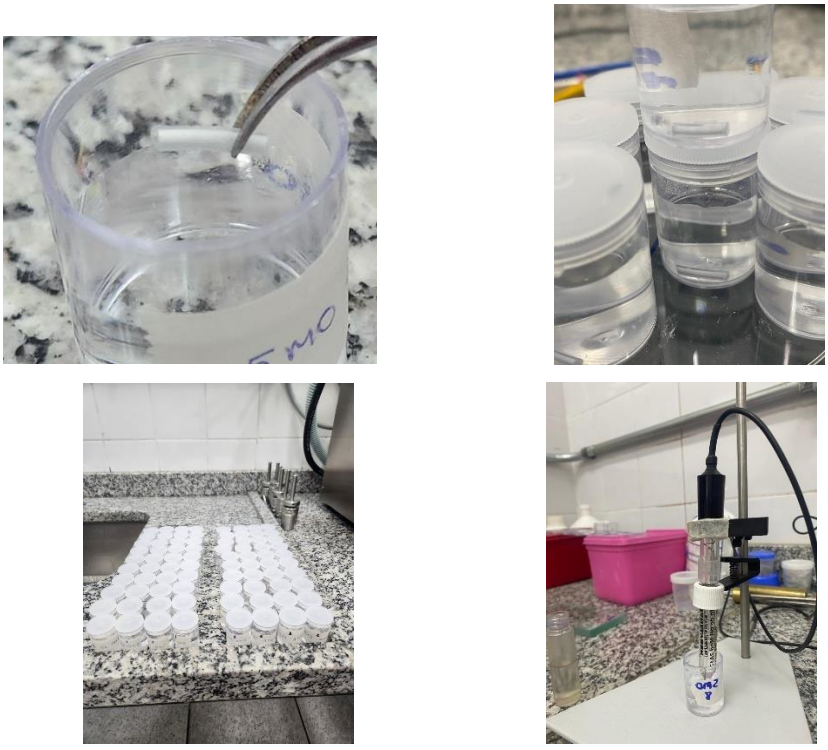
APÊNDICE A – Metodologia Detalhada da Pesquisa

pH

Tubos de polietileno de 1 mm de diâmetro foram seccionados em segmentos de 10 mm de a fim de permitir o preenchimento total pelos cimentos em análise previamente manipulados seguindo as descrições do fabricante. As amostras foram alocadas em recipientes plásticos de 10 ml preenchidos com água destilada e levados a estufa a 37°C e 95% de umidade. As análises foram feitas após 3, 12, 24, 72 horas, e 7, 14, 21 dias. Em cada período, os tubos eram removidos do recipiente plástico e colocado em outro também preenchido com água destilada. O líquido dos recipientes de plástico foram para análise de pH utilizando-se phmetro previamente calibrado Ultrabasic (Denver Instrument Company, Arvada, Colorado, USA).



Fonte: autor (2024)

Tempo de presa

Moldes de gesso (Durone-IV; Dentsply, Petópolis, Rio de Janeiro, Brazil) foram preparados contendo diâmetro de 10 mm e altura de 1 mm. Após a presa do gesso,

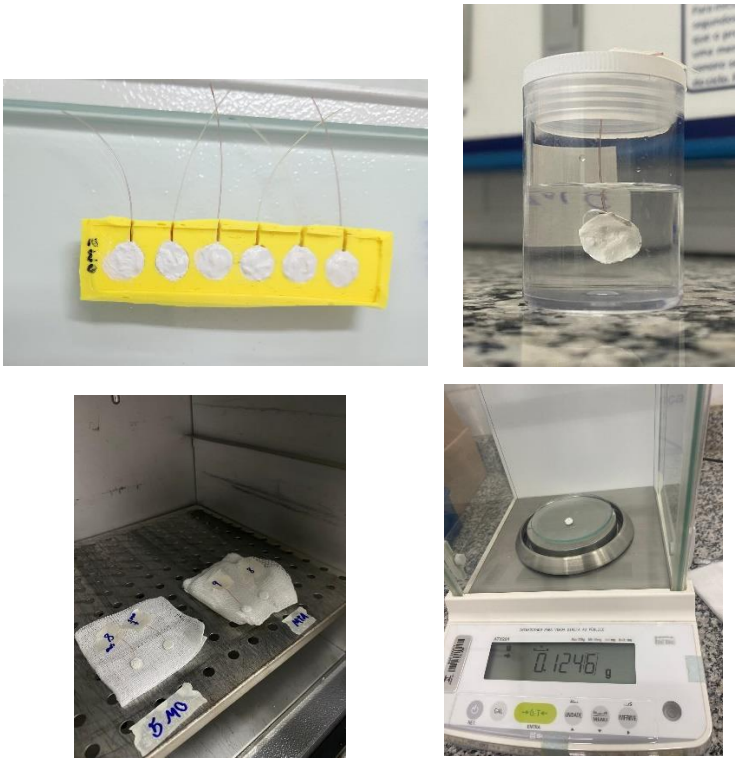
os moldes foram imersos em água destilada por um período de 24 horas antes do início do teste, para que ficassem úmidos. Em seguida, os moldes foram preenchidos com os cimentos previamente manipulados de acordo com as orientações do fabricante. Utilizou-se um recipiente de vidro para acomodar as amostras que foram colocadas em uma estufa a 37°C e 95% de umidade durante o período de análise. Periodicamente, uma agulha de Gilmore com 100 ± 0.5 g e diâmetro de 2 ± 0.1 mm era posicionado verticalmente sob a amostra para determinar o tempo de presa dos cimentos analisados. Durante o teste, o material foi mantido em estufa a 37°C e 95% de umidade e as agulhas eram limpas entre as análises. O tempo de presa foi determinado no período entre a manipulação dos cimentos e o momento em que a agulha não produzia mais marcação nas amostras.

Fonte: autor (2024)

Solubilidade

Antecipadamente, confeccionou-se modelos plásticos com dimensões de 1,5 cm de altura e 7,75 cm de diâmetro interno (Carvalho-Junior et al., 2007). Os modelos foram preenchidos com quantidade de cimento com massa inicial

previamente determinada e já espatulados de acordo com as normas do fabricante. Um fio de nylon foi integrado ao modelo de modo a ficar aderido ao cimento após sua presa. Os moldes foram posicionados em uma lamínula de vidro coberta com papel celofane e preenchidos com os cimentos analisados. Um fio de náilon foi fixado nos moldes de plástico e outra lamínula de vidro, também coberta com papel celofane, foi posicionada sobre os moldes. A montagem foi pressionada manualmente e armazenada em um forno a uma temperatura de 37°C por um período de tempo três vezes maior do que o tempo de ajuste de cada material. As amostras foram alocadas em recipiente plástico contendo 7,5 ml de solução salina tamponada com fosfato (PBS), estando suspensas pelo fio de nylon e foram mantidas em estufa a 37°C por 7 dias. Após esse período, os espécimes foram removidos da solução e levadas a um dessecador. A massa final foi medida em balança de precisão HM-200 (A & D Engineering, Inc., Bradford, MA) a cada 24 horas até que a massa se estabilizasse.



Fonte: autor (2024)

MEV/EDS

Amostras dos cimentos contendo diâmetro de 10 mm e espessura de 1 mm foram preparadas e levados a estufa a 37°C e 95% de umidade até tomar presa. Após a presa, cada amostra foi revestida separadamente com fita condutiva de carbono de dupla face e levados para análise. Para a caracterização da superfície, foi utilizada

Microscopia Eletrônica de Varredura (SEM) acoplada ao Espectrômetro por Dispersão de Energia (EDS - Sistema por Dispersão de Energia) para análise semi-quantitativa.

Ftir

Os espectros de FTIR foram obtidos utilizando pastilhas de brometo de potássio (KBr) com 0,05 g de cada material e 0,1 g de KBr. A mistura foi imediatamente transferida para um molde de prensagem de 13 mm de diâmetro, onde foi colocada sob vácuo em uma prensa de anel RIIK de 10 toneladas por 1 minuto. Os espectros foram obtidos utilizando um espectrômetro FT-IR calibrado (Vertex 70v; Bruker, Billerica, MA, EUA) operando no modo de transmitância normal, resolução espectral de 4 cm -1 e faixa de 400 a 4000 cm -1 sob vácuo. Os dados espectrais de FTIR não requerem processamento de sinal.

Análise estatística

Os dados coletados foram armazenados em banco de dados elaborados em planilha no Excel previamente à análise estatística. Todos os dados foram analisados com o programa estatístico GraphPad Prism 9.02 (GraphPad Software, Inc., CA, EUA) ($\alpha = 0,05$). A distribuição normal destes dados foi confirmada pelo teste de normalidade de Shapiro-Wilk e então as propriedades físico-químicas foram submetidas ao teste t de Student. Os valores diagnósticos foram expressos como média e desvio padrão para cada condição.

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