


A comparative evaluation of the sealing ability of two calcium silicate based sealers and a resin epoxy-based sealer through scanning electron microscopy and bond strength: an in vitro study

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Editor: Dr Altair A. Del Bel Cury

Received: January 24, 2021

Accepted: May 5, 2021

Aim: this study aimed to compare the sealing ability of two types of commercially available calcium silicate bioceramic based root canal sealers and a resin based root canal sealer.

Methods: Twenty one single-rooted teeth were used, samples (n= 21) were randomly divided into three groups according to the sealer used (group A; ADSEAL, group B; Wellroot, group C; Ceraseal). Roots were then cleaved longitudinally in the labiolingual direction; all samples were then sectioned at three, six, and nine mm from the root tip. The penetration of sealers into the dentinal tubules was examined at 1000x with a scanning electron microscope. Data were tested for normality using Shapiro Wilk test. ANOVA test was used for analyzing normally distributed data followed by Bonferroni post hoc test for pair-wise comparison. Significance level $p \leq 0.001$.

Results: groups B and C showed better sealing ability than group A in all the three sections. The coronal section showed higher sealing ability than the middle section followed by the apical section in the three tested groups. **Conclusion:** it can be concluded that both calcium silicate-based sealers had better sealing ability and higher bond strength than the resin epoxy- based sealer.

Keywords: Calcium compounds. Silicates. Epoxy resins. Root canal filling materials. Electron microscope tomography.



Introduction

Efficient filling and complete sealing of the previously cleaned and shaped root canal system are crucial steps that have an impact on the long-term success of the treatment^{1,2}. Incorporation of sealers is mandatory as gutta-percha does not adhere to the dentinal walls and thus cannot prevent leakage by it-self, accordingly sealers are used to fill the irregularities and to penetrate into dentinal tubules attempting to achieve a hermetic seal of the root canal system². Therefore, root canal sealers should attain strong adherence between gutta-percha and the dentinal walls preventing gap existence at the sealer-dentine interface^{2,3}. Thus achieving a three-dimensional seal of root canal which is essential to ensure complete prevention of reinjection of the canal and for preserving the health of the periapical tissues, consequently ensuring successful treatment^{1,4}.

There is no real chemical bond between root canal sealers and the dentinal wall of root canals; however, tubular penetration of root canal sealers may enhance the micromechanical bonding of sealers and subsequently their sealing properties⁵.

Adaptation of a sealer to the dentinal wall is evaluated using different ways; stereo-microscope, confocal laser microscopy, scanning electron microscopy (SEM), leakage tests and digital imaging³.

Currently, there is a variety of commercially available sealers. However, none of the existing sealers satisfies all the required idealistic properties³.

Calcium silicate based sealers have attracted clinicians due to their excellent biocompatibility and bioactivity as claimed by their manufacturers, combined with their ability to bond to the tooth structure^{2,6}. However, there is scarce literature on their properties and performance in vitro and in vivo.

Accordingly, this in-vitro study aimed to compare the sealing ability of two types of commercially available calcium silicate based root canal sealers and a resin based root canal sealer. The study was performed under the null hypothesis that no differences in the ability of sealing the dental tubules would be observed between the three tested root canal sealants.

Materials and methods

Materials used in this study were; ADSEAL, Well-Root ST and CeraSeal.

Table 1. materials used in this study, their manufacturer, lot number and composition.

Materials	Manufacturer	Lot number	Composition
ADSEAL	META BIOMED Korea	ADS1406171	A two paste system; - Base: • Epoxy oligomer resin. • Ethylene glycol salicylate. • Calcium phosphate. • Bismuth subcarbonate. • Zirconium oxide.

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ADSEAL	META BIOMED Korea	ADS1406171	- Catalyst: • Poly aminobenzoate. • Triethanolamine. • Calcium phosphate. • Bismuth subcarbonate. • Zirconium oxide. • Calcium oxide
Well-Root ST	Vericom, Gangwon-Do, Korea	WR8N0200	- Calcium aluminosilicate compound. - Zirconium oxide - Filler and thickening agent.
CeraSeal	META BIOMED Korea	CSL1912161	- 2g Pre-mixed syringe × 1EA Intra canal tips × 15EA

Methods

Twenty-one recently extracted human upper incisors with straight fully formed roots were selected. All teeth were inspected under stereomicroscope (Image J, Earl F, Glynn II, and Over Park, USA) at a magnification of (10x). Incisors with more than one canal, open apex, endodontically treated, Internal or external resorption, caries, cracks or fractures on the root surfaces were excluded.

Five from each group were used for push out bond strength testing. A total sample size of 15(5 sample in each group) will be sufficient to detect an effect size of 2.18, a power (1-β error) of 0.85, using a two-sided hypothesis test, significance level (α error) 0.05 for data. The remaining two samples were used for scanning.

Teeth were cleaned using ultrasonic scalar, and then placed in 2.5%NaOCl for 30 minutes for surface disinfection then stored in distilled water until use. Crowns were removed at the level of cement-enamel junction by the use of micro-saw under water cooling (IsoMet 4000 micro saw, Buehler,USA), leaving averagely 15 mm long root segments.

Working lengths were recognized for all canals by a # K file (Mani, Tochigi, Japan). Cleaning and shaping were conducted by the use of ProTaper system (DENTSPLY Maillefer, Ballaigues, Switzerland) starting by; Sx, followed by S1, S2 in a brushing motion, followed by F1,F2 F3,F4 and F5 in a non brushing motion. All root canals were irrigated during cleaning and shaping with 5ml of 25% NaOCl solution using end-perforated 27 gauge needle SUNG SHIM, Seoul, Korea) to ensure efficient cleanliness of the canal.

After complete instrumentation, all samples we placed in a glass box (n= 21), and were randomly divided by a blind technician in to three groups (n=7) according to the type of sealer used.

Samples were obturated with Protaper universal gutta percha points and the type of sealer was used according to its group (group A: ADSEAL, group B: Well-Root and group C: Ceraseal) using lateral condensation technique. Roots were then coded according to the type of sealer used and stored in a moist environment for 1 week to ensure complete setting of the sealers before testing.

1. Scanning electron microscopy:

Two roots from each group were cleaved longitudinally in the labio-lingual direction using a hammer and chisel, all samples were then sectioned at three, six, and nine mm from the root tip using a 0.3 mm disk thickness^{7,8}. The penetration of sealers into the dentinal tubules and adaptation of each sealer to the canal wall were examined at 1000x magnification with a scanning electron microscope (Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V (Netherland).

2. Push out bond strength:

Five roots from each group were tested. Each root was embedded in a centralized manner in acrylic resin using a transparent plastic mold (diameter 10 mm, length 16mm) so that the tooth surface flushes with the upper acrylic surface. After setting of the acrylic resin, coronal, middle and apical thirds were defined and a section of 1mm thickness was cut from the center of each third using water-cooled precision microsaw⁹. Test was conducted using computer-controlled universal testing machine (Instron universal testing machine model 3354 instron instruments England) with a load cell of 5-KN. A plunger with diameter of (0.9mm or 0.7mm or 0.5mm) acted as a force probe to apply a push out load at a crosshead speed of 0.5mm/min in an apical coronal direction. The selected diameter of the plunger was chosen so that it only contacts the filling to displace it downwards⁹. The maximum failure load was recorded in newtons (N) and was used to calculate the push-out bond strength in mega pascals (Mpa) according to the following formula.

Shear bond strength (Mpa) = Maximum load in (N) / Adhesion area of root canal filling (mm²)

Data were presented as mean and standard deviation. Data were tested for normality using Shapiro Wilk test. ANOVA test was used for analyzing normally distributed data followed by Bonferroni post hoc test for pair-wise comparison. Analysis was performed using IBM SPSS Statistics for Windows, Version 25.0. Armonk, NY: IBM Corp.



Figure 1. Sections in the coronal, middle and apical thirds

Results

1. Scanning electron microscopy:

The morphology of sealer/ dentin interface of the three tested sealers using a scanning electron microscope were evaluated, they showed a true hybridization and sealers tags formation inside the dentinal tubules. Group A revealed few numbers of tags having small diameters and a clear interfacial gap. While groups B and C showed numerous number of tags with large diameters protruding into the dentinal tubules with a gap at the sealer/dentin interface as shown in Figure 1.

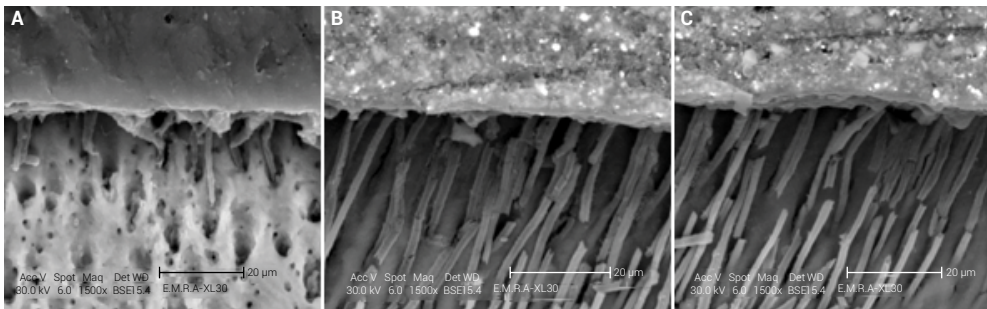


Figure 2. Scanning electron micrograph (1000X) of the interface at sealer/ dentin using A:ADSEAL, B: Well-Root and C: Ceraseal.

2. Push-out comparison between the three groups:

2a. In the coronal, middle and apical thirds between the three groups:

In the coronal section; the mean and standard deviation values of group A were (57.93 ± 1.21), while in group B were (68.49 ± 1.62) and in group C were (73.5 ± 1.88). There was no statistically significant difference between the three groups.

In the middle section; the mean and standard deviation values of group A were (44.46 ± 2.16), while in group B were (63.4 ± 1.41) and in group C were (67.59 ± 1.18). There was no statistically significant difference between the three groups.

In the apical section; the mean and standard deviation values of group A were (4.13 ± 0.71), while in group B were (10.74 ± 0.62) and in group C were (11.6 ± 0.51). There was no statistically significant difference between the three groups.

Table 2. Mean and Standard deviation (SD) and the results of ANOVA test for comparison of push-out (MPa) in the coronal, middle and apical thirds between the three groups:

Coronal	Group A	Group B	Group C	P value
Mean	57.93	68.49	73.50	<0.001
SD	1.21	1.62	1.88	
Middle				

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Continuation				
Mean	44.46	63.4	67.79	<0.001
SD	2.16	1.41	1.18	
Apical				
Mean	4.13	10.74	11.6	<0.001
SD	0.71	0.62	0.51	

Significance level $p \leq 0.001$

2b. Pair wise comparison:

The push-out in the coronal (Figure 3) and middle thirds (Figure 4) exhibited a statistically significant difference between all group pairs. In the apical third (Figure 5), a statistically significant difference was found between groups A and B and between groups A and C, while no statistically significant difference was found between groups B and C.

Table 3. Results of Bonferroni post hoc test for pair-wise comparison of push-out (MPa) in the coronal, middle and apical thirds between the three groups:

Coronal	P-value
Group A – Group B	<0.001*
Group A – Group C	<0.001*
Group B – Group C	<0.001*
Middle	
Group A – Group B	<0.001*
Group A – Group C	<0.001*
Group B – Group C	<0.001*
Apical	
Group A – Group B	<0.001*
Group A – Group C	<0.001*
Group B – Group C	<0.055

Significance level $p \leq 0.001$

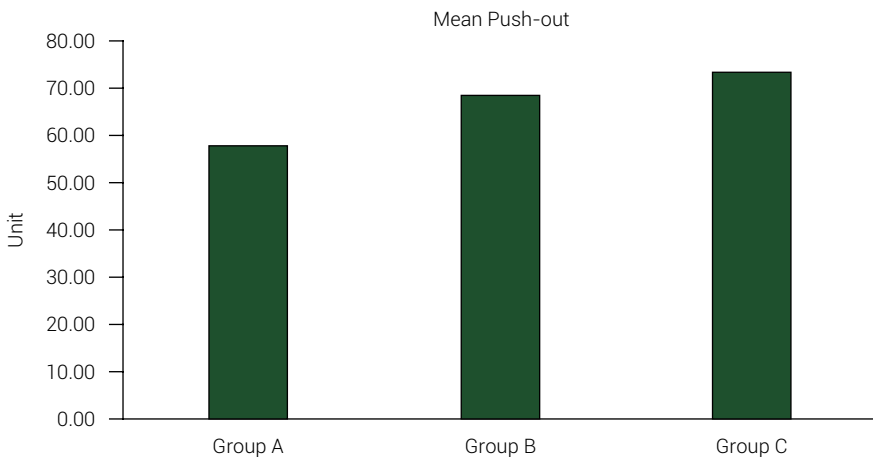


Figure 3. Bar chart representing the mean push-out in the coronal third in the three groups.

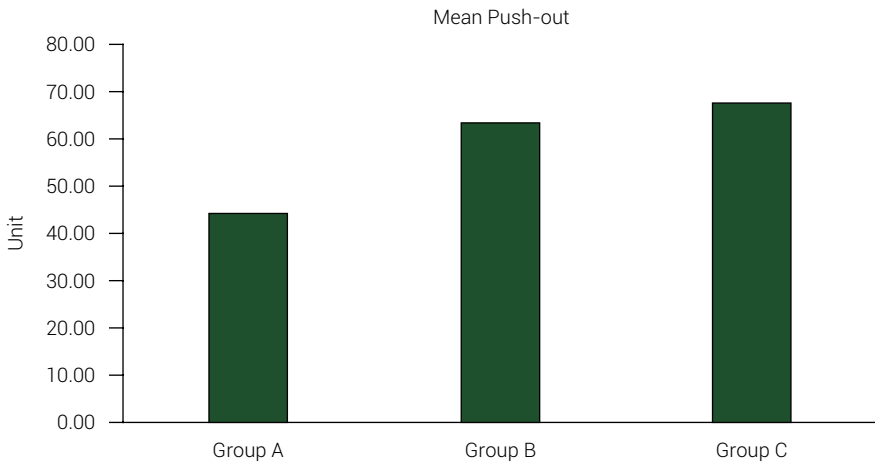


Figure 4. Bar chart representing the mean push-out in the middle third in the three groups.

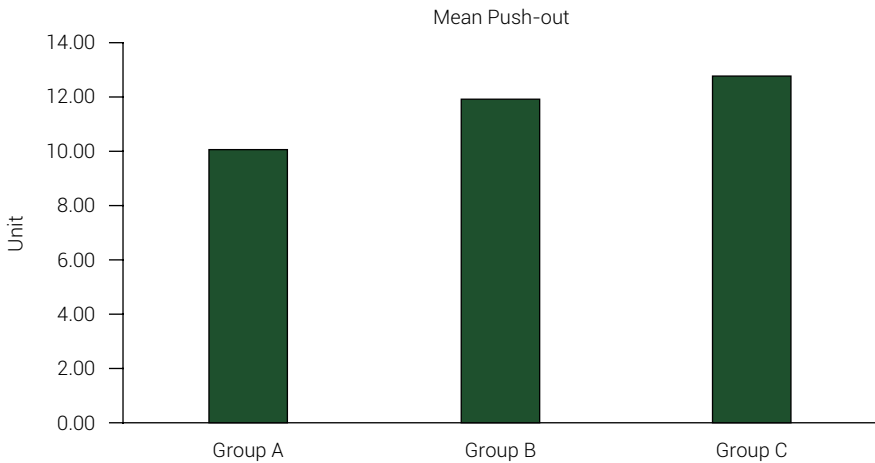


Figure 5. Bar chart representing the mean push-out in the apical third in the three groups.

Discussion

In order to achieve a successful endodontic treatment and good prognosis, it is crucial to have a tight apical seal which is dependent on proper instrumentation and cleaning of the root canal system in conjunction to adequate obturation¹⁰. Penetration of root canal sealers into dentinal tubules allows a better sealing ability thus preventing residual bacteria from re-growing within the tubular space¹¹.

Accordingly, there is a continuous improvement in the field of root canal filling materials and recently bioactive materials are becoming of high demand⁷.

Calcium silicate-based sealers are hydrophilic in nature, possess an alkaline pH, insoluble in tissue fluids and they don't shrink on setting. Moreover, the moisture environment of the tooth has an influence on the adhesion between the bioceramic sealers and root canal dentin. These sealers are characterized by their potential bio-

active properties, where calcium hydroxide and hydroxyapatite are formed once the sealer contacts water, resulting in a high alkaline pH that activates and initiates the expression of alkaline phosphatase, favoring the formation of mineralized tissue and possessing an antibacterial effect¹²⁻¹⁴.

An resin epoxy-based sealer was used in this study because it is characterized by its good adherence to the root canal dentin, outstanding biocompatibility and low risk of unfavorable postoperative inflammatory reactions¹².

Several methods have been postulated for evaluating the sealing ability of obturation materials; dye penetration, fluid filtration techniques, radioisotopes, scanning electronic microscopic analysis, electrochemical leakage tests, glucose penetration and bacterial penetration test^{13,15}.

Scanning electronic microscope was utilized in this study as it allows proper evaluation of the sealing ability and adhesiveness of the sealer to dentin walls or sealer-gutta-percha interface on the various levels of root sections¹⁵. Moreover it provides high magnification thus allows better observation of surface topography¹¹.

Improvement in the sealing ability of the sealers is achieved through the mechanical interlocking of the sealer plug inside the dentinal tubules (push-out). A strong bond is critical in order to maintain the integrity of the sealer-dentin interface¹⁶.

Therefore, this study aimed to evaluate and compare the sealing ability of two types of calcium silicate-based sealers and a resin epoxy-based root canal sealer by the use of scanning electron microscopy (SEM) and bond strength.

Results of the current study revealed that groups B and C had higher push-out bond strength than group A in the three root sections namely; coronal, middle and apical.

This can be attributed to the excellent physical properties of the calcium silicate-based sealers such as flow, low film thickness and dimensional stability. In addition to the alkaline nature of the byproducts produced by the calcium silicate-based sealers that might have denatured the dentin collagen fibers thus facilitated the sealers penetration. This was in accordance with Baruah et al.¹³ in 2018.

While, the lower bond strength exhibited by group C could be due to the incomplete polymerization and the setting shrinkage of its resinous components resulting in formation of poor microtags which consequently exhibits low adhesion properties. This was also suggested by Huang et al.⁷ in 2018 and Baruah et al.¹³ in 2018.

In the present study, results of the sealers push-out strength revealed that coronal section exhibited the highest mean value followed by the middle section while, the apical section had the lowest mean value in the three tested groups. This might result from presence of dentinal tubules with larger diameter at both coronal and middle parts when tested against the apical part¹⁶.

Moreover Wang et al.³ in 2018, stated that despite of the kind of sealers or obturation techniques used, the percentages of penetrated dentinal tubules of root canal increased from apical to coronal part due to the increased amount of eliminated smear layer in the upper-middle section of the root canal.

The fact that the degree of adhesion of the sealers to the dentin wall depends on the surface energy of the dentin, surface tension and wettability of the sealer in addition to the cleanliness of the dentin surface⁷. Dentin in the coronal, middle, and apical sections has different surface energies, in conjunction with obstacles faced during complete removal of the smear layer from the apical region might be the cause of its lower sealer penetration this was in accordance with Huang et al.⁷ in 2018 and Eid et al.¹⁶ in 2019.

These results were confirmed by the descriptive characteristics of the tags revealed by the scanning electron microscopic study as shown in figure (2). Where groups B and C demonstrated a clearly recognizable thin hybrid layer, including numerous numbers of tags with a large diameter protruding into the dentinal tubules. However, group A showed a thin hybrid layer with few numbers of resin tags having a small diameter.

The images obtained through scanning electron microscopy have some limitations; are only representative of sectioned canal levels examined. Further studies are required to evaluate the mineralogical characteristics of both Well Root and CeraSeal when it is in contact with different solutions, in addition to their physiochemical properties.

In conclusion, with the limitations of this study, it can be concluded that both calcium silicate-based sealers had sufficient sealing qualities and higher bond strength than the resin based sealer. The higher bond strength of the two tested calcium silicate-based sealers in conjunction with their bioactivity might help in improvement of the root canal system sealing. This conclusion needs further investigations.

Conflicts of interest

There are no conflicts of interest.

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