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Solubility, pH Changes and Releasing Elements of Different Bioceramic and Mineral Trioxide Aggregate Root Canal Sealers Comparative Study

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Abstract

Introduction: Bioceramic technology has been introduced recently in endodontics to benefit from the formation of hydroxyapatite during the setting reaction in the presence of tissue fluid and establish a chemical bond at the dentin interface.
Objectives: To evaluate some of physiochemical properties of new different Bioceramic (iRoot-SP, EndoSequence, Smartpastebio) versus mineral trioxide aggregate (MTA-Fillapex) root canal sealers. Adseal and ActiV-GP sealers were used as control.

Methods: Standard discs (10 for each) were prepared and immersed in 20 ml deionized water. After 1, 7, 14 and 28 days, solubility, pH changes and released elements were calculated and statistically analyzed with ANOVA test.
Results: A significantly greater solubility and higher alkalinity were displayed by the tested sealers (in descending order) Smartpastebio, iRoot-SP, EndoSequence and MTA-Fillapex ($P<0.05$). Their solubility exceeded the acceptable limit (3%). Their maximum alkaline pH was exhibited after 7 days. Adseal and ActiV-GP exhibited initial neutral and acidic pH respectively. Finally both had neutral pH. EndoSequence exhibited the significantly greatest calcium release followed by iRoot-SP, Smartpastebio and MTA-Fillapex, whereas, ActiV-GP and Adseal exhibited the significantly lowest values ($P<0.05$). There was no silicon released from iRoot-SP, Smartpastebio and MTA-Fillapex. ActiV-GP exhibited the greatest silicon, aluminum and iron release. The greatest phosphorous, manganese and magnesium content was obtained by Adseal, MTA-Fillapex and EndoSequence respectively.
Conclusion: Under the condition of this study, the prolonged alkalinity of calcium silicate sealers was synchronized with their ongoing solubility. The higher calcium ions released indicated strong alkalinity. The inequality in aluminum, iron, manganese and magnesium released by the tested sealers may expect the variance in their behaviors.

Keywords: Mineral trioxide aggregate; Calcium silicate sealers; Calcium ions

Introduction

Bioceramic and Mineral Trioxide Aggregate (MTA) root canal sealers were recently introduced into the endodontic field to benefit from the formation of hydroxyapatite during the setting reaction in the presence of tissue fluid and establish a chemical bond at the dentin interface [1]. Solubility, pH changes and released elements of root canal sealers have an impact on their clinical, biological and antibacterial behaviors. Most sealers exhibit a variable degree of solubility that affects the integrity of the apical seal of a root canal filling [2]. Therefore, insolubility is a standard property for root canal sealer. The alkaline pH is closely related to the increased hydroxyl and calcium ion (Ca^{2+}) release after root canal obturation, which inhibits growth of residual microbes, and this enhances healing of periapical pathosis [3,4].

Recent Calcium Silicate Sealers (CSS) have been claimed to be excellent sealers with alkaline pH, low solubility and providing good sealing owing to their setting expansion [5]. To date, there are not enough publications that prove their physiochemical behavior.

The aim of this study was to evaluate solubility, pH changes, calcium ions released and degradable elements of different brands of CSS. The null hypothesis is that there is no significant difference between the tested root canal sealers.

Materials and Methods

Three brands of injectable Bioceramic: EndoSequence (Brasseler, Georgia, USA), Smartpastebio (Smartseal, DRFP Ltd, UK) and iRoot-SP (Innovative BioCeramix Inc, Vancouver, Canada) with MTA-Fillapex (Angelus, Londrina, Brazil) root canal sealers were tested. Adseal (META Biomed Co., Chungbuk, Korea) and ActiV-GP (Brasseler, Georgia, USA) were used as control.

According to American Dental Association (ADA) specification number 57 for root canal filling, standard discs (15 mm diameter, 3 mm thickness) of fresh mixed sealers were prepared (10 for each material), according to manufacturer instructions. They were left in an incubator at 37°C, 100% humidity until complete setting. Each disc was tied with impermeable nylon thread and the initial weight (W_0) measured using an analytical balance machine (Balance and Scale Model AW-220, Shimadzu Corporation, Kyoto, Japan). Then they were immersed in 20 ml deionized water and incubated for 1, 7, 14 and 28 days in 100% humidity at a constant temperature of 37°C.

Solubility (%) evaluation: After each immersion time, the discs were removed from the tube, dried with blotted paper, left 24 hours for complete dryness and then reweighed (Wf_1, Wf_7, Wf_{14} and Wf_{28}). The amount of solubility (%) was calculated by the following equation:

Solubility (%) = $\frac{W_0 - W_f}{W_0} \times 100$ [6].

pH analysis: The pH of storage solution at each immersion time (1, 7, 14 and 28 days) was analysed using a pH meter (HANNA pH 211,

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UVP Inc., UpLand, USA) previously calibrated with standard solutions at pH 4.0 and 7.0 at a constant temperature (25°C).

Released elements analysis: The solutions after each immersion time were analyzed for the amount of calcium ions (Ca^{2+}) released using an Inductively Coupled Plasma (ICP) Spectrometer (model JY-Ultima 2). At the 28th day, the solutions were analyzed for other degradable elements, including phosphorus, silicate, aluminum, iron, manganese and magnesium using the ICP spectrometer.

Statistical analysis: According to the normality test, the data of solubility, pH and released elements were statistically analyzed by the One-Way ANOVA and Post-Hoc Tukey HSD tests using SPSS software (Version 16.0; SPSS, Inc, Chicago, IL) at significance level of 5%, to compare the tested materials.

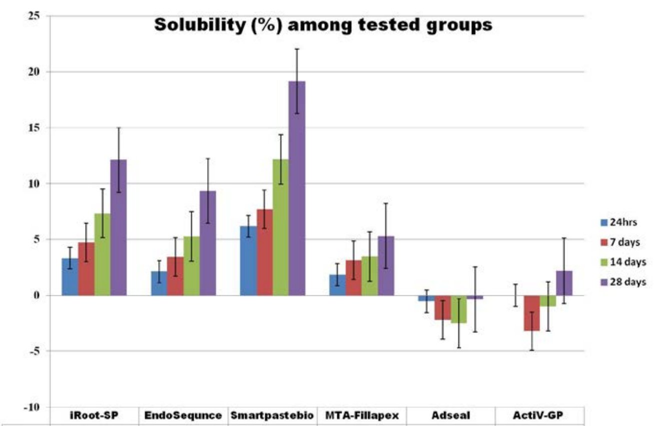
Results

Solubility (%)

Figure 1 illustrates the comparison between the mean values of solubility % of the tested materials at different immersion times. The null hypothesis for sealer solubility was rejected. The four CSS exhibited ongoing increases in solubility overtime, exceeding the acceptable limit of ADA. The significantly highest value was obtained by Smartpastebio, whereas, MTA-Fillapex displayed the most significantly lowest value at $P < 0.05$. Adseal and ActiV-GP displayed an initial increase in weight % that gradually declined with the final solubility falling within the acceptable ADA tolerance range (Figure 1).

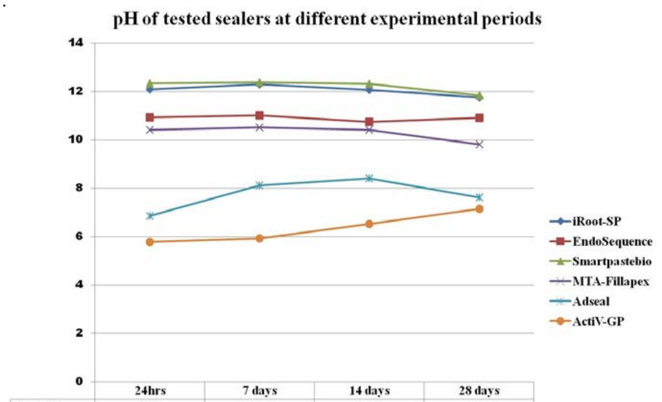
pH changes

The pH mean values of all tested materials at different immersion times are described in Figure 2. An ANOVA test revealed a statistically significant difference among groups (P=0.000). Therefore, the null hypothesis of pH changes within the groups was rejected. The four CSS exhibited high alkaline pH over time with the maximum value occurred on the 7th day. Smartpastebio and iRoot-SP exhibited significantly higher alkalinity than that obtained by EndoSequence and MTA-Fillapex ($P<0.05$). Adseal exhibited an initial neutral pH that was followed by a weak alkaline pH and then a final neutral pH. Whereas, ActiV-GP exhibited an initial acidic pH that followed by a final neutral pH (Figure 2).



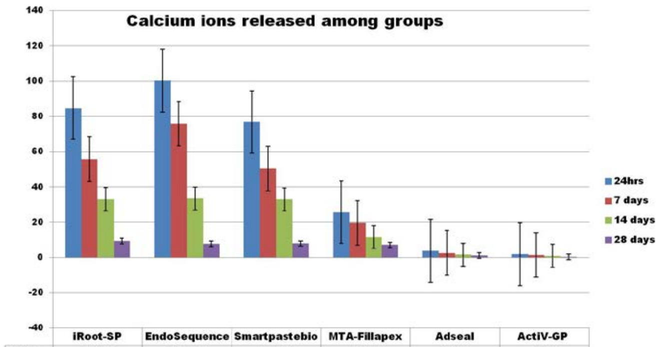
	iRoot-SP	EndoSequnce	Smartpastebio	MTA-Fillapex	Adseal	ActiV-GP
24hrs	3.32	2.12	6.17	1.85	-0.55	-0.013
7 days	4.73	3.44	7.67	3.13	-2.22	-3.22
14 days	7.33	5.27	12.14	3.46	-2.52	-0.98
28 days	12.1	9.33	19.15	5.3	-0.38	2.17

Figure 1: Error bar representing the mean values of solubility (%) over time among all tested sealers.



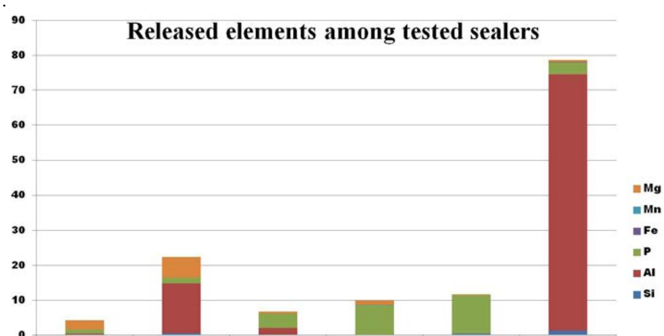
	24hrs	7 days	14 days	28 days
iRoot-SP	12.1	12.29	12.07	11.75
EndoSequence	10.94	11.02	10.75	10.92
Smartpastebio	12.34	12.38	12.32	11.84
MTA-Fillapex	10.41	10.52	10.42	9.81
Adseal	6.85	8.11	8.41	7.63
ActiV-GP	5.78	5.92	6.52	7.15

Figure 2: Plot lines representing the mean values of pH change over time for tested sealers.



	iRoot-SP	EndoSequence	Smartpastebio	MTA-Fillapex	Adseal	ActiV-GP
24hrs	84.71	100.27	76.78	25.69	3.71	1.86
7 days	55.7	75.78	50.52	19.66	2.54	1.36
14 days	32.99	33.52	32.94	11.54	1.59	0.97
28 days	9.28	7.8	7.9	7.1	1.03	0.24

Figure 3: Error bar representing the mean values of released calcium ions (mg/L) over time for the tested sealers.



	iRoot-SP	EndoSequence	Smartpastebio	MTA-Fillapex	Adseal	ActiV-GP
Mg	2.63	5.9	0.58	1.36	0.28	0.54
Mn	0.003	0	0	0.06	0	0
Fe	0.01	0.015	0.015	0.02	0.03	0.2
P	1.26	1.6	4.04	8.47	10.9	3.35
Al	0.44	14.4	2.12	0.09	0	73.3
Si	0	0.46	0	0	0.44	1.28

Figure 4: Clustered chart representing the cumulative amount of released elements (mg/L) by the tested sealers after 28 days.

Released elements

Figures 3 and 4 respectively describe the mean values of Ca^{2+} at different observation times and cumulative amounts of degradable elements released after 28 days (mg/L) by tested sealers. The null hypothesis of released elements was rejected. Regarding Ca^{2+} released, there was a statistically significant difference among groups ($P<0.05$). EndoSequence exhibited the significantly highest calcium released followed by iRoot-SP, Smartpastebio and MTA-Fillapex, whereas, ActiV-GP and Adseal sealers exhibited the significantly lowest values at $P<0.05$ (Figure 3). ActiV-GP exhibited the greatest silicon and aluminum release. The greatest phosphorous release was exhibited by Adseal (Figure 4).

With respect to the CSS, there was no silicon released from iRoot-SP, Smartpastebio nor MTA-Fillapex. The greatest amount of aluminum was released by EndoSequence. MTA-Fillapex exhibited the greatest release of phosphorous plus traces of iron and manganese. EndoSequence exhibited a greater magnesium amount than other CSS.

Discussion

In accordance to the requirements of ADA specifications No. 57 [7], the current study assessed the solubility (%), pH changes, Ca^{2+} ion released and degradable elements of three Bioceramic (iRoot-SP, EndoSequence and Smartpastebio) and MTA (MTA-Fillapex) sealers in comparison with epoxy-resin calcium phosphate-based (Adseal) and glass-ionomer-based (ActiV-GP) sealers.

Solubility is considered an undesirable property for root canal sealer because it creates gaps along the filling/dentin interface due to released ions and disintegrated particles compromising the sealer's effectiveness [8,9]. It was suggested that the root canal sealer must be insoluble or minimal when exposed to periapical tissues [10]. To prevent bacterial leakage [11].

In the current study, the four calcium silicate sealers displayed a significant greater solubility %, ongoing increasing overtime, which exceeds the ADA tolerance (3%). In descending order, the final solubility (after 28 days) was exhibited by Smartpastebio, iRoot-SP, EndoSequence, MTA-Fillapex, ActiV-GP and Adseal. EndoSequence and MTA-Fillapex have previously displayed significantly high solubility after 30 days, within the permitted limit [12,13]. In another study, the solubility of MTA-Fillapex exceeded the acceptable limit [14].

Regarding glass-ionomer- and epoxy resin-based sealers used in the current study: ActiV-GP and Adseal exhibited initial increase in weight % (for 7 and 14 days respectively) that declined with final solubility being reached after 28 days within the acceptable limit. In accordance with the current result, epoxy resin sealers previously exhibited weight gain within 4 weeks and then they became stable [15,16]. The initial weight increases of resin and glass-ionomer sealers may be attributed to their water sorption during setting [8,16]. It was found that epoxy resin exhibited a porous hydrophilic network of polymerized resin matrix that permitted initial water sorption [13], thus increasing its solubility resistance [17]. ActiV-GP previously displayed high solubility that exceeded the standard level [14,16]. The final solubility of Adseal and ActiV-GP may be attributed to polymer degradation of unreacted particles of resin [18] or ion solubilization of the glass-ionomer ActiV-GP sealer [19].

With regard to the pH of CSS, there were controversies among studies. Some studies supported our finding as strongly alkalinity (pH range 10-12) that continued for four weeks after setting [12,14,20]. Another showed that the initial pH of MTA-Fillapex was low in alkaline (pH = 9.3) that gradually declined over time to be 7.76 after 7 days [5]. It was believed that a strong alkaline pH may encourage a prolonged setting time [19] which enhances a long-lasting antibacterial effect and eliminates the residual microbes that survive on the dentinal wall [20].

The pH of the sealers varied according to various components and products of their setting reaction [3,4]. It was concluded that increases in solubility overtime enhance the ongoing alkaline effect and determine the longevity of the optimal effect of calcium hydroxide content [14,21]. In 2013, Silva et al. [5] suggested that due to high alkalinity of MTA-Fillapex, it had a strong capacity to release hydroxyl ions, thereby causing a high Ca^{2+} ion release [5,17]. The alkaline media could activate the alkaline phosphatase, neutralize the acid, inactivate the osteoclasts, prevent the further bone destruction and allow tissue repair [5] with concomitant apatite formation [22]. The extreme alkalinity, however, can induce severe tissue cytotoxicity overtime [5]. The significant difference in Ca^{2+} released from the four brands of CSS confirmed its different alkaline pH values.

On the other hand, Adseal and ActiV-GP exhibited initial neutral and acidic pH respectively that could accelerate the setting reaction [19]. The maturation of glass-ionomer sealer in the presence of humidity was characterized by the formation of polyacrylic acid resulting in acidic pH media and thus increasing the sealer's solubility [16]. In accordance with the current finding, the epoxy-resin-based sealer previously displayed a slightly neutral pH throughout the experimental period [12,23], which may attribute to its low solubility [24]. Despite Adseal displaying a neutral pH, its previously determined high antibacterial effect against black pigmented bacteria was due to its calcium phosphate content [25]. This premise was reinforced by Faria-Júnior et al. [14] who concluded that the neutral pH and low solubility of sealer would eliminate its antibacterial activity. The low amount of Ca^{2+} released by Adseal and ActiV-GP, however, confirmed that its lower pH may affect its bioactivity.

ActiV-GP released relatively greater silicon followed by EndoSequence and Adseal, whereas, no silicon was released by iRoot-SP, Smartpastebio and MTA-Fillapex. The greatest amount of phosphorous was released by Adseal, followed by MTA-Fillapex, Smartpastebio, ActiV-GP, EndoSequence and iRoot-SP. It was concluded that silicon and phosphorus were stronger inducers of dentin remineralisation and new bone growth than the effect of calcium [26,27]. On the other hand, the magnesium released may inhibit the mineralization process [28] and exhibited cytotoxicity [29]. EndoSequence released significantly high Ca^{2+}, which may enhance its bioactivity and may be considered as compensatory for the magnesium released. Otherwise, the amount of phosphorus released by Adseal, and silicon released by ActiV-GP may be considered as compensatory for their lower Ca^{2+} released in the healing process [28].

According to the manufacturer, ActiV-GP composed of barium-alumina-silicate glass powder explains its higher aluminum release versus the other tested sealers. It may play a role in its fast setting time [30] and tissue toxicity [31]. The higher iron and manganese traces were detected in ActiV-GP and MTA-Fillapex respectively. No manganese was detected in EndoSequence, Smartpastebio, Adseal and ActiV-GP.

Conclusion

Under the condition of this study, the prolonged alkalinity of all tested calcium silicate sealer was synchronized with increases in solubility. This may encourage their biological and antimicrobial behaviors over time. On the other hand, the ongoing solubility of sealer indicates permanence disintegration that may impact the sealer's ability to prevent apical leakage. The inequality in aluminum, iron, manganese and magnesium released by the tested sealers may expect the variance in their ability to help provide an acceptable apical seal after obturation. It needs further investigation to evaluate the sealing ability of Bioceramics and MTA sealers as the effect of their high solubility and releasing elements.

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