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TIME-DEPENDENT ASSESSMENT OF pH OF THREE DIFFERENT BASE ENDODONTIC SEALERS

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ABSTRACT

Aim: To measure and compare the pH values of three endodontic sealers of different bases at 7 different time intervals.

Methods: Thirty polyethylene tubes were divided into three groups: Group A (n=10): polyethylene tubes filled with MTApex, Group B (n=10): polyethylene tubes filled with AH Plus, and Group C (n=10): polyethylene tubes filled with Sealapex. All sealers were mixed according to manufacturer instructions and placed into the tubes. Samples were then immediately placed in securely sealed glass flasks containing 10 mL of distilled water with neutral pH and stored at 37°C. The pH values were measured and recorded using a pH meter immediately after immersion (0 hours) then after 6 hours, 12 hours, 24 hours, 48 hours, 96 hours, and 168 hours, the water was assessed for pH measurement. Data were collected and statistically analyzed using the Kruskal–Wallis H test, followed by the Mann–Whitney U test and Friedman test, followed by the Wilcoxon signed-rank test.

Results: Within each time interval, Sealapex sealer had statistically significantly higher pH values than MTApex and AH Plus sealers which was noticed at all evaluation periods. The pH mean values of AH Plus significantly increased at the second evaluation period (6 hours) followed by a significant decrease at 24 hours ($P < 0.05$).

Conclusions: All tested materials exhibited alkaline pH values at all evaluation time intervals. Also, Sealapex root canal sealer exhibited the highest alkaline pH values compared to MTApex and AH Plus.

KEYWORDS: Sealer, antimicrobial, alkaline pH

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INTRODUCTION

Microorganisms have been always a culprit in the endodontic disease. One of the primary goals of root canal therapy is to eradicate such germs. Endodontic sealers with antimicrobial capabilities play a critical role in the effectiveness of root canal treatment by removing remaining microorganisms from the root canal system. The antimicrobial action of several types of endodontic sealers has been related to the high pH caused by the release of the hydroxyl group. Many groups of endodontic sealers are currently available such as zinc oxide eugenol, calcium hydroxide, resin, silicone, and bioceramic-based sealers. Bioceramic sealers are characterized by multiple advantages such as alkaline pH, antibacterial action, mineralization potential, and dimensional stability [1, 2, 3].

MTApex is a recently introduced calcium silicate-based root canal sealer in a powder/liquid form. Few research studies have looked into the various properties of MTApex [4, 5]. This sealer is one of the few calcium silicate-based sealers available in powder/liquid form. When a calcium silicate-based premixed sealer is used, the hydration process does not begin until the material contacts either the remaining moisture inside the root canal or the humidity of the dentinal tubules [6].

AH Plus is an epoxy resin-based endodontic sealer, available in a paste-paste form. AH Plus is the most extensively researched root canal sealer which makes it a reliable sealer for comparison to other sealers.

Sealapex is a well-studied non-eugenol polymeric calcium hydroxide-based root canal sealer. Because calcium hydroxide-containing sealers have been in use for a long time, a literature review focusing on their different properties is adequate.

Bacterial acid contributes to the environment's acidity by lowering the pH level, which promotes bacterial growth and survival. Therefore, the ability of endodontic sealers to sustain higher pH values determines their antimicrobial efficacy and mineralization potential [7].

Therefore, the present study aimed to measure and compare the pH values of the previously mentioned endodontic sealers at 7 different time intervals.

MATERIALS AND METHODS

As test materials, three different base endodontic sealers (MTAapex, AH Plus, and Sealapex) were used to conduct this study. **Table (1)** shows the manufacturer and composition of each tested sealer. The sample size was calculated using the software G*Power 3.1 software which resulted in the sample size used (n = 10) to ensure a test power of at least 80 %.

Table (1): Manufacturer name and composition of endodontic sealers used in this study

Sealer name	Manufacturer name	Composition
MTApex	Ultradent	Tricalcium silicate, dicalcium silicate, and water-based gel.
AH Plus	Dentsply	Paste A (base): Bisphenol-A epoxy resin, Bisphenol-F epoxy resin, calcium tungstate, zirconium oxide, silica, and iron oxide. Paste B (catalyst): Dibenzydiamine, aminoadamante, trycyclodecane- diamine, calcium tungstate, zirconium oxide, silica, and silicone oil.
Sealapex	Kerr	**Base paste:** Calcium oxide, Bismuth trioxide, Zinc oxide, Sub-micron silica, Zinc stearate,Titanium dioxide, and Tricalcium phosphate **Catalyst paste:** Ethyl toluene sulfonamide, Poly (methylene methyl salicylate) resin, and Isobutyl salicylate

Classification of samples

Polyethylene tubes were cut into 30 tubes measuring 1 cm in length and 1.5 mm in diameter[8] using a Bard-Parker blade and digital caliper. The polyethylene tubes were divided into three groups:

Group A (n=10): polyethylene tubes filled with MTApex.

Group B (n=10): polyethylene tubes filled with AH Plus.

Group C (n=10): polyethylene tubes filled with Sealapex.

Preparation of samples

Every sealer was carefully mixed according to manufacturer instructions and recommendations. In Group A (MTApex): a full spoon of powder was added to 4 drops of the water-based gel for 1 min and mixed till the ideal consistency was reached. In Group B (AH Plus): pastes A and B were mixed for 1 min at a 1:1 ratio. In group C (Sealapex): Base and catalyst pastes were obtained on a mixing pad in a ratio of 1:1 and homogeneously mixed till a uniform consistency was obtained. All sealers were filled into polyethylene tubes using lentulo spiral instruments. Samples were then immediately placed in securely sealed glass flasks containing 10 mL of neutral pH distilled water and stored at 37°C.

pH readings measurement

The pH values were measured and recorded immediately following immersion (0 hours), and then the water was evaluated for pH measurement at intervals of 6 hours, 12 hours, 24 hours, 48 hours, 96 hours, and 168 hours. At each immersion time interval, the tubes were put into new, tightly sealed flasks with 10 mL of distilled water for further analysis. The pH readings were measured using a previously calibrated digital pH meter (Adwa, Szeged, Hungary). In between each recording, the tip of the pH meter was cleaned with double-distilled water (ddH_2O) to eliminate the possibility of fluid contamination during pH measurements and hence to avoid false readings.

STATISTICAL ANALYSIS

All data were subjected to statistical analysis using the Statistical Package for the Social Sciences (IBM.SPSS) software, version 26. Data were tested for normality by using the Shapiro-Wilk normality test which revealed non-normal distribution. Non-parametric Kruskal–Wallis H test, followed by pairwise comparisons by the Mann–Whitney U test, was used to compare pH between different sealers at each time interval. The level of significance was set at $P < 0.05$. Friedman test, followed by pairwise comparisons with the Wilcoxon signed-rank test, was used to compare the pH of each sealer between different time intervals.

RESULTS

The results of the study showed that the 3 tested sealers existed in the alkaline range throughout all of the evaluation periods as plotted in **Figure (1)**. The pH mean values at all time intervals for all sealers are shown in **Table (2)**.

The registered data revealed a pH value variation for the 3 tested sealers and differences in their pH values were noted over time. MTApex (group A) started with an alkaline pH mean value (9.13 ± 1.04), which increased after 96 hours and after 168 hours to (11.15 ± 0.94) and (11.34 ± 1.12) respectively. AH-plus (group B) had the lowest pH value at 0 hours compared to Sealapex and MTApex. However, it started with an alkaline pH (8.11 ± 0.72) and reached its peak after 12 hours (10.28 ± 1.13). This is followed by a gradual declination in pH values in the rest of the evaluation periods with small variations until it reached (8.05 ± 0.57) after 168 hours. Sealapex (group C) started with an alkaline pH mean value (10.36 ± 1.14), which increased gradually over time, to reach (11.75 ± 1.23) after 96 hours and (11.79 ± 1.04) after 168 hours. **Figure (2)**

Within each time interval, Sealapex sealer (group C) had statistically significantly higher pH values than MTApex (group A) and AH Plus (group B) sealers which was noticed at all evaluation periods. The pH mean values of AH Plus significantly increased at the second evaluation period (6 hours) followed by a significant decrease at 24 hours. ($P < 0.05$).

TABLE (2) Mean pH values and standard deviation of the three tested endodontic sealers at different time intervals.

Time / Group		*Group (A)*	*Group (B)*	*Group (C)*
0 hours	Mean	9.13 ^{aA}	8.11 ^{bA}	10.36 ^{cA}
	SD	1.04	0.72	1.14
6 hours	Mean	10.33 ^{aB}	10.15 ^{bB}	10.89 ^{cB}
	SD	1.27	1.09	1.37
12 hours	Mean	10.42 ^{aB}	10.28 ^{aB}	10.93 ^{bB}
	SD	1.24	1.13	1.07
24 hours	Mean	10.77 ^{aC}	8.65 ^{bC}	11.24 ^{cC}
	SD	1.18	0.58	0.95
48 hours	Mean	10.96 ^{aC}	8.54 ^{bD}	11.58 ^{cD}
	SD	1.07	0.14	1.16
96 hours	Mean	11.15 ^{aC}	8.42 ^{bD}	11.75 ^{cE}
	SD	0.94	0.93	1.23
168 hours	Mean	11.34 ^{aD}	8.05 ^{bE}	11.79 ^{cE}
	SD	1.12	0.57	1.04

Group (A): MTApex, Group (B): AH Plus, Group (C): Sealapex

SD: standard deviation

Lower case letters: horizontal analysis

Upper case letters: vertical analysis

Means sharing different letters indicate statistically significant differences (P < 0.05)

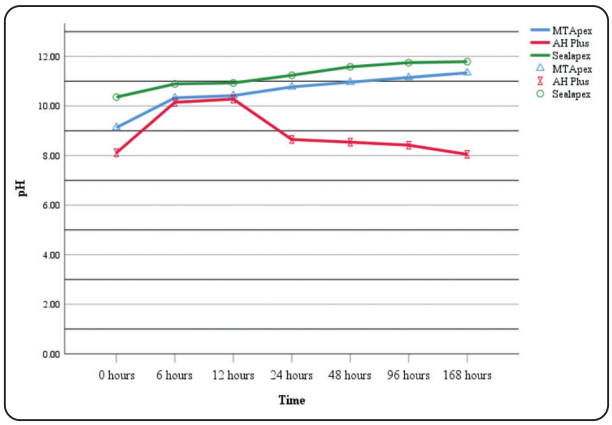

Fig. (1) Line chart depicting all pH mean values of all tested sealers at the alkaline range.

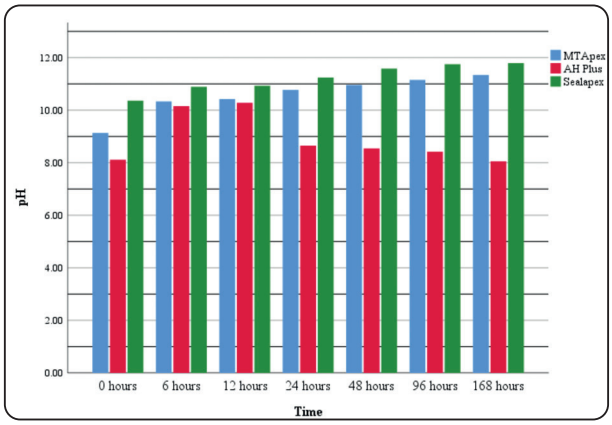

Fig. (2) Clustered bar chart showing pH mean values at different time intervals.

DISCUSSION

The pH values of endodontic sealers may have a positive impact on the formation of mineralized tissue and antimicrobial actions, contributing to the healing process[9]. The alkaline pH of root canal sealers could neutralize the lactic acid from osteoclasts and prevent the dissolution of mineralized components of teeth [10]. In other words, the rising of the pH value to a higher alkaline zone determines the material's antimicrobial properties by blocking microbial enzymes and triggering the re-mineralization processes, which in turn promotes the regeneration of damaged tissues by controlling inflammation. An acidic pH value, on the contrary, will activate the acid hydrolase, stimulating the

demineralization and resorption processes which are stimulated by osteoclastic activity [11].

On the other hand, one of E. faecalis' most distinctive traits is its resistance to alkaline pH, which typically suppresses other microorganisms[12]. It has been shown that E. faecalis can be resistant at a pH of 11.0 but gets eradicated only if the pH is 11.5[13]. Therefore, the pH of a sealer must not only be in the early alkaline range but also be as high as possible to eradicate the persistent microbes that had survived chemo-mechanical preparation.

The methodology used in the present study was similar to that used by *Duarte et al. 2000* [14] and *Duarte et al. 2003* [15]. Polyethylene tubes were used rather than extracted teeth because the placement of the materials inside root canals may result in erroneous results. Apical foramen size in extracted teeth is challenging to standardize, and root dentin may potentially affect the outcomes [16].

AH Plus showed an initial weak alkaline pH value at 0 hours which significantly increased after 6 and 12 hours then declined over time and became the lowest after 168 hours (one week). However, some studies have shown a strong initial antibacterial property of AH Plus to both planktonic and biofilm forms and its ability to possess strong but initial antibacterial effects [17, 18]. Furthermore, *Huang et al. 2019* [17] stated that AH plus had a higher initial antimicrobial activity but no inhibition activity against E. faecalis. This is consistent with the findings of the present study and could be explained by the presence of pH values of AH Plus in the weakly alkaline range which might have an anti-microbial effect but is not powerful enough to have a lethal effect on the persistent E. faecalis. It is also important to note that the pH values of AH Plus may not fully contribute toward its antibacterial property, but AH Plus, being an epoxy-resin-based sealer, can be toxic to bacteria because of its formaldehyde release during setting or Bisphenol-A diglycidyl ether component [19, 20]. According to the present study, the pH values of AH Plus existed in the early pH alkaline range. However, this is contrary to *Zordan et al. 2019* [21] who previously reported AH Plus to have an acidic pH.

Sealapex is a material that contains calcium hydroxide, which becomes biologically active only when calcium and hydroxyl ions are gradually released. This may account for the high pH during the present entire study. As previously mentioned, an increase in pH has been shown to be bactericidal, inhibit osteoclastic activity, and encourage alkalinization in the adjacent tissues [22, 23]. According to *Zhang et al. 2009* [2], Sealapex had good antibacterial action, both immediately and after several days. The effectiveness of Sealapex is partly related to its ability to sustain a high pH in the nearby medium for lengthy periods [14].

MTAapex expressed higher alkaline pH values compared to AH Plus but lower than Sealapex. However, MTApex showed a satisfactory gradual increase in pH values over time until the one-week evaluation time interval. The alkaline pH values could be explained by the calcium silicate-based chemical profile of the sealer which might obtain an alkaline pH due to calcium and hydroxyl ions release. Compared to AH Plus sealer, the findings in the present study are in line with *Janini et al. 2022* [4] who stated that MTApex sealer exhibited higher pH values in comparison to AH Plus for all evaluation periods. The results of this study are also in agreement with *Lee et al. 2017* [24] who found that the pH values of three different bioceramic-based root canal sealers were significantly higher than that of epoxy resin-based sealers including AH Plus.

CONCLUSIONS

It is possible to conclude that all tested materials exhibited an alkaline pH at all evaluation time intervals. Additionally, Sealapex root canal sealer exhibited the highest alkaline pH values compared to MTApex and AH Plus. To sum it up, paying attention to the alkalinity of root canal sealers is of prime importance. Knowing that most bacteria don't

tolerate an environment with highly alkaline pH, the use of highly alkaline sealers may be associated with efficient antibacterial properties and mineralized tissue deposition which could increase the desired success rate of endodontic treatment. Moreover, it is worth mentioning that recently introduced root canal sealers such as MTApex require further research to study and evaluate different properties of the material.

CLINICAL SIGNIFICANCE

Some root canal sealers' ability to inhibit microbial activity may be directly correlated with their pH levels. Three endodontic sealers with different bases (MTApex, AH Plus, and Sealapex) have pH values that exist in the alkaline range up to 168 hours.

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