# Adhesion of Root Canal Sealers to Dentin after Dentin Pre-treatment (An In-Vitro Study)

**Rawtiya Manjusha*, Verma Kavita*, Loomba Kapil**, Vivek Solanki***, Nikunj Patel**** , Lalit Yadav*****

*Reader, Department of Conservative Dentistry and Endodontics, Narsinhbhai Patel Dental College, Visnagar, North Gujarat.. **Professor & Head, Department of Conservative Dentistry & Endodontics, Chandra Dental College, Lucknow.*** P.G. Student, **** Senior Lecturer, Department of Conservative Dentistry and Endodontics Narsinhbhai Patel Dental College, Visnagar, North Gujarat. ***** Research Scholar, School of Medicine, The university of Adelaide, Australia

**Abstract:** Aim & Objective: Comparatively evaluate the effect of dentin pre-treatment on adhesion of various root canal sealers. Materials & Method: Single rooted human permanent teeth with single canal (n=136) were prepared using step back technique. They were divided randomly into four experimental Groups (n=34) according to the irrigant used (Group A- Smear Clear, Group B -17% EDTA, Group C -30% Citric Acid & Group D - Normal Saline). Two specimens from each group were examined under SEM. Each group was further subdivided in to four subgroups (N=8) according to the root canal sealer applied (Endomethasone, Roekoseal, Acroseal and AH Plus). Teeth were obturated using lateral condensation technique. Microleakage was evaluated by linear dye penetration study. Results:30% Citric Acid as a root canal irrigant was most effective in smear layer removal followed by Smear Clear and 17% EDTA. Endomethasone showed the maximum leakage while Roekoseal showed the minimum leakage with Smear Clear and Normal Saline ($p<0.05$). Acroseal showed maximum microleakage while AH Plus showed minimum with 17% EDTA ($p<0.05$). With 30% Citric Acid, Acroseal had the maximum value and Roekoseal had the minimum value ($p>0.3$). Conclusion: In all the combinations AH Plus in 17% EDTA (Group B) had the minimum leakage while Endomethasone-N had the maximum leakage with Normal Saline (Group D).. [Manjusha R Natl J Integr Res Med, 2019; 10(4):40-45]

**Key Words:** Citric Acid, Smear Clear, EDTA, Roekoseal, AH Plus, Acroseal

**Author for correspondence:** Dr. Manjusha Rawtiya, Reader, Department of Conservative Dentistry and Endodontics, Narsinhbhai Patel Dental College, Visnagar, North Gujarat. E-mail: manju27mona@gmail.com M: 9229783437

**Introduction:** Root canal sealers are used to attain an impervious seal between the core material and root canal walls. Good adhesion of the sealer improves the sealing ability, reduces microleakage**.** They can be classified as Zinc Oxide Eugenol, Calcium Hydroxide, Resin, Glass Ionomer, and Iodoform or Silicon.[1]

Smear layer is a surface film of debris retained on dentin or other surfaces after instrumentation with either rotary instruments or endodontic files. It consists of dentin particles, remnants of vital or necrotic pulp tissue, bacterial components and retained irrigants.[2] It acts as a physical barrier interfering with adhesion and penetration of sealers into dentinal tubules which may affect the sealing ability and efficacy of root canal obturation.[3]Smear layer removal increased bond strength and reduced microleakage.[4] Whether smear layer should be retained or removed remain a controversy.

Various irrigants such as Ethylene Diamine Tetra acetic Acid (EDTA), Ethylene Glycol Tetra Acetic Acid (EGTA), MTAD and EDTA plus Cetavlon have been used. [5]The intimacy of an irrigating solution to the dentinal walls depends on the wettability of the solution on solid dentine and low surface tension.[6] Addition of surfactants leading to better cleaning efficiency in the root canal. Smear Clear contains 17% EDTA solution along with cetrimide and an additional proprietary surfactant. [7]Citric Acid has also been recommended as a root canal irrigant because of its ability to demineralise and to remove the smear layer.[8]10%, 25% and 50% solutions of Citric Acid were all effective in removing calcium when used as a root canal irrigant.[9]

The concept of altering the dentinal substrate by removing the smear layer to increase the adhesion of sealers to canal wall must be evaluated. Hence the aim of this study was to comparatively evaluate the effect of dentin pre-treatment on adhesion of various root canal sealers.

**Material and Method:** Freshly extracted single rooted human permanent teeth (n=136) were used and stored in 10% formalin for two weeks for disinfection. They were sectioned at the cemento-enamel junction using a diamond disc on a slow speed micromotor hand piece under water cooling so as to obtain a uniform length of 15mm long root. Root Canals of all samples were instrumented by the conventional step back

technique using K-files (Dentsply, Maillefer, Tulsa, Okla) to an International Organisation of Standard (ISO) size 040. 2 ml of 5.25% sodium hypochlorite was used as the irrigation solute on after each file during instrumentation of the canal. After instrumentation and drying of the root canals with absorbent paper points, teeth were assigned to four groups (n=34). Samples were irrigated for 1 minute with 2 ml of Smear Clear (Sybron Endo) in group I, 17% EDTA (Prevest Denpro Ltd.) in Group II, 30% Citric Acid in Group III and Normal Saline in Group IV (control Group).

Two samples from each group were selected randomly and deep grooves were cut on the buccal and palatal surfaces of the roots, without perforating the root canal using carborundum discs. The roots were then split with a chisel and a mallet. One half of each tooth was randomly selected and prepared for SEM examination. The dentinal wall of the cervical, middle and apical thirds was observed at magnifications of 3000x for the presence/absence of smear layer.

Samples from each group were again divided into subgroups (n=8) according to the type of sealer applied.

**Subgroup A**- Canals were obturated with Gutta-Percha/Acroseal sealer (Septodont); An ISO size gutta-percha master cone corresponding to the prepared canal apex was tried to within 1 mm of the working length. Sealer was placed into the canal using hand lentulospirals. Master cone was coated generously with the sealer and placed into the canal. Accessory cones were placed into the canals. Lateral compaction of the cones was done with the help of finger spreaders of the ISO standardization. When the canals were fully compacted they were condensed into the canals using finger pluggers. Same procedure was carried out in Subgroup **B, C, and D** but sealer used was AH plus (Dentsply), Endomethasone (Septodont) and Roekoseal Sealer (Colene Whaledent) respectively.

After obturation the samples of the experimental subgroups were stored for 48 hours at room temperature to allow complete setting of the sealers. The teeth were coated with sticky wax at the coronal end and completely coated with two coats of nail paint to prevent any leakage inside the canal space after which they were immersed in 2% methylene blue dye for 48 hours. The teeth were then washed under running tap water and nail paint and sticky wax was scraped from the tooth surface using a scalpel. Grooves were made along the mesial and distal walls of the roots using a diamond disc on a slow speed handpeice. Then the teeth were split into two halves using a chisel and mallet. Both halves of the split samples were then evaluated under a stereomicroscope at magnification of 10 xs for visible coronal extent of dye penetration. The linear measurement of the dye penetration was noted from apical to coronal direction. Each gradation on the photomicrograph corresponded to 1 mm on a linear scale. The recorded measurements were then statistically analyzed using Wilcoxon-Signed Rank sum test.

**Results:** Stereomicroscopic evaluation showed dye penetration in all samples. Leakage of each group and the mean dye penetration values were summarized using Wilcoxon signed rank test (Table 1). Statistical significance is set at ($p<0.05$). Comparison of microleakage in different subgroups for different Sealers was done (graph 1)

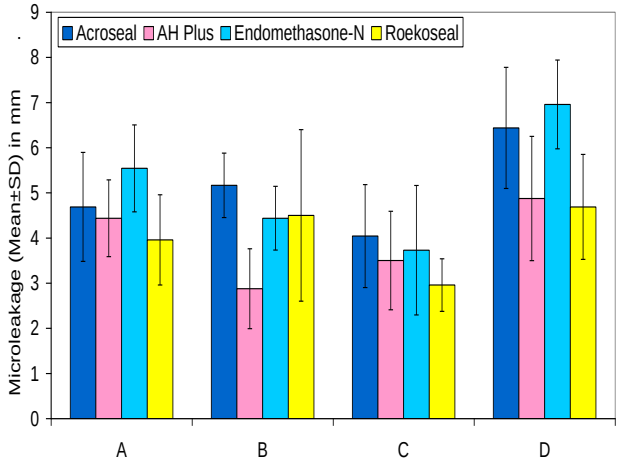
**Table I: Comparison of Microleakage in different groups for different Sealers (n=8)**

| S.N. | Group | Acroseal | | AH Plus | | Endomethasone-N | | Roekoseal | | Statistical Significance | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Mean | SD | Mean | SD | Mean | SD | Mean | SD | "$\chi^2$" | "p" |
| 1. | A (Smear Clear) | 4.688 | 1.207 | 4.438 | 0.850 | 5.542 | 0.963 | 3.958 | 0.999 | 9.082 | 0.028[a] |
| 2. | B (17% EDTA) | 5.167 | 0.713 | 2.875 | 0.885 | 4.438 | 0.707 | 4.500 | 1.900 | 10.573 | 0.014[a] |
| 3. | C (30% Citric Acid) | 4.042 | 1.140 | 3.500 | 1.091 | 3.729 | 1.434 | 2.958 | 0.582 | 3.425 | 0.331[b] |
| 4. | D (Normal Saline) | 6.438 | 1.342 | 4.875 | 1.377 | 6.958 | 0.983 | 4.688 | 1.163 | 14.334 | 0.002[c] |

**Discussion:** Different irrigation protocols introduced to remove the smear layer can create dentinal surfaces which are very different structurally. [10] The ideal purpose is to create a particular surface of dentin which is more suitable for the specific sealer used in the obturation of the root canal system. Hypothesis is that if a dentinal surface and a sealer can

complete and complement each other characteristically, ultimately they can produce a better hermetic coronal and apical seal.[11]

**Graph 1: Comparison of Microleakage in different groups for different Sealers (n=8)**




**I. Inter Group Comparison:** Minimum microleakage was observed when 30% Citric Acid was used as an irrigant. It is reported to cause more erosion of the dentinal wall creating more adhesion surface area for a resin-based sealer when compared to EDTA. [12]Smear Clear group showed less leakage than 17% EDTA which might be due to additional surfactant present in the irrigant. Smear Clear and 17% EDTA have mostly shown no significant difference in leakage values when used as an irrigant. [7]Maximum leakage was seen with normal saline (Control Group) as irrigant which does not remove smear layer and debris. The smear layer is an amorphous, non-homogeneous, weakly adherent structure and has a low density because of its higher water content, which makes it unstable and hence susceptible to dye infiltration.[13]

**II Intra Group Comparison**

Comparison of microleakage observed in different smear layer removing agents when Acroseal was used as sealer : Discussing the performance of Acroseal in presence of different irrigating agents it was observed that Acroseal showed maximum leakage when Normal Saline was used as an irrigant whereas minimum leakage was seen when 30% Citric Acid was used as an irrigant.

30%Citric Acid) ~ Smear Clear < 17% EDTA < Normal Saline: There was a statistically significant difference between mean microleakages in samples irrigated with Smear Clear and Normal Saline, 17% EDTA and 30% Citric Acid, 17% EDTA and Normal saline and 30% Citric Acid and Normal saline.

On comparison of microleakage after irrigation with 30% Citric Acid and 17% EDTA microleakage in 30% Citric Acid was less which may be due to more decalcification of peritubular dentin. The tubule orifices are enlarged because of dissolution of peritubular dentin resulting in better adhesion. In this study 30% citric acid was used. Khademi **et al**. demonstrated that 20% Citric Acid produces more erosion of dentin compared to 7% Citric Acid and 17% EDTA.[14] **Machado-Silveiro et al**. indicated that the decalcifying effect of 10% Citric Acid on dentin is more than 17% EDTA.[15]

No statistically significant difference was observed between Smear Clear (Group $A_1$) and 17% EDTA (Group $B_1$) and Smear Clear (Group $A_1$) and 30% Citric Acid (Group $C_1$). Smear Clear is a new irrigant with a surfactant resulting in smear layer removal comparable to Citric Acid.

It may be suggested that removal of smear layer may potentiate the beneficial effects of calcium hydroxide. It facilitated Ca $(OH)_2$ diffusion through the dentinal tubules and potentiates the therapeutic effect of Calcium Hydroxide Sealers in the treatment of avulsed or luxated teeth to reduce the occurrence of inflammation, surface resorption or replacement resorption.[16] Hence it may be stated that Acroseal must be used with a smear layer removing agent for better therapeutic benefits.

B. Comparison of Microleakage observed in different smear layer removing agents when AH Plus was used as sealer: AH Plus sealer has strong adhesion ability to dentine and good sealing ability. It is described as having a faster setting time. The advantage of resin-based sealers over ZOE-based sealers is that they can not only lock into open dentinal tubules but also adhere to the exposed dentinal surfaces.[17]

AH Plus sealer showed maximum leakage in presence of normal saline. This could be because of the relatively weak bond of the smear layer to the underlying dentine, approximately 5 mpa**,** which may be insufficient to withstand the shrinkage associated with the curing of resins, and the smear layer may be pulled away from the dentine and provide an avenue for microleakage.

**17% EDTA ~ 30% Citric Acid < Smear Clear ≤Normal Saline :** Minimum leakage was seen in presence of 17% EDTA may be due to alteration of the dentin surface energy as a result with pretreatment with EDTA. EDTA significantly decreased the wetting ability of dentinal wall.[18] Therefore, a suitable dentin substrate could be provided for the adhesion of materials with hydrophobic nature as the resinous AH plus. Furthermore, the effective removal of smear layer by EDTA allowed for the extension of the resin into the open dentinal tubules, creating efficient micro retention. Irrigation with 17% EDTA also resulted in higher bond strength values for AH plus**.** The dentin adhesion of endodontic sealers can be improved by dentin pretreatment with EDTAC. [19] Highest increases in adhesiveness were found for other resin based sealers like Sealer 26, and Sealapex, Apexit.

Considering the qualities of AH Plus, which was seen to be the best sealer in presence of 17% EDTA it may be stated that if the surface area of dentin exposed to this sealer is increased, the adhering and penetrating capacity of AH Plus is improved and better seal is expected. Citric Acid as irrigant showed similar performance as 17% EDTA. In this regard, irrigation solutions which cause more erosion of dentinal wall and create a porous etched surface would be a reasonable choice.

Comparing the mean microleakage of group A (Smear clear) and Group B (17 % EDTA) it was observed that AH plus showed more microleakage in presence of Smear Clear. This might be due to presence of surfactant Tween 80 which might have permitted increased dentin surface energy and wettability, hence increasing intertubular dentin permeability as well as exposure of collagen matrix and intertubular fluid which could have negatively affected the adhesion of the hydrophobic AH plus sealer**.**[20]

**Comparison of Microleakage observed in different smear layer removing agents when Endomethasone-N was used as sealer :** In this study Endomethasone-N showed maximum dye penetration in presence of normal saline whereas minimum leakage was seen in presence of 30% Citric Acid followed by 17% EDTA and Smear Clear. The chelate formed during setting reaction is known to slowly hydrolyse in presence of water to release eugenol and may be responsible for the gradual loss of its sealing ability. Also the sudden setting and debonding of the sealer from dentinal walls or cohesive fracture caused by shrinkage setting stresses might explain the higher leakage**.**[21] Further no significant difference in leakage values of Endomethasone-N was observed when either 30% Citric Acid or 17% EDTA were used. Hence it may be stated that when Endomethasone is used as a sealer the irrigant of choice may be either citric acid or 17% EDTA.

Smear Clear (Group $A_3$) had significantly higher mean value as compared to 17% EDTA (Group $B_3$) and 30 % Citric Acid (Group $C_3$.) No significant difference was observed between 17 % EDTA (Group $B_3$) and 30 % Citric Acid (Group $C_{3)}$.

**Comparison of Microleakage observed in different smear layer removing agents when Roekoseal was used as sealer:** Roekoseal Sealer showed minimum microleakage in presence of Smear Clear as an irrigant. It contains polydimethylsiloxane which gives the sealer good flowability and high penetrability into dentinal tubules. It also has excellent dimensional stability, initial expansion and low solubility contributing to the better sealing ability and minimal leakage scores.[22]

It has been suggested that the quality of the apical seal may be improved by increasing the surface contact between the root canal and the sealer. It has been shown that removal of smear layer may allow sealer to penetrate into the dentinal tubules, thereby giving a greater area of surface contact which may delay the penetration of leakage materials.[23]

Thus the order of microleakage in different groups was: **Smear Clear ~ Group 17%EDTA ~ 30% Citric Acid <Group Normal Saline :** Technique of instrumentation, type of sealer used, sealer thickness, type of filling technique, type and concentration of chelating agents used, and the technique used to produce and remove the smear layer and different laboratory procedures to check the leakage may vary the results.

The results of this study suggested that the dentinal penetration of different sealers is related to their physical and chemical properties such as dimensional changes, absorption and dissolution which may affect sealing ability of the sealers**.**

Although some studies suggest that removal of the smear layer reduces microleakage, the treatment with EDTA may leave a chelated layer of dentin at the dentin-root filling interface. Residual EDTA inside the dentinal tubules which was measured up to 38% of the originally applied volume may contribute additionally to ongoing demineralization resulting in further increase in microleakage**.**[24] Residual EDTA also may interact with the sealer, which has been demonstrated for zinc oxide eugenol sealer. However, **Madison *et al*. (1984)** could not detect any influence of the irrigation solution on the apical seal.[25]

Removal of smear layer has been suggested to improve the adaptation of root canal fillings. However, some sealers may behave differently in the absence of smear layer. Further studies are necessary to establish a correlation between endodontic smear layer and the clinical performance of root canal fillings.

**Conclusion:** 30% Citric Acid as a root canal irrigant was most effective in smear layer removal followed by Smear Clear Group, followed by 17% EDTA. All the sealers tested show minimum microleakage values in presence of 30 % Citric Acid. Acroseal in presence of normal saline show maximum leakage and minimum leakage in presence of 30% Citric Acid. AH Plus show maximum leakage in presence of normal saline whereas minimum leakage in presence of 17 % EDTA. Endomethasone-N show maximum leakage in presence of normal saline whereas minimum leakage in presence of 30% Citric Acid. Roekoseal show maximum leakage in presence of normal saline whereas minimum leakage was observed in 30% Citric Acid Group.

In presence of Smear Clear, Endomethasone showed the maximum leakage value while Roekoseal showed the minimum, In presence of 17% EDTA, Acroseal showed maximum microleakage while AH plus showed minimum microleakage. In presence of 30 % Citric Acid Acroseal had the maximum value for microleakage whereas Roekoseal had the minimum value. In presence of normal saline, Endomethasone-N had the maximum value while Roekoseal had the minimum value. Irrigants such as Citric Acid 30% EDTA and Smear Clear which remove smear layer could be used to increase the adhesion of root canal sealers. Further laboratory and also clinical studies are needed in the future to compose a clear view concerning the improvement of sealing ability following smear layer removal.

**Reference :**


1. Brainstetter J., Von Fraunhofer A. The Physical Properties and Sealing Action of Endodontic Sealer Cements : A Review of the Literature ; *Journal of Endodontics*, 1982, 8 ; 7 : 312-316.
2. Violich D.R., Chandler N.P. The Smear Layer in Endodontics – A Review; *International Endodontic Journal; 2010*43: 2-15.
3. Sen B.H., Kin B., Baran N. The Effect of Tubular Penetration of Root Canal Sealers on Dye Microleakage. *International Endodontic Journal;1996* 29, 23–8.
4. Economides N., Liolios E., Kolokouris I., Beltes P. Long Term Evaluation of the Influence of Smear Layer Removal on the Sealing Ability of Different Sealers. *Journal of Endodontics;1999,* 25, 123–5.
5. Giardino L., Ambu E., Becce C. Surface Tension Comparison of Four Common Root Canal Irrigants and Two New Irrigants Containing Antibiotic; *Journal of Endodontics*, 2006, 32(11), 1091-1093.
6. Yamaguchi M., Yoshida K., Suzuki R., Nakamura H. Root Canal Irrigation with Citric Acid Solution. *J Endod;1996,* 22:27-9.
7. Da Silva L.E., Sanguino A.C., Rocha C.T. Scanning Electron Microscopic Preliminary Study Of The Efficacy Of Smear Clear And EDTA For Smear Layer Removal After Root Canal Instrumentation In Permanent Teeth; *Journal of Endodontics*;2008 34(12), 1514-1544.
8. Yamaguchi M., Yoshida K., Suzuki R., Nakamura H. Root Canal Irrigation with Citric Acid Solution.1996, *J Endod* 22:27-9.
9. Wayman EB, Blake E, Jeremy J. An evaluation of the antimicrobial effectiveness of citric acid as a root canal irrigant *J Endod*- 1986 12(2):54-58.
10. Farhad A.R. Barekatain B. and Koushki A.R. The Effect of Three Different Root Canal Irrigant Protocols for Removing Smear Layer on the Apical Microleakage of AH26 Sealer. *Iranian Endodontic Journal;2008* 3, 62-67.
11. Saleh M., Ruyter I.E., Haapsalo D. The Effect of Dentin Pre-Treatment on the Adhesion of Root Canal Sealers; *International Endodontic Journal;2002,* 35, 859–66.
12. Torabinejad M., Khademi A.A, Babagoli J. A New Solution for the Removal of the Smear Layer. *Journal of Endodontics (2003)* 29, 170–5.

13. Torabinejad M., Handysides R., Khademi A.A. Clinical Implications of the Smear Layer in Endodontics; *Oral Surg Oral Med Oral Pat; 2002* 94:658-66.
14. Khademi A, Feizianfard M. (2004) The Effect of EDTA and Citric Acid on Smear Layer Removal of Mesial Canals of First Mandibular Molars, A Scanning Electron Microscopic Study Journal of Research in Medical Sciences; 2004 9(2)80 - 88.
15. Machado-Silveiro, LF. González-López, S. and González-Rodríguez, MP. Decalcification of root canal dentine by citric acid, EDTA and sodium citrate. International Endodontic Journal,2004, 37: 365–369.
16. Saif S, Clifton M. Carey, Tordik PA, Scott B. Effect of Irrigants and Cementum Injury on Diffusion of Hydroxyl Ions through the Dentinal Tubules Endod 2008;34:50 –52
17. Cobankara F.K., Adanir N, Belli S, Pashley DH (2002). A Quantitative Evaluation of Apical Leakage of Four Root-Canal Sealers. *International Endodontic Journal*; 2002, 35(12): 979-84.
18. Hashem AAR , Ghoneim AG, Lutfy RA, Fouda MY .The Effect of Different Irrigating Solutions on Bond Strength of Two Root Canal–filling Systems Endod 2009,1–4.
19. De-Deus G., Reis C. and Fidel S. Longitudinal and Quantitative Evaluation of Dentin Demineralization when subjected to EDTA, EDTAC and Citric Acid: A Co-Site Digital Optical Microscopy Study; *Oral Surg Oral Med Oral Pathl Oral Endod*, 2008,105: 391-7.
20. De-Deus G., Reis C. & Paciornik S. Dentine Demineralization when Subjected to EDTA with or Without Various Wetting Agents: A Co-Site Digital Optical Microscopy Study; *International Endodontic Journal* ;2008, 41, 279-287.
21. Lucena-Martin C., Ferrer-Luque C.M., Gonzalez-Rodriguez M.P., Robles-Gijon V., Navajas-Rodriguez De Mondelo J.M. A comparative study of apical leakage of endomethasone, top seal, and roeko seal sealer cements. *Journal of Endodontics*; 2002, 28 (6), pp. 423-426.
22. Gencoglu N., Turkmen C., Ahiskali R. A New Silicon-Based Root Canal Sealer (Roekoseal-Automix); *J Oral Rehabil; 2003,* 30(7):753-7.
23. Martin C.L., Ferrer-Luque C.M. (2002). A Comparative Study of Apical Leakage of Endomethasone, Topseal and Roekoseal Sealer Cements. *Journal of Endodontics;2002,* 28, 423-426.
24. Cooke HG, Grower MF, del Rio C. Effects of instrumentation with a chelating agent on the periapical seal of obturated root canals. *Journal of Endodontics*;1976, 2(10):312-4.
25. Madison S, Krell KV. Comparison of ethylene diamine tetra acetic acid and sodium hypochlorite on the apical seal of endodontically treated teeth. Journal of Endodontics; 1984 10,499–503.

Conflict of interest: None

Funding: None

Cite this Article as: Manjusha R, Verma K, Loomba K, Solanki V, Patel N , Yadav L. Adhesion of Root Canal Sealers to Dentin after Dentin Pre-treatment -An In-Vitro Study. Natl J Integr Res Med 2019; Vol.10(4): 31-33