EGYPTIAN
DENTAL JOURNAL
www.eda-egypt.org

Vol. 71, 1847:1856, April, 2025

Print ISSN 0070-9484 • Online ISSN 2090-2360

Conservative Dentistry and Endodontics

Submit Date : 31-01-2025 • Accept Date : 10-04-2025 • Available online: 25-04-02025 • DOI : 10.21608/edj.2025.356530.3364

PUSH OUT BOND STRENGTH AND ADHESIVE PATTERN OF A BIOACTIVE GLASS-BASED ROOT CANAL SEALER COMPARED TO CERASEAL® BIOCERAMIC SEALER (AN IN-VITRO STUDY)

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ABSTRACT

Aim: To compare the push out bond strength and adhesive pattern of a bioactive glass (BG) based root canal sealer with CeraSeal® Bioceramic sealer.

Materials and Methods: Sixteen extracted single-root human canine teeth were prepared using Protaper Next rotary files. During instrumentation, 2.5% NaOCl was used for irrigation to eliminate all debris and 17% EDTA as the last irrigant to eliminate the smear layer. The canals were dried using paper points. Sixteen samples were divided equally into two test groups, and one of the pre-selected root canal sealers was applied to obturate the samples using the cold lateral compaction (CLC) technique. The samples, which represent the middle third of root canals, were sectioned horizontally between 5 and 7 mm from the apex. The disc measured 1±0.1 mm in width. Both the adhesive pattern analysis and the push-out test were performed.

Results: The results of the two groups were compared by using Independent t test which showed that the BG based sealer showed insignificant higher mean push-out bond strength (PBS) compared to CeraSeal as (P=0.18).

Conclusion: Our research concluded that the prepared BG based sealer showed relatively comparable bond strength values and adhesive pattern with CeraSeal Bioceramic sealer when used in CLC technique.

KEYWORDS: Push out bond strength, adhesive pattern, Bioactive glass, CeraSeal, cold lateral condensation.

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INTRODUCTION

An effective endodontic treatment requires efficient cleaning of the infected root canal system, shape, and canal space obturation in three dimensions to prevent pulpal cavity re-infection. Insufficient sealing and microorganism clearance are the primary causes of root canal treatments failure, which might cause canal or peri-radicular space infection. [1, 2].

Despite being obturation's "gold standard, gutta-percha (GP) lacks the ability to entirely seal the canal lumen as it cannot adhere to the radicular dentin, leaving a leaky root canal [3]. Therefore, to create a strong connection between the radicular dentin and the master cone, an endodontic sealer is a must [4]. Sealers including the silicone based, methacrylate, calcium hydroxide, resin, MTA, and bioceramics (BCs), have been introduced. Their ability to form a strong connection with the radicular dentinal wall has been studied [5]. Recently bioceramic materials are perfect for endodontic treatments due to their unique physicochemical and biological characteristics. They are suitable for use in root canal therapy, as they can prevent reinfection and maintain sealing ability. BCs were first used in endodontics as retrograde filling materials in the 1990s. They are chemically and dimensionally stable, biocompatible, and non-toxic in the biological environment. Since their introduction, bioceramics hydraulic and hydration properties have generated the greatest concern [6]. When tissue fluid is present, the sealer undergoes a hydration reaction to create calcium hydroxide and calcium silicate hydrate, which then combine to form a coating of hydroxyapatite[7]. The cement's hydrophilic properties make it a great option for the root canal system. CeraSeal is a premixed calcium silicate-based sealer from Meta Biomed Co. in Cheongju, Korea. Tricalcium silicate, Dicalcium silicate, Tricalcium aluminate, Zirconium oxide, and an agent for adjusting consistency are all included in a single premixed syringe. That has "exceptional stability," the developers say. They believe that it has excellent sealing abilities as well [8].

BG is a kind of bioactive ceramic made of P2O5, SiO2, CaO5, and Na2O. It works effectively as a material for repairs in a range of endodontic procedures [9]. BG possesses sufficient strength and load-bearing capacity, as well as acceptable handling and functional qualities [10]. BG has properties called osteoconductivity and osteoinductivity [9], can greatly speed up the kinetics of tissue healing by encouraging bone cells to proliferate and repair themselves. Although BG has primarily been utilised in applications involving contact with bone tissue, it has lately demonstrated the ability to induce regeneration in soft tissues as well [10]. Since its ionic dissolution products have been shown to promote angiogenesis, it has caught the attention of numerous researchers studying soft tissues. Other BG based medicines are also available for use in peripheral nerve regeneration and wound repair [11]. These uses imply that BG is a biomaterial that is suitable and biocompatible for use on soft tissues like dental pulp and periapical tissue as well as hard tissues like dentin or cementum.

On a previous study of Huang et al, they used a BG based mixture, a new neutral BG called PSC with a chemical preparation of 10.8% P2O5, 54.2% SiO2, 35% CaO (mol.%) [12]. Based on this previous study of Huang et al, the new BG Sealer adheres well to the root dentin, can create minerals by penetrating the dentinal tubules, and can increase the roots' resistance to fracture following root canal therapy. They anticipated that it would be a bioactive root canal sealant with promising clinical applications [13].

Push-out bond strength test is mainly utilized to study endodontic sealers' resistance to dislodging from the root dentine wall [14, 15]. In this mechanical test, the root sample's is subjected to a compressive load longitudinally along the central axis until the sealer and core material are forced out or dislodged. It could be performed to assess parallel-sided samples while the bond strength is minimum since it is repeatable, and fracture takes place parallel to the adhesive attachment [16]. The test also has the advantage of being able to evaluate materials inside the root canal without the need for extremely complex equipment, making it more advantageous than other tests measuring bond strength including shear and tensile tests [17].

Since the main goal of an endodontic sealer is to create a connection between the GP with the root dentine, the quality of this adhesive connection guarantees successful outcome of the treatment [18]. The physical characteristics and chemical makeup of the sealer used, as well as humidity, primarily affect the adhesive strength [19]. Therefore, as a sealer's adhesion to the root surface is properly established, it will exhibit favourable adhesive patterns and improved push-out bond strength [20].

To the best of our knowledge, no previous research has studied the selected sealers bonding pattern to dentine walls. This study used a systematic classification to determine the type of adhesion pattern of sealer to the radicular dentin surface, offering a straightforward and useful way to evaluate the materials' adhesive pattern. Until now, there is no standardisation in the evaluation of endodontic sealers' adhesive patterns since researchers have not agreed upon any evaluation criteria. It is also essential to emphasise that sealers' adhesive qualities may be partly related to their bond strength, as demonstrated by a previous study finding [5]. Hence, the goal of our current research is to compare BG based and CeraSeal® sealers according to their push out bond strength and adhesive pattern.

MATERIALS AND METHODS

The research proposal has been registered and exempted by Institutional Review Board Organization IORG0010868, Faculty of Oral and Dental Medicine, Ahram Canadian University with the research number IRB00012891#134. A total of 16 samples (i.e., 8 samples per group) were the expected sample size (n), which was determined by using an alpha (α) level of 0.05, a beta (β) of 0.2 (i.e., power=80%), and an effect size (d) of 1.51 based on the findings of a prior study [21]. R statistical analysis software, version 4.3.2 for Windows, was used to calculate the sample size [22].

Sample selection

Sixteen freshly extracted human canines with single root canals and mature, straight roots were gathered. Teeth were checked to ensure they are free of both coronal and radicular caries, fractures, restorations, abrasions, and root resorption. The selected teeth root length was ranging between 16-17 mm. To eliminate the left-over soft tissue remnants, samples were soaked in a 2.5% solution of sodium hypochlorite (NaOCl) for 24 hours. The chosen teeth were kept at room temperature in a jar with phosphate buffered saline.

Preparation of BG based sealer

The materials used to prepare the BG used in the BG based sealer were Tetraethyl orthosilicate (TEOS); Mwt: 208.33 (Sigma Aldrich), calcium nitrate hydrate; Mwt: 236 (BioBasic- Canada) and ammonium dihydrogen phosphate; Mwt: 115.03 (Carl Roth GmbH- Germany). The previously mentioned materials were used for preparing Sol Gel (SG) BG. (Table 1)

BG preparation using the SG approach: As shown in the diagram below (Fig. 1), the ternary bioactive glass (S-BG) is composed of SiO2, CaO, P2O5 in a molar composition of 54.2: 35: 10.8. As shown in figure 1, SG processing was used. TEOS: ethanol: distilled water = 1: 1: 1 was the volumetric ratio used for hydrolysis, and nitric acid was used to keep the pH at 2 ± 0.02 with stirring continuously for one hour. After three rounds of distilled water washing and filtering, the resultant solution was centrifuged for ten minutes at 2500 rpm. Following washing, the gel was dried for two hours at 110 °C. and calcined for five hours at 600 °C. (Fig. 1)

TABLE (1) The materials used to prepare the BG containing sealer

No	Material Name	Chemical Formula	Assay	Supplier
1	Tetra ethyl orthosilicate (TEOS)	$C_g H_{20} 0_4 Si$, p=0.932 g/ml M= 208.33 g/mole	99.0%	Sigma Aldrich
2	Calcium nitrate tetra hydrated (CNT)	(Ca (N03)2.4H_2O, M=236.15 g/mole	99.0%	Bio Basic- Canada
3	Ammonium Di-hydrogen Phosphate	$NH_4H_2P0_4$, M=115.03 g/mole	99.0%	Carl Roth GmbH-Germany
4	Concentrated Nitric acid	HN03	55 wt%	El Salam chemicals
5	Ethanol absolute alcohol	C_2H_6O, M = 46.07 g/mol	95.0%	El Salam chemicals

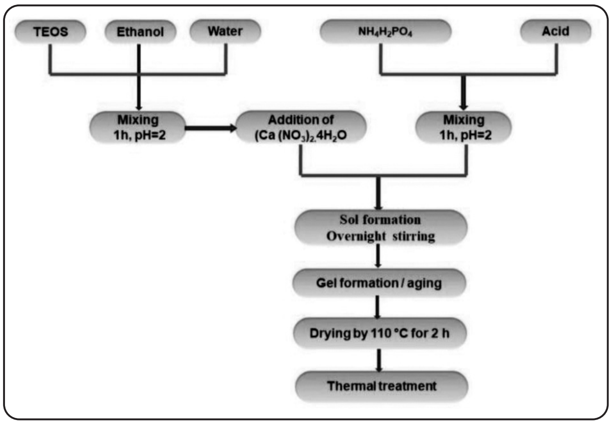

Fig. (1) A diagram showing the BG preparation by SG approach.

The prepared BG was mixed with other components according to Huang et al [23] to prepare the used BG based sealer. It was prepared by mixing powders and liquids. The powder is composed of seventy wt% of the prepared BG, and 30 wt% zirconium oxide used with nano-sized particles (ZrO2, Katana zirconia, Kuraray Noritake Dental Inc., Aichi, Japan). The liquid was composed of four mol/L phosphate buffered saline (PBS, Biowest, France) and sodium alginate (SA, Morgan chemical factories, Cairo, Egypt) with 1% mass volume fraction. To create the BG-based Sealer, a powder/liquid mixture was then made using a 1:1 powder/liquid ratio (g/mL).

Endodontic treatment

Access cavities of the sixteen teeth were done, a 10 k-file was used to guarantee canal patency, followed by working length determination using the apical foramen. ProTaper NEXT nickel titanium files (Dentsply, Maillefer, Ballaigues, Switzerland) were utilized to mechanically instrument the root space in brush strokes with a 300-rpm rotational speed, 2.0-5.2 torque, and powered by X-Smart Plus endodontic motor. Three mL of 2.5% sodium hypochlorite (NaOCl) were used for irrigation after every file, followed by 1 mL of 17% Ethylenediamine Tetra-acetic Acid (EDTA) (Prevest Denpro, Jalandhar, India) to remove the smear layer [21]. The canals were dried by thirty-sized paper points. A random allocation technique was used to divide the samples into two test groups. A pre-selected root canal sealant from the list below was applied to obturate eight samples from each test group, which was related to the sealer type.

Group 1 (n = 8)–Gutta-percha (GP) and BG based sealer

Group 2 (n = 8)–GP and CeraSeal Bioceramic sealer

The apical "tug-back" was confirmed prior to obturation by testing the GP cone size 30 and 4% taper in the root canal. The BG based sealer was mixed with 1:1 powder to liquid ratio, and CeraSeal was used directly from the tube as it is ready to be used. Obturation was carried out by cold lateral compaction technique (CLC). The excess GP was removed by using a hot condenser. Following obturation, 37% orthophosphoric acid was used to etch the coronal cavity walls for 15 seconds, followed by a 30 second rinse. Afterwards, the cavity walls were wet with bonding material and cured with a light cure device for 20 seconds. Finally, the bonded restoration was polished using a composite polishing kit. The restored teeth were incubated for 72 hours at 37°C and 100% humidity to fully set the sealers used. Following incubation, the samples were horizontally cut using a hard tissue saw. Slices were taken between 5 mm and 7 mm away from the root tip, representing the middle one third of the canal lumen. The disc thickness was approximately 1±0.1 mm [5].

Push-out bond strength test

A disc from each tooth was used in each group for conducting the bonding strength testing with the use of a Universal Testing Machine (Instron™ 3365, Massachusetts, UK). The load was applied on each disc at the obturated canal lumen part until the GP was displaced. The force was applied through the obturation materials from the smaller diameter side. The machine stainless-steel plunger was cylindrical and of 1 mm tip diameter.

The device speed was chosen as 1mm/min. and the load exerted to dislodge GP was measured in Newton (N). The push-out bond strength was determined using the following equation:

Push-out bond strength (MPa) = maximum load (N)/adhesion area (mm^2)

The bonded area was calculated by utilizing the subsequent equation: π(r1+r2) [(r1−r2)2+h2] where π = 3.14, r_1 is for the radius coronally, r_2 is for the radius apically, and h is for the thickness of the disc, which is estimated to be 1±0.1 mm. [24].

$$\pi\left(r_1+r_2\right)\left[\sqrt{\left(r_1-r_2\right)^2+h^2}\right]$$

Assessment of adhesive pattern

Using the same disc of the push-out bond strength test, the residual sealer on the radicular dentin wall was examined under a Stereo microscope (Olympus, Japan) mounted on (Canon EOS 650D) Camera to study the adhesive pattern. The pictures were taken at 40x magnification. The adhesive pattern of the two sealers was categorised into four types based on Lin et al study [5] in 2021.

Type 1: sealer was recognised in one quadrant.

Type 2: sealer was recognised in two quadrants.

Type 3: sealer was recognised in three quadrants.

Type 4: sealer was recognised in all four quadrants.

Dentinal walls devoid from sealer was considered 'non-adhesive' [5].

Statistical analysis:

Statistical analysis was done using SPSS 16 ® (Statistical Package for Scientific Studies), Graph pad prism & windows excel then presented in tables and graphs. Assessment of the provided data was carried out using the Shapiro-Wilk test and Kolmogorov-Smirnov test for normality which revealed that data originated from normal data distribution in both groups. Consequently, groups comparison was conducted by using Independent t test. The significance level was set at $P \leq 0.05$. For Adhesive pattern, groups comparison was conducted using Chi square test and the significant level was set at $P \leq 0.05$.

Assessment of Push Out Bond Strength

Table (2) presents descriptive results from comparing the push-out bond strength of the two root canal sealers: BG based sealer and CeraSeal. (Fig. 2) represents the values of the tested groups.

BG-based Sealer Results: Minimum: 26.54**,** Maximum: 76.99**,** Mean ± Standard Deviation: 58.11 **±** 15.55.

TABLE (2) Descriptive results of Push out Bond Strength of a BG-Based Root Canal Sealer Compared To CeraSeal (In-Vitro Study)

	Descriptive				**Mean Difference**	**Std. Error Difference**	**95% Confidence Interval of the Difference**		**P value**
	Minimum	Maximum	Mean	Standard Deviation			Lower	Upper	
BG-based sealer	26.54	76.99	58.11	15.55	11.31	8.10	-6.07	28.69	0.18
CeraSeal	16.17	78.03	46.80	16.83					

TABLE (3) Adhesive pattern of BG based and CeraSeal root canal sealers

	Bioactive glass		**CeraSeal**		**Chi square test**	
	Count	Column N %	Count	Column N %	Chi square	P value
Type 2	1	12.5%	1	12.5%	0.42	0.81
Type 3	2	25.0%	1	12.5%		
Type 4	5	62.5%	6	75.0%		

CeraSeal Results: Minimum: 16.17, Maximum: 78.03, Mean ±Standard Deviation 46.80 ± 16.83.

Comparison between the sealers groups was carried out using Independent t test that showed the BG-based sealer revealing insignificant higher mean push-out bond strength (58.11) compared to CeraSeal (46.80) as (P=0.18).

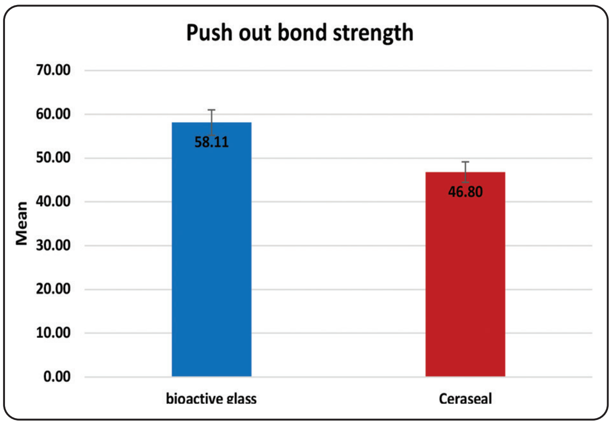

Fig. (2) Bar chart represents push out bond strength of groups.

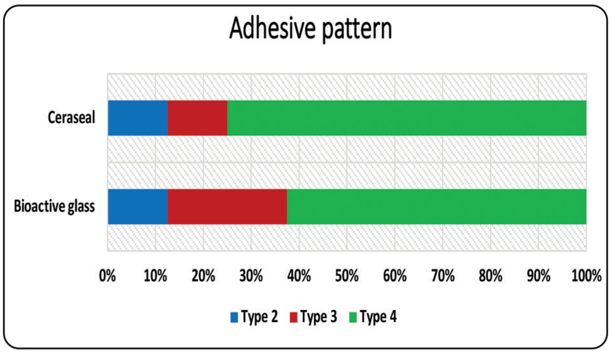

Fig. (3) Horizontal stacked bar chart representing adhesive pattern of BG based and CeraSeal root canal sealers

Evaluation of adhesive pattern:

Table (3) and (Fig. 3) represent adhesive of pattern of BG based and CeraSeal root canal sealers. Comparison between groups showed insignificant difference (P=0.81), as the highest type was type 4 (62.5, 75%) regarding BG based sealer and CeraSeal respectively. On the other hand, the least type was type 2 (12.5%) in BG, while in CeraSeal group both type 2 and type 3 revealed (12.5). Fig. 4 shows the root dentin sections observed under stereo microscope.

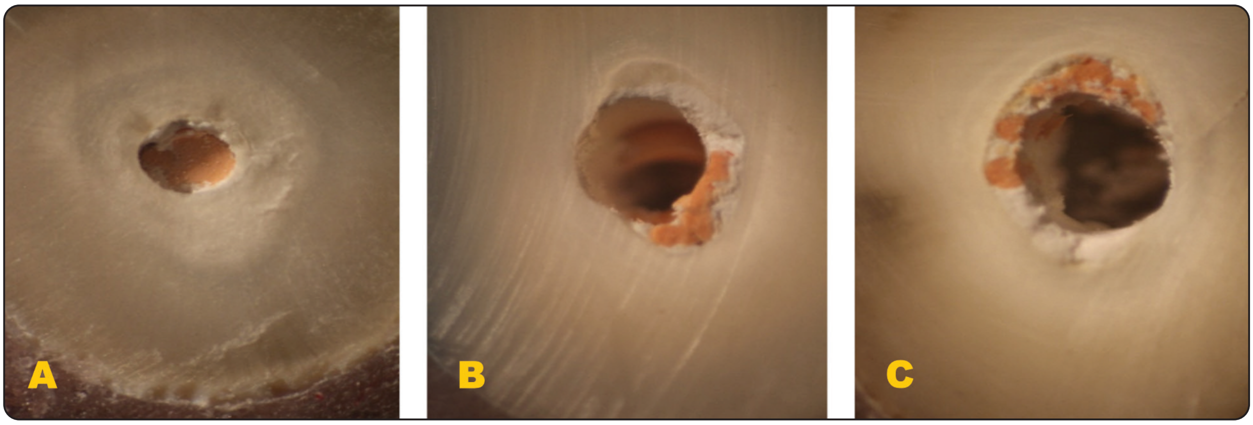

Fig. (4): Representative images showing root dentin section observed under stereo microscope at 40 × magnification. A) represents the Gutta percha dislodged from canal lumen after push out bond strength testing. (B and C) represent sealer and gutta percha adhered to root canal dentine wall.

Correlation between adhesive pattern and Push-out strength:

Correlation between Push out strength and adhesive patterns was studied by using Spearman`s Correlation Coefficient which revealed that there was a negative, moderate, non-significant correlation between them among BG group, while there was a positive moderate non-significant correlation among CeraSeal group, as presented in table 4, fig. 5 and 6.

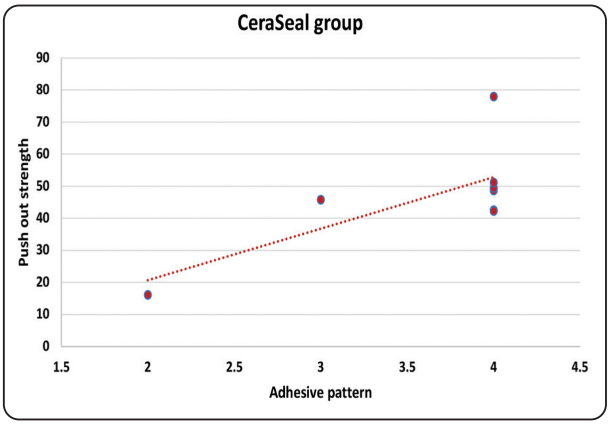

Fig. (6) Scattered chart representing correlation between Adhesive pattern and push out strength in CeraSeal group.

TABLE (4) Correlation between Adhesive pattern and push out strength in both groups:

	R	P value
Bioactive glass	-0.509	0.198
CeraSeal	0.546	0.162

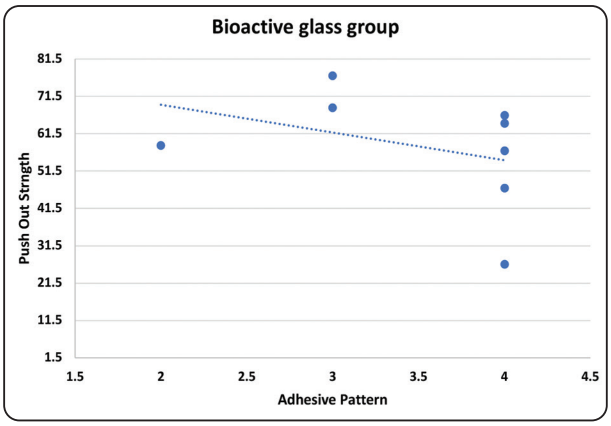

Fig. (5) Scattered chart representing correlation between Adhesive pattern and push out strength in BG group.

DISCUSSION

The primary purpose of endodontic sealer used is to establish a connection between the GP and the radicular dentin, thus enhancing the resistance to root fracture. Consequently, there has been a constant advancement in root canal sealants to improve the effectiveness of the resultant connection between the sealer and the root dentine surface. In our study, sealers utilised are a BG based and CeraSeal bioceramic sealers. The push-out test was carried out to study the dentin bond strength of the two selected sealers. Even though this test gives relevant idea by comparing different sealers, it cannot fully duplicate how well root canal sealers perform during clinical performance or show how bond strength and clinical success are related. Furthermore, this method is advantageous because it is vulnerable to minimum changes in distribution of stress while applying a load [25].

The comparison between groups in our study revealed that the BG based sealer showed insignificant push-out bond strength compared to CeraSeal. Regarding the BG based sealer material constitution, the main component is the BG powder, which comprise 70 wt% of the powder used. The remaining 30 wt% is zirconia (ZrO2) powder. This BG, known as PSC, is neutral and has the chemical constitution of 10.8% P2O5, 54.2% SiO2, 35% CaO (mol.%) [23]. The liquid is formed of phosphate solution (PS) and sodium alginate (SA) with 1% mass volume fraction. To reach the BG based sealer final composition, the ratio of powder to liquid (g/mL) was 1:1 during mixing.

Because of its high phosphorus content, PSC can quickly produce hydroxyapatite (HAP) when it reacts with PS [26]. Dentin main components are type I collagen, perfectly organized HAP with a trace quantity of non-collagenous protein. It was proven that PO_4^{3-} and Ca^{2+} emigrate from the BG sealer towards its surface to create an unorganized Ca-P layer which gradually builds up and organizes to compose a HAP layer[27]. In Huang study, it was revealed that BG sealer was capable to enter the dentinal tubules and produce minerals of apatite. This indicates that BG sealer might utilize calcium and phosphorus ions to generate a coat of minerals after entering deeply into dentinal tubules [23]. This may explain the effective push-out bond between dentin and BG based sealer.

The other utilized sealer is CeraSeal Bioceramic sealer. This calcium silicate-based sealer absorbs humidity out of the adjacent surface in the root canal and causes a certain amount of calcium hydroxide ($Ca(OH)_2$) to crystallise, forming calcium aluminate hydrate (CAH) gel and calcium silicate hydrate (CSH) gel [8]. Our findings could be justified by the previously postulated idea that as a BC sealer, it produces HA during setting and creates a chemical bond with dentine. This indicates the nature of its micromechanical interaction. Moreover, the utilization of water in the dentinal tubules completes its chemical reaction with no contraction, resulting in an interface without gaps [28].

Additionally, because of the composition of calcium silicate cement, CeraSeal can form a 'mineral infiltration zone'. The hydration of calcium silicate results in compounds that cause disintegration of the dentin forming a sieve-like surface, resulting in greater entrance of calcium and hydroxyl ions [21]. Furthermore, because of its hydrophilic nature and low contact angle, the bioceramic sealer may spread across the canal wall enabling better adaptability [29].

Our results come in accordance with Huang study who compared a BG based Sealer with iRoot SP [23]. However, Rehman et al reported that calcium silicate BG sealer has high resistance to dislodgement in comparison to Total Fill BC Sealer with superior marginal adaptation [30].

Cold lateral condensation technique was selected as it showed high bond strength values that was attributed to the condensation pressure with finger spreader to generate a vacancy that permit the insertion of auxiliary points. This improves the adaptation of the sealer to dentin surface and closes the gaps to fill in the discrepancies in the root canal[31]. Thereby, CLC reduces the sealer film thickness and enhances adhesion [32]. Although AbdelWahed et al stated that CeraSeal showed better bond strength readings with warm vertical compaction (WVC) technique [33], Mokhtari et al demonstrated that the use of single-cone technique for root canals obturation results in a lesser bond strength versus CLC [31]. Further, Nouroloyouni et al proved that push-out bond strength of CLC/ Sure Seal showed higher values than single cone obturation [34].

The second purpose of our research was to examine sealers' adhesive pattern, which indicates their bonding capability [16]. No earlier research has been done to compare the adhesive pattern of BG based sealer and CeraSeal BC sealer. Karobari discussed in his study [21], the sealer will be classified as type 1 if it is found in one quadrant and type 2 if it is found in two and followed by a similar pattern. A good adhesive nature is implied by a sealer present in all four quadrants, but superior cohesiveness is indicated by absence of sealer [5]. In our study, comparison between groups revealed insignificant difference, as the highest type was type 4 regarding BG based and CeraSeal sealers respectively (Table 2) following their bonding ability with dentine, as explained earlier [8,23,27,28]. On the other hands, the least type was type 2 in BG, while in CeraSeal group both type 2 and type 3.

The relative higher adhesiveness of BG based sealer can be justified by its higher push-out bond strength results to the radicular dentin surface (Table 2). The correlation between push out strength and adhesive patterns was assessed. Surprisingly, it was revealed that there was a negative, moderate, non-significant correlation between them among BG group, while there was a positive moderate non-significant correlation among Ceraseal group (Table 4). This could be justified by the several factors that could affect the sealer adhesion like the sealer surface tension, root surface energy and adherend cleanliness and the ability of the sealer to wet the surface [5, 35]. Also, the final irrigant has a great effect on the adherend surface energy.

Additional studies are needed to assess the used sealers' long-term clinical performance, penetration into dentinal tubules and sealing capability in several levels of the root canal. However, the findings of our study give some clinical significance on selection of the BG based sealer used and the commercially available CeraSeal based on their bonding strength and adhesive pattern.

CONCLUSION

Within the study's restrictions, it could be concluded that the prepared BG based sealer showed relatively comparable bond strength values with CeraSeal Bioceramic sealer when used in CLC technique. The prepared BG based sealer is reliable.

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