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Evaluation of The Physicochemical Properties of Ah Plus Bioceramic Sealer And Ah Plus Sealants Upon Exposure To 5.25% Sodium Hypochlorite

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Abstract:
Objective: *To evaluate the effect of 5.25% sodium hypochlorite (NaClO) on the physicochemical properties of the endodontic sealers AH Plus Bioceramic Sealer and AH Plus.*
Materials and methods: *An in vitro study was conducted using two endodontic sealers with different chemical compositions. Samples were prepared in resin molds and exposed to either 5.25% sodium hypochlorite or distilled water as a control. Solubility, pH, and surface microhardness properties were evaluated according to ISO 6876:2012. Morphological characteristics and elemental composition of the materials were also analyzed using scanning electron microscopy and energy-dispersive X-ray spectroscopy.*
Results: *The results showed that both sealants exhibited solubility values within the limits established by the standard. The bioceramic sealant showed greater solubility compared to the epoxy sealant, especially in contact with distilled water. pH values were consistently higher in the bioceramic sealant, maintaining a highly alkaline environment. Regarding microhardness, a trend toward higher values was observed in the samples exposed to NaClO. Microscopic analysis revealed changes in surface morphology and variations in the elemental composition of the materials after exposure to the irrigant.*
Conclusion: *Exposure to 5.25% sodium hypochlorite can alter some physicochemical properties of the evaluated endodontic sealers, particularly solubility, microhardness, and surface characteristics, suggesting that the interaction between the sealers and irrigating solutions could influence their clinical performance.*
Keywords: *Irrigants, NaClO, Bioceramic sealers, AH Plus Bioceramic Sealer, AH Plus, Solubility, pH, Microhardness, SEM, EDS.*

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I. Introduction

The success of endodontic treatment depends on the effective elimination of microorganisms and infected tissues from the root canal system (1). This procedure comprises three main stages: mechanical preparation, irrigation, and root canal obturation (2,3). Mechanical preparation aims to shape and enlarge the root canal to facilitate irrigation and enable adequate obturation (4). However, owing to the complex anatomy of the root canal system and the frequent occurrence of canals with an oval-shaped morphology, endodontic instruments are unable to contact all root canal walls (5). It has been reported that mechanical instrumentation affects only approximately 65% of the root canal surface, leaving uninstrumented areas that may harbor tissue remnants, bacteria, and their by-products (6). Therefore, irrigation plays a crucial role in root canal disinfection by facilitating the removal of the smear layer generated during instrumentation, while also providing lubrication and antimicrobial activity against various bacterial species present in endodontic biofilms (7,8).

Subsequently, root canal obturation aims to achieve a three-dimensional seal that prevents reinfection of the root canal system (9). This procedure is generally performed using gutta-percha cones in combination with an endodontic sealer, which fills canal irregularities and improves the adaptation of the obturation material to root dentin (10–12).

In recent years, tricalcium silicate-based bioceramic sealers have been introduced as an alternative to conventional epoxy resin-based sealers because of their bioactive properties, biocompatibility, and ability to release calcium ions, thereby promoting hydroxyapatite formation (13,14).

During endodontic treatment, irrigating solutions such as sodium hypochlorite are widely used because of their antimicrobial activity and ability to dissolve organic tissues. However, residual sodium hypochlorite within

the root canal may influence the physicochemical properties of endodontic sealers and potentially affect their clinical performance (15).

Therefore, the aim of this study was to evaluate the effect of 5.25% sodium hypochlorite on the physicochemical properties of AH Plus Bioceramic Sealer and AH Plus.

II. Material And Methods

Mold Design

The physicochemical properties of the sealers with and without contact with 5.25% sodium hypochlorite (NaOCl) were evaluated using cylindrical specimens measuring 10 mm in diameter and 1.5 mm in height. The molds were designed using Tinkercad (Autodesk, USA), exported in STL format, and processed using Chitubox software for fabrication with a Phrozen Sonic 3D printer (Phrozen Technology, Taiwan) and SUNLU 3D resin (China). Subsequently, the molds were washed with 99.9% isopropyl alcohol, UV-cured using an LC-3D Print Box (NextDent, The Netherlands), and their dimensions were verified using a digital caliper.

Specimen Preparation

AH Plus Bioceramic Sealer and AH Plus were placed into cylindrical molds, leaving only the upper surface exposed. In the experimental group, 25 µL of 5.25% NaOCl was applied for 1 min. The solution was then removed, and the specimens were incubated at 37 °C for 20 min to eliminate excess liquid. The sealers were allowed to set for 1.5 times the setting time specified by the manufacturer.

Solubility and pH Study Design

Specimens of both sealers, with and without contact with NaOCl, were prepared (n = 4). The specimens were weighed using an analytical balance and placed individually in vials containing 2.717 mL of distilled water, in accordance with the surface area-to-volume ratio specified by ISO 6876 (approximately 28.9 mm^2/mL). The vials were incubated at 37 °C and 100% humidity for 24 h, after which the pH of the immersion medium was measured using a pH meter.

The specimens were subsequently rinsed with 2 mL of distilled water and dried in a vacuum oven (DZF-6050) at 110 °C for 24 h to determine their final mass. This procedure was repeated over three evaluation periods (24 h, 5 days, and 7 days).

Microhardness Testing

The specimens were polished using 1200-grit silicon carbide abrasive paper, and Vickers microhardness was evaluated by performing three indentations on each specimen under a 100-g load applied for 10 s.

Microscopic and Elemental Analysis

The sealer surfaces were examined by scanning electron microscopy (SEM) at 100× magnification. Elemental composition was assessed by energy-dispersive X-ray spectroscopy (EDS) using a TM4000Plus II microscope (Hitachi, Japan) operated at an accelerating voltage ranging from 5 to 15 kV.

Statistical Analysis

Descriptive statistics were calculated, followed by a two-way analysis of variance (ANOVA) to evaluate the effects of the factors under investigation and their interaction. The level of statistical significance was set at $p < 0.05$. Statistical analyses were performed using IBM SPSS Statistics.

III. Result

Solubility

Descriptive analysis revealed differences in solubility among the experimental conditions across the evaluated immersion periods (1, 5, and 7 days). Overall, the AH Plus Bioceramic Sealer (AHPB) groups exhibited higher solubility values than the AH Plus (AHP) groups under all experimental conditions (Table 1).

Table 1. Descriptive statistics of the solubility of AHPB and AHP sealers under different immersion conditions and evaluation periods.

Condition	Solubility – Day 1 (Mean ± SD)	Solubility – Day 5 (Mean ± SD)	Solubility – Day 7 (Mean ± SD)
AHPB + Distilled Water	0.8696 ± 0.0261	1.7224 ± 0.0689	2.5920 ± 0.0907
AHPB + Sodium Hypochlorite	1.1092 ± 0.0333	2.2857 ± 0.0914	2.3193 ± 0.0812
AHP + Distilled Water	0.3448 ± 0.0103	0.1623 ± 0.0065	0.1826 ± 0.0064
AHP + Sodium Hypochlorite	0.3429 ± 0.0103	0.0952 ± 0.0038	0.0571 ± 0.0020

One-way analysis of variance (ANOVA) confirmed statistically significant differences among the groups at all evaluation time points (Day 1: F = 890.63; Day 5: F = 1117.62; Day 7: F = 1480.32; $p < 0.001$), indicating that sealer type and immersion medium significantly influenced solubility (Tables 2–4). Furthermore, a progressive increase in the magnitude of the differences among groups was observed over time.

Table 2. One-way ANOVA results for solubility at 24 h.

Source	SS	df	MS	F	p
Between Groups	1.556638	3.000	0.518879	890.630	<0.001
Within Groups	0.004661	8.000	0.000583	—	—
Total	1.561298	11.000	—	—	—

Table 3. One-way ANOVA results for solubility at Day 5.

Source	SS	df	MS	F	p
Between Groups	6.980037	3.000	2.326679	1117.620	<0.001
Within Groups	0.016677	8.000	0.002085	—	—
Total	6.996714	11.000	—	—	—

Table 4. One-way ANOVA results for solubility at Day 7.

Source	SS	df	MS	F	p
Between Groups	9.269493	3.000	3.089831	1480.316	<0.001
Within Groups	0.016711	8.000	0.002089	—	—
Total	9.286204	11.000	—	—	—

Multiple comparisons using Tukey's honestly significant difference (HSD) test confirmed that the AHPB groups exhibited significantly higher solubility values than the AHP groups at all evaluated time points ($p < 0.001$). Within the epoxy resin-based sealer groups, differences between distilled water and sodium hypochlorite were minimal, although slightly higher values were observed in distilled water at some evaluation time points. In contrast, the effect of the immersion medium was more pronounced for the bioceramic sealer. Sodium hypochlorite increased solubility during the initial evaluation periods, whereas greater solubility was observed in distilled water on Day 7 (Tables 5–7).

Table 5. Multiple comparisons using Tukey's HSD test for solubility at 24 h.

Comparison	Mean Difference	95% CI (Lower–Upper)	Adjusted p-value	Significant
AHP + Distilled Water vs AHP + Sodium Hypochlorite	−0.0020	−0.0348–0.0308	0.9995	No
AHP + Distilled Water vs AHPB + Distilled Water	−0.5248	−0.5576–−0.4920	<0.001	Yes
AHP + Distilled Water vs AHPB + Sodium Hypochlorite	−0.7644	−0.7972–−0.7316	<0.001	Yes
AHP + Sodium Hypochlorite vs AHPB + Distilled Water	−0.5228	−0.5556–−0.4900	<0.001	Yes
AHP + Sodium Hypochlorite vs AHPB + Sodium Hypochlorite	−0.7624	−0.7952–−0.7296	<0.001	Yes
AHPB + Distilled Water vs AHPB + Sodium Hypochlorite	−0.2396	−0.2724–−0.2068	<0.001	Yes

Table 6. Multiple comparisons using Tukey's HSD test for solubility at Day 5.

Comparison	Mean Difference	95% CI (Lower–Upper)	Adjusted p-value	Significant
AHP + Distilled Water vs AHP + Sodium Hypochlorite	0.0671	0.0228–0.1114	0.0021	Yes
AHP + Distilled Water vs AHPB + Distilled Water	−1.5601	−1.6044–−1.5158	<0.001	Yes
AHP + Distilled Water vs AHPB + Sodium Hypochlorite	−2.1234	−2.1677–−2.0791	<0.001	Yes
AHP + Sodium Hypochlorite vs AHPB + Distilled Water	−1.6272	−1.6715–−1.5829	<0.001	Yes

Comparison	Mean Difference	95% CI (Lower–Upper)	Adjusted p-value	Significant
AHP + Sodium Hypochlorite vs AHPB + Sodium Hypochlorite	−2.1905	−2.2348–−2.1462	<0.001	Yes
AHPB + Distilled Water vs AHPB + Sodium Hypochlorite	−0.5633	−0.6076–−0.5190	<0.001	Yes

Table 7. Multiple comparisons using Tukey's HSD test for solubility at Day 7.

Comparison	Mean Difference	95% CI (Lower–Upper)	Adjusted p-value	Significant
AHP + Distilled Water vs AHP + Sodium Hypochlorite	0.1255	0.0820–0.1690	0.0001	Yes
AHP + Distilled Water vs AHPB + Distilled Water	−2.4094	−2.4529–−2.3660	<0.001	Yes
AHP + Distilled Water vs AHPB + Sodium Hypochlorite	−2.1367	−2.1801–−2.0932	<0.001	Yes
AHP + Sodium Hypochlorite vs AHPB + Distilled Water	−2.5349	−2.5784–−2.4914	<0.001	Yes
AHP + Sodium Hypochlorite vs AHPB + Sodium Hypochlorite	−2.2622	−2.3057–−2.2187	<0.001	Yes
AHPB + Distilled Water vs AHPB + Sodium Hypochlorite	0.2730	0.2295–0.3165	<0.001	Yes

Overall, these findings indicate that solubility was primarily determined by the type of material, with significantly greater solubility observed for the bioceramic sealer (AHPB) than for the epoxy resin-based sealer (AHP). In contrast, the effect of the immersion medium was secondary and dependent on the duration of exposure.

pH

pH values showed clear differences between the evaluated materials throughout the experimental period. Overall, the AHPB bioceramic sealer exhibited significantly more alkaline pH values than the AHP epoxy resin-based sealer under all experimental conditions and at all evaluation time points. The AHPB groups maintained a pH of approximately 12, indicating a strongly alkaline and relatively stable environment throughout the study period. In contrast, the AHP groups exhibited considerably lower values, with a pH of approximately 9 and minimal variation between immersion media. Overall, these findings indicate that AHPB produces a more alkaline chemical environment than AHP (Table 8).

Table 8. Descriptive statistics of pH values (n = 3).

Condition	pH 1 (Mean ± SD)	pH 2 (Mean ± SD)	pH 3 (Mean ± SD)
AHPB + Distilled Water	12.00 ± 0.05	11.90 ± 0.04	11.30 ± 0.03
AHPB + Sodium Hypochlorite	11.80 ± 0.05	11.90 ± 0.04	11.30 ± 0.03
AHP + Distilled Water	9.60 ± 0.05	9.30 ± 0.04	9.10 ± 0.03
AHP + Sodium Hypochlorite	9.00 ± 0.05	9.10 ± 0.04	9.10 ± 0.03

Microhardness

Microhardness values varied according to the material and immersion solution evaluated. Overall, the groups exposed to sodium hypochlorite exhibited higher microhardness values than those immersed in distilled water. However, analysis of variance revealed no statistically significant differences among the groups (ANOVA, $p = 0.0689$).

Post hoc comparisons using the Tukey HSD test identified only one statistically significant difference, between the AH Plus Bioceramic Sealer/distilled water group and the AH Plus/sodium hypochlorite group, with the latter exhibiting greater microhardness. No statistically significant differences were observed for the remaining comparisons (Tables 9–11; Graph 1).

Table 9. Descriptive statistics of microhardness.

Group	Mean (HV 0.2)	SD
AHPB + Distilled Water	151.13	36.01
AHPB + Sodium Hypochlorite	204.33	11.35
AHP + Distilled Water	182.40	5.72
AHP + Sodium Hypochlorite	238.60	69.16

Table 10. One-way ANOVA results for microhardness.

Source	SS	df	MS	F	p
Between Groups	24,409.28	3	8,136.43	5.2145	0.0080
Within Groups	31,206.83	20	1,560.34	—	—
Total	55,616.11	23	—	—	—

Table 11. Multiple comparisons of microhardness using Tukey's HSD post hoc. test.

Group A	Group B	Mean Difference (HV)	Tukey q	Adjusted p-value	95% CI Lower	95% CI Upper	Significant (α = 0.05)
AH Plus Bioceramic / Distilled Water	AH Plus Bioceramic / Sodium Hypochlorite	53.20	3.299	0.124	−10.63	117.03	No
AH Plus Bioceramic / Distilled Water	AH Plus / Distilled Water	31.27	1.939	0.531	−32.56	95.10	No
AH Plus Bioceramic / Distilled Water	AH Plus / Sodium Hypochlorite	87.47	5.424	0.005	23.64	151.30	Yes
AH Plus Bioceramic / Sodium Hypochlorite	AH Plus / Distilled Water	21.93	1.360	0.772	−41.90	85.76	No
AH Plus Bioceramic / Sodium Hypochlorite	AH Plus / Sodium Hypochlorite	34.27	2.125	0.454	−29.56	98.10	No
AH Plus / Distilled Water	AH Plus / Sodium Hypochlorite	56.20	3.485	0.097	−7.63	120.03	No

SEM-EDS Analysis

SEM analysis revealed morphological differences between the evaluated sealers following exposure to distilled water and sodium hypochlorite.

For AH Plus Bioceramic Sealer, both experimental conditions showed a relatively homogeneous surface with a cement-like appearance. EDS analysis revealed a predominance of oxygen and zirconium, along with the presence of calcium and trace amounts of silicon and aluminum. This elemental composition is consistent with a calcium silicate-based matrix containing zirconium dioxide as a radiopacifier. Following exposure to sodium hypochlorite, a relative decrease in calcium and an increase in zirconium were observed at the surface, suggesting a modification of the surface composition of the material.

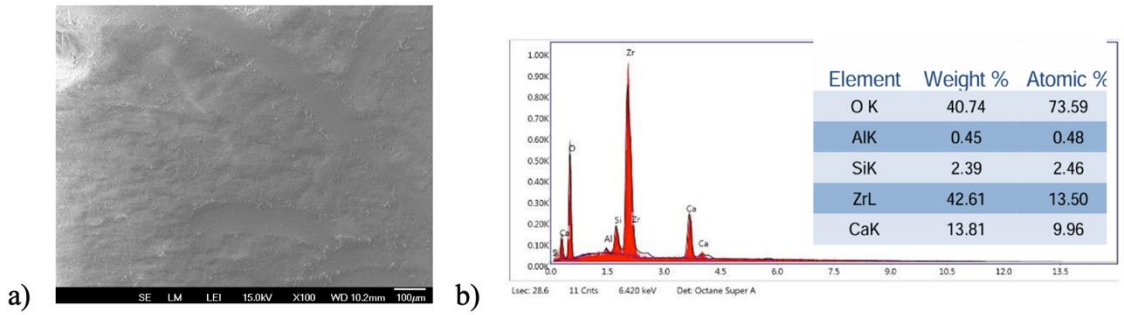


Figure 1. (a) Surface morphology of AHPB specimens exposed to distilled water, as observed by scanning electron microscopy (SEM) at 100× magnification. (b) EDS spectrum of AHPB exposed to distilled water, showing elemental composition by weight and atomic percentage.

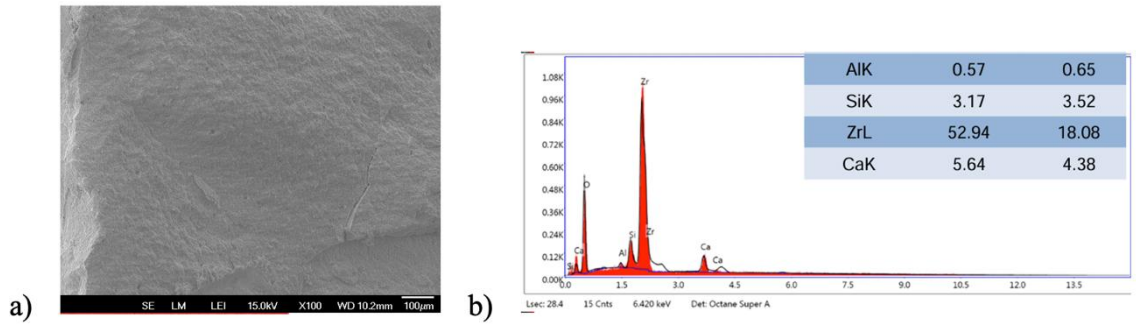


Figure 2. (a) Surface morphology of AHPB specimens exposed to 5.25% NaOCl, as observed by scanning electron microscopy (SEM) at 100× magnification. (b) EDS spectrum of AHPB exposed to 5.25% NaOCl, showing elemental composition by weight and atomic percentage.

For AH Plus, SEM micrographs showed a continuous, resin-like surface in specimens exposed to distilled water, whereas specimens exposed to sodium hypochlorite exhibited surface fractures and irregularities. EDS analysis confirmed the predominance of elements characteristic of an organic matrix (C, 64.13%; O, 25.51%; and N, 7.74%). However, specimens exposed to sodium hypochlorite exhibited greater surface exposure of radiopacifying components, primarily tungsten and zirconium.

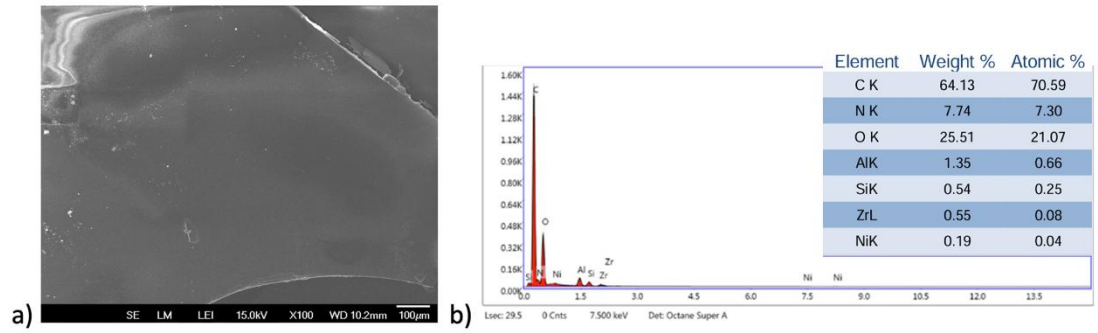


Figure 3. (a) Surface morphology of AHP specimens exposed to distilled water, as observed by scanning electron microscopy (SEM) at 100× magnification. (b) EDS spectrum of AHP exposed to distilled water, showing elemental composition by weight and atomic percentage.

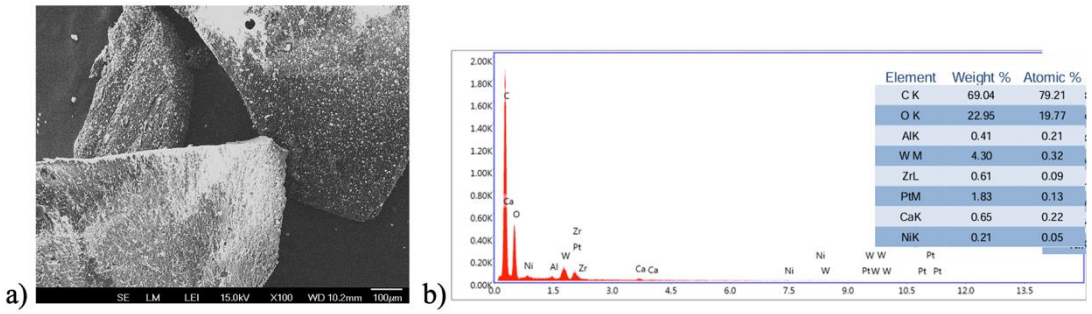


Figure 4. (a) Surface morphology of AHP specimens exposed to 5.25% NaOCl, as observed by scanning electron microscopy (SEM) at 100× magnification. (b) EDS spectrum of AHP exposed to 5.25% NaOCl, showing elemental composition by weight and atomic percentage.

Overall, the findings suggest that sodium hypochlorite induces changes in the surface composition of both sealers, although distinct patterns were observed for each material. In the bioceramic sealer, a relative reduction in surface calcium content was observed, whereas the epoxy resin-based sealer exhibited greater surface exposure of radiopacifying particles.

IV. Discussion

The aim of the present study was to evaluate the effect of 5.25% sodium hypochlorite (NaOCl) on the physicochemical properties of AH Plus Bioceramic Sealer (AHPB), using the epoxy resin-based AH Plus sealer (AHP) as a control. The findings demonstrated that NaOCl induced changes in several properties of the evaluated sealers, including solubility, pH, microhardness, and surface characteristics.

The sealers evaluated in this study have different chemical compositions. AHPB is a calcium silicate-based bioactive sealer that has demonstrated high cytocompatibility and bioactive potential compared with other sealers when in contact with human periodontal ligament stem cells (16). Furthermore, according to the manufacturer, it exhibits an adequate setting time, favorable flow characteristics, and dimensional stability (17). In contrast, AHP is an epoxy resin-based sealer that is widely used and is considered a "gold standard" in endodontics because of its adequate flow, low shrinkage, and low solubility (17–19).

Regarding solubility, all evaluated groups met the limit established by ISO 6876 (<3% mass loss at 24 h) (20). Nevertheless, AHPB exhibited significantly higher solubility values than AHP at all evaluation periods. This behavior is consistent with the bioactive nature of bioceramic sealers, which release calcium ions during the hydration process. Similar findings were reported by Hamdy et al., who observed a progressive increase in the solubility of bioceramic sealers in contact with distilled water over time (21). In the present study, contact with NaOCl showed a tendency to limit this mass loss compared with distilled water, which may be related to surface modifications induced by the oxidizing agent.

The pH analysis revealed consistent differences between the materials. AHPB maintained highly alkaline values of approximately pH 12 throughout the evaluation period, whereas AHP exhibited values of approximately pH 9. This behavior can be attributed to the release of hydroxyl ions resulting from the hydration of calcium silicates present in bioceramic sealers. Previous studies have reported similar findings, with AHPB exhibiting higher pH values than other endodontic sealers (68,80,81). This alkaline environment has been associated with bioactive properties and potent antimicrobial activity.

Regarding microhardness, no statistically significant differences were observed among the evaluated groups, although a trend toward higher values was identified in specimens exposed to NaOCl. This effect may be related to the oxidizing and dehydrating properties of sodium hypochlorite, which may induce changes in the surface structure of the materials. In the case of the epoxy resin-based sealer, this interaction could promote oxidation or cross-linking processes within the polymer matrix, resulting in a more rigid surface. Similar findings have been reported by Kapralos et al., who observed greater mechanical resistance in an epoxy resin-based sealer compared with bioceramic sealers (22).

Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) analyses revealed morphological and compositional changes in the surfaces of the sealers. In AHPB, exposure to NaOCl was associated with a relative reduction in surface calcium content and an increase in zirconium, suggesting calcium leaching induced by the oxidizing environment. This phenomenon has been described in studies of calcium silicate-based materials, in which chloride ions may interact with hydrated phases of the material and promote calcium diffusion (23,24).

In contrast, the AHP epoxy resin-based sealer exhibited a smooth and continuous surface following exposure to distilled water, whereas specimens exposed to NaOCl showed surface irregularities and fractures. EDS analysis revealed greater exposure of radiopacifying elements, such as tungsten and zirconium, suggesting possible partial degradation of the organic matrix following contact with the oxidizing agent.

Taken together, these findings indicate that sodium hypochlorite may induce surface modifications in both sealers, although through different mechanisms. In the bioceramic sealer, the observed changes appear to be associated with calcium release and leaching, whereas in the epoxy resin-based sealer, the alterations appear to be related to partial degradation of the polymer matrix and subsequent exposure of inorganic particles.

Bioceramic sealers currently represent a growing area of research in endodontics; however, information regarding their interaction with widely used irrigating solutions such as sodium hypochlorite remains limited. Therefore, future studies should further investigate these interactions under experimental conditions that more closely simulate the clinical environment to better understand their potential impact on the performance and long-term stability of these materials.

V. Conclusion

Within the limitations of this in vitro study, exposure to 5.25% sodium hypochlorite (NaOCl) induced changes in some of the physicochemical properties of the evaluated sealers, particularly solubility and microhardness. Changes in surface morphology and elemental composition were also observed, suggesting that contact with this irrigating solution may influence the surface stability of these materials. Although both sealers maintained values within clinically acceptable ranges, these findings indicate that interactions between endodontic sealers and irrigating solutions may have implications for their long-term behavior.

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