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Estabilidade volumétrica e adaptação interfacial de cimentos à base de silicato tricálcico utilizados na técnica da tampa: uma análise por Micro-CT e Microscopia Eletrônica de Varredura

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Orientador: Prof. Dr. Lucas da Fonseca Roberti Garcia

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O presente trabalho em nível de Doutorado foi avaliado e aprovado, em 20 de novembro de 2025, pela banca examinadora composta pelos seguintes membros:

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“A natureza é a arte de Deus”

Dante Alighieri

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RESUMO

A cirurgia parendodôntica com obturação retrógrada requer materiais biocerâmicos que garantam estabilidade dimensional e selamento em ambiente úmido; a técnica da tampa, que combina cimentos de diferentes consistências, surge como uma alternativa prática para otimizar o desempenho clínico. Introdução: Este estudo *in vitro* avaliou a estabilidade volumétrica e a adaptação interfacial de cimentos à base de silicato tricálcico com diferentes consistências (selador e *putty*) utilizados na técnica da tampa (*Lid technique*) para obturação retrógrada em cirurgias apicais. Trinta incisivos bovinos foram preparados, obturados e ressecados apicalmente para criação de cavidades retrógradas de 3 mm. As amostras foram distribuídas aleatoriamente em três grupos experimentais (n = 10): BioRoot Flow + BioRoot RCS, EndoSequence BC Sealer + EndoSequence RRM e Bio-C Sealer + Bio-C Repair. Os seladores fluidos foram injetados na cavidade retrógrada, seguidos pela aplicação de uma camada de cimento putty. Varreduras por microtomografia computadorizada (*micro-CT*) foram realizadas no *baseline* (D0) e após 30 dias de imersão em solução tampão fosfato (*PBS*, pH 7,4, 37 °C). As variações volumétricas foram calculadas e analisadas estatisticamente por ANOVA unidirecional e teste post hoc de *Tukey* ($\alpha = 0{,}05$). Amostras selecionadas foram avaliadas por microscopia eletrônica de varredura (MEV) para análise qualitativa da interface cimento-dentina. Todos os materiais apresentaram alterações volumétricas mínimas (<1,1%) após 30 dias, sem diferenças estatisticamente significativas entre os grupos ($P > 0{,}05$). A análise em MEV revelou íntima adaptação entre os cimentos testados e as paredes dentinárias, interfaces contínuas entre as camadas de selador e *putty*, e penetração do selador nos túbulos dentinários. *Microgaps* ocasionais foram observados, mas não comprometeram o contato interfacial geral. Dentro das limitações deste estudo *in vitro*, a técnica da tampa utilizando cimentos à base de silicato tricálcico de diferentes consistências demonstrou estabilidade volumétrica e integridade interfacial satisfatórias. A técnica mostra-se viável e clinicamente promissora para obturação retrógrada em cirurgias apicais.

Palavras-chave: Endodontia, Calcarea Silicata, Obturação Retrógrada, Microtomografia por Raio-X

ABSTRACT

Apical surgery with retrograde filling requires bioceramic materials that ensure dimensional stability and sealing in a moist environment; the Lid technique, which combines cements of different consistencies, emerges as a practical alternative to optimize clinical performance. This *in vitro* study evaluated the volumetric stability and interfacial adaptation of tricalcium silicate-based cements with different consistencies (sealer and putty) used in the Lid technique for retrograde filling in apical surgery. Thirty bovine incisors were prepared, obturated, and apically resected to create 3-mm retrograde cavities. Specimens were randomly assigned to three experimental groups (n = 10): BioRoot Flow + BioRoot RCS, EndoSequence BC Sealer + EndoSequence RRM, and Bio-C Sealer + Bio-C Repair. Flowable sealers were injected into the retrocavity, followed by placement of a putty layer. Micro–computed tomographic (micro-CT) scans were performed at baseline (D0) and after 30 days of immersion in phosphate-buffered saline (PBS, pH 7.4, 37 °C). Volumetric changes were calculated, and statistical analysis was performed using one-way ANOVA and Tukey post hoc tests ($\alpha = 0.05$). Selected specimens were evaluated under scanning electron microscopy (SEM) for qualitative assessment of the cement-dentin interface. All materials exhibited minimal volumetric change (<1.1%) after 30 days, with no statistically significant differences among groups ($P > 0.05$). SEM analysis revealed intimate adaptation between the tested cements and dentinal walls, continuous interfaces between sealer and putty layers, and sealer penetration into dentinal tubules. Occasional microgaps were observed but did not compromise overall interfacial contact. Within the limitations of this in vitro study, the Lid technique using tricalcium silicate–based cements of different consistencies demonstrated satisfactory volumetric stability and interfacial integrity. The technique appears feasible and clinically promising for retrograde filling in apical surgery.

Keywords: Endodontics, Calcarea Silicata, X-Ray Microtomography, Retrograde Obturation

SUMÁRIO

1. INTRODUÇÃO

Os altos índices de sucesso observados na terapia endodôntica moderna estão diretamente relacionados aos avanços científicos contínuos que impulsionam o desenvolvimento de novas técnicas operatórias e biomateriais capazes de promover melhores resultados clínicos e biológicos (DONG et al., 2025; PATEL et al., 2020). Apesar desses progressos, a complexidade anatômica do sistema de canais radiculares e a persistência de biofilmes multiespécie na região apical ainda representam desafios significativos que podem levar ao insucesso do tratamento endodôntico (RICUCCI et al., 2015; NEELAKANTAN et al., 2017).

Quando o retratamento endodôntico não cirúrgico se torna inviável, seja pela presença de retentores intrarradiculares, núcleos metálicos fundidos, instrumentos fraturados ou alterações anatômicas que impedem o acesso adequado ao canal principal, a cirurgia parendodôntica surge como alternativa terapêutica previsível e eficaz (HOLLAND et al., 1993; BERNABÉ et al.; 2005). Dentre as suas modalidades, a apicectomia com obturação retrógrada é amplamente indicada e consiste na ressecção da porção apical da raiz seguida da confecção de uma cavidade retrógrada que deve ser preenchida com material obturador biocompatível e dimensionalmente estável (HOLLAND et al., 1993; BERNABÉ et al.; 2005).

Historicamente, uma variedade de materiais já foi utilizada para essa finalidade, incluindo amálgama, cimento de ionômero de vidro, guta-percha, resina composta, cimentos à base de óxido de zinco e eugenol, como IRM, e reforçados com EBA (ácido etoxibenzoico) - Super-EBA - e, mais recentemente, o agregado de trióxido mineral (MTA). Este último foi originalmente desenvolvido para selamento de perfurações radiculares e de furca, e obturação retrógada (TORABINEJAD & CHIVIAN 1999; AL-KAHTANI et al., 2005; VOSOUGHHOSSEINI et al., 2008). O MTA representou um marco no campo da Endodontia, devido às suas excelentes propriedades físico-químicas e biológicas (TORABINEJAD et al., 1995; PARIROKH; TORABINEJAD, 2010). Sua biocompatibilidade e bioatividade induzem a deposição de tecido mineralizado *in situ*, promovendo excelente vedação marginal, além da elevada resistência mecânica (TORABINEJAD et al., 1995; PARIROKH; TORABINEJAD, 2010).

Originalmente, o MTA apresentava como principal constituinte em sua fórmula (% em peso) o cimento Portland (75,0), além do Bi_2O_3 (20,0), e $CaSO_4$ (5,0) (BÉLIO-REYES et al., 2009). O cimento Portland, originário da indústria da construção civil, é constituído por SiO_2 (21,2) CaO (68,1), Al_2O_3 (4,7), MgO (0,48) e Fe_2O_3 (1,89) (ASGARY et al., 2005). Com a adição de água, as partículas do pó do cimento MTA passam por um processo de hidratação formando gel de sílica hidratada (CAMILLERI & PITT FORD, 2006; ISLAM et al., 2006). A cinética de hidratação do pó de MTA tem papel fundamental na formação deste gel com característica coloidal, que por fim se solidifica em uma estrutura rígida após o término de seu tempo de presa (CAMILLERI & PITT FORD, 2006; ISLAM et al., 2006). No entanto, apesar do sucesso clínico imediato, as primeiras versões de MTA apresentavam desvantagens importantes, como tempo de presa prolongado, difícil manipulação e consistência arenosa, limitando sua aplicabilidade em cavidades retrógradas, especialmente nas de pequena dimensão e difícil acesso (TORABINEJAD et al., 1995; PARIROKH; TORABINEJAD, 2010).

Com o passar do tempo e o avanço tecnológico na produção desta classe de biomateriais, novos cimentos à base de silicato tricálcico de última geração, também conhecidos como biocerâmicos, surgiram (PRIMUS; TAY; NIU, 2019; CAMILLERI, 2020). Estes biomateriais estão disponíveis em apresentações pré-misturadas (pasta única), com bioatividade de alta performance devido à intensa liberação de íons cálcio e capacidade de formar apatita em meio fisiológico (PRIMUS; TAY; NIU, 2019; CAMILLERI, 2022). Essas formulações prontos-para-uso oferecem grande vantagem clínica por dispensarem a mistura manual, garantindo melhor homogeneidade e facilidade de inserção em locais de difícil acesso, como cavidade retrógradas, além de reduzir o risco de contaminação e falhas na proporção de mistura.

Nesse contexto, Nasseh (2015) propôs a chamada *Lid Technique*, ou técnica da tampa, uma abordagem inovadora para obturação retrógrada. A técnica consiste na injeção direta de um cimento biocerâmico de consistência fluida (*sealer*) até leve extravasamento da cavidade retrógrada, seguida pela aplicação de um cimento reparador mais denso (*putty*) sobre o primeiro, funcionando como uma “tampa”. Essa combinação visa aproveitar as propriedades individuais de cada material: a versão *sealer* garante preenchimento tridimensional e adaptação íntima às irregularidades da cavidade, enquanto a versão *putty*, de presa mais rápida e maior viscosidade, confere estabilidade e proteção mecânica durante o período de presa (NASSEH, 2015).

Embora promissora, a técnica da tampa ainda carece de evidências científicas robustas que avaliem sua efetividade e estabilidade dimensional no preenchimento de cavidades retrógradas, especialmente considerando a interação entre cimentos biocerâmicos de diferentes consistências. A literatura ainda é escassa em estudos laboratoriais que explorem a adaptação interfacial, a formação de *microgaps* e o comportamento volumétrico desses materiais em condições simuladas de umidade tecidual.

Diante disso, a microtomografia computadorizada de raios-X (micro-CT) surge como uma ferramenta metodológica de grande relevância (HAMMAD et al., 2010; LI et al., 2020). Por ser uma técnica não destrutiva, a micro-CT permite a análise tridimensional e quantitativa de espécimes com elevada precisão, possibilitando a avaliação de variações volumétricas, formação de vazios internos e alterações estruturais ao longo do tempo (HAMMAD et al., 2010; LI et al., 2020). Além disso, sua aplicação permite acompanhar a mesma amostra em diferentes períodos, sem a necessidade de cortes ou seccionamentos, o que aumenta a confiabilidade e a reprodutibilidade dos resultados.

Complementarmente, a Microscopia Eletrônica de Varredura (MEV) oferece informações essenciais sobre a microestrutura e a interface entre os materiais biocerâmicos e a dentina, permitindo observar a adaptação marginal, a continuidade interfacial e a penetração do cimento nos túbulos dentinários (ATMEH et al., 2012; GANDOLFI et al., 2010). Essa abordagem combinada fornece uma visão abrangente do comportamento físico e químico dos materiais quando empregados na técnica da tampa.

Dessa forma, a presente pesquisa *in vitro* se propôs a avaliar, por meio de análises em micro-CT e MEV, a qualidade da obturação retrógrada realizada pela técnica da tampa, utilizando cimentos à base de silicato tricálcico em suas diferentes versões: seladores (*sealers* - fluido) e reparadores (*putty* - pasta). Avaliou-se a estabilidade volumétrica dos biomateriais ao longo do tempo em condições fisiológicas simuladas e sua adaptação interfacial - tanto entre cimentos e dentina intrarradicular, como entre suas versões de diferentes consistências.

2. OBJETIVOS

2.1. OBJETIVO GERAL

Avaliar a qualidade da obturação retrógrada realizada pela técnica da tampa (*Lid Technique*) com cimentos seladores e reparadores à base de silicato tricálcico em cavidades simuladas.

2.2. OBJETIVOS ESPECÍFICOS

- Avaliar em micro-CT a alteração volumétrica dos cimentos seladores e reparadores à base de silicato tricálcico ao longo do tempo em condições fisiológicas simuladas.
- Avaliar em MEV as características da interface formada entre cimentos de diferentes consistências (fluido e pasta).
- Avaliar em MEV as características da interface formada entre cimentos de diferentes consistências (fluido e pasta) e a dentina intrarradicular.

2.3. HIPÓTESE NULA

- $H_0$1 - A obturação retrógrada pela técnica da tampa com cimentos à base de silicato de tricálcico de diferentes consistências não apresentará alteração volumétrica significativa ao longo do tempo.
- $H_0$2 - As características das interfaces formadas entre cimentos (fluido e pasta), e cimentos e dentina intrarradicular não serão diferentes.

3. ARTIGO CIENTÍFICO

Artigo científico preparado para ser submetido para análise no periódico Journal of Endodontics.

Volumetric Stability and Interfacial Adaptation of Tricalcium Silicate-Based Cements Used in the Lid Technique: A Micro-CT and SEM Analysis

ABSTRACT

Introduction: This *in vitro* study evaluated the volumetric stability and interfacial adaptation of tricalcium silicate-based cements with different consistencies (sealer and putty) used in the Lid technique for retrograde filling in apical surgery.

Methods: Thirty bovine incisors were prepared, obturated, and apically resected to create 3-mm retrograde cavities. Specimens were randomly assigned to three experimental groups (n = 10): BioRoot Flow + BioRoot RCS, EndoSequence BC Sealer + EndoSequence RRM, and Bio-C Sealer + Bio-C Repair. Flowable sealers were injected into the retrocavity, followed by placement of a putty layer. Micro–computed tomographic (micro-CT) scans were performed at baseline (D0) and after 30 days of immersion in phosphate-buffered saline (PBS, pH 7.4, 37 °C). Volumetric changes were calculated, and statistical analysis was performed using one-way ANOVA and Tukey post hoc tests ($\alpha = 0.05$). Selected specimens were evaluated under scanning electron microscopy (SEM) for qualitative assessment of the cement–dentin interface.

Results: All materials exhibited minimal volumetric change (<1.1%) after 30 days, with no statistically significant differences among groups ($P > 0.05$). SEM analysis revealed intimate adaptation between the tested cements and dentinal walls, continuous interfaces between sealer and putty layers, and sealer penetration into dentinal tubules. Occasional microgaps were observed but did not compromise overall interfacial contact.

Conclusions: Within the limitations of this *in vitro* study, the Lid technique using tricalcium silicate–based cements of different consistencies demonstrated satisfactory volumetric stability and interfacial integrity. The technique appears feasible and clinically promising for retrograde filling in apical surgery.

KEY WORDS: Endodontics; Tricalcium Silicate Cements; Retrograde Filling; Micro-Computed Tomography.

INTRODUCTION

The high success rates observed in modern endodontic therapy are directly associated with continuous scientific advances that drive the development of new operative techniques and biomaterials capable of promoting improved clinical and biological outcomes (1,2). Despite these advances, the anatomical complexity of the root canal system and the persistence of multispecies biofilms in the apical region still represent significant challenges that may lead to endodontic treatment failure (3,4).

When nonsurgical endodontic retreatment becomes unfeasible - due to the presence of fiberglass posts, cast metal cores, fractured instruments, or anatomical alterations that prevent adequate access to the main canal - apical surgery emerges as a predictable and effective therapeutic alternative (5,6). Among its modalities, apicoectomy with retrograde filling is widely indicated and consists of resecting the apical portion of the root followed by the preparation of a retrograde cavity, which must be filled with a biocompatible and dimensionally stable filling material (5,6).

Tricalcium silicate-based cements (also known as bioceramics) have been indicated for a wide range of therapeutic applications, including apexification, pulp capping, regenerative endodontic procedures, perforation repair, root canal obturation, restorative procedures, management of periodontal defects, treatment of root fractures, and retrograde filling (7). Although these materials are recognized for their excellent clinical performance and biological properties, such as bioactivity, ongoing research continues to seek formulations with properties closer to the ideal (8).

The practicality of modern ready-to-use formulations, particularly the availability of flowable sealers and putty repair cements, has enabled the development of the Lid technique (9). This technique involves injecting a tricalcium silicate–based sealer into the retrograde cavity until slight extrusion occurs, followed by the application of a putty cement over the sealer, which acts as a “lid” (9). The denser material protects the flowable sealer during its setting in a moisture-rich environment, potentially enhancing overall stability (9).

Although promising, the Lid technique still lacks robust scientific evidence evaluating its effectiveness and dimensional stability in the filling of retrograde cavities, particularly regarding the interaction between bioceramic cements of different consistencies (9). The current literature remains limited in laboratory studies investigating interfacial adaptation, microgap formation, and the volumetric behavior of these materials under simulated moisture conditions.

Accordingly, this in vitro study aimed to evaluate the quality of retrograde filling performed using the Lid technique with tricalcium silicate–based cements in their different formulations: sealers and putties. The volumetric stability of these biomaterials over time under simulated physiological conditions was assessed, as well as their interfacial adaptation—both between the cements and the intraradicular dentin, and between the different consistencies of the materials themselves.

Two null hypotheses were tested: H_{01} - retrograde filling performed using the Lid technique with tricalcium silicate-based cements of different consistencies will not exhibit significant volumetric changes over time; and H_{02} - the characteristics of the interfaces formed between the cements (sealer and putty) and between the cements and the intraradicular dentin will not differ significantly.

MATERIALS AND METHODS

Ethical Concerns and Sample Size Calculation

Thirty freshly extracted bovine incisors from animals approximately three years of age were used in this *in vitro* study. The teeth were generously provided by a local slaughterhouse. All procedures related to animal slaughter, including tooth extraction, were regulated and supervised by local health authorities. Consequently, in accordance with the institutional guidelines governing this research, ethical clearance was not required for the present study.

The sample size was calculated using G*Power software (version 3.1.9.7; Universität Düsseldorf, Germany) [10]. A pilot study was performed for the micro-CT analysis. The parameters were set at $\alpha = 0.05$, statistical power $(1-\beta) = 0.70$, and an anticipated effect size (f) = 1.0. Based on these assumptions, a minimum of ten specimens per experimental group was required for the analysis. The experimental procedures are illustrated in Figure 1.

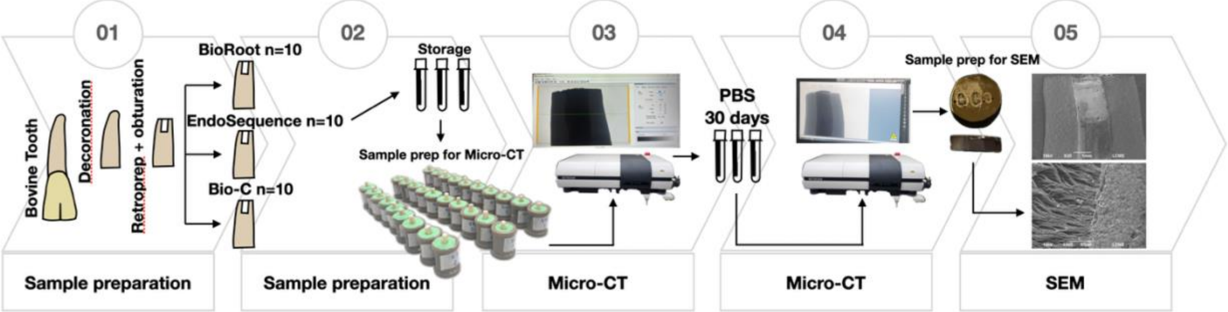

Fig. 1. Schematic representation of the experimental workflow. (1) Sample preparation: Bovine roots were decoronated, instrumented, and retrofilled using the Lid technique with tricalcium silicate-based cements (BioRoot, EndoSequence, or Bio-C). (2) Initial storage: specimens were stored at 37°C and 95% relative humidity for a period equal to three times the manufacturers' setting time before scanning. Roots were individually positioned inside polyvinyl chloride (PVC) tubes and stabilized with condensation silicone to ensure precise positioning and reproducibility during both baseline and 30-day period scans. (3) Baseline micro-CT analysis was performed to evaluate the initial volume and adaptation of the retrograde fillings. PBS storage: samples were immersed in PBS (pH 7.4) at 37°C for 30 days, simulating a physiological moist environment. (4) A second micro-CT scan was then performed to assess volumetric stability. The longitudinal and transverse sections of the specimens were embedded in epoxy resin, fixed, dehydrated in graded ethanol, sputter-coated with a gold-palladium layer, and mounted on metallic stubs for examination. (5) SEM analysis was then performed to qualitatively evaluate the interfacial adaptation between sealer-putty layers and cement-intraradicular dentin interfaces.

Specimen Selection and Initial Preparation

After visual inspection under magnifying lens (×4), single-rooted bovine teeth with straight or slightly curved roots and fully formed apices were selected. Digital periapical radiographs taken in mesiodistal and buccolingual directions were used to confirm internal morphology and to exclude teeth with fractures, cracks, excessive wear, hypoplastic/hypomineralized areas, and pulp calcifications. The roots were cleaned using Gracey curettes (Duflex 7/8, SS White, Rio de Janeiro, RJ, Brazil), followed by prophylaxis with a Robinson brush and pumice paste (Vigodent, Rio de Janeiro, RJ, Brazil). The specimens were disinfected in 0.1% thymol for 48 hours, rinsed for 24 hours, and stored in distilled water until use.

Subsequently, the tooth crowns were sectioned transversely 1 mm above the cementoenamel junction using a double-sided diamond disc (Quality Burs, São Bernardo do Campo, SP, Brazil) mounted on a slow-speed handpiece (Model 605; Kavo, Joinville, Brazil) under copious water irrigation. This procedure left a 1-mm coronal remnant and standardized the root length to 16 mm. Root length was verified with a digital caliper (Mitutoyo Corporation, São Paulo, SP, Brazil).

Root Canal Preparation

The working length (WL) was determined by inserting a size 15 K-file (Dentsply Maillefer, Switzerland) until it was visible at the apical foramen and then subtracting 1 mm. Root canal preparation was performed using the ProFile rotary system (Dentsply Maillefer) up to size 60/.04. During instrumentation, the canals were irrigated with 2 mL of 2.5% sodium hypochlorite solution (Fórmula e Ação, São Paulo, SP, Brazil) using a 30-G NaviTip needle (Ultradent Products, South Jordan, UT, USA), while aspiration was simultaneously performed with a Capillary Tip (Ultradent Products) connected to a Luer Lock suction cannula. Final irrigation consisted of 3 mL of 17% EDTA (Merck KGaA, Darmstadt, Germany) for 3 minutes, ultrasonically activated for 60 seconds during flushing with an ultrasonic insert (Irrisonic E1; Helse, Santa Rosa do Viterbo, SP, Brazil) positioned 1 mm short of the WL. Subsequently, the canals were rinsed with 3 mL of distilled water and dried with paper points (Tanari, Manaus, AM, Brazil).

Root Canal Obturation and Retrograde Cavity Preparation

The root canals were obturated with gutta-percha cones (Tanari) and an epoxy-resin sealer (AH Plus Jet; Dentsply De Trey, Konstanz, Germany) using the lateral compaction technique. A master cone was coated with the sealer and inserted to the WL using gentle circumferential motions. Accessory cones were then inserted until the canal space was completely filled. The quality of the obturation was verified using digital periapical radiographs in both buccolingual and mesiodistal directions. The entrance of the root canal were sealed with a eugenol-free provisional restorative material (Villevie, Joinville, SC, Brazil). The specimens were stored at 37°C and 100% humidity for a period equal to three times the sealer's setting time.

After completion of this period, apical resections were performed at 90° to the long axis of the root, removing 3 mm of the root tip with a Zecrya bur (Dentsply Maillefer) under copious water cooling. Retrograde cavities, 3 mm in depth, were then prepared using an ultrasonic insert (P1M; Helse) attached to an ultrasonic unit (Piezon Master 400; SEM - Electro Medical Systems, Switzerland).

After preparation of the retrograde cavities, the specimens were randomly allocated (www.random.org) into three experimental groups (n = 10): BR Group - BioRoot Flow + BioRoot RCS; ES Group - EndoSequence BC Sealer + EndoSequence RRM; and BC Group - Bio-C Sealer + Bio-C Repair. The flowable sealers were injected into the retrograde cavities using the manufacturer-provided tips until slight extrusion occurred. Subsequently, the putty cements were placed with a No. 6 spatula to cover the flowable sealer. All materials were handled according to the manufacturers' instructions. The specimens were stored at 37 °C and 95% relative humidity for a period equivalent to three times the manufacturer's specified setting time. The composition, classification, and manufacturer information of the tested tricalcium silicate-based cements are presented in Table 1.

Table 1 - Composition, classification, and manufacturer information of the tested tricalcium silicate-based cements.

Cement	Composition	Classification	Presentation	Manufacturer
BioRoot RCS	Powder: Tricalcium silicate, zirconium oxide, povidone. Liquid: Calcium chloride dihydrate, hydrosoluble polymer, purified water.	2nd Generation / Root canal sealer	Powder/Liquid	Septodont, Saint-Maur-des-Fossés, France
BioRoot Flow	Tricalcium silicate, propylene glycol, povidone, calcium carbonate, Aerosil (silica), zirconium oxide, sodium acrylamide/acryloyldimethyltaurate copolymer, isohexadecane, and polysorbate.	3rd Generation / Endodontic sealer	Ready-to-use single paste	
EndoSequence BC Sealer	Calcium silicates, zirconium oxide, tantalum oxide, monobasic calcium phosphate, and thickening agents.	3rd Generation / Endodontic sealer	Ready-to-use single paste	Brasseler, Savannah, GE, USA
EndoSequence Root Repair Material	Calcium silicates, zirconium oxide, tantalum oxide, monobasic calcium phosphate, and thickening agents.	3rd Generation / Repair cement	Ready-to-use single paste	
Bio-C Sealer	Calcium silicate, calcium aluminate, calcium oxide, zirconium oxide, iron oxide, silicon dioxide, dispersing agents.	3rd Generation / Endodontic sealer	Ready-to-use single paste	Angelus, Londrina, PR, Brazil
Bio-C Repair	Calcium silicates, calcium aluminate, calcium oxide, zirconium oxide, iron oxide, silicon dioxide, and dispersing agent.	3rd Generation / Repair cement	Ready-to-use single paste	

Micro-CT Analysis

After cements' setting, the specimens were scanned using a micro-CT system (SkyScan 1272; Bruker, Kontich, Belgium) operating at 100 kVp and 100 µA, with a 0.11-mm Cu filter and an isotropic voxel size of 5 µm. Baseline (D0) and 30-day (D30) scans were acquired using identical parameters. The datasets were reconstructed with NRecon software (Bruker) applying beam hardening and ring artifact corrections, and exported as DICOM files.

Phosphate-Buffered Saline Immersion

Subsequent to the initial scanning, the specimens were immersed in phosphate-buffered saline (PBS, pH 7.4) (Sigma-Aldrich, Barueri, SP, Brazil) at 37 °C for 30 days, with the solution renewed every 48 hours to simulate a physiological moist environment. PBS provides a phosphate source and near-physiological ionic conditions, promoting ion exchange and biomineralization at the material–dentin interface (11,12). After 30 days of PBS immersion, micro-CT scans were performed using the same parameters described previously.

Volumetric Change Calculation

The datasets were superimposed and analyzed using DataViewer (version 1.5.2.4; Bruker) and CTAn software (version 1.11.8; Bruker). A predefined volume of interest (VOI) was selected to encompass the retrocavity and the cement-dentin interface at each time interval. Adaptive histogram thresholding was applied to detect gaps ≥8.74 μm. The percentage of volumetric change was calculated as follows:

$$Volumetric\ change\ (\%) = \frac{VD0 - VD30}{VD0} x\ 100$$

3D Volumetric Reconstruction

Three-dimensional volumetric reconstruction of each specimen was performed using CTVol software (version 2.0; Bruker), providing a comprehensive visualization of the apical root section, including the retrograde cavity, dentinal walls, and filling material. This high-resolution reconstruction enabled a detailed qualitative assessment of the spatial configuration, continuity, and adaptation of the cement throughout the retrofilled region.

Scanning Electron Microscopy Analysis

After the 30-day period, the same specimens used for micro-CT analysis were longitudinally sectioned with a double-sided diamond disc (No. 11-4243; Buehler, Lake Bluff, IL, USA) on a metallographic cutting machine (Isomet 1000; Buehler) to expose the cement–dentin interface. The sectioned specimens were then dehydrated in ascending ethanol concentrations (50%, 70%, 90%, and 100%), immersing them in each solution for 10 minutes. Following dehydration, the specimens were dried in a critical point dryer for 30 minutes, mounted on metallic stubs, and sputter-coated with a 300 Å gold-palladium layer (Desk II; Denton Vacuum, Moorestown, NJ, USA) for 120 seconds. Scanning electron microscopy (SEM) (JSM-5410; JEOL, Tokyo, Japan) was performed at 10-20 kV under magnifications ranging from ×50 to ×200. The interface between cements of different consistencies (sealer and putty), their adaptation to the intraradicular dentin, and microgap formation were analyzed. In addition to the longitudinal sections, transverse sections at the level of the retrograde filling were obtained to provide a more comprehensive assessment of the cement-dentin interface. The longitudinal sections were primarily intended to examine the interaction between cements of different consistencies, although they also allowed evaluation of the cement-dentin interface.

SEM images were evaluated by two previously calibrated and blinded examiners. The findings were subjected to the Kappa test to assess intra- and inter-examiner agreement, with values greater than 0.75 - considered excellent - confirming the validity and reproducibility of the analysis. SEM evaluations were conducted at separate time points, with a 15-day interval between assessments to minimize bias.

Statistical Analysis

Statistical analysis was performed using SPSS Statistics software, version 25 (IBM Corp., Armonk, NY, USA). The normality of the data distribution was verified using the Shapiro-Wilk test, and homoscedasticity was confirmed with Levene's test. The influence of time on the volumetric changes of the different sealers was evaluated using one-way ANOVA followed by Tukey's post hoc test. The significance level was set at 5%. SEM features were evaluated qualitatively.

RESULTS

Volumetric Change

Volumetric values at baseline and after 30 days of PBS immersion are presented in Table 2. All tricalcium silicate-based cements exhibited minimal volumetric changes over time (<1.1%). Although EndoSequence showed the lowest mean change (0.40 ± 0.22), followed by BioRoot (0.72 ± 0.58) and Bio-C (1.04 ± 0.87), no statistically significant differences were observed among experimental groups (*P* > 0.05).

Table 2 - Mean volumetric change values (%) of tricalcium silicate-based cements after 30 days of immersion in PBS.

Groups	Baseline	30 days	Volumetric change
BioRoot	8.51 ± 3.21	8.44 ± 3.17	0.72 ± 0.58
EndoSequence	9.89 ± 3.75	9.84 ± 3.74	0.40 ± 0.22
Bio-C	8.46 ± 3.46	8.38 ± 3.47	1.04 ± 0.87

ANOVA and Tukey's post hoc test. No statistically significant differences were observed among groups.

The box plot in Figure 2 illustrates the distribution of volumetric data for each tricalcium silicate-based cement at baseline and after 30 days of PBS immersion. The height of each box represents the interquartile range, while the horizontal lines indicate the median values. The close overlap between the baseline (blue) and 30-day (red) box plots across all groups confirms that none of the cements exhibited substantial volumetric alterations over time. Minor variations in medians and dispersion among groups reflect sample-specific differences rather than material-related effects, consistent with the absence of statistically significant differences among the tested sealers (*P* > 0.05).

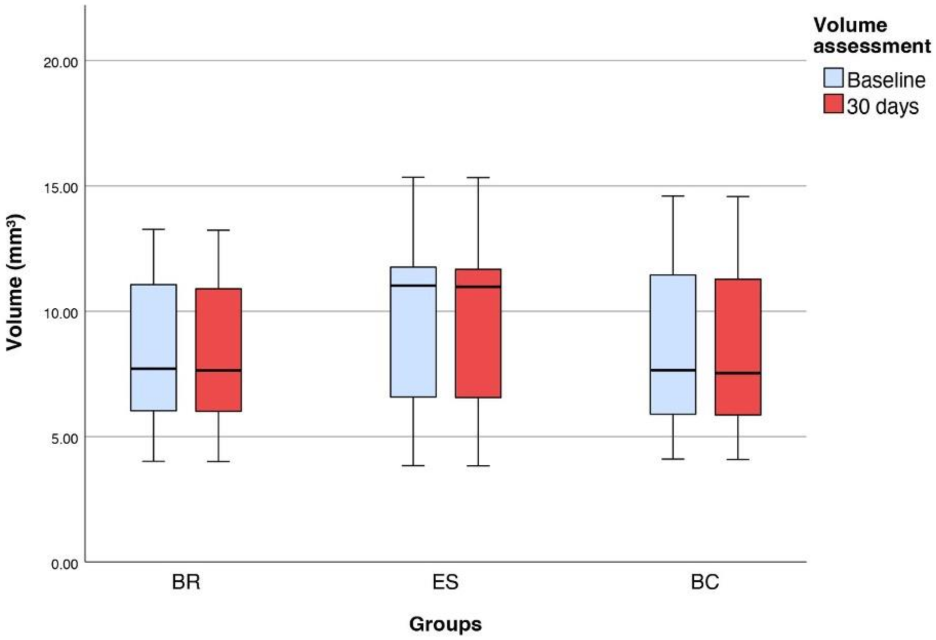

Figure 2 - Box plots showing baseline (blue) and 30-day (red) volume distributions (mm³) for BioRoot (BR), EndoSequence (ES), and Bio-C (BC).

Three-dimensional reconstructions allowed spatial visualization of the retrograde fillings and their behavior over time. The overlay of the baseline (D0) and 30-day (D30) models demonstrated a high degree of geometric correspondence between surfaces in all groups (BioRoot, EndoSequence, and Bio-C), indicating preservation of the filling volume and morphology (Fig. 3). The regions displayed in distinct colors (orange for D0 and blue for D30) showed close alignment, with minimal volumetric discrepancy between time points, confirming the stability of the retrograde sealing mass under PBS storage conditions.

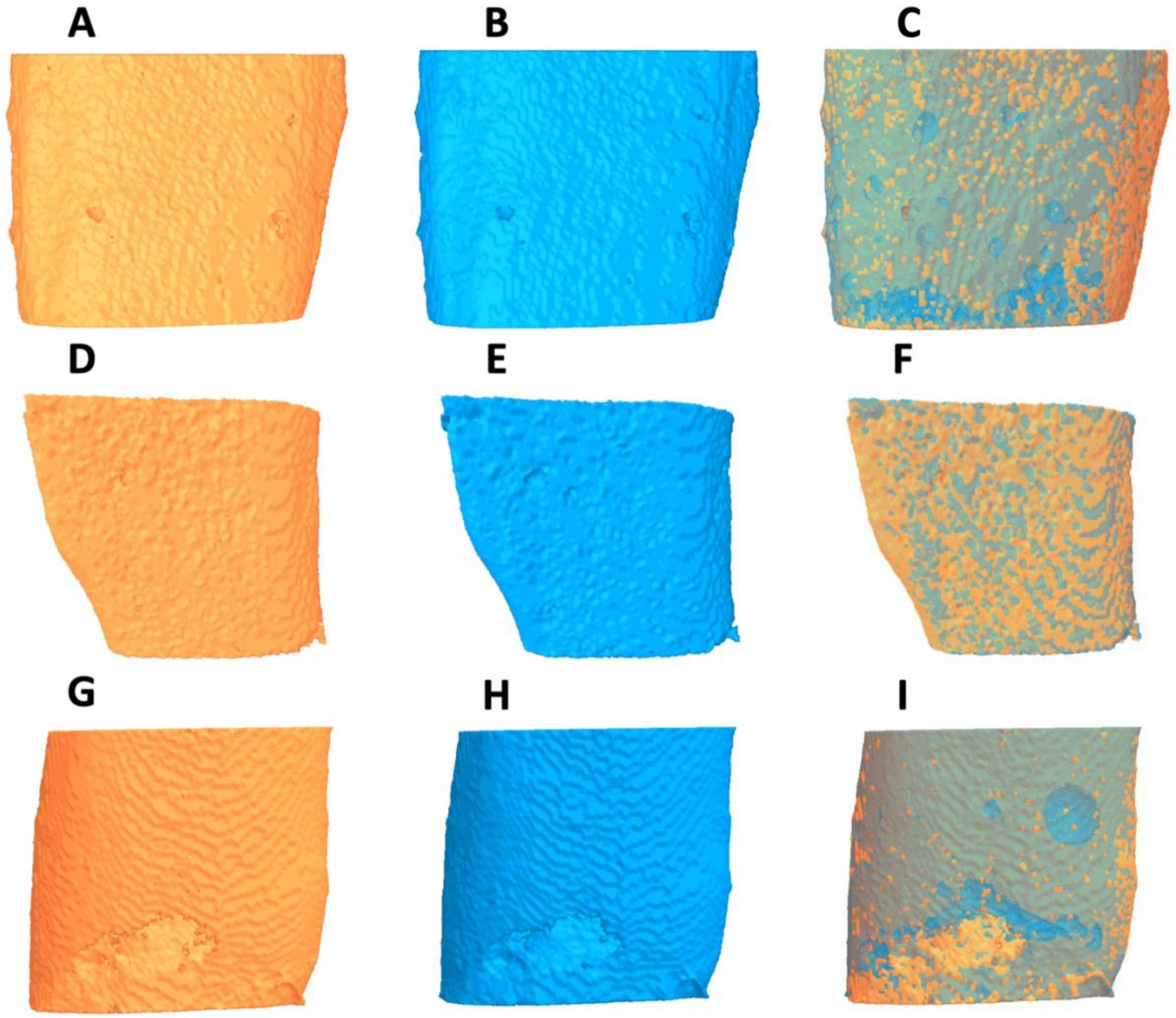

Figure 3 - Representative three-dimensional micro-CT reconstructions of the retrograde fillings for the BioRoot (A-C), EndoSequence (D-F), and Bio-C (G-I) groups. For each material, three models are shown: baseline (D0, orange), 30 days after PBS immersion (D30, blue), and the corresponding overlay (orange + blue). The models represent the retrograde filling mass.

SEM Analysis

Intra- and inter-examiner agreement was considered excellent, with Kappa values of 0.85 and 0.90, respectively. Representative SEM images of the specimens after 30 days of PBS immersion are presented in Figure 4. SEM analysis revealed intimate adaptation between all tested tricalcium silicate-based cements and the root canal walls, with continuous interfaces in most cases for both consistencies (sealer and putty). Occasional microgaps were observed at the cement–cement interface; however, these were minimal and did not compromise overall contact. Sealer penetration into dentinal tubules was evident, indicating micromechanical interlocking. In most cases, the microstructure appeared homogeneous, with no major voids or separation between layers, suggesting stable material integration in the Lid technique.

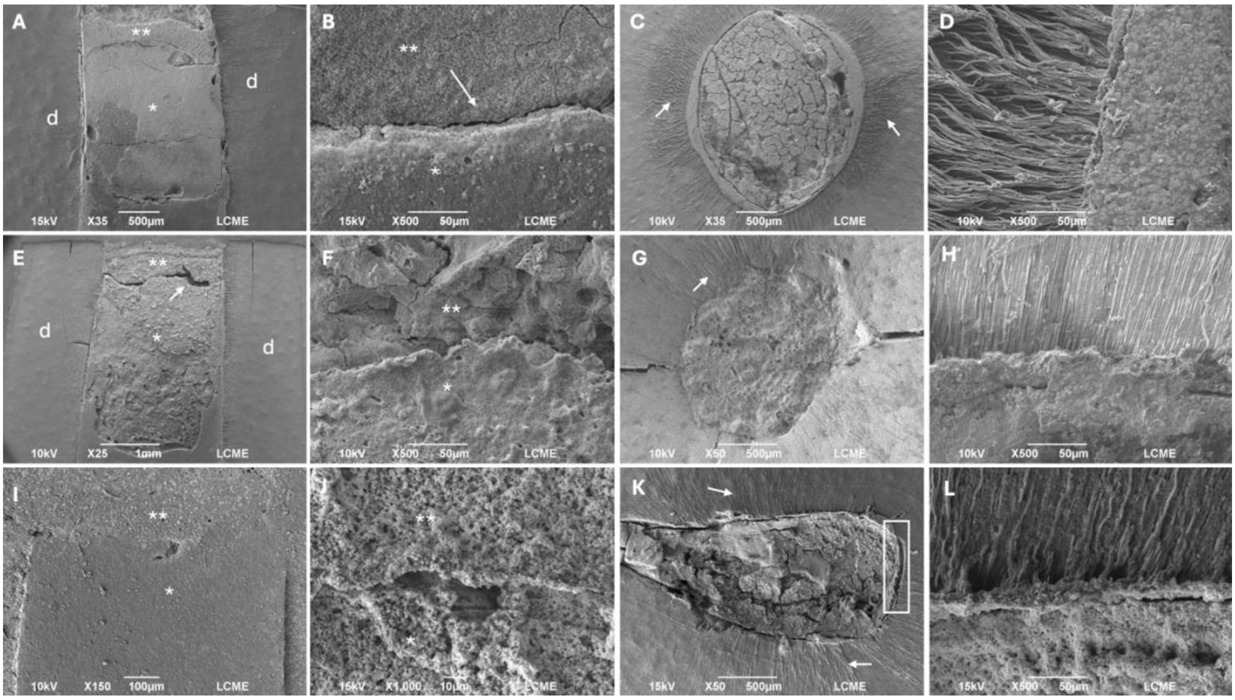

Figure 4 - Representative SEM images of the specimens after 30 days of PBS immersion. BioRoot (A-D): (A) Low-magnification overview of the retrocavity filling showing the three-dimensional adaptation of the tricalcium silicate-based cement. d, dentin; sealer (*); putty (**). (×35 magnification). (B) High-magnification view highlighting the interface between the sealer and putty layers, demonstrating continuous contact with occasional microgaps (arrow). sealer (*); putty (**). (×500 magnification). (C) Transverse section illustrating the internal structure of the retrofilling and the relationship between the cement and intraradicular dentin. Note sealer penetration into dentinal tubules (arrows). (×500 magnification). (D) Detailed view of the sealer-dentin interface, revealing intimate contact and absence of major gaps. Observe the numerous sealer tags within the dentinal tubules. (×500 magnification). EndoSequence (E-H): (E) Low-magnification overview of the retrocavity filling showing the three-dimensional adaptation of the tricalcium silicate-based cement. In this specimen, note the discontinuous interface with voids and gaps (arrow). d, dentin; sealer (*); putty (**). (×25 magnification). (F) Sealer–putty interface showing continuous adaptation and a homogeneous transition between layers. sealer (*); putty (**). (×500 magnification). (G) Transverse section of the retrofilling demonstrating the material's penetration into dentinal tubules (arrow). (×50 magnification). (H) High-magnification view of the sealer-dentin interface showing intimate adaptation but a reduced number of tags. (×500 magnification). Bio-C (I-L): (I) Low-magnification transverse view showing the complete retrofilling and its spatial relationship with the root dentin. No voids or gaps were observed at the interface. sealer (*); putty (**). (×150 magnification). (J) Cement-cement interface showing intimate adaptation between both tricalcium silicate–based layers. sealer (*); putty (**). (×1000 magnification). (K) Transverse section illustrating the internal structure of the retrofilling and the relationship between the cement and intraradicular dentin. Note the presence of gaps (box) and sealer penetration into dentinal tubules (arrows). (×50 magnification). (L) High-magnification image highlighting the cement microstructure and its interfacial adaptation to intraradicular dentin. Note the penetration of sealer tags into the dentinal tubules. (×500 magnification).

DISCUSSION

Although some studies have reported relatively high solubility for certain tricalcium silicate-based cements - a property that may compromise their long-term integrity - these materials nonetheless exhibit several highly favorable characteristics, including bioactivity, biocompatibility, and dimensional stability after setting, all of which collectively contribute to an effective apical seal and favorable clinical performance (13,14). This *in vitro* study evaluated the volumetric stability and interfacial adaptation of tricalcium silicate-based cements of different consistencies used in the Lid technique for retrograde filling. Micro-CT analysis revealed no significant volumetric differences among materials after 30 days of immersion in PBS, indicating that all tested cements maintained satisfactory dimensional stability under simulated clinical conditions. Therefore, the first null hypothesis was accepted. The second null hypothesis was also accepted because the characteristics of the interfaces formed between the cements (sealer and putty) and between the cements and the intraradicular dentin were similar.

The use of bovine teeth to simulate the retrograde cavities represented a valid methodological choice for this study. Bovine teeth are considered a suitable substitute for human dentin because of its similar tubular morphology, chemical composition, and mechanical properties, while providing easier standardization and greater specimen availability (15). Micro-CT is considered the gold standard for non-destructive, three-dimensional evaluation of endodontic materials, providing high-resolution volumetric data with excellent reproducibility and precision (16-18). This imaging technique allows quantitative assessment of dimensional changes, void formation, and interfacial adaptation without altering the specimen's structure. Unlike conventional sectioning or gravimetric techniques - which are destructive and limited to two-dimensional analysis - micro-CT enables longitudinal evaluation of the same specimen over time, offering a more reliable assessment of volumetric stability and material behavior under simulated clinical conditions. For these reasons, micro-CT was chosen as the primary method to evaluate the volumetric changes of tricalcium silicate-based cements used for retrograde filling.

However, volumetric data alone cannot fully characterize the interfacial behavior of these materials. SEM provides ultrastructural visualization of the interfaces between cements of different consistencies and between the cement and intraradicular dentin (19-21). It offers critical information about marginal adaptation, microgap formation, mineral deposition, and interfacial continuity, factors that are determinant for long-term sealing ability and clinical performance (19-21).

The SEM findings of our study provided complementary insights into the microstructural adaptation of the materials. All groups exhibited close contact between the cement and root canal walls, with occasional small gaps mainly observed at the sealer-putty interface. This observation corroborates previous studies demonstrating that tricalcium silicate-based cements can establish a chemical bond with dentin through the deposition of calcium phosphate and apatite crystals, thereby improving sealing ability and structural stability (22-26). The penetration of the sealer into dentinal tubules - particularly evident in the BioRoot and EndoSequence groups - suggests micromechanical interlocking that may enhance retention and reduce the risk of microleakage (27).

The storage of specimens in PBS between baseline and 30 days simulated the moist physiological environment encountered clinically, where retrofilling materials are exposed to tissue fluids (28-30). PBS provides a source of phosphate ions that react with calcium ions released from the cements, leading to the formation of hydroxyapatite and enhancing the sealing ability and biological performance of the materials (28-30). The presence of apatite-like deposits at the material-dentin interface improves marginal adaptation and may contribute to biomineralization, thereby supporting the biological rationale for the use of tricalcium silicate-based cements in apical surgery (28-30).

The combination of tricalcium silicate-based cements with different consistencies used in the Lid technique appears to provide synergistic benefits: the flowable sealer adapts to canal irregularities and penetrates into dentinal tubules, whereas the putty layer - with its faster setting time and higher viscosity - reinforces the interface and protects the sealer during its setting reaction (9). Our findings support the clinical rationale proposed by Nasseh (9), who introduced the Lid technique to improve handling and stability during apical surgery. The minor gaps occasionally observed between the sealer and putty layers are likely attributable to the manual nature of the placement procedure, in which the denser material is applied over the still-unset flowable sealer (9). This step may introduce small interfacial voids because of variations in handling pressure or incomplete coalescence at the interface (9). Nevertheless, since both materials share a similar tricalcium silicate-based chemical composition, a degree of chemical bonding is expected through ion exchange and the formation of calcium phosphate and hydroxyapatite at the contact surface (19). This chemical affinity likely promotes interfacial continuity and mechanical integration over time, mitigating the potential impact of minor procedural irregularities on the overall sealing effectiveness and stability of the retrograde filling (19).

The similar dimensional stability observed across all materials suggests that the Lid technique does not compromise the integrity of retrograde fillings, regardless of the sealer-putty combination used (31,32). From a clinical standpoint, volumetric stability is critical for the long-term success of retrograde fillings (22). Expansion or contraction of the material can compromise the apical seal, leading to microleakage, bacterial ingress, and eventual treatment failure (22). Therefore, the ability of tricalcium silicate-based cements to maintain their volume under physiological conditions reinforces their suitability for use in surgical endodontics.

Despite the inherent limitations of an *in vitro* study, our findings provide strong laboratory evidence supporting the dimensional stability and interfacial compatibility of tricalcium silicate-based materials when applied using the Lid technique. These results suggest that this technique may enhance the clinical manageability of retrograde obturation in confined surgical fields without compromising sealing performance or material integrity. However, future investigations should aim to validate these findings under *in vivo* conditions, incorporating biological variables such as tissue response, moisture control, and surgical handling. Long-term clinical studies are also warranted to confirm the sealing effectiveness, dimensional stability, and biocompatibility of tricalcium silicate-based cements when used in the Lid technique. Establishing clinical evidence for this approach may help optimize retrograde filling procedures in endodontic surgery, supporting predictable healing and durable apical sealing.

CONCLUSION

The Lid technique using tricalcium silicate-based cements of different consistencies demonstrated satisfactory volumetric stability over time, with no significant differences among material combinations. These findings suggest that the technique is a feasible and clinically promising approach for retrograde filling in apical surgery.

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4. CONCLUSÃO

Dentro das limitações deste estudo *in vitro*, pode-se concluir que a técnica da tampa, utilizando cimentos à base de silicato tricálcico de diferentes consistências, apresentou comportamento volumétrico estável após 30 dias, sem diferenças significativas entre as combinações avaliadas. A análise em micro-CT demonstrou que todos os cimentos mantiveram estabilidade dimensional satisfatória, o que indica adequada resistência à dissolução e bom desempenho sob condições úmidas que simulam o ambiente clínico. A análise por microscopia eletrônica de varredura (MEV) evidenciou boa adaptação dos materiais às paredes dentinárias e uma interface contínua entre os cimentos de diferentes consistências, com eventuais *microgaps* atribuídos ao procedimento manual de inserção.

A semelhança química entre os materiais, contudo, favorece a união interfacial e a integração do conjunto, reforçando o potencial da técnica da tampa como uma alternativa simples, rápida e eficaz para a obturação retrógrada em cirurgias parendodônticas, sem comprometer a estabilidade ou a vedação apical. No entanto, são necessários estudos adicionais - laboratoriais e clínicos - para avaliar o comportamento a longo prazo, a resistência mecânica e a resposta biológica desses materiais em condições mais próximas às da prática clínica.

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