The effect of etching techniques on cement penetration into dentinal tubules on fiber post cementation

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Abstract *Objective:* A combination of self-etch and additional etch techniques as pre-cementation procedures for fiber post restoration was reported able to increase the bonding of adhesive material. This research aimed to determine the effects of different etching techniques on resin cement penetration into dentinal tubules.

Materials and methods: Thirty-two endodontic treated premolars were prepared for 10 mm fiber posts and were divided into two groups (n = 16). Group one was treated with self-etch only, and the second was treated with a combination of self-etch and additional etching. The fiber posts were cemented using fluorescent rhodamine B 0.1%-colored resin cement. The samples were cut to a 2 mm thickness in the middle third of the root and were evaluated with confocal laser scanning microscopy (CLSM). Statistical analysis measurements were analyzed by an independent *t*-test and Pearson correlation test.

Results: There were statistically different in densities hybrid layer and resin tag penetration lengths. Statistically strong linear positive correlations between the hybrid layer density and resin tag penetration length in both groups were also observed. Hybrid layer density (9.68 μm), resin tag penetration depth (32.51 μm) in the self-etch group with additional etching were higher than in the other group. The Pearson correlation test results between hybrid bond density and resin tag penetration depth in the self-etch treatment group showed a value of r = 0.634 and p-value = 0.008 and in the etching group self-treatment with additional etching a value of r = 0.516 and p-value = 0.041 (p ≤ 0.05).

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Conclusion: There is a statistically significant positive correlation or linear relationship between hybrid bond density and the penetration depth of resin tags in both treatment groups: the thicker the hybrid layer density, the deeper the penetration of the resin tags. This phenomenon may increase mechanical strength regarding stronger binding of resin cement to a tooth's substrate.

1. Introduction

Endodontically-treated teeth has higher cusp fracture incidents and also cusp deflection during functional activity. Intraradicular posts and cores are required to protect the crown margins from functional deformation. Therefore, the use of intraradicular posts is necessary to increase the retention of restorations due to the quite extensive loss of tooth crown structure (Putignano et al., 2007; Tabassum & Khan, 2016; Chen et al., 2018).

Fiber posts is cemented to inter-radicular dentin via adhesive cementation creating micromechanical bond that formed by hybrid bonds and resin tags. The hybrid bond consists of an interdiffusion of liquid resin in demineralized dentine collagen fibers which the resin replace the hydroxyapatite crystals that have been removed due to the etching process. The resin tags are bonds formed by the diffusion of resin monomers into dentinal tubules (Chen et al., 2018; Machado et al., 2015; Sophia et al., 2016). The adhesive of root canal cement is obtained through an adhesive bond formed between a fiber post and resin cement by polymerization process of matrix resin (K Bitter et al., 2004).

The adhesion ability of an adhesive system can be analyzed by assess the micromechanical bonds formed which consist of hybrid bonds and resin tags. According to Ramya (2014), hybrid bonding and resin tags can increase retention and bond strengths (Lone et al., 2019). Therefore, it was necessary to conduct research using rhodamine B fluorescent dyes on adhesive material from post cementation to be assessed with confocal laser scanning microscopy (CLSM).

Many previous studies were conducted without obturation of the root canal. Any residual effects of gutta percha and sealer on microleakage of the post system were, therefore, not considered. This is important because many endodontic sealers contain eugenol, which has been shown to inhibit resin polymerization (Kahtani, 2010). The post preparation process will produce a secondary smear layer consisting of the remaining sealer and the gutta percha that became solidified due to the friction generated by the heat from the drill. The rest of the sealer and gutta percha also cannot be completely cleaned from the root canal walls; due to the cross section of oval-shaped root canals, post drills cannot shape and clean the entire surface of the root canal and thus chemical cleaning is required. Bitter (2006) reported that the use of 37% phosphoric acid was able to clean the maximum smear layer of the dentin wall, open dentinal tubules, collagen fibrils, and interfibrillar spaces, and thereby help increase the penetration of adhesives (K Bitter et al., 2004; Marigo et al., 2012; Scotti et al., 2012; Kerstin Bitter et al., 2006).

According to Martinho (2014), in a self-etch adhesive system, primary acidic material containing etching and primer proved ineffective in demineralizing dentin root canals; conse-quently, optimal bonding between the wall surface of dentin root canals and adhesive material cement resin was not achieved (Martinho et al., 2014). Hence, this research was conducted to observe whether the addition of etching to the self-etch system can help increase hybrid bonding and the penetration of resin tags on fiber post cementing.

2. Materials and methods

2.1. Tooth selection and preparation

This research used experimental laboratory testing to analyze the penetration of resin cement into the dentinal tubules at fiber post cementation by assessing the hybrid bond density and resin tag depth. This research was conducted on 32 samples divided into two groups; a total of 32 extracted human single-rooted first mandibular premolar teeth were collected with the approval of the Dental Research Ethics Committee of Dental Faculty Universitas Indonesia (ethical clearance no: 128/ethical approval/FKGUI/XII/2019). In the first group, the fiber post cementation was carried out using a self-etch adhesive system while the second group used a self-etch adhesive system with additional etching. Hybrid bond density and resin tag penetration depth were viewed using CLSM to observe the average fluorescence intensity (MFI), which was calculated automatically by ZEN 2010 software in units of μm and then compared between the two groups.

The inclusion criteria were that the tooth root was fully formed, tooth length was 20 ± 2 mm, the tooth root was straight and macroscopically intact, and the root canal shape in the middle third was round after post preparation.

Preparation was carried out using a NiTi *rotary non*-ISO (ProTaper Next, Dentsply) rotary instrument with Endomotor (X-mart, Denstply), according to a working length up to file X4 (Sophia et al., 2016). Each instrument was always lubricated with 17% EDTA gel (RC Prep), and every instrument change was always irrigated with 2.5% NaOCl and dried with paper points (Diadent). Root canal filling was carried out using lateral condensation techniques employing an epoxy-based resin sealer (AH-Plus, Denstply) that was manipulated according to the manufacturer's recommendations.. All samples were incubated at 37 °C in 100% humidity for 7 days for the setting of the root canal sealer.

2.2. Filling removal

Post core preparation was conducted by removing gutta percha using a Gates Glidden drill, leaving 5 mm of gutta percha at the apical end. A post drill was used in accordance with the shape and diameter of the fiber post (Ivoclar), producing a post chamber with a diameter of 1.5 mm in the coronal area. The root canals were irrigated with EDTA 17% 5 mL (MD

Cleanser, Meta Biomed) and rinsed with aquabidest 5 mL and then CHX 2% 5 mL. Each irrigation was carried out for 1 min, after which the irrigation fluid was activated by ultrasonic tip (Irrisafe, Acteon) and then dried with paper points.

2.3. Sample preparation and CLSM evaluation

In this study we used self etch cementation (Multilink N, Ivoclar). Group 1 (self-etch): The primers and bonding agents were mixed in a ratio of 1:1, and then applied to the root canal wall using a microbrush, left for 30 s and dried with a paper point. Next, 1 mm long resin cement was placed on a paper pad and mixed with 0.1% rhodamine B fluorescent dye, after which resin cement was inserted into the root canal and applied to the fiber post. The fiber post was inserted and polymerized with light for 10 s, and the excess post was cut with a bur.

Group 2 (self-etch with additional etching): A 37% phosphoric acid etching solution was applied to the root canal wall for 15 s with a microbrush, aquabidest (as much as 5 mL) was used as irrigation, and paper points were used for drying. Subsequently, the same treatments as on Group 1 were performed.

All teeth were implanted in an acrylic resin block (Technovit 4071, German) in a vertical position. The teeth were then cut in a horizontal plane perpendicular to the root axis using a water-spray cutting machine (Struers Accutom 2). Tooth cutting was done at the point between 7 mm and 11 mm from the apical root, and 2 sample segments were obtained with a thickness of 2 mm per segment, which represents the one-third middle area of the root canal. The samples were dried, and aluminum foil was used to protect the samples from light. Each sample was observed using CLSM (LSM 700, Carl Zeiss Microscopy, German). This study were evaluated by two observer who blindly evaluated the density of the hybrid layer and the penetration of the resin tags. And the degree of agreement of the two observers was assessed using coefficients cohen's kappa. The wavelength of the microscope to view the fluorescence of Rhodamine B was 560 nm for obtaining an accurate picture at 10x lens magnification. The hybrid bond density and length of the resin tags' penetration depth for the fiber post cementation adhesive agents entering the dentinal tubules were calculated using ZEN 2010 (Carl Zeiss Microscopy GmbH, Jena, Germany). Average fluorescence intensity (MFI) was calculated automatically by ZEN 2010 software.

2.4. Statical analysis

The data obtained from the test results in this study were statistically analyzed using SPSS Statistics 24 software. The normality test obtained normal and homogeneous data distribution. Since only two groups were compared in this experiment, an independent t-test was used.

3. Results

Value measurement the degree of agreement between assessors (inter-rater reliability) who use coefficients Cohen's Kappa on the density of the hybrid layer was strong (kappa coefficients = 0.88) and the depth of penetration of resin tags was strong (kappa coefficients = 0.83). The Pearson correlation test results between the hybrid bond density and the resin

tag penetration depth in the self-etch treatment group showed a value of r = 0.634 and p-value = 0.008 ($p \leq 0.05$) and in the self-etch with additional etching treatment group showed a value of r = 0.516 and p-value = 0.041 ($p \leq 0.05$). From these findings, it can be concluded that there is a statistically significant positive correlation or linear relationship between hybrid bond density and resin tag penetration depth in both treatment groups. The thicker the density of the hybrid bond, the deeper the resin tag penetration, hence the first hypothesis is accepted.

In Table 1, the highest mean value of hybrid bond density is for the self-etch group with additional etching, showing differences in hybrid bond density between the two groups. Based on the results of the independent t-test, there were significant differences in hybrid bond density between the two groups; thus, the second hypothesis was accepted. The mean value of resin tag penetration depth for the self-etch group with additional etching showed a difference in the penetration depth of resin tags between the groups, but based on the results of the independent t-test, there were no significant differences between the two groups. Consequently, the third hypothesis was rejected.

The images from CLSM displaying the hybrid bonds which formed and resin tag penetration in the self-etch group can be seen in Fig. 1 and in the self-etch group with additional etching in Fig. 2.

4. Discussion

The hybrid bonds and the penetration depth of resin tags can be assesed by SEM and CLSM. CLSM was chosen in this study because the sample can be visualized at various depths and did not require special specimen processing. The teeth must be completely dry but did not require the preparation of destructive specimens. CLSM can produce three-dimensional images, mainly to study the interpenetration of two different materials (adhesive material and dental hard tissue) and allow for excellent result (Marigo et al., 2012; Kerstin Bitter, Mueller, & Kielbassa, 2009).

Fiber post cementation was done using a self-etch adhesive system because of the advantage of ease in application compared to a total etch adhesive system. In this adhesive system, the primary acidic material acted as etching and priming solutions without the need for rinsing and drying (Marigo et al., 2012). According to Martinho (2014), in self-etch adhesive systems, the primary acidic materials containing etching and primers proved to be ineffective in demineralizing root canal dentine because an optimal bond between the surface of the

Table 1 The mean value of hybrid bond density (μm) and the depth of resin tag penetration (μm) in the self-etch and self-etch groups with additional etching.

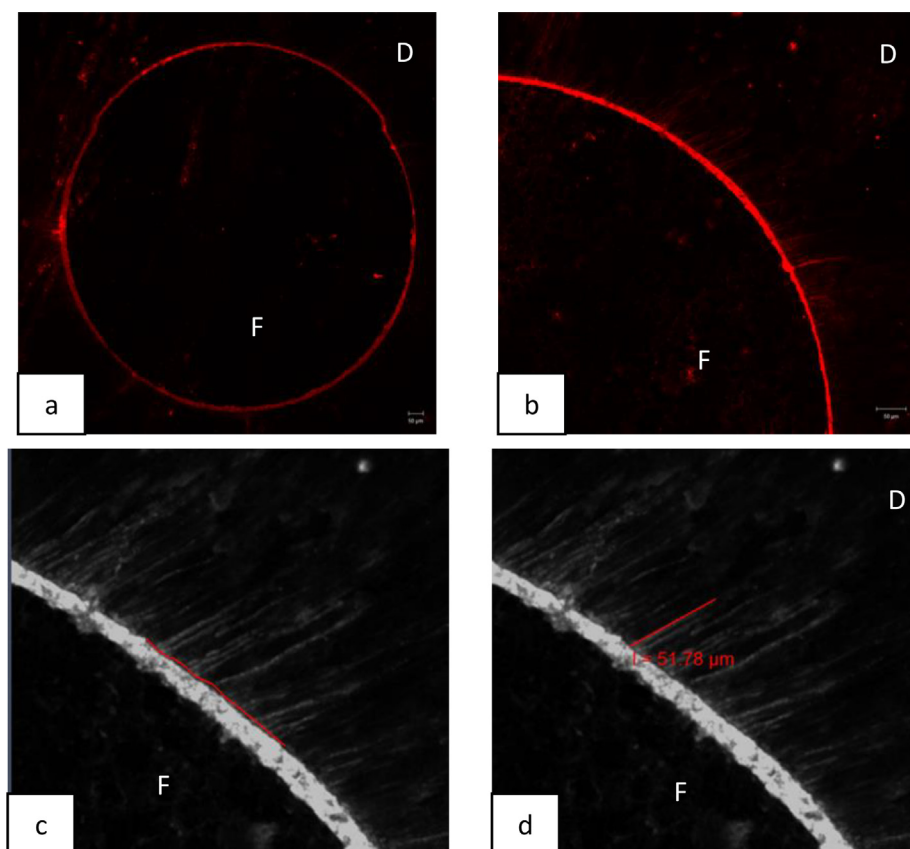| Treatment Group	Mean (SD)	p-value
Hybrid bond density		
• Self-etch	5.71 (3.36)	0.001*
• Self-etch with additional etching	9.68 (2.79)	
Penetration depth of resin tags		
• Self-etch	21.37 (15.85)	0.098
• Self-etch with additional etching	32.51 (20.72)	

* Independent t-test $p \leq 0.05$.

Fig. 1 Imaging of confocal laser scanning microscopy cross section of fiber (F) and root canal dentine (D) in the self-etch group: (a) cross-sectional imaging at 5X magnification, (b) cross-sectional imaging at 10X magnification, (c) measurement of hybrid bond density (shown on the red line) at 10X magnification, and (d) measurement of the penetration depth of resin tags (shown on the red line) at 10X magnification.

root canal dentine wall and the resin cement adhesive was not achieved (Martinho et al., 2014). Scotti (2012) reported that a self-etch technique with etching added to the tooth had the same value as push-out bond strength with a total etch technique (Scotti et al., 2012). Bitter (2006) stated that the use of 37% phosphoric acid could optimally clean the smear layer from the dentine wall, open dentine tubules, fibril collagen, and interfibrillar space, and help demineralization to increase the penetration of adhesive material (Kerstin Bitter et al., 2006; Lone et al., 2019). But in this study the application of a 37% phosphoric acid etching solution only for 15 s because if done more than 30 s, it can increase demineralization dentine zone within hybrid layer and lowering of bond strength (Marigo et al., 2012). Samples were cut from one-third of the middle area of the roots because making them as apical as possible could decrease the density of the dentinal tubules from 44,000 to 14,000 mm^2, making penetration of the adhesive more difficult to see. The samples were cut with thicknesses ranging from 1.5 to 2 mm (Marigo et al., 2012).

Table 1 and Fig. 2 show the hybrid bond density in the self-etch group with thick etching, which was 9.68 μm, and the resin tag penetration depth, which was 32.51 μm, longer than in the self-etch group. These results were consistent with research conducted by Marigo (2012) and Bitter (2004) show-

ing that the addition of etching could increase the density of hybrid bond values and the depth of penetration into the dentinal tubules (K Bitter et al., 2004; Marigo et al., 2012). The thickness of the hybrid bond density increases due to optimal cleaning of the smear layer and smear plug by phosphoric acid, demineralizing the dentine and exposing collagen fibers (Anchieta et al., 2011). Furthermore, self-etch adhesive systems have a low demineralization potential, resulting in lower bonds. A 37% phosphoric acid solution was able to demineralize dentine more optimally; hence, the adhesive material could enter the dentinal tubule (Skupien et al., 2010). The depth of resin tag penetration was influenced by the viscosity of the resin material; a higher viscosity might reduce the infiltration of resin material into the dentinal tubule (Anchieta et al., 2011).

Studies conducted by Marigo (2012) and Malyk (2010) suggested that an increasing thickness of hybrid bond density was positively correlated with bond strength (Marigo et al., 2012)·(Malyk, Kaaden, Hickel, & Ilie, 2010) According to research conducted by Anchieta (2011), the depth of resin tag penetration that was formed did not correlate with bond strength, but acted as a layer that absorbed pressure in the hybrid bond, therefore producing optimal mechanical strength. This may be the cause of a strong correlation between
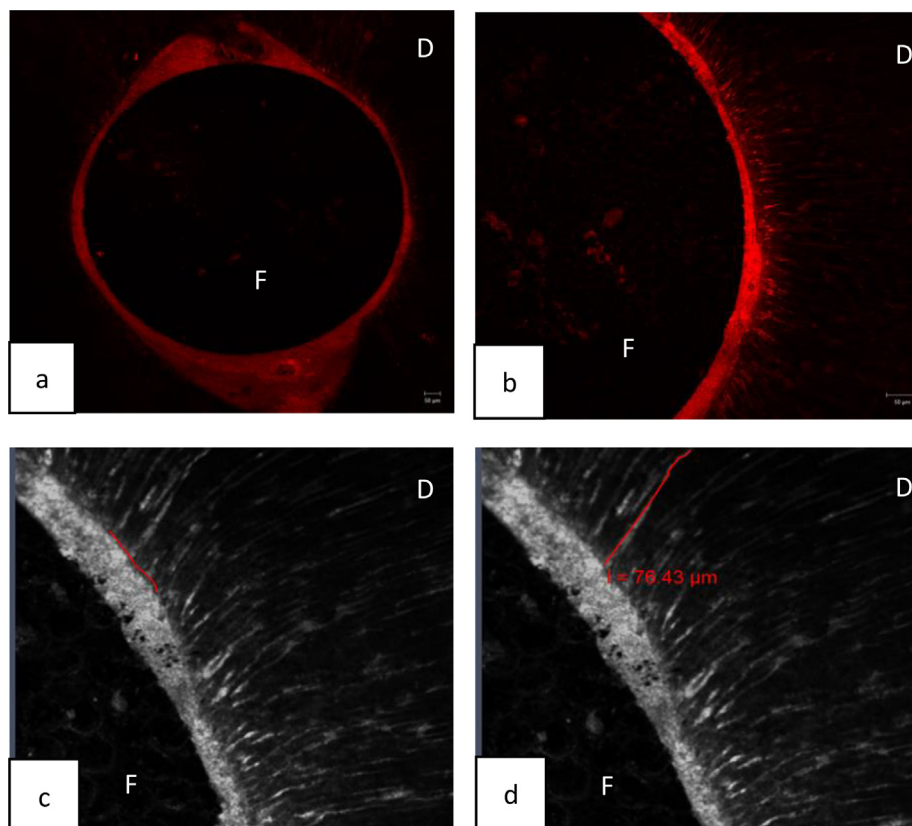
Fig. 2 Imaging of confocal laser scanning microscopy cross section of fiber (F) and root canal dentin (D) in the self-etch group with additional etching: (a) cross-sectional imaging at 5X magnification, (b) cross sectional-image at 10X magnification, (c) measurement of hybrid bond density (shown on the red line) at 10X magnification, and (d) measurement of resin tag penetration depth (indicated on the red line) at 10X magnification.

the thickness of hybrid bond density and bond strength because the hybrid bond formed from resin cement firmly bound the tooth substrate (Anchieta et al., 2011). This study supported that study's results in the form of a positive correlation between the thickness of the hybrid bond density and the depth of resin tag penetration in the two treatment groups, thus producing optimal mechanical strength because resin cement firmly binds the tooth substrate.

Oliveira (2009) found that the value of bond strength in self-etch adhesive systems was lower than in an etch-and-rinse system and that the thickness of hybrid bond density in self-etch adhesive systems was thinner compared to an etch-and-rinse system (Oliveira et al., 2009). Our research confirmed that the self-etch adhesive system with additional etching produces a better hybrid bond density and resin tag penetration depth than the self-etch technique.

5. Conclusion

The density of hybrid bonds in the self-etch group with additional etching was thicker compared to the self-etch group, and the penetration of resin tags in the self-etch group with additional etching had a deeper penetration than in the self-etch group. There was a strong positive linear correlation between hybrid bond density and depth of resin tag penetration in both treatment groups: the thicker the hybrid bond density, the longer the resin tag penetration, thereby increasing the mechanical strength of resin cement to the tooth substrate.

CRediT authorship contribution statement

Anita Erdiani: Conceptional, data curation, formal analysis, investigation, methodology, writing original draft. Writing-review and editing. **Ratna Meidyawati:** Funding acquisition, project administration, supervision. validation, visualization, writing-review. & editing. **Aditya Wisnu Putranto:** Software, supervision, validation, Visualization. writing-review & editing. **Dicky Yudha:** Data curation, writing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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