BASIC RESEARCH – TECHNOLOGY

"Effect of Heat Generated by Endodontic Obturation Techniques on Bond Strength of Bioceramic Sealers to Dentine"

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ABSTRACT

Introduction: Gutta-percha combined with an endodontic sealer remains the most widely used obturation technique. Bioceramic sealers (BS) were developed for root canal obturation in combination with gutta-percha cones using the cold single-cone technique. Few studies have assessed the effect of thermal treatment on the performance of BS. The present study evaluated the effect of heat on BS adhesion to root dentine in the apical third of the root canal of extracted human lower premolars. **Materials and Methods:** Three BS combined with a hydraulic condensation technique, a warm vertical compaction technique, and a carrier-based technique were evaluated. Sixty three lower premolars were prepared following the same surgical protocol to standardize root canal shape at the level of the apex, randomly assigned to one of nine groups, and obturated accordingly. One millimeter-thick sections were subjected to a push-out test using a universal testing machine and classified according to mode of failure. Two-way ANOVA was applied using SPSS software (IBM Corp). **Results:** No significant differences in maximum load or failure mode were observed among BS, techniques, or when considering the interaction between sealers and techniques. **Conclusions:** The heat generated by the obturation techniques used here did not affect BS adhesion to the dentinal wall. *(J Endod 2023;■:1–5.)*

KEY WORDS

Bioceramic sealers; bond strength; hydraulic cements; obturation techniques; push-out test

SIGNIFICANCE

Bioceramic sealers (BS) were promoted for use with the single gutta-percha cone technique. We found that the heat generated by warm obturation techniques did not affect BS adhesion to the dentinal wall.

The main goal of endodontic obturation is to provide complete three-dimensional (3D) filling of the root canal system using inert, antiseptic, bioactive materials that promote − or do not interfere with − the healing process[1]. Adequate coronal restoration is also essential to impede bacterial microfiltration from the oral cavity[2,3].

Gutta-percha combined with an endodontic sealer remains the most widely accepted and used obturation technique[4]. Endodontic sealers were developed to overcome the physical limitations of rigid materials (gutta-percha) to completely obturate the root canal space. They are essential in endodontic therapy to fill the irregularities of the root canal system and minor discrepancies between the gutta-percha and the dentinal wall[5].

The first mineral trioxide aggregate-based bioceramic cement (MTA, ProRoot Dentsply) for sealing accidental perforations of the root canal was released in 1993[6,7]. Biodentine® (Septodont, Saint-Maur-des-Fosses, France), a novel tricalcium silicate-based cement designed as a dentine substitute material with improved handling properties and shorter setting time than MTA and that causes no tooth staining, was introduced in 2009[8,9]. Bioceramic sealers (BS) were later developed for root canal obturation in combination with gutta-percha cones using the hydraulic condensation (HC) or the lateral condensation technique[10]. Unlike conventional sealers, which are negatively affected by humidity, BS are hydrophilic[11]. Bioceramics are not resorbable and do not shrink but expand slightly during setting[12]. Nevertheless, penetration of the material into the dentinal tubules can block them, making endodontic retreatment challenging and affecting repair of the dentinal wall[13].

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The single cone technique has often been considered inadequate due to the potential for apical leakage[14]. However, with the recent advent of BS, also known as "hydraulic cements", acceptance of HC has increased due to its improved properties[15-17]. Although BS were proposed for use with HC, they can be applied with any obturation method, including cold and thermoplastic techniques. Thermoplasticized gutta-percha is widely used with specially designed systems such as Guttacore, a carrier-based technique consisting of a cross-linked gutta-percha core obturator coated with alpha-phase gutta-percha that is highly flowable and has good adhesive properties and a melting temperature of 56°C. As to the quality of obturation, similar results are achieved with obturator-based techniques and the vertical compaction method[18].

Few studies have assessed the effect of thermal treatment on the performance of BS[19,20]. **Camilleri et al** compared the effect of heat on 3 resin-based sealers and a tricalcium silicate-based sealer (experimental version); the authors concluded that the experimental tricalcium silicate cement was suitable for obturation using cold laterally condensed gutta-percha[11]. **Atmeh et al**, however, observed no significant effect of thermal treatment on the chemical structure of calcium silicate-based sealers, despite microstructural changes that can occur due to loss of water[21]. **Donnermeyer et al and Dewi A et al** found that most of the sealers they assessed could be considered suitable for warm obturation techniques[22,23].

The present study sought to compare 2 thermoplastic obturation techniques and the hydraulic condensation technique in order to evaluate the effect of heat on BS adhesion to root dentine in the apical third of the root canal of extracted human lower premolars.

MATERIALS AND METHODS

Two premixed (Bio C Sealer (BCS) Angelus, Brazil, CeraSeal (CS) MetaBiomed, Korea) and ,one powder/liquid (BioRoot (BR) Septodont, France) BS, each combined with a hydraulic condensation (HC) a warm vertical compaction (WVC), and a carrier-based technique, Guttacore (G), were evaluated.

The study comprised 63 lower premolars extracted for orthodontic or periodontal reasons unrelated to the present study at the Dental School Surgery Department (Catholic University of Córdoba, Argentina). All patients gave their informed consent for tooth extraction. Only lower premolars with a single root, complete root formation, a permeable apical foramen, and an initial apical file K #10 or #15 were included.

Chamber opening was performed using a high-speed drill under irrigation. Apical patency of the root canal was confirmed with a #10 file. Working length was determined visually under an operative microscope, subtracting 1 mm from the outer limit of the apical foramen.

All 63 specimens were prepared following the same surgical protocol to standardize root canal shape at the level of the apex. Preparation diameter was increased gradually to working length using a primary (#25.07), a medium (#35.06), and a large (#45.05) WaveOne Gold file under reciprocation motion. The final apical taper was standardized using a Protaper Gold rotary file F5 (#50.05) (Dentsply-Maillefer, Ballaigues, Switzerland). Irrigation was performed with 2 ml of 2.5% sodium hypochlorite after each instrument, verifying patency to prevent apical blockage. Before obturation, the samples were irrigated with sonically activated EDTAC 17% followed by sonically-activated 2.5% sodium hypochlorite. Biomechanical preparation was completed using a final rinse with 5 ml of sterile distilled water. The canals were dried with #50 sterile paper points (MetaBiomed). An F5 Protaper gutta-percha cone (Dentsply-Maillefer, Ballaigues, Switzerland) matching the master apical file was selected and tested. All the samples were prepared by a single calibrated operator.

The samples were then randomly assigned to one of the following 9 groups ($n = 7$) HC-**Bio C Sealer** Group, HC-**CeraSeal** Group, HC-**BioRoot** Group, WVC-**Bio C Sealer** Group, WVC-**CeraSeal** Group, WVC-**BioRoot** Group, G-**Bio C Sealer** Group, G-**CeraSeal** Group, and G-**BioRoot** Group.

For the HC technique, the BS was placed in the root canal from the apex to the canal orifice. The master cone was then coated with a thin layer of sealer and slowly inserted to the working length. The cone was seared off at the canal orifice using a Calamus plugger (Dentsply-Maillefer, Ballaigues, Switzerland) and vertically compacted with a #3 Machtou plugger (Dentsply-Maillefer, Ballaigues, Switzerland). For the warm vertical compaction technique, the cone was cut off at the canal orifice, and a Calamus Dual heated plugger M (Dentsply Maillefer, Balagues, Switzerland) was inserted into the obturation mass 5 mm from the working length. The master cone was cut off at that length, and the filling material was vertically compacted with a cold Machtou #1 plugger. The middle and coronal thirds were backfilled (thermoplasticized gutta-percha) using the Calamus dual device. Obturation using Guttacore involved applying the BS to the canal wall in the middle and coronal portions of the canal, inserting a #50 Guttacore obturator heated in a Therma-Prep oven (Dentsply-Maillefer, Ballaigues, Switzerland) following the manufacturer's instructions, removing excess carrier and gutta-percha above the orifice, and trimming the obturation mass at the orifice.

After confirming proper obturation on radiovisiography images, the samples were wrapped in saline-soaked gauze and stored in a calibrated oven at 37°C and 100% humidity for 2 weeks to allow the sealer to set completely.

To test bond strength, the samples were sectioned to obtain a 1mm-thick section 3 mm from the working length using a MicroDisc NH 6P cutting machine under water cooling. The obtained sections were subjected to a push-out bond test using a universal testing machine. Push-out pin diameter was selected and confirmed using a caliper to ensure it was adequate for the tested root canal. The force needed to dislodge the filling material from the dentin wall is expressed in Newtons (N); results were recorded in an Excel file. The tested sections were then examined under a light microscope at 100 magnification, photographed, and classified according to mode of failure as follows.

1. **Adhesive**: when the sealer detached completely from the dentine (Fig. 1*A*).
2. **Non adhesive**: when failure occurred inside the sealer (cohesive failure: Fig. 1*B*), or there was a combination of adhesive and cohesive failure (mixed failure: Fig. 1*C*).

The Push out test data were statistically analyzed using two-way ANOVA with materials and technique as independent variables and force as the dependent variable, establishing an α value of 5% to determine the effect of each variable and their interaction. SPSS software (IBM Corp) was used for the analysis. Failure mode was analyzed using Chi-square test to determine significant differences at an α value of 5%.

RESULTS

Figure 2 shows the results of the Push out test in terms of maximum recorded force and standard deviation. Interestingly, Bio C sealer with all 3 techniques, CeraSeal combined with HC, and BioRoot used with WVC showed the highest values. The high standard deviation in all the experimental groups is noteworthy (Fig. 2).

Multifactorial analysis of variance showed no significant differences in maximum recorded force among sealers, methods, or

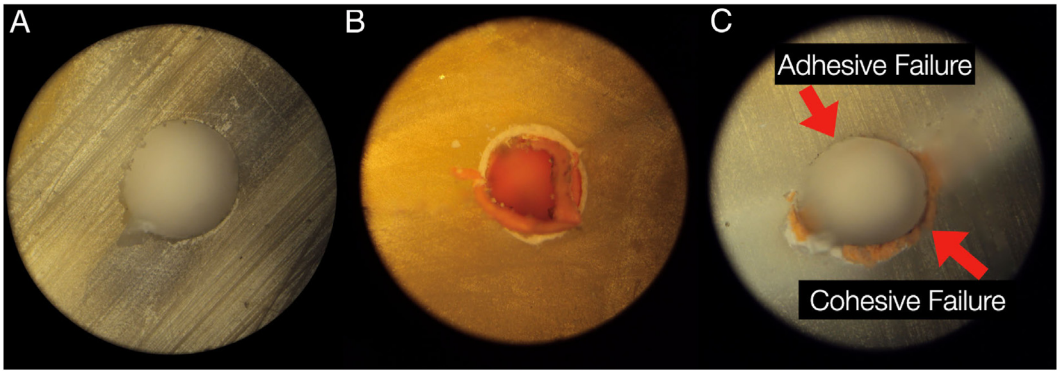

FIGURE 1 – A: Adhesive failure; B: Cohesive failure; C: Mixed failure.

when considering the interaction between sealers and techniques ($P > .05$) (Table 1).

DISCUSSION

The development of bioceramic materials established a new trend in endodontics, making them the material of choice for many dental professionals. As described by **Guivarc'h et al**, the use of these materials increased sharply in the last decade[15]. However, in their study on an experimental bioceramic combined with WVC, Camilleri et al concluded that the choice of the sealer had to be considered when selecting the filling technique[11]. Tricalcium silicate-based sealers are recommended for obturation with a single cold gutta-percha cone or hydraulic condensation. Although there is little information on the behavior of these materials on applying heat during obturation, new tricalcium silicate-based sealers for use in combination with warm obturation techniques have been introduced on the market.

Hadis and Camilleri compared premixed hydraulic sealers for application with warm and cold obturation techniques similar to those used in our study. They found that both BC sealer (Brasseler) and HIFLOW were resistant to heat and had identical chemical properties, except for modifications to the organic component[24]. **Eid et al** compared dentinal tubule penetration of Bio -C- Sealer (Angelus) and HiFlow (Brasseler) combined with WVC and HC. The authors observed that WVC enhanced penetration of the calcium silicate-based sealers into the dentinal tubules and found no differences between HiFlow and Bio-C Sealer. Although BS are recommended for HC, the authors concluded that it would be of interest to reconsider using them with warm obturation techniques[25]. In line with the report by **Eid et al**, the present results showed that adhesion to the dentine wall of the sealers studied here was not affected by the heat generated during warm obturation.

Castagnola et al compared the frequency and volume of void inside root canals obturated with HC or Guttacore using micro-computed tomography (micro-CT). The carrier-based method generates hydraulic forces that move the flowable gutta-percha that covers the core so that the root filling presents mostly gutta-percha and only a thin layer of sealer or none at all[26,27]. This characteristic could decrease bonding of the sealer to the dentine, as shown by the thinner layer of sealer observed in the samples studied here as a result of the obturation technique. The same occurs when using the WVC. Nevertheless, this behavior may not be clinically significant if the root canal is hermetically sealed - a prime objective of endodontic obturation procedures.

The push-out test is commonly used to evaluate push-out resistance to dislodgement of different materials. It has been posited that

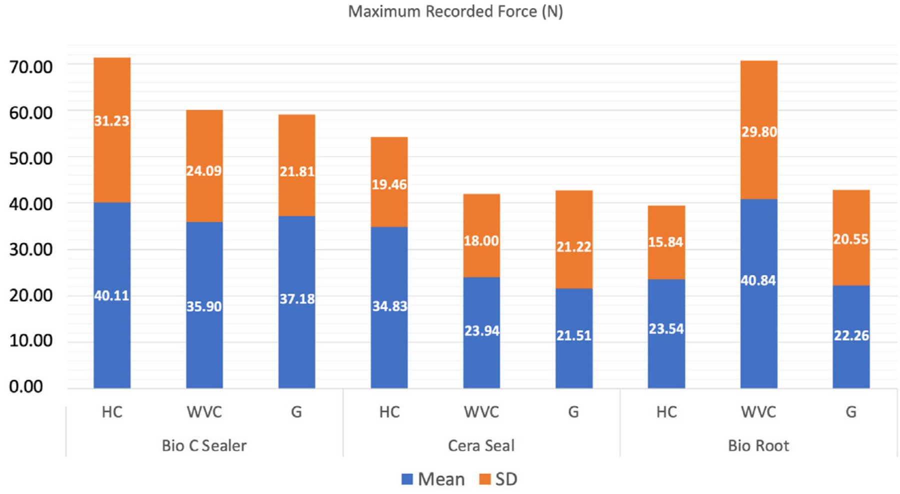

FIGURE 2 – Push-out test results: *blue bars* show mean maximum recorded force for each group expressed in Newton, and *orange bars* show standard deviation. Numeric values are shown *inside bars*. Chi-square test for failure mode showed no significant differences in mode of failure (Chi-square (df 2) = 0.86; $P > .05$).

TABLE 1 - ANOVA of Push-Out Bond Strength. Source as in Independent Variables. Value: Maximum Recorded Force. Df: Degrees of Freedom. Sig: *P* Value

Source	Dependent variable		Sum of type III squares	df	Quadratic mean	F	Sig.
Sealer	dimension 1	Value	2860452.667	2	1430226.333	1.273	.288
		Failure	.381	2	.190	.216	.807
Technique	dimension 1	Value	2375521.810	2	1187760.905	1.057	.355
		Failure	.286	2	.143	.162	.851
Sealer * Technique	dimension 1	Value	3353860.667	4	838465.167	.746	.565
		Failure	9.048	4	2.262	2.560	.049
Error	dimension 1	Value	6.067E7	54	1123565.190		
		Failure	47.714	54	.884		
Total	dimension 1	Value	8.084E7	63			
		Failure	286.000	63			
Corrected total	dimension 1	Value	6.926E7	62			
		Failure	57.429	62			

a. R squared = .124 (adjusted R squared = −.006)
b. R squared = .169 (adjusted R squared = .046)

the test does not entirely replicate the clinical performance of root canal filling materials. Despite its limitations, however, the push-out test is a suitable, widely validated method to compare different materials and obturation techniques[28]. **Chen et al** concluded that the most crucial step when performing this sort of test is determining the ratio of the pin diameter to the diameter of the specimen[29]. In keeping with Chen et al., we measured the diameter of the push-out pin using a digital caliper and calibrated it to correspond with the diameter of the root canal preparation. **In their 2021 study, Retana-Lobo et al** used the push-out test to evaluate four endodontic sealers: a resin-based sealer (AHPlus) and three BS (EndoSequence BC Sealer, ProRoot Endo Sealer, and Bio-Root RCS). The authors sought to evaluate the push-out bond strength of premixed and powder-liquid BS with and without a gutta-percha cone. Interestingly, they found that push-out strength of the hydraulic sealers was greater when used without a gutta-percha cone[30]. **Dewi et al** investigated the effect of a heat-based obturation technique on the bond strength of a classic BS and the new HiFlow sealer in single-rooted human lower premolars using the push out test[23]. As in the present study, they observed no significant difference in push-out bond strength among the 4 groups of bioceramics and found mixed failure to be the most common mode.

The most prevalent failure mode in the study by **Al-Hiyasat et al** mentioned above was mixed failure, regardless of the sealer and obturation technique[31]. **Renata-Lobo** found that bond strength was significantly higher in samples obturated with BioRoot than in those filled with EndoSequence BC Sealer, irrespective of the obturation technique, and that adhesive failure was the most frequent failure mode[30]. Our results showed no significant differences in mode of failure among sealers or among the obturation techniques. Of note, however, 32 of the 63 samples displayed adhesive failure and 31 nonadhesive failure (26 showed mixed failure, and only 5 exhibited cohesive failure).

Chen et al and Pane et al showed that the mode of failure is associated with the type of material, among other factors, and underscored the relevance of calibrating the punch diameter with the diameter of the tested root canal[27,29]. The present results indicate that the more accurately the punch diameter coincides with the diameter of the root canal, the higher the likelihood of adhesive and mixed failure. An inadequate fit resulting in the punch plunging in the center of the gutta-percha or slightly grazing the inner perimeter of the sealer increases the likelihood of cohesive failure. The central part of the obturation is dislodged, and the sealer remains attached to the canal wall because it is stronger than the sealer/gutta-percha interface. Our observations are in agreement with those reported by the authors cited above regarding the importance of properly calibrating the push-out tip according to the diameter of the root canal to be tested before conducting the final tests, as we did in the pilot study we performed before this research.

CONCLUSIONS

Under the experimental conditions used in the present study, the heat generated by the obturation techniques did not affect adhesion of the studied bioceramic sealers to the dentinal wall.

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The authors deny any conflicts of interest related to this study.

In memory of my dear mentor Dra. Mini Baroffi (1948-2023).

REFERENCES

1. Soares I, Goldberg F. Endodoncia: Técnicas y fundamentos. 2 ed. Editorial Médica Panamericana; 2012. p. 225–342.
2. Siqueira JF Jr, Rôças IN. Present status and future directions: microbiology of endodontic infections. Int Endod J 2022;55(Suppl 3):512–30.
3. Ng YL, Mann V, Gulabivala K. A prospective study of the factors affecting outcomes of nonsurgical root canal treatment: part 1: periapical health. Int Endod J 2011;44:583–609.
4. Goldberg F, Araujo J. Estudio comparativo de la penetración de la gutapercha en las irregularidades del conducto radicular. Sistema Guttacore y técnica de cono único. Rev Asoc Odontol Argent 2016;104:4–8.
5. Cohen S, Hargreaves K. Pathways of the pulp. 11 ed. Elsevier; 2016.
6. Lee SJ, Monsef M, Torabinejad M. Sealing ability of a mineral trioxide aggregate for repair of lateral root perforations. J Endod 1993;19:541–4.

7. Zafar K, Jamal S, Ghafoor R. Bio-active cements-mineral trioxide aggregate based calcium silicate materials: a narrative review. J Pak Med Assoc 2020;70:497–504.
8. Darvell BW, Wu RC. "MTA"- an hydraulic silicate cement: review update and setting reaction. Dent Mater 2011;27:407–22.
9. Kohli MR, Yamaguchi M, Setzer FC, Karabucak B. Spectrophotometric Analysis of Coronal Tooth Discoloration Induced by Various Bioceramic Cements and Other Endodontic Materials. J Endod 2015;41:1862–6.
10. Camilleri J, Sorrentino F, Damidot D. Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, Biodentine and MTA Angelus. Dent Mater 2013;29:580–93.
11. Camilleri J. Sealers and warm gutta-percha obturation techniques. J Endod 2015;41:72–8.
12. Viapiana R, Flumignan DL, Guerreiro-Tanomaru JM, et al. Physicochemical and mechanical properties of zirconium oxide and niobium oxide modified Portland cement-based experimental endodontic sealers. Int Endod J 2014;47:437–48.
13. Zhou HM, Shen Y, Zheng W, et al. Physical properties of 5 root canal sealers. J Endod 2013;39:1281–6.
14. Pommel L, Camps J. *In vitro* apical leakage of system B compared with other filling techniques. J Endod 2001;27:449–51.
15. Guivarc'h M, Jeanneau C, Giraud T, et al. An international survey on the use of calcium silicate-based sealers in non-surgical endodontic treatment. Clin Oral Investig 2020;24:417–24.
16. Heran J, Khalid S, Albaaj F, et al. The single cone obturation technique with a modified warm filler. J Dent 2019;89:103181.
17. Zavattini A, Knight A, Foschi F, Mannocci F. Outcome of root canal treatments using a new calcium silicate root canal sealer: a Non-randomized Clinical Trial. J Clin Med 2020;9:782.
18. Migliau G, Palaia G, Pergolini D, et al. Comparison of two root canal filling techniques: obturation with guttacore carrier based system and obturation with Guttaflow2 Fluid Gutta-Percha. Dent J 2022;10:71.
19. Alberdi J, Martin G. Selladores biocerámicos y técnicas de obturación en endodoncia. Refo 2021;1:17–23.
20. Camilleri J, Atmeh A, Li X, Meschi N. Present status and future directions: hydraulic materials for endodontic use. Int Endod J 2022;55(Suppl 3):710–77.
21. Atmeh AR, AlShwaimi E. The effect of heating time and temperature on epoxy resin and calcium silicate-based endodontic sealers. J Endod 2017;43:2112–8.
22. Donnermeyer D, Ibing M, Bürklein S, et al. Physico-chemical investigation of endodontic sealers exposed to simulated intracanal heat application: hydraulic calcium silicate-based sealers. Materials 2021;14:728.
23. Dewi A, Upara C, Sastraruji T, Louwakul P. Effect of a heat-based root canal obturation technique on push-out bond strength of the classical bioceramic and new HiFlow sealer. Aust Endod J 2022;48:116–20.
24. Hadis M, Camilleri J. Characterization of heat resistant hydraulic sealer for warm vertical obturation. Dent Mater 2020;36:1183–9.
25. Eid D, Medioni E, De-Deus G, et al. Impact of warm vertical compaction on the sealing ability of calcium silicate-based sealers: a confocal microscopic evaluation. Materials 2021;14:372.
26. Castagnola R, Marigo L, Pecci R, et al. Micro-CT evaluation of two different root canal filling techniques. Eur Rev Med Pharmacol Sci 2018;22:4778–83.
27. Pane ES, Palamara JE, Messer HH. Critical evaluation of the push-out test for root canal filling materials. J Endod 2013;39:669–73.
28. Collares FM, Portella FF, Rodrigues SB, et al. The influence of methodological variables on the push-out resistance to dislodgement of root filling materials: a meta-regression analysis. Int Endod J 2016;49:836–49.
29. Chen WP, Chen YY, Huang SH, Lin CP. Limitations of push-out test in bond strength measurement. J Endod 2013;39:283–7.
30. Retana-Lobo C, Tanomaru-Filho M, Guerreiro-Tanomaru JM, et al. Push-out bond strength, characterization, and ion release of premixed and powder-liquid bioceramic sealers with or without gutta-percha. Scanning 2021;6:6617930.
31. Al-Hiyasat AS, Alfirjani SA. The effect of obturation techniques on the push-out bond strength of a premixed bioceramic root canal sealer. J Dent 2019;89:103169.