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ORIGINAL ARTICLE

Evaluation of Void Volume and Blood Contamination–Induced Changes in Surface Microhardness of Calcium Silicate-Based Cement, Sealer, and Their Combination (Lid Technique) in Retrograde Filling

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ABSTRACT

Objective: This study aimed to compare the effect of blood contamination on the surface microhardness and void volume of a calcium silicate-based cement, sealer, and a combination of the two (Lid technique) when used for retrograde filling.

Methods: Thirty mesial roots of extracted human mandibular molars were divided into three groups: iRoot BP Plus (cement), iRoot SP (sealer), and a combination of the two (Lid technique). The root ends of the teeth were resected and prepared, and the roots were filled. The void volumes were evaluated using micro-computed tomography. Sixty clear resin replicas were created to assess the microhardness after exposure to phosphate-buffered saline and blood; measurements were obtained after 4 and 30 days.

Results: All groups exhibited similar void volumes. The groups retrofilled with iRoot BP Plus and the Lid technique had higher microhardness values than the iRoot SP group. The iRoot SP group displayed similar microhardness values after exposure to PBS and blood. Blood contamination reduced the microhardness values in the iRoot BP Plus and Lid technique groups; however, no significant differences were observed between the measurement times (p=0.203).

Conclusion: In multi-canal roots, the Lid approach is as effective as traditional root-end filling. Blood contamination reduced microhardness emphasizing the significance of handling materials with care during endodontic microsurgery.

Keywords: Apicoectomy, bioceramic, endodontic microsurgery, micro-computed tomography, root-end filling

HIGHLIGHTS

- Mandibular molars retrofilled with either iRoot BP Plus, iRoot SP, or the Lid technique exhibited no difference in void formation, but iRoot BP Plus and Lid technique resulted in higher microhardness values.
- Blood contamination had a detrimental effect on the surface microhardness of both iRoot BP Plus and the Lid technique.
- The Lid technique offers both efficiency and simplicity, making it a practical approach for retrograde filling in mandibular molars.

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INTRODUCTION

Filling root canals with complex anatomies presents a significant challenge, as remnants of pulp tissue and debris can impede the process (1). A study on failed endodontically treated molars reported that 83% of lower first molars had isthmuses in their mesial roots that lacked filling materials (2). Thus, complex root canal anatomy is one of the most common causes of endodontic treatment failure

(3). Endodontic retreatment can be performed using either a nonsurgical or surgical approach. The surgical method has evolved from traditional techniques to modern endodontic microsurgery. This contemporary approach involves resecting the root apex, preparing a root-end cavity, and filling it with retrograde materials to eliminate any remaining pathology within the complex root canal system (4, 5).

Most retrograde filling materials, such as mineral trioxide aggregate (MTA; Dentsply, Tulsa Dental Specialties, Johnson City, TN, USA), Biodentine (Septodont, Saint-Maur-des-Fossés, France), and EndoSequence Root Repair Material (ERRM; Brasseler USA, Savannah, GA, USA), are bioceramics that possess ideal properties, including biocompatibility, non-toxicity, excellent sealing ability (6, 7), low solubility, and the ability to slightly expand within a suitable environment (8, 9). Most importantly, they support both hard tissue conduction and induction (10, 11). MTA is currently regarded as the gold standard material for retrograde filling in endodontic surgery (12, 13). However, it has notable drawbacks, such as a prolonged setting time and challenging handling characteristics (14, 15). In addition, issues such as void formation and blood contamination can occur when using MTA in molars with complex root canal anatomies. To address these challenges, a novel retrograde filling approach known as the "Lid technique" has been proposed. This technique combines a calcium silicate-based cement with a calcium silicate-based sealer and has shown promise in overcoming the limitations associated with traditional materials (16–19).

During surgical procedures, it is essential to maintain a dry prepared cavity and achieve effective bleeding control. Numerous studies have reported the adverse effects of blood contamination on retrograde filling materials. For example, blood exposure during the setting process can interfere with the setting reaction and lead to reduced surface microhardness (20, 21). However, other studies have found no significant differences in gap formation among various retrograde materials, suggesting that certain materials may be more tolerant to contamination (22, 23).

According to the literature, void formation and blood contamination can negatively impact the outcomes of endodontic treatment (24–26). However, studies evaluating void volume in teeth retrofilled using the Lid technique have been limited to single- and two-rooted canals (19, 27). To date, no research has assessed void volume in molars retrofilled with the Lid technique, nor have any studies investigated the effects of blood contamination on the surface microhardness of this technique. Therefore, the present study aimed to compare the void volumes and evaluate the effects of blood contamination on the surface microhardness of a calcium silicate-based cement, a calcium silicate-based sealer, and their combination (Lid technique) when used as retrograde filling materials in mandibular molars.

MATERIALS AND METHODS

The study protocol was approved by the Human Experimentation Committee of Chiang Mai University, Thailand (protocol number 38/2023) and adhered to the Declaration of Helsinki. The materials used for the retrograde fillings included a calcium silicate-based cement (ERRM Putty, also known as iRoot BP Plus; Innovative BioCeramix Inc., Vancouver, Canada) and a calcium silicate-based sealer (iRoot SP; Innovative BioCeramix Inc.). Sample size calculations were performed for both the microhardness and void volume evaluations. With a type I error (α) of 0.05 and a type II error (β) of 0.20 (80% power), the required sample size was determined to be 5 specimens per group for void volume assessment and 8 specimens per group for the microhardness test. Consequently, a total of 10 samples per group were used in this study to ensure adequate statistical power (28–30).

Tooth Preparation

Thirty extracted human mandibular molars were collected and immediately stored in 0.1% thymol solution. The inclusion criteria were as follows: mesial roots with two separate root canals, root curvature of less than 20° (as determined by Schneider's method (31), and complete root formation. Exclusion criteria included visible cracks, fractures, or resorption; incomplete root formation; and previously treated teeth. The mesial root length was standardized to 12 mm. Canal patency was confirmed, and working length was established 1 mm short of the apex using a size 10 K-file (Dentsply Maillefer, Tulsa, OK, USA). Canal preparation was performed using rotary nickel-titanium files (VDW.ROTATETM, VDW GmbH, Munich, Germany) from size 15/0.04 to 30/0.04 at 300 rpm and 2.3 Ncm torque. The canals were irrigated with 17% ethylenediaminetetraacetic acid (EDTA) followed by 2.5% sodium hypochlorite (NaOCl), then dried with paper points. The iRoot SP sealer was injected into the coronal one-third of each canal using a syringe, followed by the insertion of matched size 30/0.04 gutta-percha cones (VDW.ROTATETM Gutta-Percha, VDW GmbH) into the mesio-buccal and mesio-lingual canals using the single-cone technique. The gutta-percha cones were trimmed 2 mm below the canal orifices, and the orifices were sealed with temporary filling material (Cavit; 3M ESPE, St. Paul, MN, USA). The specimens were then stored in a humidified incubator at 37°C for 7 days to allow complete setting of the sealer.

The specimens were subsequently embedded in self-curing epoxy resin. The root ends were resected 3 mm perpendicular to the long axis of the root using a slow-speed saw (Isomet 1000, Buehler, Lake Bluff, IL, USA), followed by a 3-mm root-end cavity preparation using an ultrasonic tip (AS3D; Satelec, Acteon, Merignac, France). One of the prepared root canals was scanned to create a three-dimensional (3D) reconstruction, and 60 clear resin replicas were fabricated to serve as retrograde filling models (Fig. 1). These standardized models were used for evaluating surface microhardness under both phosphate-buffered saline (PBS) and blood-contaminated conditions.

Sampling

The tooth specimens were randomly assigned to three groups (n=10 per group): Group 1 was retrofilled with iRoot BP Plus, Group 2 with iRoot SP, and Group 3 with a combination of the two materials using the Lid technique. All retrograde procedures were performed under a dental operat-

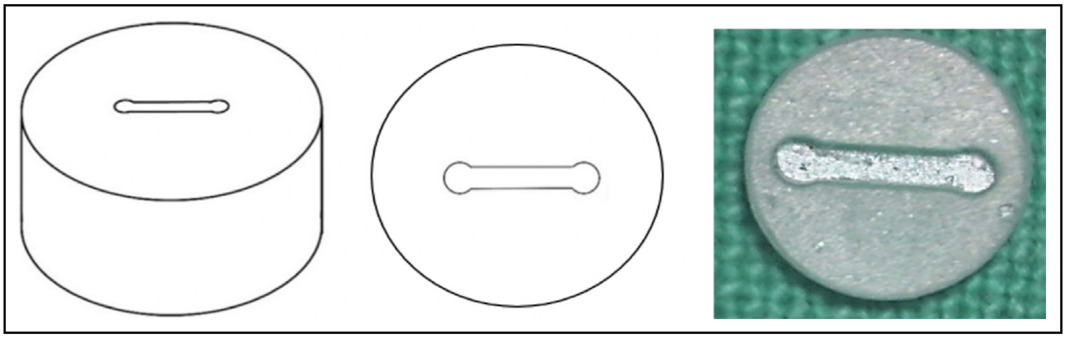

Figure 1. Clear resin replicas used for microhardness test. The molds were designed by 3D scanning a prepared root canal, with the design featuring two root-end cavities containing isthmuses. Each cavity has a depth of 3 mm

ing microscope (DOM; OPMI pico, ZEISS, Jena, Germany) at 40× magnification. The experimental model was designed to closely simulate clinical conditions (Fig. 2).

In Group 1, iRoot BP Plus was incrementally placed into the root-end cavity and condensed using a microplugger. In Group 2, iRoot SP was introduced into the cavity using a sterile 27-gauge needle. In Group 3 (Lid technique), iRoot SP was first introduced to the root-end cavity, followed by iRoot BP Plus placed at the outermost portion and condensed with a microplugger. Radiographs were taken to assess the condensation quality of the retrograde filling materials. The filled specimens were then placed in cylindrical plastic tubes containing PBS and stored in a humidified incubator at 37°C for 7 days. Following the storage period, the specimens underwent micro-computed tomography (micro-CT) (Skyscan 1275; Bruker, Kontich, Belgium) scanning to evaluate the void volume within the retrograde fillings.

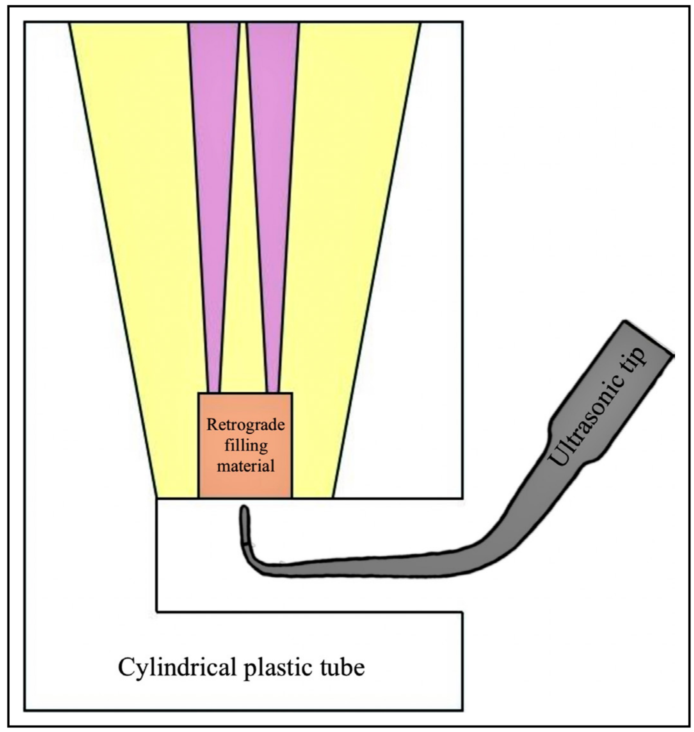

Figure 2. The experimental model. The specimens were adhered to cylindrical plastic tubes. Root-end cavities were prepared with an ultrasonic tip and subsequently filled with retrograde filling materials. The procedure was performed using a retrograde approach throughout the study

Void Volume Evaluation

The evaluation method followed the protocol described in a previous study (16). The 30 tooth specimens were scanned using a micro-CT scanner to assess void volume. Scanning parameters included an X-ray source voltage of 55 kV, a source current of 125 µA, a pixel size of 9 µm, a 1 mm thick aluminum filter, an exposure time of 500 milliseconds, and a rotation step of 0.2°. The scanned images were reconstructed into three-dimensional (3D) models using CTvox software (Bruker) (Fig. 3). Voids within the retrograde filling materials and at the interface between the materials and root canal walls were analyzed using CTAn software (version 1.18.1, Bruker). The percentage of total void volume was calculated using the following equation:

$$\%\ \text{Total void volume} = \frac{\text{Total void volume} \times 100}{\text{Retrograde cavity volume}}$$

Surface Microhardness Test

The 60 resin replicas were divided into three groups based on the retrograde filling materials used (n=20 per group). Each group was further subdivided into two subgroups (n=10 each) according to the experimental conditions: Subgroup A was exposed to PBS, and Subgroup B was exposed to human blood, which was obtained by phlebotomy from one of the authors. All specimens were stored in a humidified incubator at 37°C for 30 days.

After 4 days, the surfaces of the 60 resin replicas exposed to either PBS or blood were wet-polished using silicon carbide sandpaper with grits of 1000, 1200, and 2000 for 30 seconds. The Vickers microhardness test was then performed for each subgroup using a microhardness tester (STARTECH SMV-1000, Guiyang Sunproc International Trade Co., Ltd., Guiyang, China) with a 100 g load applied for 15 seconds. Four indentations were randomly made on each specimen—specifically at the main canals and isthmus—ensuring that indentations were adequately spaced from each other and from the specimen's border to avoid interference. The Vickers hardness (HV) was calculated based upon the equation below, in accordance with the method described in the previous study (28).

$$HV = 1.854 \times \frac{F}{d^2}$$

F is the applied force (Kgf), and d is the area of the indentation (mm). After the initial testing, the specimens were returned to cylindrical plastic tubes filled with PBS and stored in a humid-

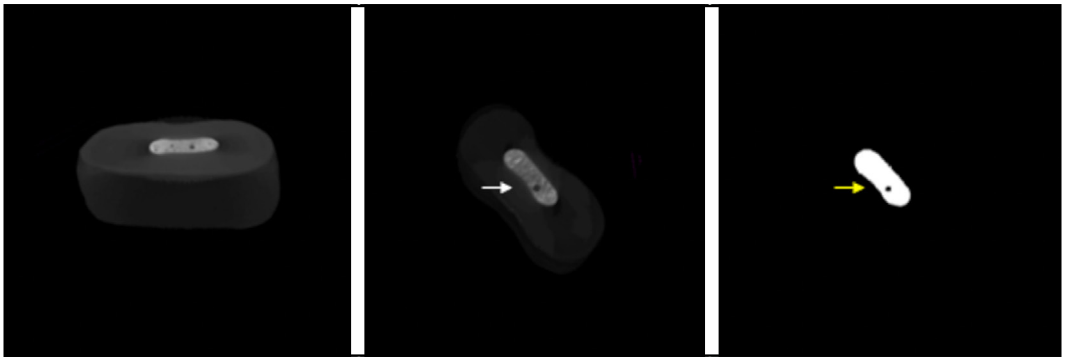

Figure 3. 3D and cross-sectional raw images of retrograde filling material in mandibular first molar (left and center) reconstructed using NRecon software (SkyScan NRecon v.2; Bruker). The white arrow represents void within the material. The binary images (right) were created using CTAn (CTAn v.1.18.1; Bruker). The yellow arrow represents void (black) within the material (white)

ified incubator at 37°C. After a 30-day storage period, the microhardness values were re-evaluated using the same Vickers microhardness testing protocol as previously described.

Statistical Analysis

The data were statistically analyzed using SPSS software version 26.0 (SPSS Inc., Chicago, IL, USA). One-way analysis of variance was used to identify significant differences among groups and a post-hoc test was used to compare data between each subgroup at a significance level of $p<0.05$. An interaction effect between three variables was determined by three-way analysis of variance. Intra-observer reliability was assessed using Cohen's Kappa coefficient.

RESULTS

Void Volume Evaluation

All groups exhibited comparable percentages of total void volume, with no statistically significant differences observed among them ($p=0.998$; Fig. 4).

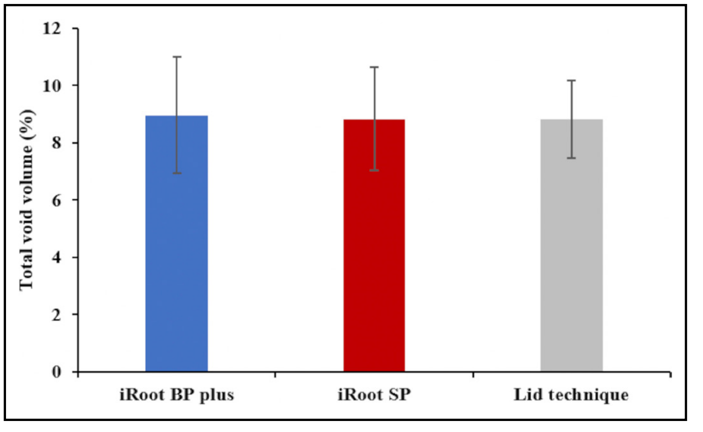

Figure 4. Graph demonstrates mean and standard error (SE) of the percentages of total void volume of calcium silicate-based cement, sealer, and their combination (Lid technique) when using as retrograde fillings in mandibular first molars ($p<0.05$)

Surface Microhardness Test

The results indicated that the groups retrofilled with iRoot BP Plus and the Lid technique demonstrated higher microhardness values compared to the group retrofilled with iRoot SP. However, no significant difference was found between the iRoot BP Plus and Lid technique groups ($p=0.081$). The microhardness values of iRoot SP remained consistent across all conditions. Moreover, blood contamination significantly reduced the microhardness values in the iRoot BP Plus and Lid technique groups ($p<0.001$). No significant differences were observed between the measurements taken at 4 and 30 days in any of the experimental groups ($p=0.203$; Fig. 5).

DISCUSSION

The difficulty in eliminating biofilm within complex root canal anatomies—such as the presence of an isthmus—is a common cause of endodontic treatment failure, primarily due to reinfection and microleakage (3), which can ultimately necessitate retreatment or extraction. To address this challenge, mandibular first molars with isthmuses were selected for this study to compare the void volumes associated with different calcium silicate-based materials used for retrograde fillings.

The application of iRoot SP sealer prior to iRoot BP Plus, as in the Lid technique, may enhance the adaptation of the retrograde material to the dentinal walls by generating hydraulic condensation pressure. However, in the present study, all tooth specimens demonstrated similar total void volumes, regardless of the isthmus length or the type of retrograde filling material used. This finding may be attributed to the incremental condensation technique employed with iRoot BP Plus, as well as the favorable flow properties of the iRoot SP sealer, which likely facilitated thorough distribution of the material within the prepared root-end cavity.

The findings of this study are consistent with those reported by Jung et al. (16), who found no significant difference in void volume between groups retrofilled with calcium silicate-based cement alone and those retrofilled using a combination of calcium silicate cement and a calcium silicate-based sealer. In contrast, Dong et al. (32) reported a significantly lower void fraction in the group retrofilled using the Lid technique compared to the group retrofilled with iRoot BP Plus alone. The discrepancies in findings may be attributed to several factors, including tooth type, sample size differences, and variations in methodology. In our study, mandibular molars with two separate canals were

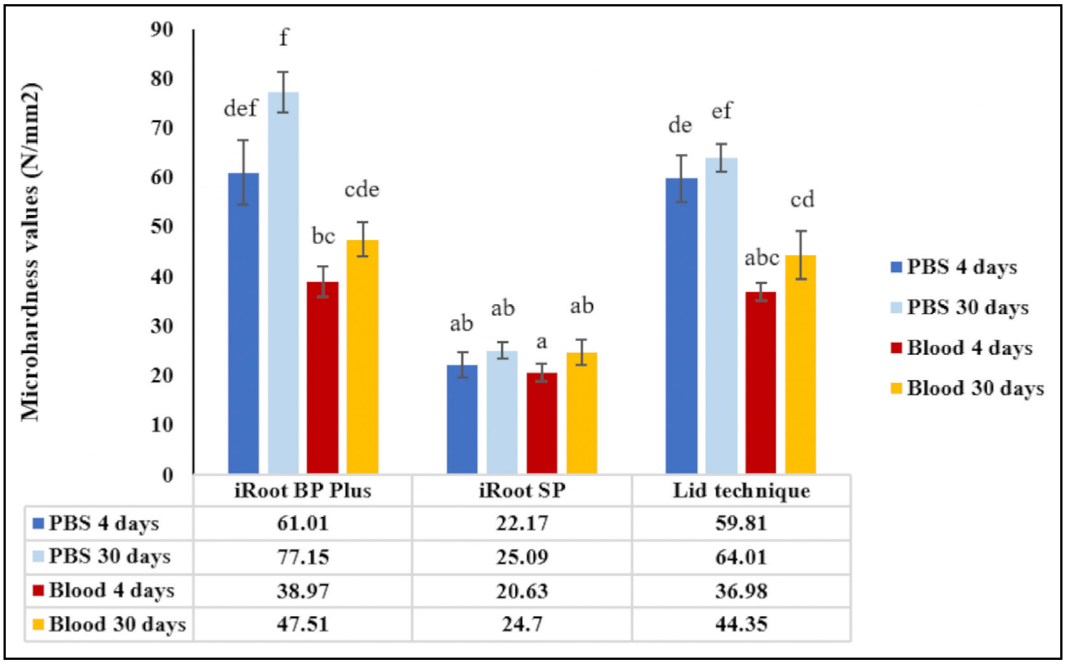

Figure 5. Graph demonstrates mean and standard error (SE) of the surface microhardness values of different calcium silicate-based materials after 4-day and 30-day settings in PBS and blood. Different letters indicate significant differences among the experimental groups ($p<0.05$)

PBS: Phosphate-buffered saline

used, whereas Dong et al. (32) utilized lower premolars. The presence of an isthmus in molars adds complexity to the procedure compared to lower premolars. Additionally, our experimental model was designed to simulate clinical conditions, necessitating indirect root-end filling using a micro-mirror. Given the complexity of the procedure, materials were carefully applied under 40× magnification using a dental operating microscope. However, our study had a smaller sample size than that of Dong et al. (32), which may have also contributed to the differences in results.

Evaluation of the void volume using micro-CT was recently recommended in some studies (33, 34). Micro-CT can be used to inspect the entire depth of the root structure to visualize and evaluate the material and void volume. The technique is noninvasive, repeatable, and enables 3D reconstruction with high resolution. Despite these advantages, micro-CT has certain limitations. Artifacts caused by radiopaque materials can interfere with image quality, and the accuracy of density measurements depends heavily on scanning parameters such as voltage, current, and filter settings (35, 36). Additionally, distinguishing between materials with similar densities remains a significant challenge, potentially affecting the accuracy of void detection and material differentiation.

The second part of this study aimed to compare the effects of blood contamination on the surface microhardness of materials used in different retrograde filling techniques. Resin molds were used instead of natural teeth to eliminate potential confounding factors, such as variations in cavity shape or material thickness, that could influence microhardness measurements. The results demonstrated that blood contamination adversely affected the surface microhardness of bioceramic materials, similar to the previous findings (20, 21). Three-way ANOVA revealed a significant interaction between the type of solution in which the material was immersed and the type of retrograde filling material used. Several studies reported similar microhardness results when calcium silicate-based cements were exposed to an acidic environment (37–39). Blood and acid could interfere with the hydration reaction and crystal formation and negatively affect microhardness. This highlights the importance of minimizing blood contamination during retrograde filling procedures to preserve the integrity of the materials and optimize treatment outcomes.

In the present study, the iRoot SP group exhibited the lowest surface microhardness values, with no significant differences between the 4-day and 30-day measurements or between exposure to PBS and blood. This outcome may be attributed to the material's inherently low mechanical strength, which, when exposed to fluids, could result in incomplete setting. Previous studies have reported that iRoot SP sealer is prone to washout and dissolution when immersed in fluid environments, further compromising its physical properties (40, 41). The washout of the root-end filling was not assessed, as it was beyond the scope of this study. Additionally, prior to microhardness testing, the specimens were polished in accordance with the standard protocol for this methodology. The washout of material may be related to material's properties such as surface microhardness and solubility. Future studies are needed to confirm this hypothesis. In the Lid technique group, iRoot SP was introduced into the root-end cavity first, followed by the application of iRoot BP Plus at the end of the cavity and plugging with a microplugger. As the materials were compacted, any excess sealer was pushed to the surface. Consequently, the indentations made at random could penetrate either the iRoot BP Plus or the iRoot SP sealer. For that reason, the study was worked with the assumption that some indentations in the Lid technique group may have a decrease in microhardness values due to the penetration of the diamond indenter into the sealer. However, in the current study, the iRoot BP Plus and Lid technique groups demonstrated superior microhardness values, with no significant difference.

The final setting time of iRoot SP has been reported to exceed 72 hours (42), while iRoot BP Plus sets in approximately 4 hours (28). Therefore, the fourth day of the experiment was chosen to ensure that all materials had fully set. Studies by Guo et al. (28) and Bayraktar et al. (43) have reported that the microhardness of ERRM putty increases over a 30-day period and that these materials may require approximately 7–10 days for complete setting. Based on these findings, we selected an extended storage period of 30 days to ensure complete setting and to allow for potential increases in material hardness. Our findings indicated a slight, though statistically non-significant, increase in microhardness over time for both the iRoot BP Plus and Lid technique groups. This result is in agreement with the study by Nekoofar et al. (20), which found no significant difference in the microhardness of MTA over an extended period from 4 to 180 days. As current evidence on the long-term behavior of set bioceramic materials remains limited, further studies are warranted to provide a more comprehensive understanding of their durability and performance over time.

The methodology used in this study was designed to simulate endodontic microsurgery in the clinical context. During the procedures, the prepared cavity may come into direct contact with blood or contain voids within the retrograde filling materials. Our results demonstrated that both the iRoot BP Plus and Lid technique groups achieved superior filling quality compared to the group retrofilled with iRoot SP sealer alone. The use of iRoot SP sealer alone for retrograde filling poses technical difficulties, particularly in molars, where it is necessary to bend the syringe tip and position it as close as possible to the root canal filling to minimize void formation. However, this technique also offered the advantage of the shortest working time, as the injectable, premixed sealer rapidly filled the retrocavity within seconds. A specially designed needle may be necessary to optimize the bioceramic sealer's application into the retrograde cavity. However, our findings strongly indicated that the use of iRoot SP sealer alone as a retrograde filling material is not recommended in clinical practice. In contrast, retrofilling with iRoot BP Plus alone required the longest working time, as the putty needed to be incrementally placed throughout the entire length of the retrocavity. Extended working time may increase the risk of blood contamination. This highlights the importance of effective bleeding control during the placement of bioceramic putty to ensure optimal material integrity and clinical outcomes. Based on the findings of this study, we recommend the use of the Lid technique in cases where time constraints are a concern or where there is an increased risk of contamination due to significant bleeding during the surgical procedure. This technique may help reduce operating time and potentially enhance treatment outcomes.

This study was designed to introduce blood contamination only once after the completion of retrograde filling, aiming to evaluate its effect on the surface microhardness of retrograde filling materials in both the short and long term, representing incompletely and fully set materials. In clinical scenarios, the prepared cavity may come into direct contact with blood both before and after the placement of the retrograde filling material due to inadequate hemostasis or patient-related factors, such as systemic medications that impair proper hemostasis. As a result, a blood clot may adhere to the canal wall or material surface, potentially interfering with the material's setting process. Therefore, thorough preoperative evaluation and effective hemostasis are essential. Furthermore, although real human blood and PBS were used in this study to simulate the oral environment, it remains an *ex vivo* study and cannot fully replicate clinical conditions. Future clinical studies are necessary to validate and expand upon these findings.

The success of endodontic treatment can be influenced by a variety of factors. Although a recent study has suggested that the long-term dimensional stability of endodontic sealers is the primary determinant of treatment outcomes (44), evaluating parameters such as void volume and surface microhardness remains valuable. These assessments provide insight into the filling quality and allow for meaningful comparisons between different retrograde filling techniques.

CONCLUSION

Retrofilling with iRoot SP, iRoot BP Plus, or Lid technique resulted in similar void volumes. Both iRoot BP Plus and Lid technique provided higher microhardness values than iRoot SP alone. Blood contamination had a negative impact on the microhardness of the iRoot BP Plus and Lid technique. Our research findings support the efficacy of the Lid technique and highlight the importance of careful manipulation of root-end filling materials, especially in mandibular molars.

Disclosures

Ethics Committee Approval: The study was approved by the Chiang Mai University, Thailand Human Experimentation Ethics Committee (no: 38/2023, date: 16/06/2023).

Informed Consent: Informed consent was obtained from all participants.

Conflict of Interest Statement: The authors have no conflicts of interest to declare.

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REFERENCES

1. De-Deus G, Reis C, Beznos D, de Abranches AMG, Coutinho-Filho T, Paciornik S. Limited ability of three commonly used thermoplasticized gutta-percha techniques in filling oval-shaped canals. J Endod 2008; 34(11):1401–5. [CrossRef]
2. Von Arx T. Frequency and type of canal isthmuses in first molars detected by endoscopic inspection during periradicular surgery. Int Endod J 2005; 38(3):160–8. [CrossRef]
3. Song M, Kim HC, Lee W, Kim E. Analysis of the cause of failure in nonsurgical endodontic treatment by microscopic inspection during endodontic microsurgery. J Endod 2011; 37(11):1516–9. [CrossRef]
4. Setzer FC, Shah SB, Kohli MR, Karabucak B, Kim S. Outcome of endodontic surgery: a meta-analysis of the literature-part 1: comparison of traditional root-end surgery and endodontic microsurgery. J Endod 2010; 36(11):1757–65. [CrossRef]
5. Floratos S, Kim S. Modern endodontic microsurgery concepts: a clinical update. Dent Clin North Am 2017; 61(1):81–91. [CrossRef]
6. Roberts HW, Toth JM, Berzins DW, Charlton DG. Mineral trioxide aggregate material use in endodontic treatment: a review of the literature. Dent Mater 2008; 24(2):149–64. [CrossRef]
7. Torabinejad M, Parirokh M. Mineral trioxide aggregate: a comprehensive literature review-part II: leakage and biocompatibility investigations. J Endod 2010; 36(2):190–202. [CrossRef]
8. Storm B, Eichmiller FC, Tordik PA, Goodell GG. Setting expansion of gray and white mineral trioxide aggregate and Portland cement. J Endod 2008; 34(1):80–2. [CrossRef]
9. Zedler JA. Dimensional changes of ProRoot white mineral trioxide aggregate, Endosequence root repair material, and Biodentine during setting using digital image correlation [dissertation]. Univ Minnesota; 2016.
10. Moretton TR, Brown Jr CE, Legan JJ, Kafrawy A. Tissue reactions after subcutaneous and intraosseous implantation of mineral trioxide aggregate and ethoxybenzoic acid cement. J Biomed Mater Res 2000; 52(3):528–33. [CrossRef]
11. Parirokh M, Torabinejad M. Mineral trioxide aggregate: a comprehensive literature review-part I: chemical, physical, and antibacterial properties. J Endod 2010; 36(1):16–27. [CrossRef]
12. Caron G, Azérad J, Faure MO, Machtou P, Boucher Y. Use of a new retrograde filling material (Biodentine) for endodontic surgery: two case reports. Int J Oral Sci 2014; 6(4):250–3. [CrossRef]
13. Paños-Crespo A, Sánchez-Torres A, Gay-Escoda C. Retrograde filling material in periapical surgery: a systematic review. Med Oral Patol Oral Cir Bucal 2021; 26(4):e422–9. [CrossRef]
14. Lee ES. A new mineral trioxide aggregate root-end filling technique. J Endod 2000; 26(12):764–5. [CrossRef]
15. Camilleri J, Montesin F, Di Silvio L, Pitt Ford T. The chemical constitution and biocompatibility of accelerated Portland cement for endodontic use. Int Endod J 2005; 38(11):834–42. [CrossRef]
16. Jung J, Kim S, Kim E, Shin SJ. Volume of voids in retrograde filling: comparison between calcium silicate cement alone and combined with a calcium silicate-based sealer. J Endod 2020; 46(1):97–102. [CrossRef]
17. Rencher B, Chang AM, Fong H, Johnson JD, Paranjpe A. Comparison of the sealing ability of various bioceramic materials for endodontic surgery. Restor Dent Endod 2021; 46(3):e35. [CrossRef]
18. Azim A, Albanyan H, Azim K, Piasecki L. The Buffalo study: outcome and associated predictors in endodontic microsurgery-a cohort study. Int Endod J 2021; 54(3):301–18. [CrossRef]
19. Eskandar RF, Al-Habib MA, Barayan MA, Edrees HY. Outcomes of endodontic microsurgery using different calcium silicate-based retrograde filling materials: a cohort retrospective cone-beam computed tomographic analysis. BMC Oral Health 2023; 23(1):1–10. [CrossRef]
20. Nekoofar MH, Oloomi K, Sheykhrezae M, Tabor R, Stone DF, Dummer PMH. An evaluation of the effect of blood and human serum on the surface microhardness and surface microstructure of mineral trioxide aggregate. Int Endod J 2010; 43(10):849–58. [CrossRef]
21. Song M, Yue W, Kim S, Kim W, Kim Y, Kim JW, et al. The effect of human blood on the setting and surface micro-hardness of calcium silicate cements. Clin Oral Invest 2016; 20(8):1997–2005. [CrossRef]
22. Bolhari B, Yazdi KA, Sharifi F, Pirmoazen S. Comparative scanning electron microscopic study of the marginal adaptation of four root-end filling materials in presence and absence of blood. J Dent (Tehran) 2015; 12(3):226–34.
23. Saker MA, Khalefa MM, El-Sheikh HM. Effect of blood contamination on marginal adaptation of MTA and Endosequence root repair material as a retrograde filling. Al-Azhar J Dent 2020; 7(3):383–9. [CrossRef]
24. Villa-Machado P, Botero-Ramírez X, Tobón-Arroyave S. Retrospective follow-up assessment of prognostic variables associated with the outcome of periradicular surgery. Int Endod J 2013; 46(11):1063–76. [CrossRef]
25. Stabholz A, Shani J, Friedman S, Abed J. Marginal adaptation of retrograde fillings and its correlation with sealability. J Endod 1985; 11(5):218–23. [CrossRef]
26. Taschieri S, Bettach R, Lolato A, Moneghini L, Del Fabbro M. Endodontic surgery failure: SEM analysis of root-end filling. J Oral Sci 2011; 53(3):393–6. [CrossRef]
27. Shinbori N, Grama AM, Patel Y, Woodmansey K, He J. Clinical outcome of endodontic microsurgery that uses EndoSequence BC root repair material as the root-end filling material. J Endod 2015; 41(5):607–12. [CrossRef]
28. Guo YJ, Du TF, Li HB, Shen Y, Mobuchon C, Hieawy A, et al. Physical properties and hydration behavior of a fast-setting bioceramic endodontic material. BMC Oral Health 2016; 16:23. [CrossRef]
29. Roizenblit RN, Soares FO, Lopes RT, Dos Santos BC, Gusman H. Root canal filling quality of mandibular molars with EndoSequence BC and AH Plus sealers: a micro-CT study. Aust Endod J 2020; 46(1):82–7. [CrossRef]
30. Torres FFE, Jacobs R, EzEldeen M, Guerreiro-Tanomaru JM, Dos Santos BC, Lucas-Oliveira É, et al. Micro-computed tomography high resolution evaluation of dimensional and morphological changes of 3 root-end filling materials in simulated physiological conditions. J Mater Sci Mater Med 2020; 31(2):14. [CrossRef]
31. Schneider SW. A comparison of canal preparations in straight and curved root canals. Oral Surg Oral Med Oral Pathol 1971; 32(2):271–5. [CrossRef]
32. Dong X, Xie Q, Xu X. *In vitro* evaluation of the sealing ability of combined use of iRoot BP Plus and iRoot SP for root-end filling. Clin Oral Invest 2023; 27(6):2969–77. [CrossRef]
33. Hammad M, Qualtrough A, Silikas N. Evaluation of root canal obturation: a three-dimensional *in vitro* study. J Endod 2009; 35(4):541–4. [CrossRef]
34. El-Ma'aita AM, Qualtrough AJ, Watts DC. A micro-computed tomography evaluation of mineral trioxide aggregate root canal fillings. J Endod 2012; 38(5):670–2. [CrossRef]
35. Orhan K, Jacobs R, Celikten B, Huang Y, de Faria Vasconcelos K, Nicolielo LFP, et al. Evaluation of threshold values for root canal filling voids in micro-CT and nano-CT images. Scanning 2018; 16:9437569. [CrossRef]
36. Torres FFE, Jacobs R, EzEldeen M, de Faria-Vasconcelos K, Guerreiro-Tanomaru JM, Dos Santos BC, et al. How image-processing parameters can influence the assessment of dental materials using micro-CT. Imaging Sci Dent 2020; 50(2):161–8. [CrossRef]
37. Lee YL, Lee BS, Lin FH, Lin AY, Lan WH, Lin CP. Effects of physiological environments on the hydration behavior of mineral trioxide aggregate. Biomaterials 2004; 25(5):787–93. [CrossRef]
38. Namazikhah M, Nekoofar MH, Sheykhrezae M, Salariyeh S, Hayes SJ, Bryant ST, et al. The effect of pH on surface hardness and microstructure of mineral trioxide aggregate. Int Endod J 2008; 41(2):108–16. [CrossRef]
39. Wang Z, Ma J, Shen Y, Haapasalo M. Acidic pH weakens the microhardness and microstructure of three tricalcium silicate materials. Int Endod J 2015; 48(4):323–32. [CrossRef]
40. Formosa L, Mallia B, Camilleri J. Mineral trioxide aggregate with anti-washout gel-properties and microstructure. Dent Mater 2013; 29(3):294–306. [CrossRef]
41. Falkowska J, Chady T, Dura W, Droździk A, Tomasik M, Marek E, et al. The washout resistance of bioactive root-end filling materials. Materials (Basel) 2023; 16(17):5757. [CrossRef]
42. Juntha S, Tungsawat P, Wongwatanasanti N, Suksaphar W, Lertnantapanya S. Evaluation of setting time, flowability, film thickness, and radiopacity of experimental monocalcium silicate-based root canal sealers. Int J Dent 2024; 20:8541653. [CrossRef]
43. Bayraktar K, Basturk FB, Turkaydin D, Gunday M. Long-term effect of acidic pH on the surface microhardness of ProRoot mineral trioxide aggregate, Biodentine, and total fill root repair material putty. Dent Res J (Isfahan) 2021; 18:2. [CrossRef]
44. De-Deus G, Souza EM, Silva EJNL, Belladonna FG, Simões-Carvalho M, Cavalcante DM, et al. A critical analysis of research methods and experimental models to study root canal fillings. Int Endod J 2022; 55(Suppl 2):384–445. [CrossRef]