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EVALUATION OF WATER SORPTION, WATER SOLUBILITY AND PUSH OUT BOND STRENGTH OF DIFFERENT BIOCERAMIC ENDODONTIC SEALERS

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ABSTRACT

Aim: The research was to detect the difference in water sorption, water solubility and push out bond strength between three bioceramic endodontic sealers (**CeraSeal, NeoSEALER Flo, Well Root ST**) with epoxy resin based sealer (AH plus).

Materials and methods: For water sorption and solubility, we prepared thirty-two discs and incubated them until the sealers set completely. According to the type of sealer used, they were divided into four groups (n=8). The percentage of water sorption was calculated utilizing this formula: percentage of water sorption= (weight gained-initial weight /initial weight) x100. Percentage of water solubility **=** (water solubility / Dry weight) x100. For push out bond strength we selected forty single rooted teeth with fully formed apices. The teeth working length was measured after the teeth were decoronated. Then we performed instrumentation and irrigation. Four groups were created depending on the sealer used. Cross- sectional cuts were made perpendicular to the root's long axis for each specimen. The peak load observed was divided by the surface area to get the bond strength using the formula (A= (3.14 h (r1+r2))**.**

Results: For solubility Ah plus showed lower solubility after 1 and 3 days but Ceraseal showed lowest solubility after 7 days. For water sorption Neosealer Flo showed lowest water sorption after 1 day but AH plus showed lowest water sorption after 3 and 7 days. For push out bond strength, for coronal section, Ah Plus and Well-Root ST showed a significant highest push out bond strength in comparison with Ceraseal and Neosealer Flo**.**

Conclusion: The bioceramic sealers had high water solubility and water sorption which allowed release of biologically relevant ions. All the sealers bond strength meets the required standards.

KEYWORDS: Bioceramic, Sealers, Water sorption, Solubility, Push-out

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INTRODUCTION

A properly filled root canal is a critical component for achieving success in root canal therapy. The most commonly utilized method for filling the root canal involves utilization of sealer in the root canal in combination with gutta-percha (GP). This filling helps to maintain a clean root canal and provides a secure seal against bacteria and fluid. Utilization of gutta percha and a root canal sealer together ensures an appropriate fit within the root canal anatomy, which results in a strong and long-lasting seal. The root canal sealer ensures a strong bond between the wall of the root canal and the gutta-percha, preventing microleakage and reducing the risk of re-infection. [1,2]

Recently, bioceramic sealers (CSBSs) have gained widespread usage in endodontic treatments. Their calcium silicate formulation makes them biocompatible and bioactive, enables them to create an apatite layer upon contacting the tissue and chemically bond with dentin. The micromechanical interlock of the bioceramic sealers and root dentin promotes stability of the sealer-dentin interface even when under functional stress [3,4].

These sealers can penetrate dentinal tubules interacting with moisture of dentin, establishing bonds between the filling material and dentin. This results in low shrinkage and improved dimensional stability [5]. Bioactive substances released by these sealers encourage the development of a tag-like connection between sealer and dentin. When gutta-percha and bioceramic sealer are used for obturating the root canal, secondary monoblock adhesion occurs, strengthening the root [6].

Of these calcium silicate sealers (bioceramics) are **NeoSEALER® Flo** which is a premixed bioceramic root canal sealer that features superior handling properties and supports the healing process by promoting the formation of hydroxyapatite. As a biocompatible and antimicrobial sealer, it is dimensionally stable and does not contain any resins. Its bioactive components include tricalcium silicate and dicalcium silicate [7].

Well-Root ST (Vericom, Chuncheon-si, Gangwon-Do, Korea) is a new premixed calcium silicate sealer, composed of calcium aluminosilicate acting as reactive agent[8] [Yang, D.K 2018]. This sealer can set in presence of moisture in just 25 minutes [9].

CeraSeal (Meta Biomed Co., Ltd., Republic of Korea) is a newly developed bioceramic sealer, available in form of a flowable paste that can be easily placed in the root canal. Its manufacturer claims that it has exceptional stability, neither shrinking nor expanding, and boasts outstanding sealing capabilities, enabling obturation using single cone technique[10]. (CeraSeal Pamphlet, 2019). The BC sealer chemically bond with dentin as a result of releasing hydroxyapatite during the setting process by "mineral infiltration zone", occurring through the micromechanical interaction nature. This sealer takes advantage of the moisture in the dentinal tubules to set without shrinking, resulting in a gap-free interface between dentin and obturation materials [11]. Additionally, it creates a good adaptation and hermetic seal as it readily diffuses into the dentinal tubules, and this accounts for its high bond strength. The observed result may be due to the Ceraseal Bioceramic sealer bioactivity, hydrophilic properties, and low contact angle allowing the easy spread over the canal walls, resulting in thorough coverage, excellent adaptation, and an effective, tight seal [12].

These sealers were compared to AH Plus sealer, which is an epoxy resin that has long been regarded as the gold standard, and it comes in a paste-paste form [13-15]. Immediately after the two pastes are mixed, a thermal curing poly-addition reaction occurs, resulting in a polymer with a high molecule weight. As several studies have assessed the physio-chemical properties of this sealers[16,17] so, this material has been used to compare to other newly developed materials. Therefore, in this study we compared these bioceramic sealers with the epoxy resin sealer regarding water sorption, solubility and push out bond strength.

Materials and methods Materials: (Fig.1)

AH plus (the epoxy resin sealer) comes in a paste-paste form. Paste A [bisphenol-F epoxy resin, bisphenol-A epoxy resin, zirconium oxide, calcium tungstate, , silica and iron oxide pigments] and paste B [aminoadamantane, dibenzyldiamine, calcium tungstate, tricyclodecane-diamine, zirconium oxide, silica and silicone oil].

Fig 1: AH plus sealer, CeraSeal sealer, Well Root ST sealer, and NeoSEALER Flo sealer

The bioceramic sealers were:

CeraSeal (Meta Biomed Co., Ltd., Republic of Korea) is a newly developed bioceramic sealer, available in form of a flowable paste, which is composed of Zirconium dioxide, dicalcium silicate, tricalcium silicate, tricalcium aluminate, polyethylene glycol, thickening agents.

NeoSEALER® Flo is a premixed bioceramic root canal sealer, which is composed of Tantalite, calcium aluminate, tricalcium silicate, dicalcium silicate, calcium sulfate, tricalcium aluminate, polyethylene glycol, grossite.

Well-Root ST (Vericom, Chuncheon-si, Gang-won-Do, Korea) is a premixed Calcium silicate seal-er, this material comes in a premixed syringe which has intra- canal tip which allow easy application of the sealer. The sealer composed of calcium silicate, filler, zirconium oxide and thickening agents.

METHODS:

Sample size was calculated by adopting an alpha (α) level of 0.05, a beta (β) level of

0.20; i.e.power =80%, indicated that the predicted sample size was a total of (30) samples.

Water sorption:

Thirty-two discs, each diameter measures 10±1mm and thickness is 2±0.1mm, were created and separated into 4 groups of 8 each based on the sealer utilized. The discs were fabricated with a Teflon mold that had a 10mm diameter and 2mm thik central hole.

To synchronize the setting times of sealers and ensure complete setting, the discs were placed in an incubator (37°C, 95% relative humidity) and allowed to set for 4 hours.

The specimens were placed in a container made of glass with dehydrated silica gel (from Fischer Scientific in Leicester, UK), kept at 37±1°C and allowed to sit for an hour. After that, they were stored for an additional hour at 23±1°C. The weight of the samples was measured with a four-digit precision electronic balance from ***(Sartorius, Biopharmaceutical and Laboratories, Ger).*** The process was repeated up till a steady weight was attained, either the dry weight or the original weight. Subsequently, every sample was placed in its own container filled with distilled water at 37±1°C. Water sorption was evaluated through changes in weight, which were measured daily for three days, on the fifth day, and once a week until a state of balance was achieved (over a one-week period). The weight percentage of water sorption was reported as %. The wet weight was calculated using the methods outlined in ADA specification No. 27 for resin-based filling materials. The samples were taken out of water, paper filter was used to dry them, and allowed to air dry for 15 seconds to eliminate visible moisture. The final weight was measured one minute after being removed from water.

The calculation of the percentage of water sorption was performed using the following formula:

Percentage of water sorption= (weight gained-initial weight /initial weight) x100 Where, m2= weight gain, m1= initial weight

As the resin is placed in water, certain elements as unreacted monomers have dissolved and leached out from the sample, causing a decrease in weight that can be quantified as solubility. To determine actual water sorption, it was necessary to measure the solubility first.

Water Solubility:

The water solubility was determined by monitoring water release from the samples after they had absorbed it for one week. The process of removing the water, known as desorption, was carried out by placing the samples in a tightly sealed container with silica gel. The specimens were weighed repeatedly until a stable weight was achieved. The weight obtained after 1 week was used to calculate the percentage of water solubility, indicating the material amount that had been leached from the specimens.

Water solubility **=** weight before immersion (dry weight (m1)- weight after desiccation (m3). Percentage of water Solubility **=** (water solubility / Dry weight) x100 Push-out test.

These tests were performed using Bluehill lite Software from Instron.

Push-out test procedure

Forty extracted single rooted teeth were chosen. The crowns were decoronated at the cemento-enamel junction utilizing a water-cooled diamond bur. Instrumentation was done using ProTaper Next rotary instrument until F4. Irrigation was performed by utilizing 5 ml of 2.5% sodium hypochlorite. Paper point was used to dry all root canal. Teeth were distributed into 4 group (n=10) according to the sealer used. Group 1: AH plus, group 2: Ceraseal, group 3: Neosealer Flo, group 4: Well Root ST. To ensure that the shear bond strength test was not affected by the gutta-percha/cement interface, the canals were filled solely with endodontic cement, without using the gutta-percha points. All specimens were wrapped in gauze soaked in phosphate buffered saline and stored in incubator at 37°C for two weeks. For acquiring a transverse section of each specimen with a thickness of 2 mm ± 0.1 from the root thirds, perpendicular to the root's long axis, a water-cooled Isomet saw was employed. The thickness was determined utilizing a digital caliper (Pachymeter, Electronic Digital instruments, China). Each section was labeled and photographed from both the coronal and apical surfaces utilizing a stereomicroscope (SZ-PT; Olympus, Tokyo, Japan) at an original magnification of 45x. The "Set Scale" tool within the software of image analysis (Image J: NIH, Bethesda, MD) was utilized to allow calibration which was carried out by contrasting an item of known length, in this study was a ruler. Subsequently, the radius was computed after measuring the filling's diameter. ."Each tooth slice was placed in a specially made loading fixture, consisted of a metal block with a circular cavity in the center, this cavity was used to house specimens and had a central opening to help the displacement of extruded filling material. Subsequently, it was exposed to compressive loading using a computer-controlled material testing machine, with a crosshead speed of 1 mm/min. (Model 3345; Instron Industrial Products, Norwood, MA, USA).

Load was exerted by plungers with diameters of 1.2, 0.9, and 0.8 mm corresponding to the radicular part to be examined. The plunger tip was sized and positioned so that it contacted the filling material, without putting stress on the surrounding dentin. The plunger was pushed in the apical-coronal direction, to move the filling towards a wider diameter, preventing any restrictions on its movement that might have been caused by the taper of the canal. This ensured that the overlying dentin was supported adequately during the loading process.

Highest possible failure load was calculated in Newton and converted to MPa. The recorded peak load divided by the computed surface area was used to calculate the bond strength using the following formula: (A= (3.14 h (r1+r2)), where r1 apical radius, r2 coronal one and h is the thickness of the sample in millimeters.

The failure was demonstrated by the filler piece's extrusion, which was further corroborated by a sharp decline seen along the load-deflection curve that Bluehill's software had recorded. Every root slice had its push-out bond strength evaluated.

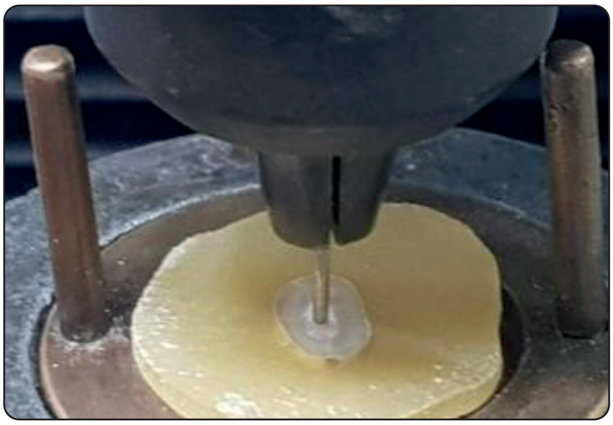

Fig. (2) Push out bond strength test.

Statistical analysis

Data checked for normality utilizing Kolmogorov Smirnov test and showed normal distribution. For water sorption and solubility, one-Way ANOVA utilized to compare between tested groups within follow-up periods. One-Way ANOVA utilized to compare follow-up periods within each tested group. For push-out bond strength, one-Way ANOVA utilized to compare between tested groups within follow-up periods. One-Way ANOVA utilized to compare follow-up periods within each tested group. Significant level was set at p=0.05.

RESULTS

After 1 day, AH plus resulted in a significant lowest solubility% compared to all other sealers. Insignificant difference resulted between Ceraseal, NeoSEALER Flo, and Well-Root ST. After 3 days, AH plus resulted in a significant lowest solubility% compared to Ceraseal, NeoSEALER Flo. Well-Root ST showed an insignificant difference with all other sealers. After 7 days, Ceraseal resulted in significant lowest solubility% followed by AH plus followed by NeoSEALER Flo, and Well-Root ST, with insignificant difference between each other's.

For CeraSeal, 3 days showed a significant decrease in solubility % followed by a further substantial decrease after 7 days. For NeoSEALER Flo, 3 days showed an insignificant decrease in solubility% followed by a substantial decrease after 7 days. For Well-Root ST, 3 days showed significant decrease in solubility% followed by an insignificant decrease after 7 days. For AH Plus, 3 days showed an insignificant decrease in solubility% followed by an insignificant decrease after 7 days. However, 3 days showed a significant decrease in solubility% compared to 7 days.

Sorption % results

After 1 day, NeoSEALER Flo resulted in a significant lowest Sorption% compared to CeraSeal and Well-Root ST. Insignificant difference resulted between Ah plus and all other sealers. After 3 days, AH plus resulted in significant lowest Sorption% compared all other sealers followed by Well-Root ST which showed a significant difference with all other sealers. After 7 days, AH plus resulted in significant lowest sorption% followed by Well-Root ST followed by CeraSeal and NeoSEALER Flo. For CeraSeal, 3 days showed a significant increase in Sorption% followed by an insignificant decrease after 7 days. For NeoSEALER Flo, 3 days showed a significant increase in Sorption% followed by a substantial increase after 7 days. For Well-Well Root ST, 3 days showed significant increase in sorption% followed by a substantial increase after 7 days. For AH Plus, 3 days showed an insignificant decrease in sorption% followed by a substantial increase after 7 days.

TABLE (1) Solubility % results for different tested groups.

		CeraSeal		NeoSEALER Flo		Well-Root ST		AH plus		p-value
		Mean	SD	Mean	SD	Mean	SD	Mean	SD	
Solubility %	1 Day	12.5^{bC}	0.7	13.2^{bB}	3.1	11.9^{bB}	0.4	-1.0^{aA}	1.1	<0.001*
	3 Days	9.9^{bB}	7.3	6.1^{bB}	3.2	5.4^{abA}	3.2	0.4^{aAB}	1	0.002*
	7 Days	0.0^{aA}	1.4	8.0^{cA}	0.4	6.5^{cA}	1.1	2.0^{bB}	2.7	<0.001*
p-value		<0.001*		<0.001*		<0.001*		0.015*		

****=significant, NS=non-significant, Different lowercase letters indicate significant differences between tested groups. Different uppercase letters indicate significance within each column.***

TABLE (2) Sorption % results for different tested groups.

		CeraSeal		NeoSEALER Flo		Well-Root ST		AH plus		p-value
		Mean	SD	Mean	SD	Mean	SD	Mean	SD	
Sorption %	1 Day	3.2^{bA}	2.5	1.0^{aA}	0.5	3.3^{bA}	0.4	1.4^{aAB}	0.1	0.001*
	3 Days	17.6^{cB}	2.3	15.5^{cB}	3.6	11.7^{bB}	1.1	0.2^{aA}	0.9	<0.001*
	7 Days	19.0^{bcB}	9.0	21.3^{cC}	0.6	13.5^{bC}	0.4	2.8^{aB}	2.8	<0.001*
p-value		<0.001*		<0.001*		<0.001*		0.017*		

****=significant, NS=non-significant, Different lowercase letters indicate significant differences between tested groups. Different uppercase letters indicate significance within each column.***

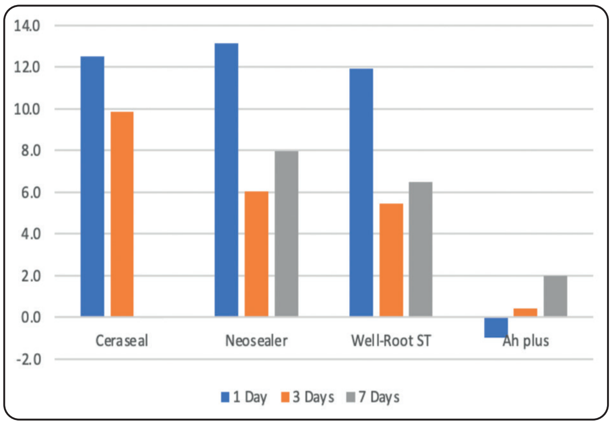

Fig. (3) Bar chart showing the mean solubility % for different tested groups.

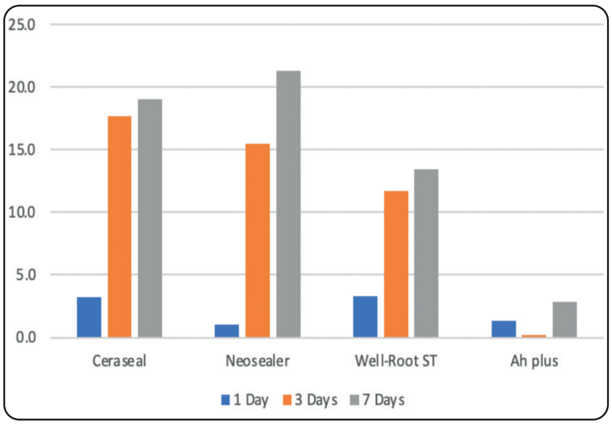

Fig. (4) Bar chart showing the mean sorption % for different tested groups.

Push out bond strength

For coronal section, AH Plus and Well-Root ST resulted in a significant highest Push out bond strength (MPa) compared to CeraSeal and NeoSEALER Flo. For middle and apical sections, insignificant difference between tested sealers resulted.

Insignificant difference between root sections for all sealers.

TABLE (3) Push out Bond Strength (MPa) results for different tested groups.

		Ceraseal		Neosealer Flo		Well-Root ST		Ah plus		p-value
		Mean	SD	Mea n	SD	Mea n	SD	Mea n	SD	
Push out Bond Strength (MPa)	Coronal	1.93[ab]	0.56	1.63[a]	0.32	2.13[b]	0.26	2.41[b]	0.53	0.003*
	Middle	2.51[a]	1.11	1.92[a]	0.48	2.51[a]	0.91	2.43[a]	0.47	0.290NS
	Apical	2.56[a]	1.29	1.98[a]	0.53	2.87[a]	0.72	2.16[a]	0.54	0.093 NS
p-value		0.335 NS		0.203 NS		0.074 NS		0.429 NS		

=significant, NS=non-significant, Different lowercase letters indicate significant differences between tested groups.

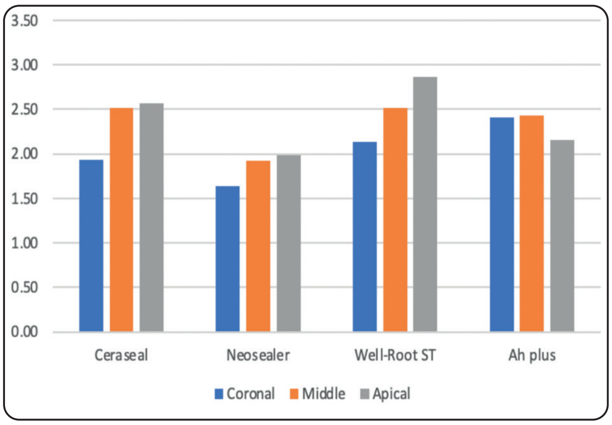

Fig. (5) Bar chart showing the mean Push out Bond Strength (MPa) for different tested groups.

DISCUSSION

The sealing of the root canal is largely determined by how well the material used in obturation adhere to the canal walls, in the stationary and dynamic conditions. Under stationary conditions, its function is to impede the leakage of fluids between canal wall and the filling material, hence preventing buildup of microorganisms and stagnant tissue fluids that can result in periapical disease. While in dynamic condition, it improves the resistance of the filling material to dislodgement [18]. Sealers are deemed necessary due to gutta-percha's weak adhesive properties to root canal walls. Endodontic sealers primarily aim to establish a connection between the obturating material and the canal walls, thereby enhancing tooth resistance to fracture. This has driven a constant advancement in the development of root canal sealers.

For numerous years, epoxy resin sealers have been viewed as the gold standard and were extensively employed in dental field and being the subject of many scientific studies [13-15]. Bioceramic materials, also known as calcium silicate (CS) materials, can set in moist environments and are frequently regarded as a ground-breaking development in dentistry due to their versatility in treating various endodontic conditions [15].

Jeong et al [19] studies have indicated that calcium silicate-based sealers can infiltrate dentinal tubules without the use of compression forces commonly employed in alternative obturation methods. Additionally, a recent scanning electron microscopy investigation found that bioceramic sealers exhibited superior adaption to canal walls compared to AH Plus sealers [20].

So, in this study we compared between bioceramic sealers and AH plus regarding water solubility, water sorption, and push out bond strength.

Solubility is the measure of the amount of mass lost by a material after being immersed in water for a specific period[21]. AH Plus sealer displayed less solubility compared to other sealers, possibly due to the robust cross-linking of its epoxy resin-based composition[22]. Conversely, it has been proposed that the hydrophilic nanoparticles present in bioceramic sealers lead to a greater surface area, resulting in more liquid molecules interacting with the sealer, thereby accounting for the high solubility of NeoSEALER Flo [21].

Furthermore, the elevated solubility of bioceramic sealers in contrast to AH Plus may be attributed to the bio-interactivity resulting from the release of biologically relevant ions. This bio-interactivity is due to the significant open pore volume that generates a network of water-filled pores internally, resulting in a significant surface area implicated in the leaching process. [23,24]. The discharge of ions is contingent upon the network structure of the sealer, which governs its capacity for solubility and water absorption, in addition to the material's permeability to water diffusion [23,24]. Consequently, sealers that possess a greater open pore volume can allow more water absorption, leading to increased solubility and possibly more ion release, commensurate with the presence of reactive agents CaSi particles. Nevertheless, higher solubility in vitro of the tested material in water does not necessarily imply a harmful effect in vivo, as apatite and carbonate nucleation could occur, potentially mitigating the behavior of the sealer.

Bioceramic sealers exhibited a higher degree of water sorption than epoxy resin- based sealers owing to their hydrophilic nature, which results in superior wettability, potentially influencing their adhesion to dentin, as stated by (Kapralos, V in 2021) [25]. On the other hand, Ah Plus displayed lower sorption due to its hydrophobicity resulting from the presence of resin.

Push-out bond strength tests were conducted to assess the adhesive properties of root canal filling materials to the root dentin. In PBS testing, shear stress is generated at the interface of dentin-cement, which is similar to the clinical stress conditions, as noted by Pane ES in 2013[26]. The PBS test is considered superior to other tests in evaluating adhesion, as it creates parallel fractures in interfacial region of the dentin bond, according to Sudsangiam S in 1999[27]. The highest bond strength was with AH plus and Well Root ST and this was in accordance with [Kharouf, N 2023 [28]). The presence of epoxy resin in the composition of AH Plus was believed to contribute to its excellent bond strength. This was due to the effect of epoxy resin on its compression modulus, as well as forming covalent connections between the resin and collagen as a result of its capacity to interact with exposed amino groups in collagen. This is supported by [Lee KW 2002] [29]. For Well Root ST, this may be due to its bonding mechanism. As several studies have reported that the high alkaline effect of Well Root ST may cause denaturation of collagen fibers, which allows for the infiltration of the sealer's mineral content in the inter-tubular dentin, which results in the formation of a mineral infiltration zone [30,31]. This, in conjunction with the hydration reaction of the calcium silicate sealer, generates calcium hydroxide that interacts with phosphate to form hydroxyapatite. This, in turn prompts the development of a mineral infiltrationzone which fosters a chemical bond between dentinal walls and calcium silicate- based materials. (32). This was in disagreement with other study that showed that Ceraseal showed higher bond strength than AH plus [33].

In our study, to simulate the clinical conditions of the unstandardized root canal anatomy, we implemented filling the whole canal with sealer and analysis of the different root sections of extracted teeth, rather than using dentin slices with standardized holes to test the push out bond strength. This technique might have a disadvantage due to lack of standardization of comparisons.

Within the limitations of our study, it can be concluded that the relatively high water sorption & solubility of the tested bioceramic sealers might be clinically beneficial due to the superior wettability and also the chemical bond formed by creating apatite deposits at the sealer-dentin interface, explaining the comparable or even higher bond strength to the epoxy resin sealer AH Plus.

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