Original Article

Effect of chelating agents on the wettability of BioRoot RCS and AH Plus sealers

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Abstract

Introduction: To evaluate the effect of different chelating agents on the wettability of AH Plus and BioRoot RCS sealers on intraradicular dentin.

Materials and Methods: Fifty single-rooted premolars were split longitudinally and divided into five groups according to irrigant regimen. Group 1: 2.5% sodium hypochlorite (NaOCl) – 17% ethylenediaminetetraacetic acid (EDTA); Group 2: 2.5% NaOCl – 7% maleic acid (MA); Group 3: 2.5% NaOCl – SmearOFF™ (containing chlorhexidine and EDTA); Group 4: 2.5% NaOCl – Dual Rinse® hydroxyethylidene diphosphonic acid (HEDP); Group 5: 2.5% NaOCl – distilled water. Specimens were treated with controlled volume of experimental sealers and subjected to contact angle measurement.

Results: Wettability of both sealers on intraradicular dentin was found to be best with MA. For AH Plus sealer, wettability was better with SmearOFF™, followed by EDTA, Dual Rinse® HEDP, and distilled water. However, there was no statistical difference between SmearOFF™ and EDTA ($P > 0.05$). For BioRoot RCS, wettability was better with SmearOFF™ and EDTA, with the former showing better results ($P < 0.001$). This was followed by Dual Rinse® HEDP and distilled water.

Conclusion: Within the limitations of the study, it can be concluded that MA when used as a final irrigant showed better wettability of both AH Plus and BioRoot RCS sealers compared to the other tested solutions.

Keywords: AH Plus sealer, BioRoot RCS sealer, chelating agents, contact angle, wettability

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INTRODUCTION

Success of root canal treatment relies on meticulous cleaning of the root canal system.[1] Maximum portions of canal walls remain unblemished by manual or rotary instrumentation during instrumentation of the root canals as shown in the previously done studies.[2] Thus, comes into the highlight, the importance of the use of chemical debridement in the cleaning and shaping of root canal system. The mechanical instrumentation of the root canal creates an amorphous smear layer that covers the intraradicular dentinal tubules and walls.[3] Eradication of the smear layer has been shown to enhance the fluid-tight

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seal of root canal system.[4] The penetration of irrigants, intracanal medicaments, and sealers into the dentinal tubules is also hindered by the presence of smear layer over the dentinal walls of the root canals.[5] Hence, removal of smear layer is mandatory in root canal therapy.

Various chelating agents such as ethylenediaminetetraacetic acid (EDTA), citric acid, BioPure® MTAD® (Dentsply Sirona, York, PA, USA), and QMix 2in1 (Dentsply Sirona) have been used for the removal of canal wall smear layer.[6,7] For an effective eradication of smear layer, the sequential use of EDTA and sodium hypochlorite (NaOCl) has been commonly advocated. However, there exist various drawbacks with the use of EDTA such as diminished efficacy in the removal of smear layer in the apical third of the canal space,[8] diminution in dentin microhardness,[9] and cytotoxicity.[10] In addition, the bond strength of resin cements to radicular dentin is also reduced after application of EDTA.[11]

Maleic acid (MA) is a mild organic acid used as a conditioner in adhesive dentistry. In a study done by Ballal *et al*., MA has been found to possess a higher ability to remove smear layer as compared to 17% EDTA, especially in apical third of root canal system.[8,12] Recently, a novel chelating agent called SmearOFF (Vista Dental Inc., USA) consisting of a mixture of EDTA and chlorhexidine has been marketed. Manufacturers claim it to be effective in smear layer removal. Moreover, there is no precipitate formation on mixing with NaOCl. It has been shown to have better calcium suspension and 27% more dentinal tubules clearance in comparison with 17% EDTA. The efficiency of SmearOFF in eradication of smear layer from the canal system has been well demonstrated in a recent study.[13] A recent study has shown that Dual Rinse® hydroxyethylidene diphosphonic acid (HEDP) (Medcem, GmbH, Weinfelden, Switzerland) also known as etidronic acid, (1-hydroxyethylidene-1,1-diphosphonic acid) which is a soft chelator can also be used in combination with NaOCl. The use of NaOCl and Dual Rinse® HEDP as a single irrigant during the instrumentation of the root canal has been well demonstrated in the study. There has been a significant reduction in the amassing of the hard tissue debris in the isthmus area when irrigated with freshly mixed NaOCl and Dual Rinse® HEDP.[14] Dual Rinse® HEDP (9%) is a medical device approved for use in the root canal therapy. It is dispensed as a capsule carrying etidronate powder weighing 0.9 g, which should be stirred instantly with 10 mL of the NaOCl solution of choice before the procedure. The so-formed combination of Dual Rinse® HEDP and NaOCl inhibits the formation of smear layer formation during root canal instrumentation as well as conditions the root canal walls for subsequent obturation.[15]

AH Plus, an epoxy-based root canal sealer, has got several advantages such as good mechanical properties, high radiopacity, reduced polymerization shrinkage, low solubility, and a high degree of stability on storage. It has also been proven to improve the fracture resistance of endodontically treated teeth.[16,17] Recently, a novel sealer, BioRoot RCS, has been marketed by Septodont (Saint Maur Des Fosses, France). It has an active biosilicate technology and forms a strong void-free seal with outstanding adhesion to gutta percha points and root dentin.[18] It has also shown to induce angiogenesis and osteogenesis, the prerequisites of tissue regeneration.[19] An experimentally measured contact angle acts as a good indicator of the spreading nature of the liquid on the solid surface as it measures the wetting behavior of a liquid on a solid surface. The wetting angle so formed has a three-phase boundary where there is intersection between liquid, gas, and solid. Higher the values of contact angle, poorer are the wetting. The existence of smear layer on the root canal walls affects the wettability of the sealer.[20] Hence, proper wetting of sealer can be achieved by complete eradication of smear layer from the radicular dentin. To date, the effect of Dual Rinse® HEDP and SmearOFF on the wettability of sealers has not been adequately investigated. Hence, the aim of the study was to compare the effect of 17% EDTA, 7% MA, SmearOFF, and Dual Rinse® HEDP on the wettability of BioRoot RCS and AH Plus sealers.

MATERIALS AND METHODS

Specimen preparation

Extracted human teeth were used for the study after obtaining ethical clearance from the institutional review board (IEC 860/2016). Fifty human single-rooted premolars with single canal and fully formed apex were collected and cleaned with ultrasonics to remove the debris and calculus. Teeth with fractures, caries, resorption, and calcification were excluded. Samples were disinfected using 0.2% sodium azide (Sigma, Aldrich, Germany) and stored at 4°C. Decoronation of the samples was done with a low-speed diamond disk (Horico, Berlin, Germany) under water coolant and the teeth were split longitudinally into hundred sections. Later, split sections were flattened and polished with silicon carbide paper 100 grit (30 strokes per teeth) to obtain fine surface for analysis. Specimens were then divided arbitrarily into five groups ($n = 20$) depending on the irrigation regimen as follows:

1. EDTA group: 5 mL of 2.5% NaOCl for 1 min – 5 mL 17% EDTA (Vista Dental Inc., USA) solution for 1 min – final rinse of 5 mL distilled water for 1 min

2. MA group: 5 mL of 2.5% NaOCl for 1 min – 5 mL 7% MA (Sigma, Aldrich, Germany) for 1 min – final rinse of 5 mL distilled water for 1 min
3. SmearOFF group: 5 mL of 2.5% NaOCl for 1 min – 5 mL SmearOFF for 1 min – final rinse of 5 mL distilled water for 1 min
4. Dual Rinse® HEDP group: 5 mL of 2.5% NaOCl for 1 min – 5 mL of Dual Rinse® HEDP for 1 min – final rinse of 5 mL distilled water for 1 min
5. Distilled water group (control): 5 mL of 2.5% NaOCl for 1 min – 5 mL of distilled water for 1 min – final rinse of 5 mL distilled water for 1 min.

In Dual Rinse® HEDP group, one capsule of HEDP was mixed with 10 mL of NaOCl and was continuously stirred until the granules dissolved completely in NaOCl. On the basis of the sealer being used, the specimens under each category/group were further subdivided into two subgroups ($n = 10$).

Contact angle measurement

After drying using blotting paper, each specimen was placed on a flat glass surface in a contact angle instrument (Holmarc Opto-Mechatronics Pvt. Ltd., Kochi, Kerala, India). The equipment consisted of a horizontal stage to mount a solid or liquid sample, a motor-controlled syringe pump to dispense the liquid droplet, an illumination source, and an imaging camera. A sessile droplet of known volume was dispensed onto the substrate of interest, and equilibrium contact angle of the droplet at the two or three phase contact points was estimated by fitting the tangent to the droplet image. The mixing of each sealer (BioRoot RCS or AH plus) was carried out as per the manufacturer's directions. Herein, a controlled-volume droplet (0.1 mL) of each sealer was put over the surface of a specimen from each group. A dispensing syringe pump was used to regulate the volume of each sealer. Two drops of the same sealer were evaluated for each irrigant group, and the spreading process was recorded for 60 s. Software (Contact Angle Meter Version 5.0.0.0) was then used to record the images of the droplets to measure the static contact angles formed between the sealer and the radicular dentin. Data from each sealer were analyzed using one-factor analysis of variance to assess the effects of various irrigating solutions on the wettability of the sealer on root canal dentin.

Statistical analysis

Statistical analysis was performed using IBM SPSS statistics version 20.0 software (IBM Corp., Armonk, NY, USA). The intragroup comparison among the irrigants was done by one-way ANOVA test, and intergroup comparison between the sealers was done by Tukey's honestly significant difference test. $P < 0.05$ was considered statistically significant (95% confidence level).

RESULTS

Comparison of the sealers

When HEBP and EDTA were used, there was a high statistical difference between the contact angles made by AH Plus and BioRoot RCS ($P < 0.001$), in which AH Plus showed better wettability than BioRoot RCS. However, no statistical differences were found in wettability of AH Plus and BioRoot RCS when MA, SmearOFF, and distilled water were used ($P > 0.05$) [Table 1].

Comparison of the irrigants

Irrigants used as the final rinse had a highly significant effect on the wettability of AH Plus ($P < 0.001$) and BioRoot RCS sealers ($P < 0.001$) on root canal dentin.

Wettability of AH Plus sealer was better when root canal dentin was treated with SmearOFF, EDTA, and MA when compared to Dual Rinse® HEDP and distilled water. On comparison among SmearOFF, MA, and EDTA, MA showed the best results. However, there was no difference between SmearOFF and EDTA ($P = 0.998$). When comparing Dual Rinse® HEDP and distilled water, Dual Rinse® HEDP showed better wettability of AH Plus sealer ($P = 0.125$) [Table 2].

When BioRoot RCS was evaluated, it showed better wettability when root canal dentin was treated with MA, SmearOFF, and EDTA compared to Dual Rinse® HEDP and distilled water. On comparison among the former, MA showed the best results. However, there was a significant difference between SmearOFF and EDTA ($P = 0.241$), in which SmearOFF showed better results followed by EDTA, Dual Rinse® HEDP, and distilled water [Table 2].

Table 1: Intragroup comparison of wettability between AH plus and BioRoot RCS sealers

Irrigants	Sealers	*n*	Mean	SD
DW	AH plus	10	58.871	10.756
	Bio RCS	10	57.895	8.315
DR HEDP	AH plus	10	49.275	9.367
	Bio RCS	10	68.173	9.454
SO	AH plus	10	44.535	6.265
	Bio RCS	10	51.499	8.996
EDTA	AH plus	10	45.738	8.931
	Bio RCS	10	59.217	6.786
MA	AH plus	10	40.298	8.161
	Bio RCS	10	43.831	7.374

SD: Standard deviation, DW: Distilled water, DR: Dual Rinse, SO: SmearOFF, EDTA: Ethylenediaminetetraacetic acid, MA: Maleic acid, HEDP: Hydroxyethylidene diphosphonic acid

Table 2: Multiple comparisons of irrigants tested

Sealers	Irrigants	Irrigants	Mean
AH plus	DW	HEBP	9.59
		SO	14.33
		EDTA	13.13
		MA	18.57
	DR HEDP	SO	4.74
		EDTA	3.53
		MA	8.97
	SO	EDTA	-1.20
		MA	4.23
	EDTA	MA	5.440
Bio RCS	DW	HEBP	-10.27
		SO	6.39
		EDTA	-1.32
		MA	14.06
	DR HEDP	SO	16.67
		EDTA	8.95
		MA	24.34
	SO	EDTA	-7.71
		MA	7.66
	EDTA	MA	15.38

DW: Distilled water, DR: Dual Rinse, SO: SmearOFF, EDTA: Ethylenediaminetetraacetic acid, MA: Maleic acid, HEDP: Hydroxyethylidene diphosphonic acid, HEBP: Hydroxyethylidene bisphosphonate

DISCUSSION

For optimal wettability, the liquid should have the lowest possible contact angle with the surface.[21] The surface possessing a lower contact angle or greater surface free energy presents with high wettability, which means that the spreading and interaction of sealer is better in a solid presenting with high surface energy, thus resulting in the formation of a lower contact angle.[22] Because root canal sealers used in the present study are in liquid form, its wettability on the root canal dentin can be evaluated in terms of contact angle measurement.

The results of this study demonstrated that both AH Plus and BioRoot RCS sealers showed the best wettability when MA was used to treat the root canal dentin. This could be due to the better chelating action of 7% MA when compared to 17% EDTA and Dual Rinse® HEDP.[8,23] Moreover, the acidic nature of MA is very high, thus demonstrating its better demineralizing effect over a shorter duration of time.[24] It has been demonstrated that removing the smear layer from root dentin surface leads to increase in surface roughness due to more pronounced opening of dentinal tubules.[25,26] As per the reports of previously done studies, an increase in surface roughness reduces the contact angle,[27] which could be another reason for both the sealers showing better wettability when irrigated with MA.

Followed by MA, both AH Plus and BioRoot RCS sealers showed good wettability with SmearOFF and EDTA with no statistical difference between the two. SmearOFF is a combination of EDTA, chlorhexidine, and surfactants. The presence of surfactants and surface modifiers in its composition could have attributed to the results. The rationale of adding surfactants is to lower surface tension and increase wettability,[28] which also enables better penetration of irrigant into the root canal.

When compared to MA, EDTA showed poor results in wettability of both the sealers. However, the wetting property of EDTA was better than Dual Rinse® HEDP and the control group (distilled water). The presence of excessive amount of OH^- due to high pH in EDTA leads to low dissociation of smear layer hydroxyapatite.[29] This phenomenon reduces the number of calcium ions which EDTA can chelate, resulting in limited effectiveness of EDTA on the wettability of sealers. Furthermore, the increased pH value (compared to MA) negatively affects and decreases the breakdown of hydroxyapatite, in spite of being fully deprotonated and having good affinity for calcium ions. EDTA showed equally efficient results as SmearOFF which could be due to gradual dissolution of inorganic and organic matrix of intertubular and peritubular dentin, leading to increased surface roughness and hence higher surface free energy and good wetting behavior.

Dual Rinse® HEDP showed poor results with both the sealers when compared with the other chelating agents used in this study. Previous studies have shown that HEDP produces minute changes in surface roughness compared to other commonly used chelating agents such as EDTA due to its weak chelating property.[30] AH Plus sealer showed better wettability with Dual Rinse® HEDP than distilled water. This could be because Dual Rinse® HEDP is a bisphosphonate and is highly adsorbed to hydroxyapatite surface, leading to increase in surface free energy.[31] The rise in surface free energy leads to increase in wettability.[32]

In the present study, when AH Plus sealer was used, EDTA showed better results than Dual Rinse® HEDP, which is in accordance with the literature.[33,34] Better penetrability of the sealer into the microirregularities could be another reason for showing good wetting property.

Controlled volume (0.1 mL) of each sealer was used for recording all the measurements in this study and the reason for use of the controlled volume of sealer being volumetric changes that could affect the value of contact angle.[35,36] Because the surface tension coefficient of liquid is influenced by change in temperature and humidity, the entire experimental procedure was carried out under standard environmental conditions.[37]

A captive bubble or sessile drop technique can be used for the measurement of contact angle, the latter of which was used in the present study. The advantage of using this approach was to maintain a dry environment during the measurement of contact angle of a liquid drop on flat surfaces.

BioRoot RCS sealer was used in this study because it is one of the novel bioceramic sealer which has shown to have good biological properties.[18,19] Because AH plus sealer is known to be the gold standard,[38] it was compared with BioRoot RCS for its wettability.

Because the hydration state of the dentin surface has also shown to affect the contact angle,[39] the samples in the current study were dried using blotting paper. The dentin surfaces were polished thoroughly to achieve reduction in the influence of roughness on the surface energy of root dentin wall, thus leading to the reduction of its influence on the measurement of contact angle.[21]

CONCLUSION

Within the limitations of the study, it can be concluded that MA when used as a final irrigant showed better wettability of both AH Plus and BioRoot RCS sealers compared to the other tested solutions.

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Conflicts of interest
There are no conflicts of interest.

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