

RESEARCH AND EDUCATION

## Effects of different peracetic acid formulations on post space radicular dentin



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Endodontically treated teeth may require intracanal posts for restoration, especially when there is extensive loss of tooth substrate.<sup>1,2</sup> In these situations, the endodontic filling is partially removed with manual and/or rotary instruments.<sup>3-6</sup>

During the preparation phase, there is the risk of microbial contamination of the post space, which may lead to future periodontal complications.<sup>7</sup> Additionally, the smear layer that is formed may compromise the adhesive system of the post in the post space.<sup>8,9</sup>

After preparation of the post space, an irrigation protocol that includes both antimicrobial and root dentin cleaning properties is recommended.<sup>7,9</sup> Sodium hypochlorite is typically recommended for endodontic irrigation; however, it does not remove the smear layer

### ABSTRACT

**Statement of problem.** The optimal irrigating solution with antimicrobial and dentin cleansing properties for post space preparation for fiber posts is unclear. Peracetic acid is one option but is available in various chemical formulations that require evaluation.

**Purpose.** The purpose of this in vitro study was to evaluate dentin surface cleanliness based on the presence of a smear layer and the number of open dentin tubules. It also investigates the chemical composition of residues after canal irrigation with a 1% peracetic acid solution (PA) at low or high concentration of hydrogen peroxide during the preparation of intracanal fiber posts.

**Material and methods.** After filling the root canals of 40 mandibular incisors, a rotary instrument was used for intracanal preparation to place fiber posts. The teeth were divided into 4 groups (n=10) according to the post space irrigation protocol as follows: CG (control): distilled water; NA (NaOCl): 2.5% sodium hypochlorite; LH: PA with low concentration of hydrogen peroxide; and HH: PA with high concentrations of hydrogen peroxide. After irrigation, the teeth were sectioned, and the intracanal dentin surface was subjected to analysis using energy dispersive spectroscopy to evaluate chemical composition and to scanning electron microscopy (×500) to evaluate the presence of the smear layer. The number of open dentin tubules was measured by scanning electron microscopy analysis (×2000) using photo-editing software. ANOVA and the Tukey test ( $\alpha=.05$ ) were used to evaluate the data, except for the presence of a smear layer, for which the Kruskal-Wallis and Dunn tests were used ( $\alpha=.05$ ).

**Results.** The highest concentrations of oxygen in the dentin residues were detected in LH and HH ( $P<.05$ ); CG and NA showed similar oxygen concentrations ( $P>.05$ ). NA had a higher concentration of chlorine ( $P<.05$ ), whereas LH had a lower amount of smear layer and a larger number of open dentin tubules than the other groups ( $P<.05$ ). These were equivalent to each other ( $P>.05$ ), except for HH, which also had a larger number of open dentin tubules than CG and NA ( $P<.05$ ).

**Conclusions.** PA 1% with a low concentration of hydrogen peroxide yielded a lower amount of smear layer and a larger number of open dentin tubules in the dentin of the post space when compared with PA 1% with a high concentration of hydrogen peroxide, despite maintaining a similar oxygen concentration in these dentin residues. (J Prosthet Dent 2018;120:92-8)

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## Clinical Implications

The post space should be irrigated with solutions that have antimicrobial activity and also reduce the smear layer. Peracetic acid addresses these needs, although the effects of different formulations on the root dentin have yet to be determined.

completely and interferes with the bonding of fiber posts.<sup>10-13</sup>

Other solutions including chlorhexidine digluconate, phosphoric acid, and ethylenediaminetetraacetic acid (EDTA) have been proposed to achieve the same goals.<sup>14-22</sup> However, these solutions also have adverse effects and/or limited antimicrobial activity, which makes them less than ideal for irrigating the post space before cementation.<sup>23</sup>

Peracetic acid has been proposed as an alternative solution for irrigating root canals, given that it has antimicrobial activity, satisfactory surface cleaning capability, and dentin penetrability.<sup>24-30</sup> The recommended solution for endodontic use is composed of hydrogen peroxide and acetic acid.<sup>29</sup> However, hydrogen peroxide can interfere with the dentin cleaning of endodontic irrigation protocols.<sup>31</sup>

Commercial peracetic acid solutions are available with both high and low concentrations of hydrogen peroxide.<sup>32</sup> However, the effects of these solutions on the post space dentin before intracanal post cementation are unknown.

The purpose of this study was to use scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) analysis to evaluate the cleaning capacity of 1% peracetic acid solution with low or high concentrations of hydrogen peroxide to chemically analyze the residues, to examine the presence of a smear layer, and to count the number of dentin tubules in the space prepared for intraroot post placement. The null hypothesis was that the final irrigation of the canal space with solutions with different concentrations would not present statistically significant differences.

## MATERIAL AND METHODS

This study was approved by the research ethics committee of the Araraquara Dental School (registration number 44018715.9.0000.5416). Forty maxillary central incisors were obtained from patients who needed extractions for periodontal disease and were stored in 1% thymol solution at 4°C. All teeth were radiographed in the buccolingual and mesiodistal directions to verify the presence of a single root canal with similar endodontic anatomy. Those that did not meet these requirements were discarded and replaced.

**Table 1.** Mean  $\pm$  standard deviation of frequency distribution (%) of oxygen and chlorine present in smear layer on dentin surface of intracanal space prepared for fiber post

Element	CG	NA	LH	HH
Oxygen	96.35 $\pm$ 1.33 <sup>b</sup>	96.16 $\pm$ 1.44 <sup>b</sup>	98.23 $\pm$ 1.19 <sup>a</sup>	98.64 $\pm$ 1.45 <sup>a</sup>
Chlorine	2.33 $\pm$ 1.36 <sup>b</sup>	3.98 $\pm$ 1.22 <sup>a</sup>	1.75 $\pm$ 1.18 <sup>b</sup>	1.16 $\pm$ 1.11 <sup>b</sup>

CG, distilled water (control); HH, peracetic acid solution with high concentration of hydrogen peroxide; LH, peracetic acid solution with low concentration of hydrogen peroxide; NA, 2.5% sodium hypochlorite; SD, standard deviation. Different superscript letters in same row signify statistical difference ( $P < .05$ ).

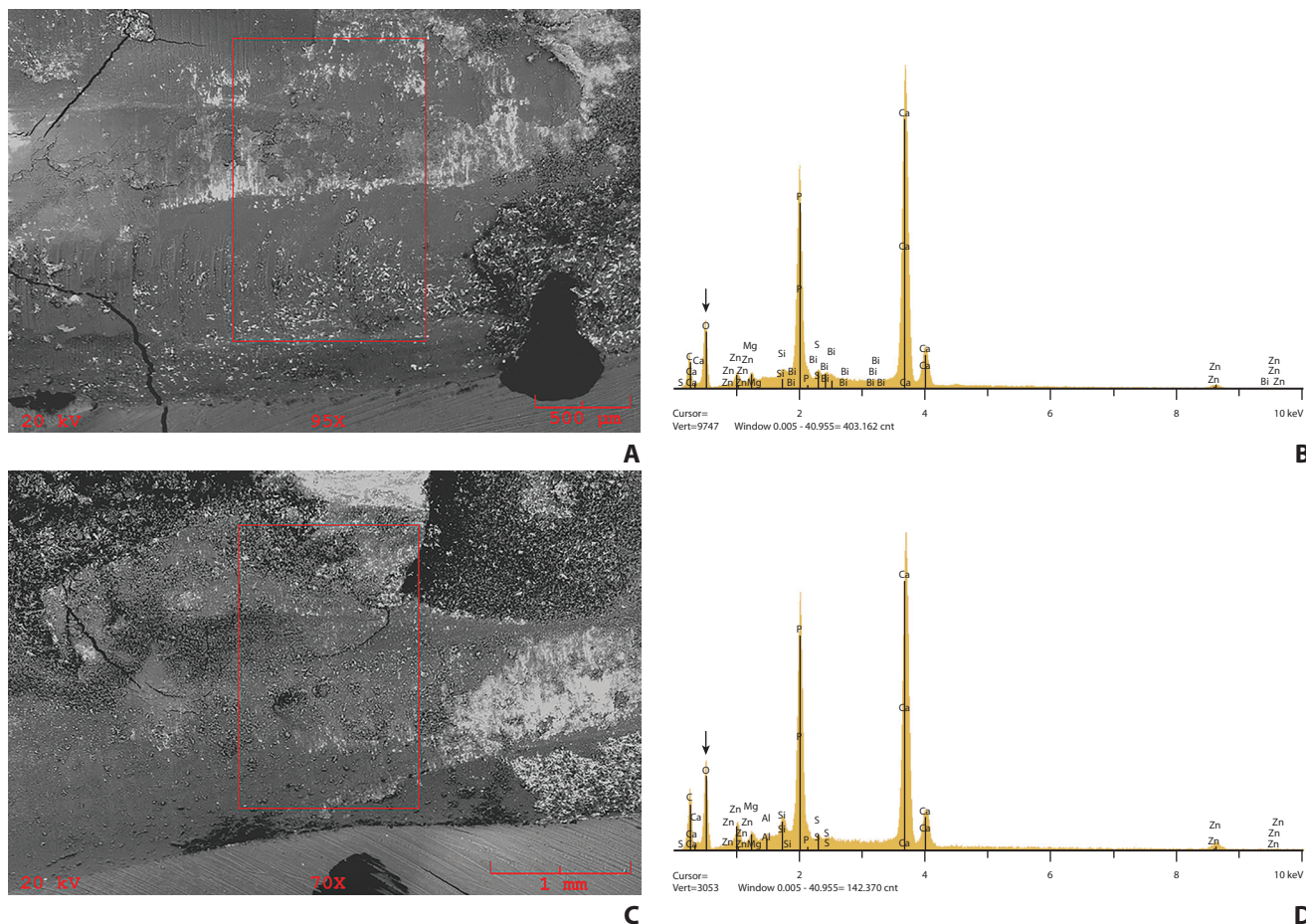
The teeth were sectioned by using a double-sided diamond disk (KG Sorensen) at 17 mm from the root apex. The root canals were prepared with a series of shaping and finishing files (ProTaper; Dentsply Sirona) up to the F2 size to achieve a standardized working length of 16 mm, according to the manufacturer's recommendations.

At each change of the instruments, the root canal was irrigated with 5 mL of 2.5% sodium hypochlorite solution (Asfer). At the end of the chemical-mechanical preparation, the root canal was irrigated with 5 mL of 17% EDTA (Biodinâmica) and maintained inside the canal for 3 minutes. The final irrigation was carried out with 10 mL of 2.5% sodium hypochlorite solution. The root canal was aspirated and dried with absorbent paper points, size F2 (ProTaper; Dentsply Sirona).

The root canals were obturated with size F2 gutta percha points (ProTaper; Dentsply Sirona) and epoxy resin-based cement (AH Plus; Dentsply Sirona) by using a single-cone technique (Touch'n Heat; Sybron Endo) sectioned with a warmed spatula 1 mm short of the cervical root surface. The cervical opening of the root canal was restored with glass ionomer cement (Vitreon; SS White), and the roots were immediately immersed in distilled water and stored at 37°C for 7 days.

After the immersion period, the restoration with glass ionomer cement was removed with a diamond rotary instrument (N. 1012; KG Sorensen). The post space was prepared and the gutta percha removed with a rotary instrument (Post Preparation; Hets) with 1.05 mm (apical) and 1.8 mm (cervical) dimensions at low speed (8000 rpm) without cooling, with a cervical extension of 12 mm toward the root apex. One longitudinal groove was made externally in the buccal and lingual root surfaces.<sup>33-37</sup>

The roots were divided into 4 groups (n=10) according to the irrigation protocol of the prepared post space as follows: CG (control) distilled water; NA (NaOCl), 2.5% sodium hypochlorite solution (Asfer); LH (LCPA), 1% peracetic acid solution (Sigma; Aldrich) with a low concentration of hydrogen peroxide (40% peracetic acid and 8% hydrogen peroxide); and HH (HCPA), 1% peracetic acid solution (Peresal; Profilática) with a high concentration of hydrogen peroxide (4% peracetic acid and 26% hydrogen peroxide).



**Figure 1.** A, C, Representative scanning electron microscopy images of site of dentin residues. B, D, Energy dispersive spectroscopy analysis showing presence of oxygen. Black arrow indicates presence of oxygen. A, B, Control distilled water; 2.5% sodium hypochlorite. C, D 40% peracetic acid and 8% hydrogen peroxide; 4% peracetic acid and 26% hydrogen peroxide. A, Original magnification,  $\times 95$ . C, Original magnification,  $\times 70$ .

The post space was irrigated with 5 mL of the solution for 1 minute and kept for 3 minutes without stirring. After irrigation, the post space was aspirated with a 0.48-mm-diameter suction tube (Capillary Tips; Ultradent Products, Inc) and dried with size F2 absorbent paper points (Pro-Taper; Dentsply Sirona). The roots were then split longitudinally in a buccolingual direction with a chisel.

The distal surface of each root was selected for microscopic analysis. The specimens were mounted on metal stubs, oven-dried at 37°C for 5 days, and stored for 2 days in a closed chamber containing silica gel. The smear layer was chemically analyzed using chemical SEM-EDS microanalysis (EVO 50; Carl Zeiss) at  $\times 75$  and  $\times 95$  magnification within an area measuring 250  $\mu\text{m}^2$  of the middle third of the post space. The relationship between the presence of oxygen and chlorine was measured as a percentage of the smear layer surface through EDS and submitted to ANOVA and the Tukey test ( $\alpha=.05$ ).

The specimens were sputter-coated with metal and assessed under SEM (EVO 50; Carl Zeiss) in the same area in which the images were obtained for EDS. Four different fields were analyzed, and a representative image

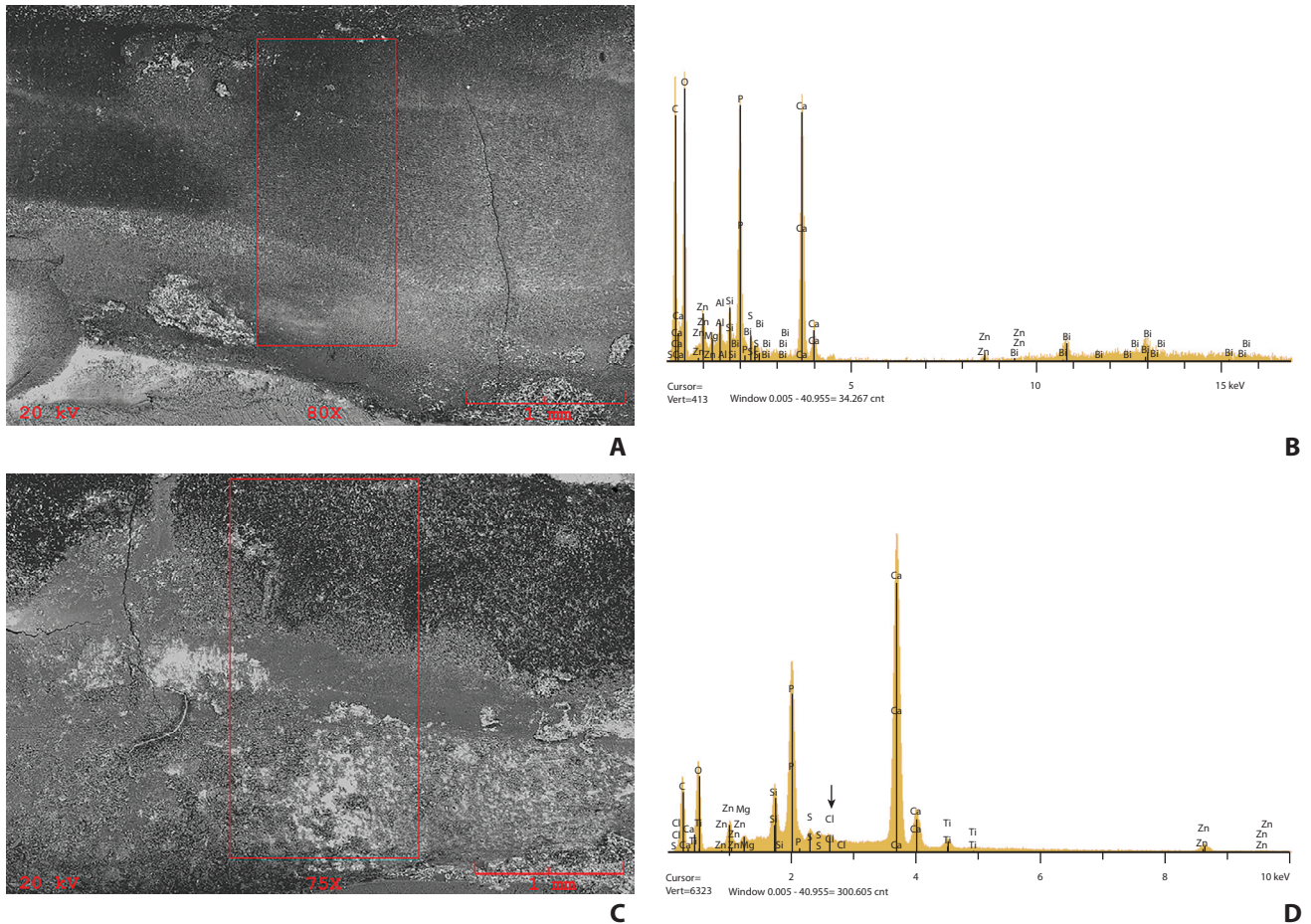
for each specimen was obtained at  $\times 500$  and  $\times 2000$  magnification.

The images obtained at  $\times 500$  magnification were used to evaluate the presence of residues (smear layer) on the dentin surface. Scores ranging from 0 to 4 were assigned according to the degree of residue observed by following the parameters described by Aranda-Garcia et al.<sup>38</sup> The data were submitted to the Kruskal-Wallis test followed by the Dunn test for multiple comparisons ( $\alpha=.05$ ).

The images obtained at  $\times 2000$  magnification were used to quantify the presence of open dentin tubules through visual counting using photo-editing software (Adobe Photoshop CS6; Adobe Systems), according to the parameters described by Arslan et al.<sup>39</sup> The data were submitted to ANOVA and the Tukey test ( $\alpha=.05$ ).

## RESULTS

The presence of calcium and phosphorus was identified in all specimens. LH and HH showed higher concentrations of oxygen in the smear layer than did the other groups ( $P<.05$ ), but similar concentrations to each other



**Figure 2.** A, C, Representative scanning electron microscopy images of site of dentin residues. B, D, Energy dispersive spectroscopy analysis showing presence of chlorine. Black arrow indicates presence of chlorine. A, B, Control distilled water; 2.5% sodium hypochlorite). C, D 40% peracetic acid and 8% hydrogen peroxide; 4% peracetic acid and 26% hydrogen peroxide. A, Original magnification,  $\times 80$ . C, Original magnification,  $\times 75$ .

**Table 2.** Median, maximum, and minimum scores, first and third quartiles of scores attributed to presence of smear layer on dentin surface of intracanal space prepared for fiber post

Scores	CG	NA	LH	HH
Median	4 <sup>b</sup>	4 <sup>b</sup>	2 <sup>a</sup>	3.5 <sup>b</sup>
Minimum	4	2	2	3
Maximum	4	4	3	4
Q1	4	4	2	3
Q3	4	4	2	4

CG, distilled water (control); HH, peracetic acid solution with high concentration of hydrogen peroxide; LH, peracetic acid solution with low concentration of hydrogen peroxide; NA, 2.5% sodium hypochlorite; Q1, first quartile; Q3, third quartile. Different superscript letters in same row signify statistical difference ( $P < .05$ ).

( $P > .05$ ). No difference was found between CG and NA ( $P > .05$ ). NA showed a higher concentration of chlorine in the smear layer than did the other groups ( $P < .05$ ), which were similar to each other ( $P > .05$ ). Table 1 shows the mean and standard deviation of the oxygen and chlorine percentage in the smear layer.

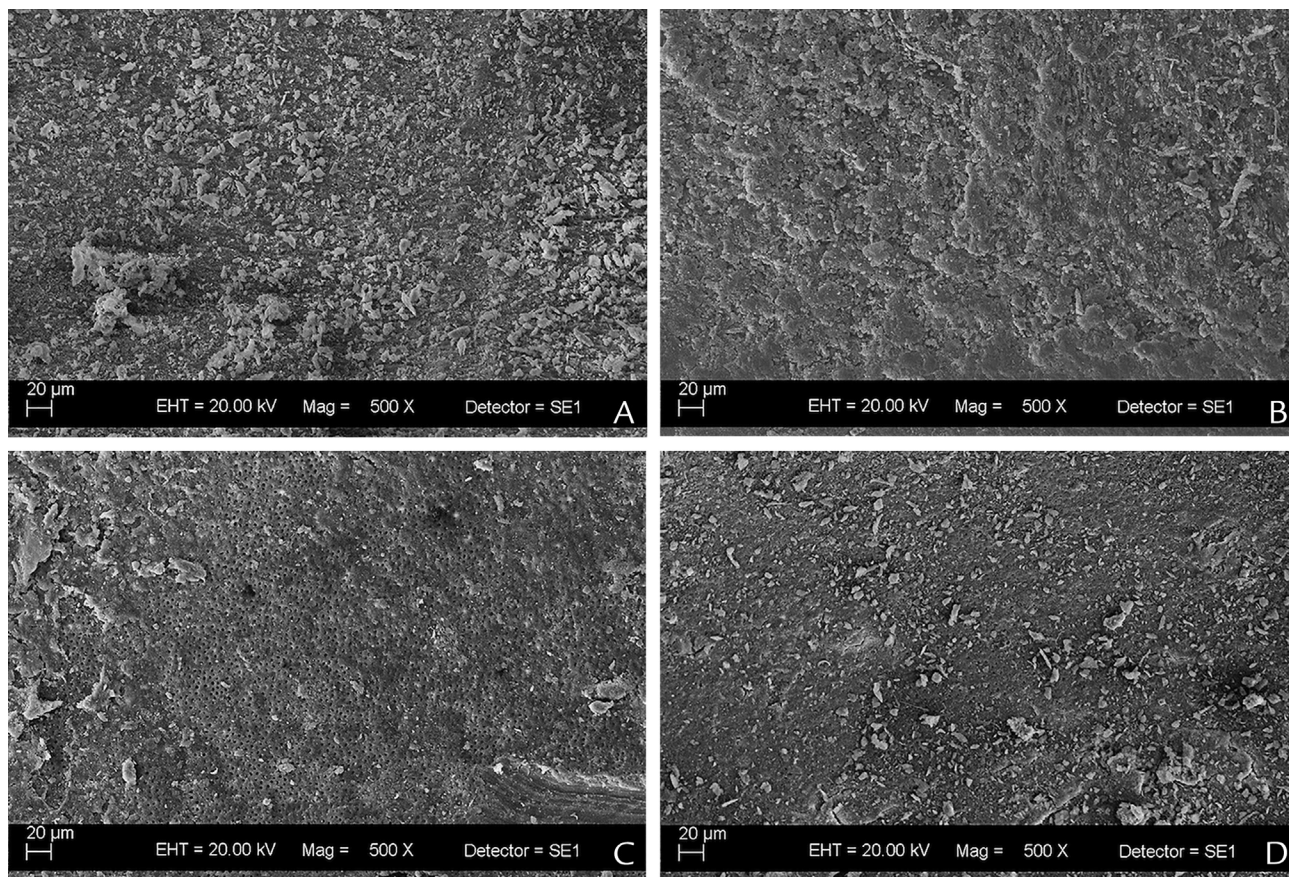
Figure 1 illustrates the site of the residues and the profile of the presence of oxygen. Figure 2 illustrates the site of the residues and the profile of the presence of chlorine.

LH showed a lower incidence of smear layer on the dentin surface of the post space than did the other groups ( $P < .05$ ), which did not differ from each other ( $P > .05$ ). Table 2 shows the median, maximum, and minimum scores and the first and third quartiles of the experimental groups. Figure 3 shows the characteristic of the smear layer present in each of the experimental groups.

LH had a greater number of open dentin tubules than the other groups ( $P < .05$ ). HH had a greater number of open dentin tubules than CG and NA ( $P < .05$ ), which, showed a similar number of open dentin tubules ( $P > .05$ ). Table 3 shows the arithmetical mean, standard deviation, minimum, and maximum number of open dentin tubules for each of the experimental groups. Figure 4 shows the characteristic of the open dentin tubules in the experimental groups.

**DISCUSSION**

The preparation of the post space for intracanal posts may leave residues on the post space surface because of the mechanical action of rotary instruments. However,



**Figure 3.** Representative image of smear layer present. A, Control distilled water. B, 2.5% sodium hypochlorite. C, 40% peracetic acid and 8% hydrogen peroxide. D, 4% peracetic acid and 26% hydrogen peroxide. (Original magnification,  $\times 500$ .)

**Table 3.** Mean  $\pm$  standard deviation, and maximum and minimum scores of number of dentin tubules

No Tubules	CG	NA	LH	HH
Mean $\pm$ SD	21.45 $\pm$ 10.98 <sup>c</sup>	39.81 $\pm$ 13.91 <sup>c</sup>	130.81 $\pm$ 69.10 <sup>a</sup>	84.45 $\pm$ 29.86 <sup>b</sup>
Vmax–Vmin	42–9	58–16	310–60	130–51

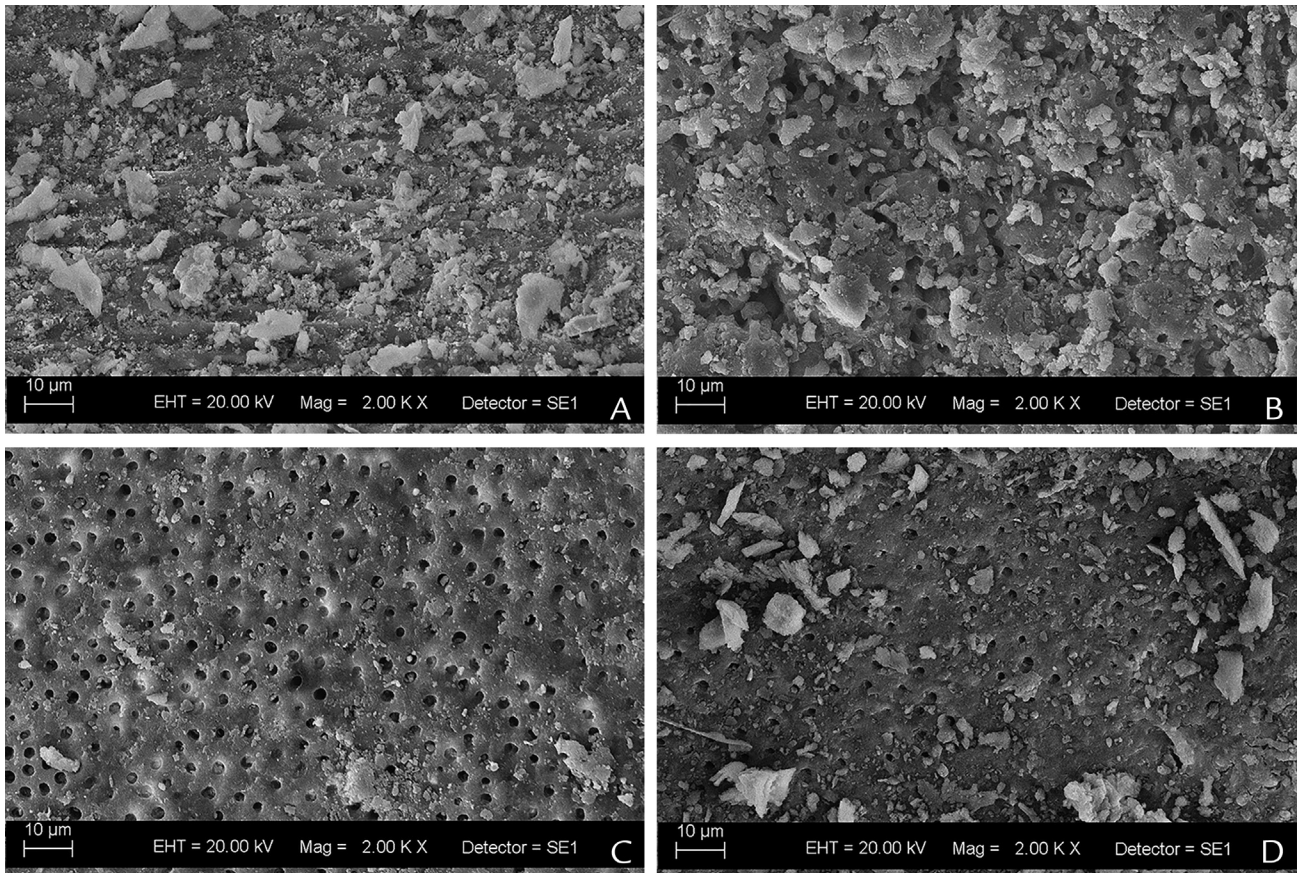
CG, distilled water (control); HH, peracetic acid solution with high concentrations of hydrogen peroxide; LH, peracetic acid solution with low concentration of hydrogen peroxide; NA, 2.5% sodium hypochlorite; SD, standard deviation; Vmax, maximum score; Vmin, minimum score. Different superscript letters in same row signify statistical difference ( $P < .05$ ).

these residues are different from the smear layer caused by chemical-mechanical preparation, which also contains endodontic cement debris.<sup>15</sup> As a result, final irrigation solutions routinely used in endodontic treatment are not recommended for post space irrigation, given that they do not possess satisfactory dentin cleaning and antimicrobial properties.<sup>27,31,38</sup> Peracetic acid was used in this study because it has both properties.<sup>16,25,26</sup> However, it had different effects on the root dentin of the space prepared for the placement of intracanal posts, depending on the chemical formulations with different concentrations of hydrogen peroxide.

Although oxygen was present in all specimens, the concentration was significantly higher in groups in which

the peracetic acid solution was used. The solution comprised hydrogen peroxide, acetic acid, and acetyl hydroperoxide, which release hydroxyl and free radicals during the chemical decomposition process, resulting in oxygen and acetic acid.<sup>17,25,29</sup> The highest chlorine concentration was observed in NA, because the solution yields hypochlorous acid and sodium hydroxide.<sup>13,26</sup> Moreover, the peracetic acid solution has no chlorine in its chemical composition. The possible presence of this ion in the smear layer of some specimens finally irrigated with peracetic acid solution may be a by-product of the sodium hypochlorite used during the chemical-mechanical preparation of the root canals.<sup>10,13,18</sup>

The ability of peracetic acid to clean the dentin surface is satisfactory, regardless of its concentration in the solution, and is similar to that of EDTA.<sup>19,25</sup> In this study, the solution with a low concentration of hydrogen peroxide was more effective at removing the smear layer, suggesting that the presence of residues is directly related to the concentration of hydrogen peroxide present in the solution. This finding agrees with that of Scelza et al,<sup>31</sup> who reported that endodontic irrigation associated with hydrogen peroxide was less effective in removing the smear layer of root dentin.



**Figure 4.** Representative image of presence of open dentin tubules. A, Control distilled water. B, 2.5% sodium hypochlorite. C, 40% peracetic acid and 8% hydrogen peroxide. D, 4% peracetic acid and 26% hydrogen peroxide. (Original magnification,  $\times 2000$ .)

Peracetic acid cleared more of the dentin tubules than did other irrigating solutions because of its demineralizing action, irrespective of the hydrogen peroxide solution concentration.<sup>24</sup> Because hydrogen peroxide concentration is directly associated with the presence of residues on the dentin wall, the solution with the lowest concentration (Sigma) also yielded the highest number of open tubules.

As the bonding of adhesive resin-based cements used for fiber posts involves interaction with the collagen fibers of the root dentin, reducing the residue between them to a minimum would be desirable.<sup>14,20-22</sup> Also, as the smear layer produced by rotary instruments is distributed both on the intertubular dentin surface and over the openings of the dentin tubules, peracetic acid solution (1%) with a low concentration of hydrogen peroxide was the most effective final irrigation process<sup>30</sup>; furthermore, this solution has satisfactory antimicrobial activity.<sup>16,27,28</sup>

Although 1% peracetic acid solution was the most effective final irrigation method, it also showed high residual concentrations of oxygen, which would interfere with the adhesion of any resin cement by inhibiting complete polymerization.<sup>29</sup> The findings reported here reinforce those of Pereira et al,<sup>33-36</sup> who demonstrated

that the highest bond strength is provided by glass ionomer cements. This may make conventional glass ionomer cement the most suitable for intracanal post cementation, even when peracetic acid is used to clean the post space. In the present study, the post space was prepared with rotary instruments without water cooling. The use of copious irrigation might prevent the development or accumulation of debris, though this has yet to be demonstrated.<sup>37</sup>

The presence of residues containing calcium and phosphorus on the root dentin was observed in all specimens. These chemicals are also routinely detected in the smear layer after the chemical-mechanical preparation of post spaces.<sup>10</sup> This fact supports the use of glass ionomer, which bonds to the tooth substrate by means of chemical bonds from its carboxylic radicals to the calcium ions existing either in the enamel, dentin, or cement structures.<sup>33</sup>

Even though 1% peracetic acid solution at low hydrogen peroxide concentration demonstrated adequate cleaning of the preparation for fiber post placement, further studies are needed to evaluate the impact of this solution, as well as the effects of hydrogen peroxide on the bond strength of resin-based adhesive systems.

## CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. Final irrigation of the canal space with 1% peracetic acid produced a higher concentration of oxygen in the smear layer than did the remaining solutions tested ( $P < .05$ ).
2. The solution with a low hydrogen peroxide concentration yielded less residue on the root dentin and a higher incidence of open dentin tubules when compared with other solutions ( $P < .05$ ).

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