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Characterization and Assessment of Physical Properties of 3 Single Syringe Hydraulic Cement–based Sealers

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ABSTRACT

Introduction: A number of sealers with different chemistries are badged as Bioceramic, implying biological activity, but have dissimilar properties, which has implications on the sealer properties and will affect the quality and outcome of root canal treatment. This study aimed to assess the physical and chemical properties of 3 hydraulic cement–based sealers, namely BC Universal sealer compared with Totalfill BC sealer and AH Plus Bioceramic. **Methods:** The microstructure and composition of the sealers were assessed using scanning electron microscopy and energy dispersive spectroscopy after setting. The crystalline phases were assessed by X-ray diffraction analysis and the leachates were tested using inductively coupled plasma. All testing was performed at 0, 7, and 28 days. The physical properties of film thickness, flow, radiopacity, and solubility were evaluated using ISO 6876:2012 standards. **Results:** All 3 sealers contained calcium, zirconium, and silicon. Totalfill BC had the highest calcium release at 7 and 28 days followed by AH Plus Bioceramic and BC Universal sealer. All 3 sealers adhered to the ISO standard in terms of flow and radiopacity. BC Universal sealer was slightly over the range (>50 μm) for film thickness. All sealers exceeded the solubility range set by ISO 6876:2012. **Conclusion:** Although these hydraulic cement sealers had similar components and delivery, the properties varied significantly. The testing of material properties to confirm the suitability for clinical use is necessary. *(J Endod 2024;50:381–388.)*

KEY WORDS

Bioceramic; characterization; hydraulic calcium silicate cements; hydraulic sealers; physico-chemical properties; solubility

SIGNIFICANCE

Hydraulic cement–based sealers may have similar compositions but the materials properties may vary, thus affecting the clinical use and treatment outcomes.

The complexity of the root canal anatomy results in challenges in obturation. Several techniques using gutta-percha and sealer have been developed with the latter being more important when using sealer-based obturation techniques[1]. A plethora of endodontic sealers are available in the market varying in composition and physical, mechanical, and biological properties[2,3]. An ideal root canal sealer will reduce inflammation, seal the canal space, and prevent further growth of bacteria[4]. The composition and properties of root canal sealers directly affect the quality and outcome of nonsurgical root canal treatment[5]. Epoxy resin–based sealers are commonly used endodontic sealers[6] but do not possess desirable biological properties[7]. Most of the sealers create a hermetic seal but lack antimicrobial effects. On the other hand, hydraulic calcium silicate cement–based sealers produce biologically active by-products that interact with the environment contributing to adequate elimination of microorganisms[8,9], and are also biocompatible[10]. Sealers in contact with periradicular tissues can exhibit adverse biological reactions depending on what is released and hence, the biocompatibility is crucial for an endodontic sealer[4].

The hydraulic cement–based sealers are collectively known as bioceramics. Sealers that are classified as bioceramic have a wide range of chemistries, the quantity of the active components can vary,[3] and they all vary in their physical, chemical, and biological properties[11-13]. The calcium hydroxide formed as a by-product of hydration is mainly responsible for enhancing the antibacterial properties of these materials[14,15]. Hydraulic calcium silicate–based sealers require water/moisture to set to produce

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calcium hydroxide. The antimicrobial effect produced by the free hydroxyl ions is due to the high pH, and this results in better healing and repair[16]. The hydroxyl ions can disrupt the bacterial structure by inactivating bacterial enzymes when exposed to a high pH[17,18].

This study aimed to assess the microstructure and chemical and physical properties at 3 time points of 3 hydraulic sealers using the test methods and requirements of ISO 6876:2012 standard[19].

MATERIALS AND METHODS

Three sealers were selected for the study: BC Universal sealer (FKG Dentaire, Chaux de Fonds, Switzerland: WR320100), Totalfill BC sealer (FKG Dentaire: 22003SP), and AH Plus Bioceramic (Dentsply, Tulsa, OK, USA: K1230516). The sealer details are shown in Table 1. No information was found pertaining to BC Universal sealer.

Characterization of Materials

Sealer Microstructure

Specimens measuring 10 mm in diameter and 2 mm thick were prepared using molds. All tests were conducted in triplicate. The sealers were allowed to set in a humidity chamber at 37°C covered with moist gauze and sealed in a plastic bag to avoid evaporation and drying out. After setting, the samples were embedded in epoxy resin (Epofix; Struers, Ballerup, Denmark) and were ground under a continuous flow of water using progressively fine grit diamond polishing discs (Struers) starting with MD Piano 250, 500, 1000, and 2500, and then polished using polishing cloths and diamond-impregnated polishing pastes (Struers; MD Largo, MD Dac, MD Nap with polishing pastes impregnated with 9 μm, 3 μm, and 1 μm diamond particles). Before examination in the scanning electron microscope, samples were coated with a thin layer of gold. Images were taken using a scanning electron microscope (EVO MA 10; Carl Zeiss Ltd., Cambridge, UK) using backscatter mode and magnifications ranging from 1K to 2.5 K. Elemental analysis of the samples was also carried out using X-ray energy dispersive analysis (Oxford Instruments, High Wycombe, UK).

Phase Analysis

X-ray diffraction was performed immediately after setting and also after immersion in Hank's balanced salt solution (HBSS; H6648, Sigma-Aldrich, Darmstadt, Germany) for 7 and 28 days. All testing was conducted in triplicate. After removal from the solution and desiccation, the materials were ground to a fine powder using an agate mortar and pestle. The specimens were placed in a sample holder and phase analysis was performed with an X-ray diffractometer (Panalytical Empyrean X-ray Diffractometer; Malvern Panalytical, Malvern, UK) using a copper source (Kα – 1.54 Å). The X-ray patterns were acquired in the 2θ (10–60°) with a step of 0.02° and 0.6 seconds per step. Phase identification was established using a search-match software and the ICDD database (International Centre for Diffraction Data, Newtown Square, PA).

Ion Release

Three specimens of each sealer type measuring 10 mm in diameter and 2 mm thick were prepared and allowed to set. They were then immersed in 5 ml of HBSS, and leachates extracted at 7 and 28 days. The solutions were analyzed for elements of interest that were detected in the energy dispersive spectroscopy (EDS) analyses: calcium, zirconium, aluminum, silicon, and lithium. This was performed using inductively coupled plasma optical emission spectrometry (Optima 8000 ICP-OES; Perkin Elmer, Waltham, MA). Before assessment, leachates were acidified to a 2% HNO_3 in a falcon tube and diluted 100-fold. Four calibration solutions were used and HBSS was acidified with 2% HNO_3, which served as the blank sample.

Evaluation of the Physical Characteristics of the Materials

The assessment of radiopacity, flow, film thickness, and solubility was undertaken based on ISO 6876:2012,[19] as described in the following sections.

Radiopacity

The sealers were allowed to be set in rubber molds of 10 mm diameter and prepared to a height of approximately 1 mm. Three specimens of each sealer type were placed

TABLE 1 - Endodontic Sealers Used in the Study Along With Composition as Declared by the Manufacturer

Material	Lot number	Composition	Website	Manufacturer
BC Universal sealer	WR320100	/	https://www.fkg.ch/products/endodontics/obturation/bc-universal	FKG Dentaire, Chaux de Fonds, Switzerland
Totalfill BC sealer	[10]22003SP	Zirconium oxide – 35%–45% Tricalcium silicate – 20%–35% Dicalcium silicate – 7%–15% Calcium hydroxide – 1%–4%	https://www.fkg.ch/sites/default/files/FKG_TotalFill%20BC%20Sealer_IBC_Safety%20data%20sheet_20230815.pdf	FKG Dentaire, Chaux de Fonds, Switzerland
AH Plus Bioceramic	K1230516	Zirconium dioxide - 50%–75% Tricalcium silicate - 5%–15% Dimethyl sulfoxide - 10%–30% Lithium carbonate - <0.5% Thickening agent - <6%	https://www.dentsplysirona.com/content/dam/master/product-procedure-brand-categories/endodontics/product-categories/obturation-materials/sealers-root-repair/ah-plus-bioceramic-sealer/ifu/END-IFU-AH-PLUS-BIOCERAMIC-SEALER-TDS-V00-WEB-NAM-EN-2021-05.pdf	Dentsply, Tulsa, OK, USA

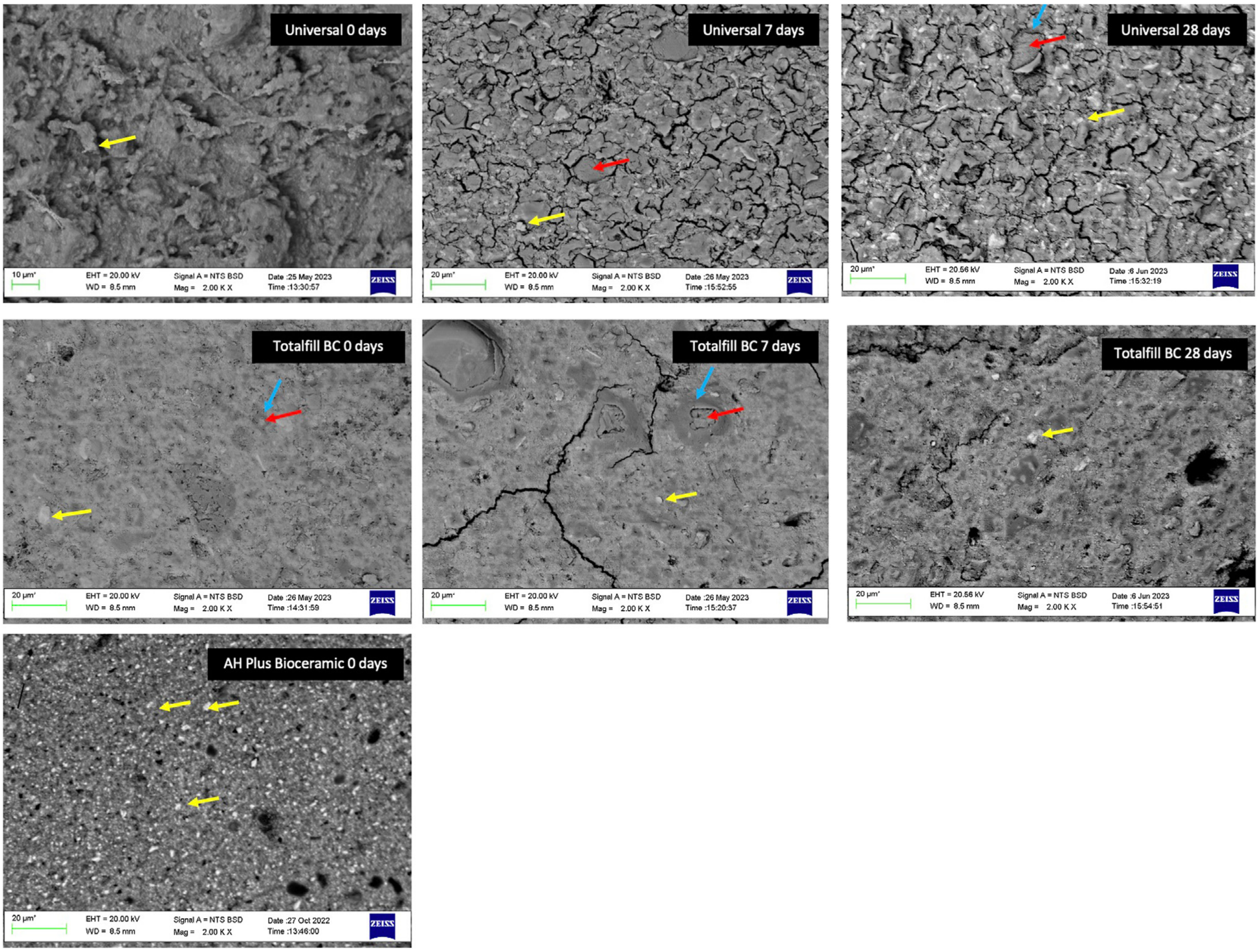


FIGURE 1 – Scanning electron micrographs of the sealers immediately after setting and after storage for 7 and 28 days in Hank's balanced salt solution (HBSS) showing the microstructural changes over time (magnification $\times$2.5k). *Yellow arrows* indicate the particles rich in zirconium. *Blue arrows* show hydration rims and *red arrows* show the cement particles. The AH Plus Bioceramic disintegrated after immersion in HBSS and thus could not be imaged.

next to an aluminum step wedge having a thickness from 0.5 mm to 9.0 mm in equally placed steps of 1 mm on an intra-oral X-ray occlusal digital film and irradiated at 65 kV ($\pm$ 5 kV) at a distance of 300 mm to 400 mm for sufficient time that the exposed film under the 1-mm-thick section of the step wedge has an optical density in the region of 0.5 to 2.5. Image J analysis was used to express the radiopacity equivalent of the specimens in millimetres of aluminum.

Flow

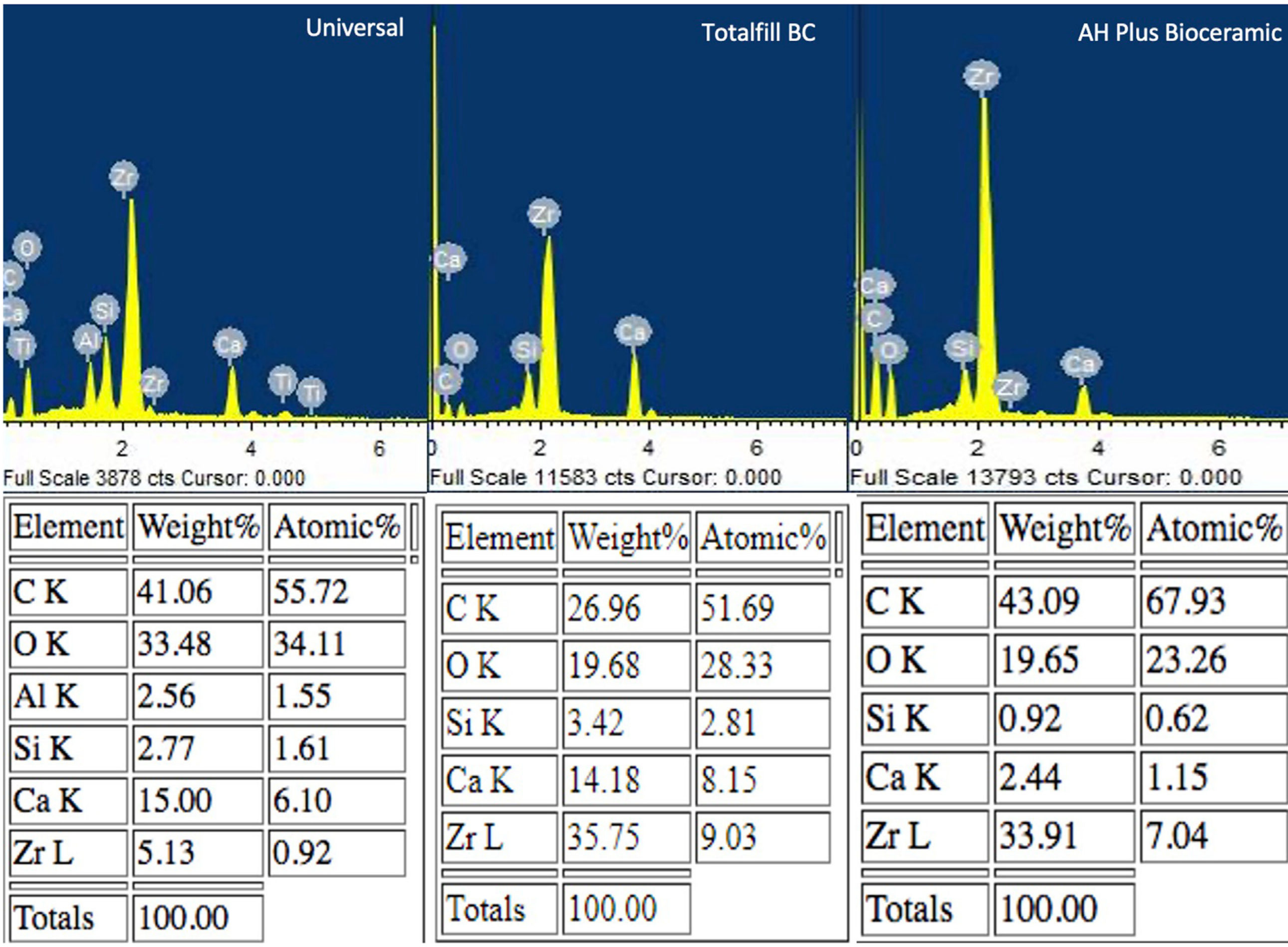
Using the pre-mixed material syringes, 0.05 ml ($\pm$ 0.005) of sealer was dispensed onto a glass plate measuring 40 $\times$ 40 mm, of 5 mm thickness and a mass of 20 g using a graduated syringe. A second glass plate of the same dimension was placed centrally on top of the sealer and compressed with a mass of 100 g. The maximum and minimum diameters of the compressed sealer discs were measured 10 minutes after the placement. The test was repeated if the 2 diameters were not within a tolerance of 1 mm to each other. This test was repeated 3 times for each sample.

Film Thickness

The combined thickness of 2 glass plates, each 5 mm in thickness, were measured using a micrometer to an accuracy of 1 μm. Sealer of 0.05 ml ($\pm$ 0.005) in volume was deposited centrally between the 2 glass plates. After 180 seconds ($\pm$ 5 seconds) from the dispensing, a loading device (MTS Criterion, Eden Prairie, MN) was used to evenly load 150 N ($\pm$ 3 N) vertically on the top plate so that the sealer filled the area between the glass plates. The combined thicknesses of the 2 glass plates and the film of sealer were measured using a micrometer after 10 minutes from the dispensing. The thickness of the film of sealer was calculated by determining the difference in thickness of plates with and without sealer. The experiment was conducted 3 times for each sample and a mean value was calculated.

Solubility

The sealers were dispensed to prepare 6 specimens 20 mm in diameter and 1.5 mm in height in plastic molds. Once they were set, the specimens were weighed to the nearest 0.001 g (TS400D; Ohaus Corporation, Florham Park, NJ). Two samples were placed in a shallow dish, and 50 $\pm$ 1 ml of water was added. The container was covered and kept in the incubator for 24 hours at 37°C before transferring all contents to a second dish. The liquid was evaporated at 110 $\pm$ 2°C and the containers were transferred to a desiccator at room temperature to cool and dry before weighing. The percentage difference of the final mass compared with the original mass of the material expressed the material solubility in the



Element	Weight%	Atomic%
C K	41.06	55.72
O K	33.48	34.11
Al K	2.56	1.55
Si K	2.77	1.61
Ca K	15.00	6.10
Zr L	5.13	0.92
Totals	100.00	

Element	Weight%	Atomic%
C K	26.96	51.69
O K	19.68	28.33
Si K	3.42	2.81
Ca K	14.18	8.15
Zr L	35.75	9.03
Totals	100.00	

Element	Weight%	Atomic%
C K	43.09	67.93
O K	19.65	23.26
Si K	0.92	0.62
Ca K	2.44	1.15
Zr L	33.91	7.04
Totals	100.00	

FIGURE 2 – Energy dispersive spectroscopic plots showing the elements present. All materials contained calcium, silicon, oxygen, and zirconium with BC Universal sealer, including titanium and aluminum.

solution used for immersion. The experiment was repeated 3 times for each material.

Statistical Analysis

The Kolmogorov-Smirnov test was used to test if the data were normally distributed. One-way analysis of variance was followed by Tukey post hoc tests ($\alpha = 0.05$) for parametric data; the Kruskal-Wallis test was done for nonparametric data.

RESULTS

Characterization of Materials

The scanning electron micrographs of the materials immediately after setting and after 7 and 28 days stored in HBSS are shown in Figure 1. The energy dispersive spectroscopic plots are shown in Figure 2. The particle size of the AH Plus Bioceramic was finer compared with Totalfill BC, with BC Universal sealer displaying the coarsest particle size. The AH Plus Bioceramic was mainly composed of opaque particles (marked in yellow arrows) that were rich in zirconium. These particles were also observed in the Totalfill BC and BC Universal sealers but less (marked in yellow arrow). All sealers exhibited pores at all the different time points. The 7- and 28-day analysis could not be undertaken as the specimens disintegrated on immersion in HBSS.

The other particles were rich in calcium and silicon and varied in degree of hydration. This is evident from the presence of reaction rims (marked in blue arrows) around the cement particle (marked in red arrows). The Totalfill BC sealer exhibited a high degree of hydration even when tested after setting. By 28 days, most of the cement particles had hydrated and the material was composed of by-products of the reaction. Some cracks were evident in the aged specimens, which is an artifact caused by the dehydration process required for the coating and imaging. The BC Universal sealer hydrated with time and some reaction rims (blue arrows) could be seen at 28 days. The material exhibited unreacted cement particles (marked in red) in the aged specimens.

The X-ray diffraction (XRD) plots with identified peaks are shown in Figure 3. The BC Universal sealer was composed of monoclinic tricalcium silicate, monoclinic zirconium oxide, rhombohedral calcium carbonate, orthorhombic calcium aluminum silicate, hexagonal calcium hydroxide, and tetragonal titanium oxide. The tricalcium silicate peaks marked at $\sim$29, 32, 33°2θ reduced in intensity after 28 days, and the calcium hydroxide peaks ($\sim$18°2θ) increased in intensity showing sealer hydration with the formation of calcium hydroxide. The calcium carbonate peak also reduced in intensity on hydration.

The Totalfill BC sealer was composed of monoclinic zirconium oxide orthorhombic dicalcium silicate and rhombohedral calcium carbonate. The calcium hydroxide peaks ($\sim$18°2θ) increased in intensity as the sealer hydrated. The calcium carbonate peaks reduced in intensity on hydration. The AH Plus Bioceramic sealer was composed of monoclinic zirconium oxide with traces of monoclinic tricalcium silicate shown in Figure 3. Further analysis of the sealer at 7 and 28 days could not be performed because the

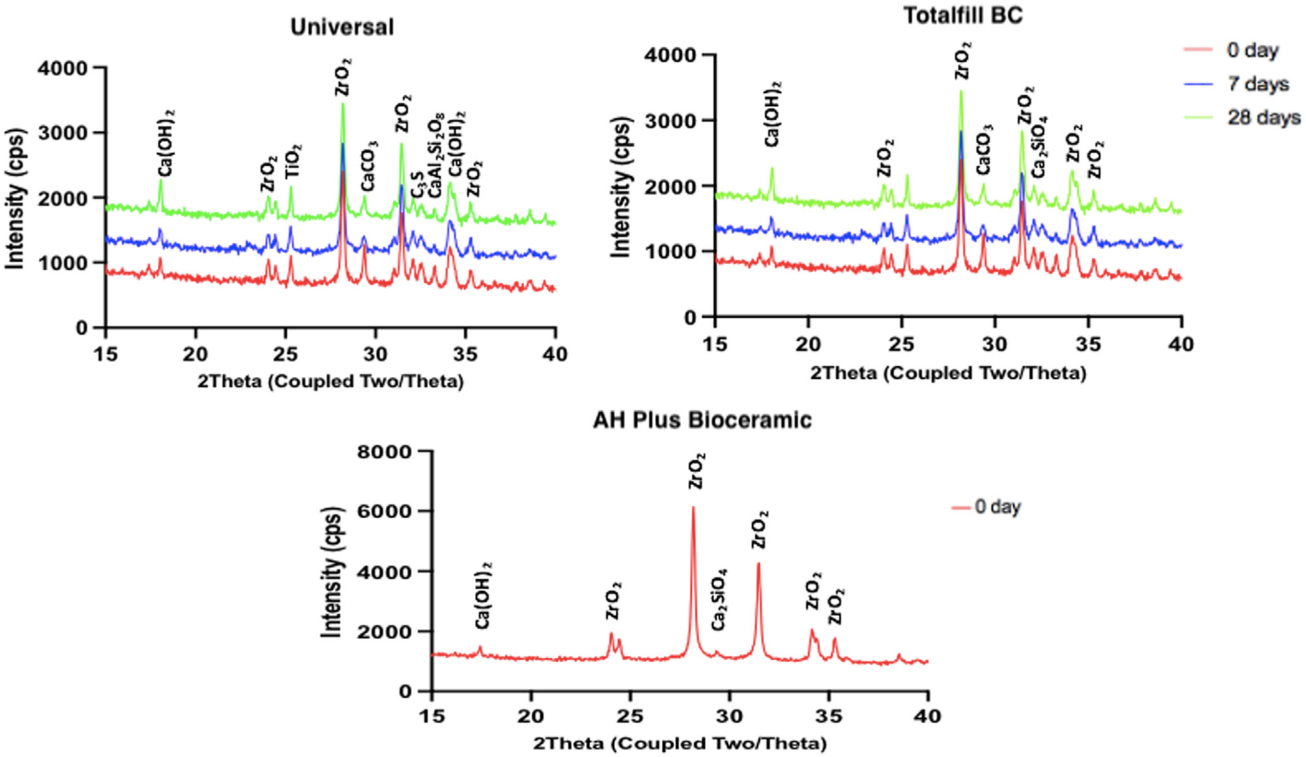


FIGURE 3 – X-ray diffraction plots of the 3 sealers tested immediately after setting and after 7 and 28 days immersed in Hank's Balanced Salt Solution showing the main phases present.

sealer disintegrated in contact with the storage solution.

The leaching profile over the 28-day time point is shown in Table 2. Calcium was released in solution for all sealers tested in the current study. Totalfill BC sealer exhibited the highest calcium release compared with both AH Plus Bioceramic ($P = .013$ and $P < .0001$ for 7 and 28 days, respectively) and BC Universal sealer ($P = .003$ and $P < .0001$ for 7 and 28 days, respectively) and was increasing in intensity after 28 days. The lowest calcium release was from AH Plus Bioceramic, which reduced further after 28 days ($P < .01$). BC Universal sealer also exhibited leaching of aluminum in small amounts both at 7 and 28 days. AH Plus Bioceramic leached lithium in solution at both time points. Zirconium was not detected in any of the leachates.

Determination of the Physical Properties of the Sealers

Table 3 shows the results of the physical properties of the sealers compared with the ISO 6876:2012 standard[19]. All the sealers exhibited a flow of greater than 17 mm ranging from about 20 to 24 mm. The AH Plus Bioceramic exhibited the lowest film thickness compared with the Totalfill Bioceramic and BC Universal sealer ($P < .001$), with the latter being slightly too high as the film thickness was greater than the 50 mm as specified in the ISO standard.

All the sealers had a radiopacity higher than 3 mm aluminum thickness with the AH Plus Bioceramic being the most radiopaque sealer.

None of the sealers complied with ISO 6876:2012 in terms of solubility, all sealers exceeded the standard value.

DISCUSSION

Currently no endodontic sealers fulfill all the properties an ideal sealer should possess and hence a need exists to improve currently available materials[1]. Hydraulic tricalcium silicate cements have some enhanced antibacterial effects and satisfactory physico-chemical properties compared with sealers with different chemistries that are nonhydraulic[20,21]. Three calcium-based hydraulic sealers were selected for this study due to their similarity in chemical composition and a similar delivery method. Although all sealers included tricalcium silicate, BC Universal sealer's cement phase is Portland cement, as indicated by the presence of an aluminum phase[22] unlike the Totalfill BC, which was composed of tricalcium silicate. The AH Plus Bioceramic was primarily composed of zirconium dioxide with small amounts of dicalcium silicate.

In vitro studies such as this contribute to understanding the physical and chemical properties of commercial sealers for clinical use. The tested sealers varied in their surface structure, with AH Plus Bioceramic sealer displaying finer particle size and more densely packed structure (Fig. 1) compared with BC Universal and Totalfill BC. The difference in particle size could alter the hydration of the Totalfill BC and the BC Universal sealers. The fine particle size of the Totalfill BC sealer provided a large surface area for the cement particles to hydrate resulting in a high degree of hydration when compared with the BC Universal sealer, which at the same time point exhibited less hydration by-products as seen in the scanning electron micrographs.

EDS revealed the elemental contents of the materials. All the sealers indicated the presence of calcium, silicon, and zirconium with an additional titanium content in BC Universal sealer as shown in Figure 2. AH Plus Bioceramic had the highest zirconium content on EDS analysis, which is in accordance with the manufacturer's manual (50%–75%) as presented in Table 1. AH Plus Bioceramic exhibited the leaching of lithium in solution. Lithium or compounds based on lithium were not found in the EDS analysis and this can occur because lithium is a light element and can be detected only by windowless

TABLE 2 - Chemical Composition of the Storage Solution in Contact With Each Sealer Assessed by Inductively Coupled Plasma, Showing the Main Elements Leached Over a Period of 28 days (Mean ± Standard Deviation)

Material	Element ppm									
	Ca		Si		Zr		Al		Li	
	7 days	28 days	7 days	28 days	7 days	28 days	7 days	28 days	7 days	28 days
BC Universal sealer	414.87 ± 48.1	537.36 ± 59.7	2.21 ± 1.4	3.15 ± 0.7	BDL	BDL	0.21 ± 0.0	0.55 ± 0.3	BDL	BDL
Totalfill BC	836.26 ± 51.8	1034.64 ± 51.7	0.36 ± 0.2	1.07 ± 0.2	BDL	BDL	BDL	BDL	BDL	BDL
AH Plus Bioceramic	521.28 ± 70.2	217.75 ± 10.2	1.22 ± 0.1	3.74 ± 1.5	BDL	BDL	BDL	BDL	7.98 ± 1.0	2.24 ± 0.2

BDL, Below detection limit.

detectors. There also were no lithium-based phases in XRD analysis. This could be due to its presence in low amounts or it being amorphous phases.

An aluminum-based phase was detected in BC Universal sealer on XRD analysis. Calcium carbonate peak was present in both Totalfill BC and BC Universal sealers after setting but was reduced following day 7 immersion. Carbonation of the specimens during preparation can occur by interaction of atmospheric carbon dioxide with the calcium hydroxide produced by hydration[22]. BC Universal sealer contained a titanium oxide peak detected in the EDS analysis. Titanium oxide could be added to enhance the whiteness of the material.

All sealers exhibited high calcium ion release at all time points. BC Universal and Totalfill BC sealers released calcium ions even after a prolonged period, whereas AH Plus Bioceramic exhibited 50% less calcium ion release as it aged. This could be because of a lesser calcium component (5%–15%) present (Table 1). Calcium release is beneficial for clinical use as it promotes antibacterial effects[21].

HBSS was used as the immersion medium to mimic an *in vivo* environment. All the tested hydraulic sealers exceeded 3%, indicating high solubility. Calcium silicate–based sealers are shown to produce high solubility and this result aligns with previous studies[9,23]. AH Plus Bioceramic discs disintegrated after the seventh day, indicating rapid high solubility as evaluated in a previous study[24]. The disintegration is due to the lower percentage (5%-15%) of tricalcium silicate cement present in the sealer. An ideal endodontic sealer should not exhibit high solubility, as it deteriorates the quality of the root canal filling and treatment[25]. This defect can encourage formation of gaps between the sealer and canal walls[26], reinfection, and inflammatory reaction when the sealers come in direct contact with the periapical tissues[27].

The ISO 6876:2012 was used to determine the solubility, and this standard indicates that the sealer solubility should not exceed 3% after immersing the samples in water. Previous research has shown that water is not a suitable medium to test the solubility of hydraulic cements because the values are not clinically translatable,[28] as the value of solubility varied with the media used.

All the tested sealers met the ISO 6876:2012[19] requirements for radiopacity. AH Plus Bioceramic was the most radiopaque (9.3 mm Al thickness), due to the high content of 50% to 75% of zirconium dioxide that is present in the sealer. Maintaining adequate radiopacity is crucial while evaluating root-filled

TABLE 3 - Results for the Determination of Physical Characteristics of the Sealers Compared With the Values Specified in the ISO 6876 Standard (Mean ± Standard Deviation)

	Property			
	Flow	Film thickness	Radiopacity	Solubility
Material	mm	μm	mm Al	%
BC Universal sealer	23.3 ± 0.7	53.3 ± 0.1	3.2 ± 0.1	5.9 ± 0.0
Totalfill BC Sealer	19.9 ± 0.5	46.7 ± 0.0	4.8 ± 0.6	4.94 ± 0.0
AH Plus Bioceramic	23.7 ± 1.0	13.3 ± 0.0	9.3 ± 2.5	4.3 ± 0.0
ISO standard value	>17	<50	>3	<3

teeth to distinctly differentiate between the sealer and surrounding structures[29].

AH Plus Bioceramic and Totalfill BC sealer met the ISO standards; however, BC Universal sealer exhibited slightly higher film thickness (53.3 μm). The film thickness of AH Plus Bioceramic sealer was much lower and this may have clinical implications when the material is used in the single cone obturation technique in which a large volume of sealer is required to compensate for the lower volume of gutta-percha used in this technique. All the sealers exhibited a similar flow complying with the standard. Previous literature[30,31] on Totalfill BC and AH Plus Bioceramic reported different flow values but all values were higher than the 17 mm specified in the ISO standard. Maintaining flow in accordance with the standards is essential, as this property allows the materials to reach complex anatomical structures of the root canal system, which could otherwise risk extrusion and cause adverse reactions to the tissues[32]. The ISO values for flow and film thickness are significantly important factors for obturation techniques using more gutta-percha than sealer, such as the warm vertical compaction and laterally condensed gutta-percha techniques.

Overall, the tested hydraulic sealers displayed adequate physical properties with high calcium release; however, they all indicated high solubility exceeding the level set by ISO 6876:2012. This *in vitro* study provides preclinical assessment of the sealers, which should be undertaken before clinical use and distribution. Although the ISO standards are used frequently to assess materials, these methods are not clinically translatable and give a rough method to compare materials *in vitro*.

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