



INVITRO COMPARATIVE EVALUATION OF CONTACT ANGLE OF THREE ROOT CANAL SEALERS

Dental Science

Dr vinodha Chandrasegaran

Assistant Professor, Department Of Dental Surgery, PSG Institute Of Medical Science And Research

ABSTRACT

AIM: The aim of this study was to evaluate the contact angle of three endodontic sealers zinc oxide eugenol, AH plus [epoxy resin based] and bioceramic [calcium silico phosphate based] sealers.

METHODOLOGY: Working length of 21 samples of human mandibular premolars were determined followed by chemomechanical preparation. The specimens were then decoronated, split longitudinally, flattened, smoothed and stored in distilled water. Half of the dentin discs were treated with 5ml of 3%NaOCl followed by 17%EDTA and 2%chlorhexidine for 1min each. The samples were air dried and divided into 3 groups based on the experimental sealers used. A total of 14 samples were assigned for each group with 7 samples per subgroup. In each sample 1 drop [0.1 ml] of sealer was deposited and the contact angle at 0,5,10,15,20,25,30 seconds after the initial application of the sealers were recorded at room temperature.

RESULT: contact angle values were low for zinc oxide eugenol followed by AH Plus and Bioceramic sealer.

CONCLUSION: The present study revealed that lower contact angle along with smear layer removal enhanced the wettability of root canal sealers.

KEYWORDS

Wettability, Contact Angle, Smear Layer, dentin

INTRODUCTION

In endodontic practice, the success of root canal therapy mainly depends on the elimination of pathogenic microorganisms and their byproducts followed by three dimensional obturation of the root canal space. To achieve this challenging task, a biologically acceptable solid or semisolid obturating material in conjunction with root canal sealers has been advocated in endodontic literatures.

Guttapercha is the most widely used core material for obturation till date. Its property of inertness, plasticity and solvent solubility have made it an ideal candidate for root canal filling. However it has a major disadvantage of not adhering to dentinal walls of the root canal space. This necessitates the use of endodontic sealer mandatory [5,19]

Adhesion of root canal sealers is highly dependent on the wettability of dentin. Wettability is a measure of surface reactivity or adhesiveness of substrate. It can be expressed in terms of angle between the liquid drop and the plane surface of solid on which it rests. This angle is referred to as **contact angle** [4,10,12,14,22,32]. Contact angle has inverse relationship with wettability, that is, the lower the contact angle, the greater the surface free energy and hence the greater the adhesion [8,12,32]

There are a number of factors which influence wettability of endodontic sealers. They are

1. The physicochemical properties of root dentin (surface free energy). [3,6,32]
2. The influence of irrigant solutions (sodium hypochlorite, EDTA, chlorhexidine etc) that are used during the chemomechanical preparation. [2,26,32]
3. The dentin moisture condition and dentinal tubular density. [20]
4. Presence of smear layer due to biomechanical preparation of root canal. [17,29]
5. Hydrophobicity, Hydrophilicity, viscosity of sealers, film thickness, obturation technique and the temperature at which the procedure is performed. [3]

For good adhesion of root canal sealers, wettability [surface free energy] of dentin should be high and contact angle of sealers should be low.

Different endodontic sealers have different contact angle. In the present day, a plethora of sealers are made available in the market, one may find it difficult to choose a good sealer. [33] Every manufacturer claims that their product is the ideal one, but till date none of the sealers have proved to be the ideal except for a few which can come closer to be ideal.

The aim of this present study was to compare and evaluate the contact angle of three endodontic sealers zinc oxide eugenol, AH plus [epoxy resin based] and bioceramic [calcium silico phosphate based] sealers

in the presence and absence of smear layer at 0,5,10,15,20,25,30 seconds after initial application of the sealers with goniometer.

MATERIALS AND METHODS

MATERIALS USED

SEALERS:

1. ZINC OXIDE EUGNOL SEALER (grossman formula)
2. AH PLUS SEALER (DENTSPLY)
3. BIOCERAMIC SEALER (SMART PASTE BIO)

IRRIGANTS:

Distilled water, 3% sodium hypochlorite (PRIME DENTAL PRODUCTS), 2% chlorhexidine (Asep-RC), 17% EDTA (Den tm ark), 2% sterile paper points (Dentsply)

ARMAMENTARIUM

carborundum disk, Laboratory Lathe (Suguna Enterprises), Sand paper (3M ESPE., 1000 grit roughness), Glass slab, Cement spatula, Plastic spatula, K-files (MANI, INC., size 15 - 45), Finger spreaders (MANI, INC., size 15-30),

SPECIAL EQUIPMENTS:

Goniometer (Easy Drop - KRUSS DSA 204)

SAMPLE SIZE

60 decoronated human mandibular premolars were included in this study. 21 decoronated human mandibular premolars were longitudinally sectioned to obtain 42 root dentin samples for contact angle measurement.

SAMPLE SELECTION

INCLUSION CRITERIA:

Single rooted mandibular premolars with single root canal, complete root formation and intact root surface.

EXCLUSION CRITERIA:

Teeth with root resorption, Root canal treated teeth, Teeth with root fracture

STUDY METHOD

PREPARATION AND GROUPING OF THE SAMPLE FOR CONTACT ANGLE MEASUREMENT

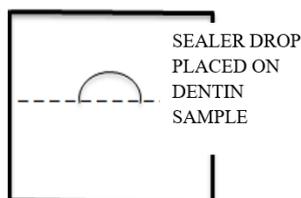
From the stored collection of human mandibular premolars extracted for orthodontic purpose, 21 samples were assigned for contact angle measurement. Working length was determined. Chemomechanical preparation of the specimens was done using K-files till apical size 30, the specimens were irrigated with initial rinse protocol using 5ml of 3% NaOCl between each instrument, then the specimens were decoronated. The remaining root segments were then split longitudinally through the root canal in buccolingual direction using carborundum disk at low speed under water coolant. Buccal and lingual

surface of the samples were further flattened and smoothed using 1000 grit sand paper to obtain smooth and flat surface for contact angle measurement. The samples were then stored in distilled water until use. In one half of the specimens, canal space of the dentin sections were treated with the following irrigation protocol: 5ml of 3% sodium hypochlorite followed by 5ml of 17% EDTA and 5ml of 2% chlorhexidine as a final rinse. The dentin sections were rinsed with each irrigant solution for 1min. The samples were gently air dried before contact angle measurement. The samples were then divided into 3 groups based on the experimental sealers used. A total of 14 samples were assigned for each group with 7 samples per subgroup. The contact angles were measured at varied time intervals of 0.5, 10, 15, 20, 25, 30 seconds in room temperature. The groups were divided as follows

MEASUREMENT OF CONTACT ANGLE OF SEALERS ON DENTIN SURFACE

The samples were mounted flat on glass slab for measurement of contact angle.

In each sample 1 drop [0.1 ml] of sealer [mixed as per manufacture instructions] was deposited on the dentin surface and then with help of software, images of the drop was captured and analysed to provide values of contact angles at time intervals of 0.5, 10, 15, 20, 25, 30 seconds after the initial application of the sealers and the values were recorded.



STATISTICAL ANALYSIS

Kruskal - Wallis and Mann Whitney tests were used for intra and inter group analysis

CONTACT ANGLE MEASUREMENT

The contact angle values (in degrees) were measured using goniometer. The observation revealed that the values decreased upto 30 seconds from the time of application in all the groups.

Table 1 shows the mean standard deviation Contact angle value in degrees for all the groups

Table 2 shows inter group p-value comparison for contact angle measurement in the presence of and after the removal of smear layer.

Table 3 shows intra group p-value comparison for contact angle measurement.

DISCUSSION

For better penetration of sealer and adhesion into dentinal tubules, the wettability should be high. Wettability of sealer is high when the contact angle formed by the sealer on the dentinal surface is low. The moisture content, temperature, the contents present in the dentinal tubules also influences the wettability and sealer penetration. [35]

In the current study contact angle of Zinc oxide eugenol, AH plus and bioceramic sealers were compared. Bioceramic sealer was compared with Zinc oxide eugenol and AH plus sealers because Zinc oxide eugenol sealer has been the gold standard for all the sealers and AH plus sealer is found to be a benchmark amongst the recently introduced resin based sealers. [33]

Middle third of root dentin is used in this study to measure contact angle, as the dentin tubule density is high in this region. From the literature it is evident that the diameter of open dentinal tubules in the middle third of root dentin was large enough to promote deeper sealer penetration. [3,9,35] Additionally greater forces may be expected to be applied during lateral condensation in this part of dentin when compared to coronal and apical third of root dentin. [35] Apical third of the root section may not be ideal to perform the present study because it contains less tubules with smaller diameter and few of them frequently found obliterated. [3]

Irrigation regime used in this study was similar to the clinical situation as recommended by Zehnder 2006 where intermittent rinse with saline was used between the irrigants - 3% sodium hypochlorite, 17% EDTA and 2% Chlorhexidine gluconate. [2,26,32]

It has been proved in the literature that 3% sodium hypochlorite has antimicrobial activity and pulp dissolving capacity, hence it was recommended as a routine irrigant in the irrigation protocol. [1,25] Moreover it also has the collagen dissolving property which enhances the adhesion of smear layer to dentinal wall. Because of this reason subsequent irrigation with 17% EDTA, a chelating agent has been recommended to favour the removal of smear layer. [11,21,29]. Smear layer is defined as layer of irregular, amorphous microcrystalline and organic particle debris that is found on root canal walls and in dentinal tubules after root canal instrumentation. It was first described by McComb and Smith in 1975. It has two layers, superficial layer and deep layer [smear plugs] where the material is packed into dentinal tubules. Smear layer if present will impede the sealer adhesion and penetration by decreasing the wettability of the sealers and also favours microleakage contributing to endodontic treatment failure. [17]

Final rinse was carried out with 2% Chlorhexidine gluconate, a cationic bisbiguanide because of its antimicrobial property that acts by absorbing onto or disrupting the cell wall causing leakage of intracellular components [1], additionally it exhibits substantivity in the root canal for some time after being used as final rinse [25]. It was found from the previous studies that chlorhexidine gluconate when used as the final irrigant increases the surface free energy of dentin and this ensures better wetting, flow and penetration of the endodontic sealers [23].

From the literature search, it was observed that clinical significance of the precipitate (HEDTA)₂(H₂CHX)₃ formed by the interaction of EDTA with chlorhexidine has not been established [23]. From the wettability data obtained in the previous studies (de Assis, Bohn & Ilie), it was confirmed that this precipitate decreases the wettability effect of EDTA. In the current study dentin was irrigated with each irrigant for one minute [31].

The contact angle was measured for 30 seconds (with 5 seconds interval) from the initial application of the sealer to dentin using sessile drop method [30]. This represents the clinical time frame necessary to apply a freshly mixed sealer to the root canal wall prior to obturation (Bohn & Ilie 2014). It is worth mentioning here that contact angle measurement was carried out on a flat dried dentinal surface. Contact angle measurements require a solid surface with a clean, rigid and highly smooth surface [2, 12]. From the previous studies it is known that blot drying dentin with paper points lead to the formation of irregular fluid film on the dentinal surface making the measurement critical [16, 20]. Therefore, in this study dentin discs were gently air dried to create a standard surface condition while maintaining the hydration in the deeper layers of dentin to facilitate the contact angle measurement. The dentin discs were not totally desiccated as it is well known that on desiccation, the exposed collagen matrix can even collapse [24].

Any volumetric change could affect the contact angle value. To avoid this, sealer droplet volume was standardized with micropipette. 0.1 ml of the sealer was used to calculate the contact angle [8, 13, 15, 18, 34]. Room temperature was maintained at 25°C during the measurement of contact angle in air conditioned room [22]. Roughness created by the instrumentation technique also influences the contact angle values [18, 22]. To prevent the influence of the instrument used during the canal preparation, dentin surfaces were smoothed using 1000 grit sand paper.

In the present study dentin sections treated with distilled water was used as control group because of the presence of smear layer in it. All the sealers were mixed individually according to manufacturer instructions before the measurement. Contact angle was measured in the presence and absence of smear layer in all the groups. As the irrigation regime and time was standardized, it was expected that surface free energy of dentin is the same for all the groups and surface tension of the sealer has major influence on the contact values obtained [4, 31].

The contact angle measurement decreased from the time of application to final measurement in all the groups (Table 1, 2). This may be related

to the spreading and setting properties of the sealers used in the study[6].The inter and intra group comparison was highly significant expect for inter group comparison between zinc oxide eugenol and AH plus sealers at 10 and 15 seconds in the presence of smear layer. This may be attributed to the long working time of zinc oxide eugenol and AH plus sealers.

The contact angles measurement obtained in the current study reveals that zinc oxide eugenol has the least values followed by AHplus and Bioceramic sealer for both the test and control groups.From the values obtained (Table 1,2), it was clear that absence of smear layer in the test groups has favoured the wettability which was in accordance with the previous studies[8,12,22,28,35].The results were consistent with the surface tension values of the sealers.The contact angle of bioceramic sealer obtained in the study was high compared to other two experimental sealers which could be attributed to the viscosity and spreading properties of the sealers.

Bioceramic sealer sets in the presence of dentinal tubule fluid.It was also found that Bioceramic sealer exhibited better adaptation to dentinal walls . They produce non resorbable hydroxyapatite during setting reaction forming a chemical bond with dentin walls.This may be the cause for the better adaptation and penetration of bioceramic sealer over zinc oxide eugenol and AH plus sealers observed in the current study.

CONCLUSION

Within the limitations of this in-vitro study, it can be concluded that:

1. Zinc oxide Eugenol sealer showed the lowest **contact angle values** followed by AH Plus and Bioceramic sealer(ZnoEu< AH plus <Bioceramic sealer).
2. For all the sealers, smear layer removal from the canal influenced the reduction of the contact angle

From the current study, it is evident that lower contact angle with smear layer removal improves the wettability of sealers on dentin substrate.The role of other factors like dentin moisture condition and roughness, obturation technique, viscosity, film thickness,capillary action of sealers etc which influence the wettability were not evaluated in the current study. Hence further clinical studies relating to the above factors are required for the identification of ideal sealer to achieve long term endodontic success.

Table1 the mean standard deviation of contact angles in degrees for all the groups

Group	Sealer	Initial Rinse	Storage	Final Rinse
I A	Zinc oxide eugenol	5ml of 3%NaOCl	Distilled water	Distilled water
I B	Zinc oxide eugenol	5ml of 3%NaOCl	Distilled water	5ml of 3%NaOCl 5ml of 17%EDTA &5ml of 2%CHX
IIA	AH plus	5ml of 3%NaOCl	Distilled water	Distilled water
IIB	AH plus	5ml of 3%NaOCl	Distilled water	5ml of 3%NaOCl 5ml of 17%EDTA &5ml of 2%CHX
IIIA	Bioceramic	5ml of 3%NaOCl	Distilled water	Distilled water
III B	Bioceramic	5ml of 3%NaOCl	Distilled water	5ml of 3%NaOCl 5ml of 17%EDTA &5ml of 2%CHX

Table2 Inter group pvalue comparison for contact angle measurement in the presence and after the removal of smear layer

Seconds	Zinc oxide eugenol and AH plus		Bioceramic and zinc oxide eugenol		Bioceramic and AHplus	
	Smear layer	No smear layer	Smear layer	No smear layer	Smear layer	No smear layer
0	0.033	0.002	0.002	0.002	0.002	0.002
5	0.002	0.001	0.001	0.001	0.001	0.001
10	0.18	0.001	0.002	0.001	0.002	0.001
15	0.68	0.013	0.001	0.002	0.002	0.001
20	0.001	0.002	0.001	0.002	0.001	0.002
25	0.001	0.002	0.001	0.002	0.001	0.001
30	0.001	0.002	0.002	0.002	0.001	0.001

p-value <0.05 significant

Table 3: Intra group comparison for contact angle values p-value

sealer	Subgroups	p – value						
		0sec	5sec	10sec	15sec	20sec	25sec	30sec
Zinc oxide eugenol	Irrigant	.000	.000	.000	.000	.000	.000	.000
	Control							
AH plus	Irrigant	.000	.000	.000	.000	.000	.000	.000
	Control							
Bioceramic	Irrigant	.000	.000	.000	.000	.000	.000	.000
	Control							

p-value<0.05 is significant

REFERENCES

1. Akisue, Viviane S.Tomita, GiulioGavini, and Jose Antonio Poli de Figueiredo.Effect of the combination of sodium hypochlorite and chlorhexidine on dentinal permeability and scanning electron microscopy precipitate observation.Journal of Endodontics 2010;36:847-50. 1
2. Attal, Erik Asmussen, Michael Degrange. Effects of surface treatment on the free surface energy of dentin. Dent Mater 1994;10:259-264. 2
3. Balguerie, Lucas van dresluis, Karen valleys, Marie gurgel –georgelin, and Franck diemer.Sealer penetration and adaptation in the dentinal tubules: A scanning electron Microscopic study.Journal of Endodontics2011;37:1576-79. 3
4. Bohn and N.IlieWetting behaviour of silicone – and resin based root canal sealers. International Endodontic Journal2014;47:542-549. 4
5. Branstetter and J.A. Von Fraunhofer The physical properties and sealing action of endodontic sealer cements: a review of the literature. Journal of Endodontics 1982; 8: 126-30.5
6. Chadha, Sonalitanej, Mohitkumar, Sumitgupta.An in vitro comparative evaluation of depth of tubular penetration of three resin- based root canal sealers. Journal of conservative dentistry 2012; 15 :18-21. 6
7. Chandra,Padmanabhan Shankar and Rajamani Indira.Depth of Penetration of Four Resin Sealers into Radicular Dentinal Tubules A Confocal Microscopic Study. Journal Of Endodontics 2012;38:1412-1416. 7
8. DeAssis,Maira do Prado and RenataA.Simao.Evaluation of the Interaction between Endodontic Sealers and Dentin treated with Different Irrigant Solutions. Journal Of Endodontics 2011;37:1550-1552. 8
9. De-Deus,Karina Di Giorgi and Rival Antonio. Interfacial adaptation of Epiphany self – adhesive sealer to root Dentin.Oral surgery, oral medicine, oral pathology,oral radiology and Endodontology2011;111:381-386. 9
10. Dilipkumar,Shafath Ahmed,M.Dhivya.Comparison of efficiency of various cleansing techniques on dentin wettability using contact angle test. Indian Journal Of Interdisciplinary Dentistry 2012; 1:250-254. 10
11. Dotto, Rosanamaria Coelho travassos, Elias pandonortmocy de olivira, ManoelDurado de lima machado and Jose luiz martins.Evaluation of ethylenediaminetetraacetic acid (EDTA) solution and gel for smear layer removal.Australian Endodontic Journal 2007;33: 62-65. 11
12. Extrand. Contact angles and their hysteresis as a measure of liquid –solid Adhesion.Langmuir 2004;20:4017-4021. 12
13. Good and M.N.Koo.The Effect of Drop Size on Contact Angle.Journal of Colloid and Interface Science 1979;71:283-292.13
14. Hu,Junqi Ling and Yan Gao.Effects of Irrigation Solutions on Dentin Wettability and Roughness. Journal Of Endodontics 2010;36:1064-1067. 14
15. Iwanami, Takatomo Yoshika, Mitsuhiro Sunakawa, Chihiro Kobayash. Spreading of root canal irrigants on root dentine. Australian Endodontic Journal 2007;33:66-72. 15
16. Khorasani, H.Mirzadeh, Z. Kermani.Wettability of porous polydimethylsiloxane surface morphology Study.Applied Surface Science 2005;242:339-345. 16
17. Kokkas,Asteriosch,Boutsioukis, Leonidas P.Vassiliadis and Christos K.Stavrianos. The influence of smear layer on the Dentinal Tubule Penetration Depth by Three Different Root Canal Sealers:An In Vitro Study. Journal Of Endodontics 2004;30:100-102. 17
18. Kontakiotis, Giorgos N. Tzanetakis and Alexios L.Loizides.A comparative study of contact angles of four different root canal Sealers. Journal Of Endodontics 2007;33(3):299-302. 18
19. Lee, Michael C. Williams, Jean J.Camps and David.Pashley.Adhesion of Endodontic Sealers to Dentin and Gutta-Percha. Journal Of Endodontics 2002;28:684-688. 19
20. Nagas, M.OzgurUyanik, AyhanEymirli, Cehereli ,PekkaK. Vallittu,Lippo V.J. Lassila and VelDurmaz. Dentin Moisture Conditions Affect the Adhesion of Root Canal Sealers. Journal Of Endodontics 2011:1-5. 20
21. Nakashima and Rintaro Terata. Effect of pH modified EDTA solution to the properties of Dentin. Journal Of Endodontics 2005;31:47-49. 21
22. Danielle Ferreira de Assis, Brenda P.F.A Gomes and Renata A.Simao.Effect of disinfectant solutions on the surface free energy and wettability of filling material. Journal Of Endodontics 2011;37:980-982. 22
23. Rasimick, Michelle Nekich, Megan , Barry L. Musikant, and Allan S. Deutsch.Interaction between Prado chlorhexidinedigluconate and EDTA. Journal of endodontics 2008; 34:1521-3. 23
24. Rosales, G.W. Marshall, S.J. Marshall, L.G.Watanabe ,M. Toledano, M.A. Cabrerizo and R.Osorio.Acid –etching and Hydration influence on dentin roughness and Wettability.J Dent Res 1999;78:1554-1559. 24
25. Rosenthal, LaizSpanberg and Kamran Safari .Chlorhexinesubstantivity in root canal dentin. Oral surgery, oral medicine, oral pathology,oral radiology and Endodontology2004; 98:488-92. 25
26. Saleh, I.E.Ruyter, M.Haapasalo and D.Ostavik. The effects of den tine pretreatment on the adhesion of root –canal sealers. International Endodontic Journal 2002;35:859-866. 26
27. Sen, B. Piskin & N.Baran.The effect of tubular penetration of root canal sealers on dye Microleakage. International Endodontic journal -1996; 29: 23-28. 27
28. Shafrin and William A.Zisman.Constitutive relations in the wetting of low energy surface and the theory of the retraction method of preparing monolayers.1960;64:519-524. 28
29. Shahravan, Aliakbar Haghdooost, Alireza Adl, Hessamrahimi and Fahimehsh adifa r.Effect of smear layer on sealing ability of canal obturation : A systematic review and meta –analysis. Journal of endodontics 2007; 33:96-105. 29
30. Shang, Markus Flurry James B.Harsh, Richard L.Zollars.Comparison of different methods to measure contact angles of soil colloids. Journal of Colloid and Interface Science 2008;328:299-307. 30
31. Teixeira, M.C.S.Felippe and W.T.Felippe. The effect of application time of EDTA and NaOCl on intracanal smear layer removal :an SEM analysis. International Endodontic Journal 2005;38:285-290. 31
32. Tuncer and Safa Tuncer. Effect of Different Final Irrigation Solutions on Dentinal Tubule

- Penetration Depth and Percentage of Root Canal Sealer Journal Of Endodontics 2012;38:860-863. 32
33. Tyagi,Priyesh Mishra,ParimalaTyagi.Evolution of root canal sealers: an insight story. European Journal Of General Dentistry 2013;2:199-218. 33
34. Vafei,M.Z.Podowski.Analysis of the relationshipbetween liquid droplet size and contact angle. Advances in colloid and Interface Science 2005;113:133-146. 34
35. Vassiliadis,Spyros A. Sklavounos and Christos K.Stavrianos. Depth of Penetration and Appearance of Grossman Sealer in the Dentinal Tubules:An In Vivo Study.Journal OfEndodontics 1994;20: 373-376. 35
36. Wu,A.J.Degee and P.R.Wesselink.Effect of tubule orientation in the cavity wall on the seal of dental filling materials :an in vitro study.International Endodontic Journal 1998;31:326-332. 36