ISSN No:-2456-2165

Adhesive Strength of Endodontic Sealer Epoxy Resin with Addition of Nano Chitosan High Molecular in Root Canals

Gari Wijaya[1] ,Trimurni Abidin[1], Dennis,[1] Harry Agusnar,[2] Basri A. Gani[3]
[1] Department of Conservative Dentistry, Faculty of Dentistry, Universitas Sumatera Utara Medan, Indonesia
[2]Departemenof Chemistry, Faculty of Mathematics and Natural Science, Universitas Sumatera Utara, Medan, Indonesia
[3.] Department of Oral Biology, Dentistry faculty, Universitas Syiah Kuala, Darussalam, Banda Aceh, Aceh, Indonesia

Abstract:- Root canal treatment is performed to repair and increase the resilience of the tooth. Using a suitable sealer material can increase penetration and adhesion to the root canal wall. Epoxy and chitosan resins have been reported to act as endodontic sealers with good penetration and adhesive properties. The evaluation of the penetration and adhesion properties of epoxy resin sealer and nano chitosan high molecular on the tubules dentin in root canal. The absorption and penetration assay of sealer used to the methylene blue dye assessment, while the confirmation the adhesive strength using SEM (Scanning electron microscope). The addition of chitosan nanoparticles 1% allowed the epoxy sealer group to have better penetration and absorption capabilities in tooth root canals. Inaddition, this group increased the adhesion of the root canal wall compared to other groups because it increased its density and adapted well to the entire dentine tubules on the root canal wall. The sealer penetration value was significantly different between the treatment groups ($p<0.005$);0.001) with a strong relationship ($r=0.89$). Epoxy resin sealer with the addition of 1% chitosan high molecule nanoparticle has excellent absorption and penetration properties in root canal walls, increasing adhesion to root canal walls.

Keywords:- *Adhesive, Chitosan, Root Canal, Sealer Endodontic.*

I. INTRODUCTION

Root canal treatment highly depends on the endodontic triad (Access, Clean & Shape, Obturation). Root canal treatment has a high level of difficulty because it is difficult to observe the shape of the root canal. At the filling stage of the root canal system, guttapercha is used as the primaryobturation material, which is applied with an endodontic sealer, which functions as a material to cover the gap that exists between the guttapercha and the treated root canal (Tabassum and Khan, 2016). The density between the sealer and the root canal wall is significant to know microscopically because the success of root canal treatment is also influenced by the sealing ability of the filling material to the dentine tubules of the root canal to binding of dentine collagen and its penetration in dentine tubules (Mamootil and Messer, 2007).

In endodontic treatment, root canal obturation/filling aims to prevent recontamination of root canals filled with guttapercha as the primary filling material and endodontic sealer (Jin et al., 2021). The endodontic sealer acts as a lubricant and assists in compacting the filling by bonding the guttapercha to the dentinal walls and filling any voids that the primary filling material cannot load. The success of endodontic treatment in the long term depends on the solid filling after root canal obturation (Al-Askary et al., 2013). The failure of sealer penetration can lead to the development of *Enteroccus faecalis, fusobacterium nucleatum, Actinobacillus actinomycetemcomitans,* which can interfere with sealer density and endodontic treatment failure (Huang etal., 2018 and Darmawi et al. 2022).

Dimensional changes and failure of sealer adhesion with guttapercha are the main problems in increasing the reliability of the root canal surface. Therefore, the adaptability of sealers to dentin is a significant factor in micro fissures and root canal reinfection (Russell, 2017). Roth (2011) reported that sealers based on epoxy resin did not experience volumetric shrinkage (shrinkage) during the polymerization process compared to sealers based on methacrylate. Epoxy sealers have disadvantages such as polymerization that lasts 24-36 hours, contains silver powder, causes tooth discoloration, and is highly cytotoxic to fibroblast cells (Roth, 2011).

Chitosan is a natural ingredient consisting of glucosamine and N-acetyl-glucosamine. In general, Chitosan is biocompatible, non-toxic, biodegradable, and antibacterial. In dental conservation, high molecular Chitosan has been developed, which has been applied to reversible pulpitis, antibacterial, and endodontic regeneration (Adiana et al., 2018). Sutrisman et al. (2013) reported the addition of high molecular chitosan nanoparticles with a weight of 0.015% w/v in the glass ionomer variant (SIKMR and SIKMRn) was able to increase the attachment between the material to dentin (Sutrisman et al., 2013).

The application of an epoxy resin sealer is expected to increase penetration into the dentinal tubules and seal all ramifications and dentinal tubules in the apical 1/3 of the root. The combination of an epoxy resin sealer with the addition of high molecular chitosan nanoparticles is expected to increase the adhesive power to the root canal wall.

ISSN No:-2456-2165

II. MATERIAL AND METHODS

A. Research Materials

The study used premolar Teeth and Epoxy resin as a sealer material. The divided in three groups (Sealer Epoxy resin (AH Plus) as control, Sealer Epoxy resin + nano chitosan high molecule 1%, and sealer epoxy resin + nano chitosan high molecule 2%. Also used to the irrigation of NaOCl 2,5 % + EDTA 17% as control, Irrigation of NaCl 2.5 % + nano chitosan hight molecule 0.1%, Irrigation of NaCl 2.5 % + nano chitosan hight molecule 0.2%. These materials were then assessed for absorption and penetration properties inthe dentine tubules of the root canal. It is a reference to measure the quality and quantity of potential chitosan nanoparticles as a sealer in endodontic treatment.

B. Preparation sample of tooth

A total of 30 mandibular premolars that had been extractedwere immersed in saline solution and divided into three groups, Group A (epoxy resin sealer with irrigation of NaOCl 2,5 % + EDTA 17%, B (epoxy resin sealer with 1% Chitosan nanoparticles and Irrigation of NaCl 2.5 % + nano chitosan hight molecule 0.1%.), C (epoxy resin sealer with 2% Chitosan nanoparticles and Irrigation of NaCl 2.5 % + nano chitosan hight molecule 0.2%.. Each group consisted of 6 dental samples. Furthermore, all samples were cut with a disc burr onthe crown of the teeth and the cementoenamel junction boundary with the help of a bais. Next, endodontic treatment modeling was carried out. Each sample was measured fortooth length to determine the working distance. Root canals were negotiated to apical patency and working length using file #10. Then, the initial irrigation of 5 mL of irrigation solution was carried out. Then The root canal was prepared using a crown down technique using a Protaper Gold rotary instrument file and an endo motor speed of 300 rpm. The root canal preparation was started with file S1, followed by files S2, F1, and F2 according to the working length. 3 mL, while for the final irrigation, it is given 5 mL of irrigation solution for 1 minute. Then the root canals were dried using paper points. Furthermore, according to a predetermined group, the root canal was obturated with guttapercha and a sealer (Sornkul and Stannard, 1992).

C. Sealer Penetration Assay

The sealer penetration test into the dentinal tubules used a dye approach with 2% Methylene Blue. The samples made endodontic treatment. The teeth were coated with nail polish twice over the entire sample surface, except for 1mm at the apical portion. The samples were put into test tubes according to their respective groups, adding 5 mL of 2% Methylene Blue. Then incubated at 37 0C for 48 hours and allowed to stand for 15 at room temperature. A total of 150 L of dye was put in a 96 Well Plate, then examined at 515 nm OD spectrophotometry. Methylene Blue color change indicates dye penetration activity in dentinal tubules (Souza et al., 2009).

D. Scanning Electron Microscope of Root Canal

Examination of the adhesive sealer on the root canal wall was examined by SEM (Scanning Electron Microscope). The treatment started by cutting the tooth crown longitudinally from the cementoenamel junction to the tip of the apex. The sample was cleaned to remove dirt and form a good sample surface structure, then polished. Afterward, the samples were examined using an SEM tool (Hitachi TM 3000, Japan). The samples were placed in a vacuum chamber in the SEM and performed at 1200x, 1500x, and 2000x magnification. For 1500x magnification, the image will be divided into nine observation areas assessed using the scoring method through double-blind observations by two different people. They measured the level of density (adhesive) of the sealer on the root canal wall (Virdee et al., 2018). Qualitative data from SEM images were analyzed descriptively.

E. Statistical Analyses

The absorption and penetration data between the epoxy and chitosan nanoparticle sealer groups were analyzed by a Simple T-test. The comparison data between the treatment groups were examined by One Way Anova with a limit of the significance of $p<0.05$ and $r=1$, and there was a strong relationship.

III. RESULTS AND DISCUSSION

In Figure 1, it is shown that the epoxy sealer group with the addition of 1% chitosan nanoparticles had better adhesion to the root canal than the other groups. The indication is that there is an increase in the penetration of the sealer in the root canal and a decrease in the absorption value. The reduction in sealer absorption indicates that the sealer penetration into the root canal has been evenly distributed.

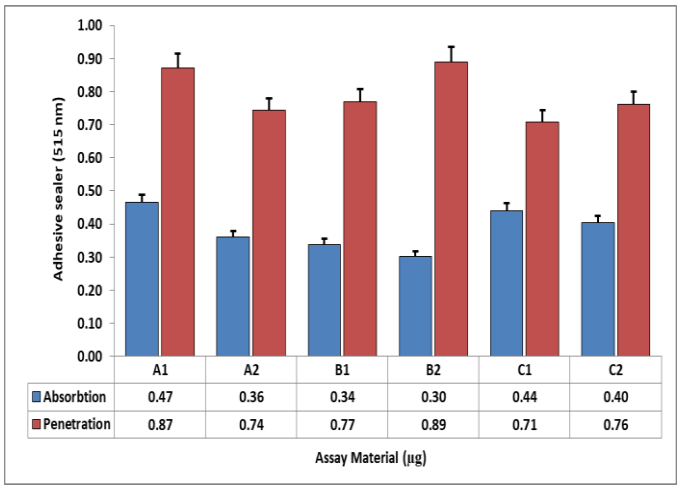

Fig. 1:- The adhesive power of sealer and chitosan nanoparticles in root canals. The absorption and penetration of methylene blue in the root canal as a reference for the adhesive strength of the sealer in the root canal. A (epoxy resin sealer), B (epoxy resin sealer with 1% Chitosan nanoparticles),C (epoxy resin sealer with 2% Chitosan nanoparticles). Bar (Adhesive sealer) Bar Error (Standard error)

Figure 2 shows the sealer density. Line 1, (a) sealer and (b) residual smear layer. Both images show the sealer density with the root canal wall and the presence of a residual smear layer. Line 2, (a) SEM profile of root canal density sealer with the addition of 1% chitosan, which adapts well to the entire

root canal wall. Line 3, (a) remaining guttapercha attached to the sealer with 2% chitosan. (b) microleakage between sealer with 2% chitosan on the root canal wall. Based on the adhesion, the sealer with adding 1% chitosan had better adaptability in increasing the adhesive on the root canal.

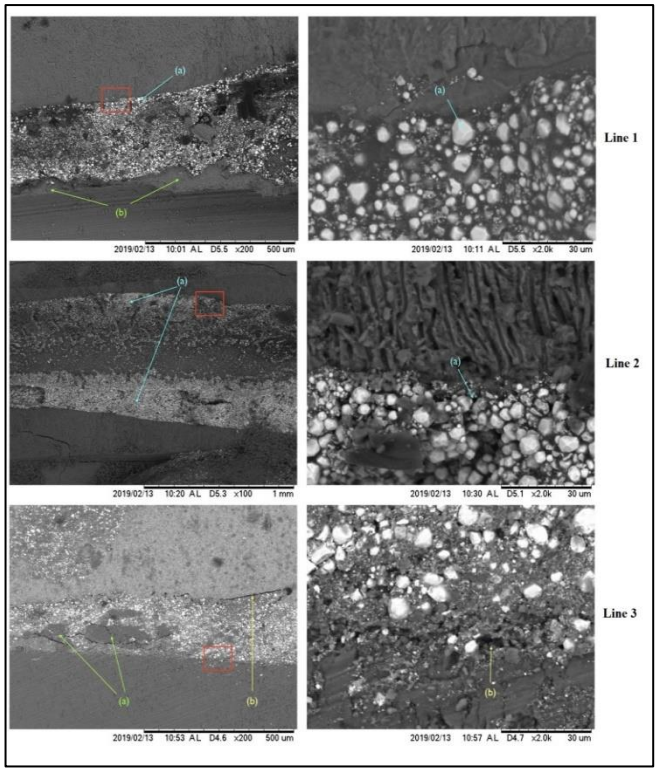

Fig.2:- SEM profile of adhesive sealer in root canals. Line 1 (Root canal with epoxy sealer). Line 2. (Root canals treated with epoxy sealer with 1% Chitosan nanoparticles). Line 3 (Root canals treated with epoxy sealer with Chitosan nanoparticles 2%). 200x (left) and 1500x (right) magnification

Based on the sample test, the T. test showed a significant difference in sealer absorption ($p<0.05$; 0.04) and sealer penetration ($p<0.05$; 0.003). The intensity of absorption and penetration has a relatively strong relationship ($r=0.79$). One Way Anova analysis data between the absorption and penetration groups had a significant relationship ($p<0.005$;0.001) with a strong relationship ($r=0.89$).

This study evaluated the potential of high molecular chitosan nanoparticles combined with epoxy resin as a sealer in root canal treatment. Chitosan has been applied in dentistry as an antibacterial, anti-oxidant, and adhesive in tooth root canals. (Gutmann et al., 2010) Its use as a sealer in obturation treatment has become the choice of researchers to compare its effectiveness with sealers. Epoxy resins that are often used in dentistry, such as AH plus

In this study, it was reported that the epoxy sealer group added with 1% chitosan nanoparticles could increase the attachment to the root canal better, which indicated an increase in the penetration of the sealer in the root canal and a decrease in the absorption value of the sealer. (Image 1). Absorption of sealers and other materials is absorption which means the uptake or transfer of substances into or through tissues or tubules, such as the biological removal of an extruded sealer (Fisher et al., 2008). The increase in sealer activity on the material depends on the strength of the absorption power of the sealer material when interacting with the adhesion opponent. The absorption of the sealer material is highly dependent on changes in time and the content of the adhesive compound. In addition, sealers play an essential role in repairing root canals of teeth (Skrtic and Antonucci, 2016).

Kim (2010) reported that using root canal sealers to provideadhesion can fill irregularities and minor differences between the core filling material and the root canal wall. In addition, the sealer also acts as a lubricant during the absorption adaptation process (Jang et al., 2010). Henston (2012)revealed that root canal sealers and other materials have the main determinants of adhesion, where each sealer. Its advantages and disadvantages. Like AH, plus, zinc oxide eugenol is the most commonly used sealer and is the standard in many studies for comparison with other sealers (Henston et al., 2012). The penetration of the sealer into the dentinal tubules can indicate the degree of smear layer shedding (Ernani et al., 2015 Puspita et al., 2017). The ability of the sealer to penetrate the dentinal tubules depends on many factors, such as humidity and temperature. In addition, the presence of a smear layer can serve as a barrier and prevent the penetration of the sealer into the dentinal tubules (Turner-Walker, 2008).

In this study, a root canal sealer containing a mixture of Chitosan and AH Plus resin was found to increase the penetration in dentin tubules of the root canal. General conditions such as Chitosan's pH, Chitosan, the chemical structure of Chitosan, and the type of ion can determine the interaction with other solutions (Aranaz et al., 2009). Chitosan has been used to repair bone and has proven to be one of the most promising dental biomaterials (Cheung et al., 2015). Another study on Chitosan in dentistry has been shown to reduce the microhardness of dentin when used as an irrigating solution in root canals, and the effect is more significant at higher concentrations (Ernani et al., 2015; Praveenet al., 2017).

The research results reported in Figure 2 are to study and observe the density and strength of the sealer, smear layer, microleakage, and root canal wall profile. The research results in the figure show that the AH Plus sealer can provide good density, but the SEM profile shows the presence of a smear layer. The smear layer that is formed is generally the remnant of the sealer interaction process with the dental material on the root canal wall because AH plus is not biodegradable, so the residual material tends to be scattered throughout the root canal wall with different morphological forms (Debelian and Trope, 2003). 2016). Endodontic irrigation influenced the effect of the smear layer development. The produce smear layer will help the growth of pathogenic bacteria and thus thwart root canal obturation (Zargar et al., 2015).

Ormiga (2016) and Rasendren (2021) compared the AH plus product withEndoREZ, both used as sealers in endodontic therapy. In contrast, EndoREZ presented a higher ability to fill the root canal system in conjunction with guttapercha when compared to AH Plus and Pulp Canal Sealer. (Ormiga et al.,

2016), but the addition of Chitosan can increase the strength of the push-out guttapercha bond ($p<0.05$). (Aydin et al., 2016) From the results of the study, it was found that AH plus (epoxy resin) had a failure of adhesion and the formation of a sear layer associated with its attachment to the dentinal root (Fig. 2). the polymerization of the sealer interface sealer on the root canal wall may have been affected by oxygen which can inhibit the polymerization of the vinyl in the composite resin and 40-60% of the carbon bonds remain unsaturated (Franco et al., 2002).

Nunes (2008) reported that higher sealer flow would allow more significant contact with the target material (primary) and hence more outstanding mechanical interlocking with dentin (Nunes et al., 2008). An SEM study showed that primers were found in all thirds of the root canals (cervical, middle and apical), which reinforces the assertion that failures noted at the sealer/dentine interface were more likely to be related to the sealer and not tothe primary application (Tay et al., 2005). . In this study, Chitosan has an excellent effect as a sealer, this has something to do with its excellent biocompatibility, biodegradability, non-toxicity, and also has bio-adhesion (Silva et al., 2013).

IV. CONCLUSION

Epoxy resin sealer with the addition of 1% chitosan high molecule nanoparticle has excellent absorption and penetration properties in root canal walls, increasing adhesion to root canal walls.

ACKNOWLEDGMENT

The author extends gratitude to Laboratorium Oral Biology Dentistry Faculty, Universitas Syah Kuala, Darussalam, Banda Aceh, Aceh, Indonesia.

REFERENCES

[1]. Adiana ID, Abidin T and Syafiar L.Effect of High Molecular Nano-Chitosan Addition on The Impact Strength of Heat Polymerized Polymethyl Methacrylate Denture Base Resin, in International Dental Conference of Sumatera Utara 2017 (IDCSU 2017) pp 127-129, Atlantis Press.
[2]. Al-Askary RA, Ismail SA, Al-Sabawi NA. Bond strength of endodontic sealers to gutta percha and dentin. Al-Rafidain Dental Journal 2013;13:282-289.
[3]. Aranaz I, Mengíbar M, Harris R, Paños I et al. Functional characterization of chitin and chitosan. Current chemical biology 2009;3:203-230.
[4]. Aydin U, Aksoy F, Tosun S et al. The effect of combined use of Chitosan and PIPS on push-out bond strength of root canal filling materials. Journal of adhesion science and Technology 2016;3**0**:2024-2031.
[5]. Cheung R, Ng T, Wong J Chan W. Chitosan: an update on potential biomedical and pharmaceutical applications. Marine drugs 2015;13:5156-5186.
[6]. Imelda Darmawi, Trimurni Abidin, Harry Agusnar, Basri A. Gani. In Vitro Study of Irrigation solution of Chitosan Nanoparticles to Inhibit the Adhesion and Biofilm Formation of Enterococcus faecalis in the Root Canal. Research Journal of Pharmacy and Technology 2022; 15(6):2691-6.
[7]. Debelian G and Trope M The use of premixed bioceramic materials in endodontics. Giornale Italiano di Endodonzia 2016;30:70-80.
[8]. Ernani E, Abidin TM, Indra I. Experimental comparative study and fracture resistance simulation with irrigation solution of 0.2% chitosan, 2.5% NaOCl and 17% EDTA. Dental Journal (Majalah Kedokteran Gigi) 2015;48(3):154-8.
[9]. Fisher JS, Ahari F, Hronowski LJ. Biodegradable polymer for marking tissue and sealing tracts, Google Patents 2018;18;29
[10]. Franco EB, Lopes LG, D'alpino PH et al. Evaluation of compatibility between different types of adhesives and dual-cured resin cement. Journal of Adhesive Dentistry 2002; 4:12-16.
[11]. Gutmann JL, Kuttler S, Niemczyk SP. Root canal obturation: An update. Academy of Dental Therapeutics and Stomatology, a division of PennWel 2010;2010
[12]. Henston J, Sarma N, Subhash C. Root Canal Sealers & its Role in Successful Endodontics-A Review. Annals of Dental Research 2012;2:68-78.
[13]. Huang Y, Orhan K, Celikten B, Orhan AI et al. Evaluation of the sealing ability of different root canal sealers: a combined SEM and micro-CT study. Journal of Applied Oral Science 2018;26:15-25.
[14]. Jang J-A, Kim H-L, Her M-J et al. effect of moisture on sealing ability of root canal filling with different types of sealer through the glucose penetration model. Journal of Korean Academy of Conservative Dentistry 2010;35:335-343.
[15]. Jin H-R, Jang Y-E, Kim Y. Comparison of Obturation Quality between Calcium Silicate-Based Sealers and Resin-Based Sealers for Endodontic Re-treatment. Materials 2021;15:72.
[16]. Mamootil K, Messer H. Penetration of dentinal tubules by endodontic sealer cements in extracted teeth and in vivo. International endodontic journal 2007;40:873-881.
[17]. Nunes VH, Silva RG, Alfredo E et al. Adhesion of Epiphany and AH Plus sealers to human root dentin treated with different solutions. Brazilian Dental Journal 2008;19:46-50.
[18]. Ormiga F, Ferreira de Assis D, de Andrade Risso P. Ability of Three Endodontic Sealers to Fill the Root Canal System in Association with Gutta-Percha. The open dentistry journal 2016;10:12-18.
[19]. Puspita A, Prawati G, Fatimah I. Chitosan-modified smectite clay and study on adsorption-desorption of urea. Chemical Engineering Transactions 2017 56:1645-50.
[20]. Praveen M, Aarthi G, Meenapriya P, Kumar SS et al. A comparative evaluation of intraradicular smear removal efficacy of 2% chitosan (low molecular weight), 4% Chitosan Citrate, and 10% Citric Acid when Used as Final Rinse in Irrigation Protocols: A Field Emission Scanning Electron Microscopic Study. Journal of pharmacy & bioallied sciences 2017;9:S73.

[21]. Rasendren K, Abidin T. Effect of 0.2% chitosan high molecule nanoparticle on the push-out bond strength of resin cement in post-restoration (An in-vitro study). Journal of Syiah Kuala Dentistry Society 2021;6(2):75-80.
[22]. Roth KA. Proliferation of pathogenic biofilms within sealer-root dentin interfaces is affected by sealer type and aging period, University of Toronto 2011.p.12-19
[23]. Russell A. The Butterfly Effect: An investigation of sealer penetration, adaptation and apical crack formation in filled root canals, University of Otago 2017;p25-35.
[24]. Silva P, Guedes DFC, Nakadi FV et al. Chitosan: a new solution for removal of smear layer after root canal instrumentation. International endodontic journal 2013;46:332-338.
[25]. Skrtic D, Antonucci JM Polymeric dental composites based on remineralizing amorphous calcium phosphate fillers. Current trends in polymer science 2015;17:1-10
[26]. Sornkul E, Stannard JG. Strength of roots before and after endodontic treatment and restoration. Journal of Endodontics 1992;18:440-443.
[27]. Souza EM, Pappen FG, Shemesh H et al. Reliability of assessing dye penetration along root canal fillings using methylene blue. Australian Endodontic Journal 2009;2009;35:158-163.
[28]. Sutrisman H, Abidin T, Agusnar H. Effect of additional high molecular chitosan nanoparticles on cement nanoparticle glass ionomers on the survival ability of pulp cells: effect of high molecular chitosan nanoparticle addition in gic ellnoparticle to viabilsity of pulp. Dentika: Dental Journal 2013;17:255-259.
[29]. Tabassum S, Khan FR. Failure of endodontic treatment: The usual suspects. *Eur J Dent* 2016;10:144-147.
[30]. Tay FR, Loushine RJ, Weller RN, Kimbrough WF, Pashley DH, Mak Y-F, Lai C-NS, Raina R and Williams MC (2005) Ultrastructural evaluation of the apical seal in roots filled with a polycaprolactone-based root canal filling material. *Journal of endodontics* **31**:514-519.
[31]. Turner-Walker G. The chemical and microbial degradation of bones and teeth. Advances in human palaeopathology 2008;592:20-35