

**Effect of application of chelating agents and organic acids on push out bond strength of fibre post to root dentin:an in vitro study.**

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**Abstract**

**Background:** Fibre posts are most commonly used as it induces less stress on to dentin compared to other posts. Elastic modulus is almost similar to that of dentin and is effective corrosion. Another challenge in adequate adhesion of fibre post is presence of smear layer which is produced during instrumentation or post space preparation. Chelating agents were introduced in dentistry to help in preparation of narrow and calcified root canals. liquid formulations are used for removal of smear layer

**Materials And Methods**

75 mandibular premolars were decorated to standardise the working length. working length was determined with the help of 15K file followed by which cleaning and shaping was done using proper gold Niti files following normal irrigation protocol. Canals were obturated by cold lateral compaction technique wit AH plus sealer. Stored for 1 week at 37<sup>0</sup> C after which post space preparation was done using #3pees reamer. After post space preparations the canals were treated with different chelating agents G1- 17%DTPA, G2- 10% Citric acid and G3-17%EDTA.

After using the irrigants fibre post was luted using self etch self adhesive resin cement and push out test was performed. Failure modes were analysed using stereomicroscope.

**Result:** Inter group comparison using Tukey post hoc test revealed that push out bond strength was statistically significant when DTPA ( $1.10 \pm 0.58$ ) was compared with EDTA( $0.35 \pm 0.10, p=0.000$ ) and citric acid group( $0.35 \pm 0.85, p=0.000$ )

**Conclusion:** DTPA can be considered as an alternative to commercially available chelating agents as the smear layer removing capability is significantly better than other agents.

**Keywords:** DTPA, EDTA, NAOH, NVA, Liquid.

### Introduction

Micro organisms play a key role in development of pulpal and periapical diseases. Its always a challenging task to eliminate micro organism from the root canal owing to its complex anatomy<sup>1</sup>. Various techniques and measures have been taken to eliminate micro organisms from the root canal including use of various instrumentation technique, irrigation regimens and intracanal medicaments<sup>2</sup>. Literature suggests that mechanical instrumentation alone cannot eliminate the bacteria in root canal system, hence it is recommended to use irrigants and intracanal medicaments to eliminate micro organisms<sup>3</sup>. Non surgical endodontic therapy has been described as two fold treatment procedure involving both biological and mechanical objective<sup>4</sup>. Biological objective is to eliminate micro organism from the root canal system and mechanical objective is to have a proper coronal and apical seal<sup>4</sup>.

Rehabilitation of Endodontically treated teeth depends on the amount of tooth structure present<sup>5</sup>. If the remaining coronal tooth structure is less than 50% or less, rigid or non rigid post can be used for retention of the crown and

facilitate function of the tooth<sup>6</sup>. Fibre posts are most commonly used as it induces less stress on to dentin compared to other posts. Elastic modulus is almost similar to that of dentin and is effective corrosion<sup>7</sup>. However placement of fiber post demands clinical skills and is technique sensitive process<sup>8</sup>.

Self etch self adhesive resin cements are most commonly used for luting fibre post as it has strengthening effect on tooth and bonds well to root dentin<sup>9</sup>. Resin based cements improves adhesion between matrix of post and collagen fibres of dentin along with formation of smear layer<sup>23</sup>. The adhesiveness of fibre post also depends on presence of moisture in root canal and polymerisation shrinkage of the cement<sup>10</sup>.

Another challenge in adequate adhesion of fibre post is presence of smear layer which is produced during instrumentation or post space preparation<sup>11</sup>. Smear layer mostly composed of inorganic dentin and some amount of organic matter like odontogenic process, micro organism and necrotic pulp material<sup>11</sup>. This smear layer has to be removed to improve adhesion of fibre post to root dentin. Various irrigant activation system have been introduced to remove smear layer<sup>12</sup>. Most commonly used irrigants are sodium hypochlorite and 17% EDTA<sup>12</sup>.

However after post space preparation sodium hypochlorite is not used due to its increased oxidising property which leaves behind a oxygen rich layer on the dentin surface. This property of sodium hypochlorite is responsible for decreasing the bond strength of adhesive cement<sup>13</sup>.

Chelating agents were introduced in dentistry to help in preparation of narrow and calcified root canals<sup>15</sup>. A liquid preparation of chelating agents chemically softened the root canal dentin and dissolved the smear layer thereby enhancing the dentin permeability<sup>14</sup>. EDTA is most commonly used chelating agent as it is non toxic and less irritant in weak solution. Effect of EDTA on dentin

depends on concentration of irrigant used and duration of irrigation<sup>14</sup>.

Pentetic acid or Diethylene triamine penta acidic acid (DTPA) is another chelating agent whose molecule can be viewed as an expanded version of EDTA and has similar uses<sup>16</sup>. There is a scarce literature available on the effect of various chelating agents on the push out bond strength of the post, hence the main objective of the present study was to evaluate the push out bond strength of fibre post to root dentin using various chelating agents for removal of smear layer.

### Materials and Method

**Tooth selection and preparation:** Seventy five freshly extracted human mandibular single rooted premolar teeth with type I classification that was confirmed by taking radiograph were collected from patients undergoing therapeutic extraction. The collected samples were caries free and devoid of any anomalies. Samples were washed in running water and soft tissue remnants, debris or calculus were removed. They were placed in hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) solution for 1 hour after which it was transferred to normal saline until use. The teeth were decoronated using diamond disc below Cemento enamel junction to standardize working length to 14mm. In order to simulate the periodontium, root sections were dipped in wax to depth of 1mm below the coronal opening. It was then mounted on self cure acrylic resin and allowed to set. Once set root was removed and the space was filled with elastomeric impression material and root was re inserted into the acrylic cylinder. 15K file was used to negotiate the teeth upto working length, Biomechanical preparation was done using NiTi rotary files upto F3(Protaper gold, Denstply) following normal irrigation protocols 3% NaOCl & 17% EDTA(Prime dental pvt It) and final irrigation of the samples were done with 5ml-distilled water. After final rinse canals were dried using absorbent paper points.

Canals were coated with resin sealer (AH plus dentsply) and obturated by cold lateral compaction technique. Roots were stored at 37<sup>0</sup>C until the sealer sets<sup>17</sup>.

**Post space preparation:** Once the sealer was set post space preparation was done with #3 peeso reamer mounted to slow speed handpiece leaving behind apical 3mm of GP. After post space was prepared fit of size 3 fibre post (Reforpost-Angelus dental product) was checked by radiograph<sup>17</sup>.

**Experimental groups:** After post space preparation samples were divided into 3 groups (n = 25) based on dentin treatment.

**Group 1: 17% DTPA**

**Group 2: 10% Citric acid (Citra wash-Prime dental pet ltd)**

**Group 3: 17% EDTA (Prime dental pvt It)** 17% DTPA solution was prepared by dissolving 17 gram DTPA powder(AVA Chemicals ) in 150ml distilled water and NaOH was added to adjust the pH 8.0. Mixture was swirled with magnetic stirrer until powder dissolved. Then 20ml phosphate buffered saline was added and solution was filtered. All the groups were irrigated with 10ml of the respective solution for 40 seconds and final rinse was done with 5 ml of normal saline<sup>18</sup>. After irrigation, the post space was dried with paper points and fibre post was luted with self etch self adhesive resin cement( 3M relyx X)

**Preparation of root dentin slices and push out test:**

Acrylic slices with the roots were prepared using slow speed diamond disc and was sectioned perpendicular to fibre post. Thin slices of 2mm thickness were obtained from coronal, middle and apical region of the fibre post. Push out bond strength was evaluated with the help of instron machine( INSTRON <sup>1</sup>3382). Fibre posts were pushed with the help of plunger of 1.5mm diameter. It was made sure that the tip of the plunger contacted only the

fibre post and avoided contact with adjacent dentin. Cross head speed of 1mm/min was applied on the post in apico coronal direction with the help of plunger until bond failure occurred<sup>21</sup>. Bond strength in Mpa was calculated using formula:

**Bond strength = bond failure in N / area of bonded interface**

(area of bonded interface =  $2\pi rh/4$  where  $p = 3.14$ ;  $r$  - radius of post and  $h$ - thickness of slice in mm)

After push out bond strength the acrylic slices were visualised under stereomicroscope and modes of failure were studied for the individual group.

### Statistical analysis

For statistical analysis of data, multiple comparisons were performed using one-way analysis of variance (ANOVA) followed by Tukey post hoc analysis. Statistical significance was accepted at a level of  $P < 0.05$ . Data were analyzed using SPSS (version 22.0).

### Results

The push out bond strength of all the three experimental group are presented in Table 1. One way ANOVA results showed that there was significant difference in bond strength between groups ( $F=35.45, p=0.000$ ). Inter group comparison using Tukey post hoc test revealed that push out bond strength was statistically significant when DTPA ( $1.10 \pm 0.58$ ) was compared with EDTA ( $0.35 \pm 0.10, p=0.000$ ) and citric acid group ( $0.35 \pm 0.85, p=0.000$ ). There was statistically significant difference between EDTA and Citric acid groups ( $p=0.769$ ). Graph 1 shows that bond strength value of DTPA group was more.

The stereomicroscope images showed that DTPA group showed cohesive mode failure whereas EDTA and Citric acid groups showed more of adhesive failure (Fig 3).

Figure 1: Specimen Preparation

- A: Preparation of acrylic resin block
- B: biomechanical preparation using rotary files
- C: drying the canal with paper point
- D: obturation
- E: cementation of fibre post

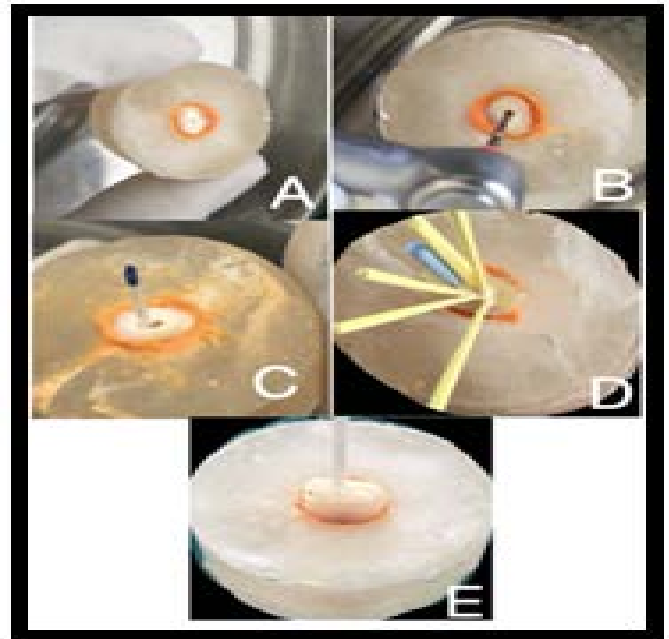


Figure 2: Push out test Acrylic slices placed on Instron machine for push out strength

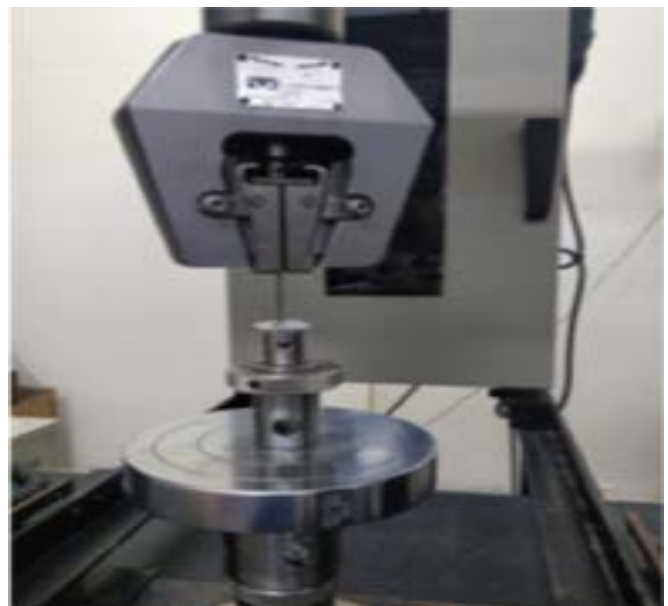
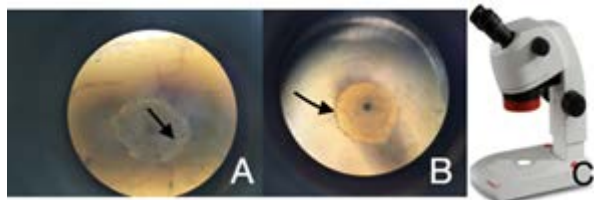


Fig 3: Failure Modes

A-Cohesive failure in DTPA samples

B-Adhesive failure in EDTA and Citric act sample

C- Stereomicroscope -40x magnification



Graph 1: Push out bond strength of fibre post among three experimental groups.

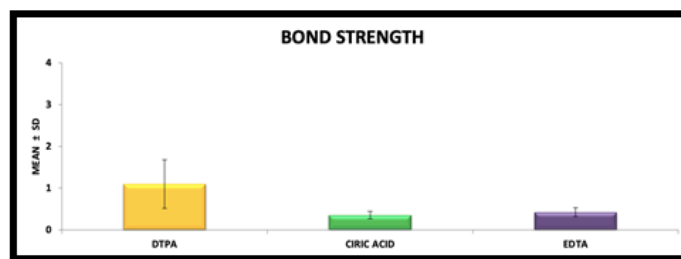


Table 1: Statistical analysis, mean and SD of push out bond strength of fibre post in all experimental group and intergroup comparison using turkeys test.

Group	Mean	Std. Deviation	F	Sig	Group	Mean Difference	Std. Error	Sig	95% Confidence Interval For Mean	
(I)	(I) Group	(I) Group			(J)	(I-J)			Lower Bound	Upper Bound
Dtpa	1.103816	.5837876	35.455	.000	Citric Acid	.7444336	.0977703	.000	.510457	.978410
					EDTA	.6767392	.0977703	.000	.442763	.910716
Citric Acid	.359383	.0858729			DTPA	-.7444336	.0977703	.000	-.978410	-.510457
					EDTA	-.0676944	.0977703	.769	-.301671	.166282
Edta	.359383	.1013992			DTPA	-.6767392	.0977703	.000	-.910716	-.442763
					Citric Acid	.0676944	.0977703	.769	-.166282	.301671

The mean difference is significant at the 0.05 level.

### Discussion

Glass fibre post is one of the most popular choice of post for restoring badly broken down tooth. In the present study, Angelus glass fibre post was used as it has serrated parallel sided shape which favours mechanical interlocking with the root canal walls<sup>19</sup>. Its conical tip allows less tooth preparation at the apical third which saves dentin in this area<sup>20</sup>. Pretreatment of fibre post with 37% phosphoric acid and silane coupling agent opens the resin matrix to expose the glass fibres which enhances bonding. The undercuts present on the post improves retention with the resin cement<sup>22</sup>.

For cementation of fibre post, resin cements is commonly preferred which are tooth coloured or translucent cement composed of diacrylate resin which requires a bonding agent for improving adhesion to the tooth structure<sup>23</sup>. Adhesive resin cements have adhesive monomers which

improves bonding without the need for application of adhesive monomer but requires application of primer<sup>10</sup>. In the present study self etch self adhesive resin cement is preferred as it is composed of adhesive component which eliminates the need for using etchants and primer for bonding to tooth<sup>9</sup>,

Smear layer is formed during post space preparation which is mainly composed of gutta percha remnants and mineralised collagen matrix<sup>24</sup>. Thickness of smear layer may vary and the moisture content interferes with polymerisation of resin cement<sup>25</sup>. This smear layer has to be removed to improve adhesion of fibre post to root dentin. Various organic acids and chelators are used as irrigant for removal of smear Layer<sup>26</sup>.

Chelates are stable complexed metal ions along with organic substance to form ring shaped bonds<sup>27</sup>. It binds with inorganic component of dentin and causes demineralisation of dentin surface<sup>14</sup>. EDTA in pure form



will have low surface tension compared to sodium hypochlorite and saline solution<sup>28</sup>. It has 2 amine and 4 carboxyl group which binds with calcium of root canal dentin and forms chelates<sup>29</sup>. It has self limiting action as the solution can dissolve only certain amount of dentin, an equilibrium will be reached once all the chelating ions have reacted<sup>30</sup>. EDTA in its pure form already has a lower surface tension than 1 or 5% sodium hypochlorite (NaOCl), saline solution or distilled water<sup>31</sup>. EDTA removes non collagen and hydroxyapatite in a selective manner which prevents major changes in collagen structure<sup>32</sup>. It retains the interfibrillar minerals which improves resistance to dehydration and significantly improving infiltration of resin cement<sup>33</sup>.

Citric acid is an organic acid with chelating property when it binds with metals to form non ionic chelates<sup>34</sup>. However it is a weak acid and leaves behind precipitate in the canal which hinders bonding of post to root dentin<sup>35</sup>.

In August 2004, the US Food and Drug Administration (USFDA) determined zinc-DTPA and calcium-DTPA to be safe and effective for treatment of those who have breathed in or otherwise been contaminated internally by plutonium, Americium or curium<sup>16</sup>. In dentistry, the cleaning effect of various chelating preparations was compared by Pawlicka et al(1981) and found that by adding detergent (EDTAC and DTPAC), the efficiency of EDTA and DTPA could be improved. DTPA has 3 amine and 5 carboxyl groups which binds with calcium by forming 8 bonds. The formation constant is 100 times better than EDTA which results in improved chelation and better smear layer removal<sup>36</sup>.

Push out test was done to check the bond strength of fibre post as it is easy to align the samples for testing. Thin slices can be placed and after load is applied even the minute variations can be recorded<sup>17</sup>. Stereomicroscope

was used to visualise the mode of failure as it provides three dimensional perspective of root surface<sup>37</sup>.

In this study bond strength of DTPA was significantly better compared to EDTA and citric acid as it is a better chelate than other groups. Intergroup comparison revealed that bond strength of EDTA and citric acid groups were not statistically significant. Stereomicroscope images confirmed that in DTPA samples the failure mode was more cohesive whereas in EDTA and citric acid group it was more of adhesive failure.

A limitation of this present study is that micro hardness of root dentin was not checked. Further studies are required to evaluate the effect of DTPA on micro hardness of root dentin<sup>17</sup>.

### Conclusion

Within the limitations of the study it can be concluded that use of DTPA significantly improved the push out bond strength of fibre post. It can be considered as an alternative to commercially available chelating agents as the smear layer removing capability is significantly better than other agents.

### References

1. Good, M.-L., Karim, I.E., and Hussey, D. (2012). Endodontic 'solutions' part 1: a literature review on the use of endodontic lubricants, irrigants and medicaments. *Dental Update* 39, 239–246
2. Abbott, P. (2012). Endodontics - Current and future. *J Conserv Dent* 15, 202.
3. Wright, P.P., and Walsh, L.J. (2017). Optimizing Antimicrobial Agents in Endodontics. In *Antibacterial Agents*, R. N. Kumavath, ed. (InTech).
4. Cavalleri, G., Cuzzolin, L., Urbani, G., and Benoni, G. (1989). Root Canal Microflora: Qualitative Changes after Endodontic Instrumentation. *Journal of Chemotherapy* 1, 101–102.

5. Composite filling or single crown? The clinical dilemma of how to restore endodontically treated teeth (2014). *Quintessence International* 45, 457–466.
6. Venugopal, S. (2013). Contemporary restoration of endodontically treated teeth. *Br Dent J* 215, 632–632.
7. Machado, J., Almeida, P., Fernandes, S., Marques, A., and Vaz, M. (2017). Currently used systems of dental posts for endodontic treatment. *Procedia Structural Integrity* 5, 27–33.
8. Ijar, and a, S. (2018). CONSIDERATIONS IN RESTORING AN ENDODONTICALLY TREATED TEETH- A REVIEW. *IJAR* 6, 874–877
9. Silva, S.M. de A. e, Carrilho, M.R. de O., Marquezini Junior, L., Garcia, F.C.P., Manso, A.P., Alves, M.C., and Carvalho, R.M. de (2009). Effect of an additional hydrophilic versus hydrophobic coat on the quality of dentinal sealing provided by two-step etch-and-rinse adhesives. *J. Appl. Oral Sci.* 17, 184–189.
10. Cadenaro, M., Breschi, L., Rueggeberg, F., Agee, K., Dilenarda, R., Carrilho, M., Tay, F., and Pashley, D. (2009). Effect of adhesive hydrophilicity and curing time on the permeability of resins bonded to water vs. ethanol-saturated acid-etched dentin. *Dental Materials* 25, 39–47.
11. Torabinejad, M., Khademi, A., Babagoli, J., Cho, Y., Johnson, W., Bozhilov, K., Kim, J., and Shabahang, S. (2003). A New Solution for the Removal of the Smear Layer. *Journal of Endodontics* 29, 170–175.
12. Craigbaumgartner, J., and Mader, C. (1987). A scanning electron microscopic evaluation of four root canal irrigation regimens. *Journal of Endodontics* 13, 147–157.
13. Barkhordar, R.A., Watanabe, L.G., Marshall, G.W., and Hussain, M.Z. (1997). Removal of intracanal smear by doxycycline in vitro. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology* 84, 420–423.
14. Hülsmann, M., Heckendorff, M., and Lennon, Á. (2003). Chelating agents in root canal treatment: mode of action and indications for their use. *International Endodontic Journal* 36, 810–830.
15. Kuah, H.-G., Lui, J.-N., Tseng, P.S.K., and Chen, N.-N. (2009). The Effect of EDTA with and without Ultrasonics on Removal of the Smear Layer. *Journal of Endodontics* 35, 393–396.
16. Pelerin, J.J. (54) DENTAL BONDING FORMULATION. 4.
17. Moura, A.S., Pereira, R.D., Rached Junior, F.J.A., Crozeta, B.M., Mazzi-Chaves, J.F., Souza-Flamini, L.E., and Cruz Filho, A.M. (2017). Influence of root dentin treatment on the push-out bond strength of fibre-reinforced posts. *Braz. oral res.* 31.
18. common-stock-solutions-buffers-and-media-2001.pdf.
19. Perdigão, J. ed. (2016). *Restoration of Root Canal-Treated Teeth* (Springer International Publishing).
20. Nagasiri, R., and Chitmongkolsuk, S. (2005). Long-term survival of endodontically treated molars without crown coverage: A retrospective cohort study. *THE JOURNAL OF PROSTHETIC DENTISTRY* 93, 7.
21. Aleisa, K., Alghabban, R., Alwazzan, K., and Morgano, S.M. (2012). Effect of three endodontic sealers on the bond strength of prefabricated fiber posts luted with three resin cements. *The Journal of Prosthetic Dentistry* 107, 322–326.
22. Rocca, G.T., and Krejci, I. Crown and post-free adhesive restorations for endodontically treated posterior teeth: from direct composite to endocrowns. *CLINICAL RESEARCH*, 25.
23. Mazzitelli, C., Monticelli, F., Toledano, M., Ferrari, M., and Osorio, R. (2010). Dentin treatment effects on the bonding performance of self-adhesive resin

- cements. *European Journal of Oral Sciences* 118, 80–86.
24. An In Vitro Study of Smear Layer Removal And Microbial Leakage Along Root Canal Fillings (2010). *Australian Endodontic Newsletter* 22, 18–18.
25. Foster, K.H., Kulild, J.C., and Weller, R.N. (1993). Effect of smear layer removal on the diffusion of calcium hydroxide through radicular dentin. *Journal of Endodontics* 19, 136–140.
26. Violich, D.R., and Chandler, N.P. (2010). The smear layer in endodontics – a review: Smear layer in endodontics. *International Endodontic Journal* 43, 2–15.
27. Grovescookeiii, H. (1976). Effects of instrumentation with a chelating agent on the periapical seal of obturated root canals. *Journal of Endodontics* 2, 312–314.
28. Gu, X.-H., Mao, C.-Y., and Kern, M. (2009). Effect of Different Irrigation on Smear Layer Removal after Post Space Preparation. *Journal of Endodontics* 35, 583–586.
29. Scelza, M.F.Z., Pierro, V., Scelza, P., and Pereira, M. (2004). Effect of three different time periods of irrigation with EDTA-T, EDTA, and citric acid on smear layer removal. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology* 98, 499–503.
30. Caron, G., Nham, K., Bronnec, F., and Machtou, P. (2010). Effectiveness of Different Final Irrigant Activation Protocols on Smear Layer Removal in Curved Canals. *Journal of Endodontics* 36, 1361–1366.
31. Fabiani, C., Franco, V., Covello, F., Brambilla, E., and Gagliani, M.M. (2011). Removal of Surgical Smear Layer. *Journal of Endodontics* 37, 836–838.
32. Prado, M., Gusman, H., Gomes, B.P.F.A., and Simão, R.A. (2011). Scanning Electron Microscopic Investigation of the Effectiveness of Phosphoric Acid in Smear Layer Removal When Compared with EDTA and Citric Acid. *Journal of Endodontics* 37, 255–258.
33. Prado, M., Gusman, H., Gomes, B.P.F.A., and Simão, R.A. (2011). Scanning Electron Microscopic Investigation of the Effectiveness of Phosphoric Acid in Smear Layer Removal When Compared with EDTA and Citric Acid. *Journal of Endodontics* 37, 255–258.
34. Machado-Silveiro, L.F., González-López, S., and González-Rodríguez, M.P. (2004). Decalcification of root canal dentine by citric acid, EDTA and sodium citrate. *International Endodontic Journal* 37, 365–369
35. Haznedaroğlu, F. (2003). Efficacy of various concentrations of citric acid at different pH values for smear layer removal. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology* 96, 340–344.
36. Hülsmann, M., Heckendorff, M., and Lennon, Á. (2003). Chelating agents in root canal treatment: mode of action and indications for their use. *International Endodontic Journal* 36, 810–830.
37. Yamaguchi, M., Yoshida, K., Suzuki, R., and Nakamura, H. (1996). Root canal irrigation with citric acid solution. *Journal of Endodontics* 22, 27–29.