

# A scanning electron microscope-based comparative evaluation of resin tag formation at the resin dentin inter-diffusion zone

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## ABSTRACT

Aim of study was evaluate and compare the formation of the resin dentin interdiffusion zone (RDIZ) at the radicular dentin and resin interface after etching with phosphoric acid (PA) and glycolic acid (GA), using either light-cure and self-cured adhesives. Twenty extracted teeth were root canal treated. Post space preparation was done, and the samples were randomly divided into two main groups depending upon the etchant. Each group was further divided into two subgroups depending on the type of adhesive used. One group was etched with 37% PA (Prime® Dental Products PVT LTD, India) and the other with 35% GA (Glycolic 35 Peel, Rejsol, India). Etching was followed by the application of light-cured adhesive (3M ESPE Adper Single Bond, USA) or self-etch (3M ESPE Single Bond Universal, Germany). Fibre posts were then placed and after a coronal build-up, the teeth were sectioned longitudinally and observed under SEM. Statistical analysis: Data was analyzed by descriptive statistics as a percentage of resin tag density for all the groups. The Kruskal-Wallis test was applied for overall comparison and a Mann-Whitney U test for inter-group analysis. Resin tag density was found to be the highest in the coronal third followed by the middle third of radicular dentin and was the least in apical thirds for both groups. Resin tag density was similar for light cure and self-etch adhesives in the coronal, middle, and apical thirds of both groups. In conclusion this study concluded that GA could be thought of as a potential etchant for the radicular dentin.

**Keywords:** resin tag, resin dentin inter-diffusion zone, scanning electron microscopy, phosphoric acid, glycolic acid

## INTRODUCTION

Post-endodontic restoration is crucial for the survival of endodontically treated teeth, as they are structurally weakened and more susceptible to fracture. The human dentin is a complex structure with a mineralized organic matrix, consisting of 50% inorganic components, 30% organic components, and more type I collagen. The adhesion of hydrophilic and hydrophobic components of resin cement to the

non-vital dentin substrate is complex and technique-sensitive [1,2].

Various adhesive systems have gained popularity for cementing an endodontic intra-radicular post, with resin-based luting agents used for fiber posts [3]. The goal is to create an effective bond between the radicular dentin and the adhesive system, avoiding microbiological activity at the interface. Post-space preparation involves a smear layer, which is mainly composed of remnants of gutta-percha,

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sealer, and debris, which affects bonding at the interface [4]. Surface conditioning is a required step for the micromechanical interlocking of resin cement into the tooth substrate [5]. Etch-and-rinse adhesive systems create micro-porosities in the tooth substrate, leading to the formation of resin tags, also known as the Resin-Dentin Interdiffusion Zone (RDIZ) [6,7].

Phosphoric acid (PA) is the most commonly used etchant in dentistry, with concentrations ranging from 30% to 40%. PA increases surface roughness and wettability demineralizes the inter-tubular and peri-tubular dentin matrix, and exposes type I collagen fibers [8]. However, PA is highly acidic and can cause structural changes in the dentinal matrix. Glycolic acid (GA) is a naturally occurring organic acid that is widely used in dermatology due to its ability to elevate collagen synthesis and fibroblast proliferation [9]. The effectiveness of adhesives can be assessed by evaluating the RDIZ and penetration of tags in the underlying tooth substrate under a microscope [10].

This study aims to evaluate and compare resin tag density at the radicular dentin-resin interface after etching with either 37% PA or 35% GA followed by using an adhesive. The uniqueness of this study is that glycolic acid is used as an etchant and the RDIZ is evaluated in radicular dentin after using GA.

## MATERIALS AND METHODS

This experimental study focuses on the extraction of extracted teeth from a dental institution. Sample preparation was done in the department. The study was conducted at Icon Analytical Equipment Pvt Ltd in Worli, Mumbai. The sample size was 20 (5 in each group) and the inclusion criteria included intact molar or mandibular teeth with normal morphology. Exclusion criteria included teeth with developmental defects, multiple canals, or apical curvature.

The selected samples were randomly divided into two main groups with ten samples each which was further divided into two subgroups with five samples in each subgroup.

**Group 1:** Etching with 37% PA (Prime® Dental Products PVT LTD, India)

- Group 1A: Bonding with Light-cure adhesive (3M ESPE Adper Single Bond, USA)
- Group 1B: Bonding with Self-etch adhesive (3M ESPE Single Bond Universal Adhesive, Germany)

**Group 2:** Etching with 35% GA (Glycolic 35 Peel, Rejsol, India)

- Group 2A: Bonding with Light-cure adhesive (3M ESPE Adper Single Bond, USA)
- Group 2B: Bonding with Self-etch adhesive (3M ESPE Single Bond Universal Adhesive, Germany)

## Method of Preparation

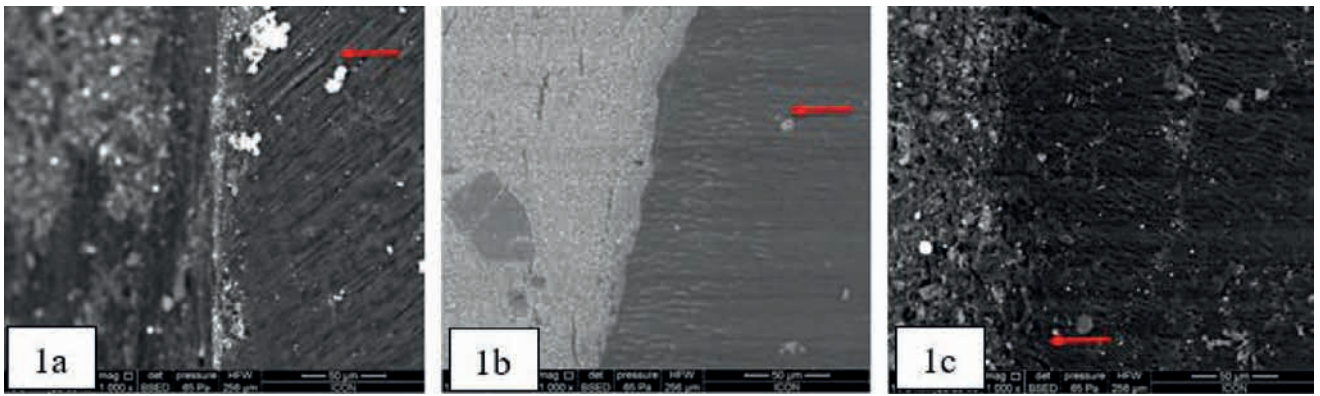
The study involved selecting teeth and preparing them for root canal treatment. Chemo-mechanical preparation was performed using Gates-Glidden (GG) drills for orifice enlargement, and irrigation was performed using 3% sodium hypochlorite solution and 17% EthyleneDiamene Tetraacetic acid (EDTA). The canals were dried with paper points. Obturation was done using resin-based sealer AH Plus and gutta-percha. Post-space preparation involved enlarged orifices with GG drills and Peso Reamers, leaving the apical plug intact. Posts were etched with 37% PA and 35% GA, followed by self-etch adhesive and Hi-Rem posts. The coronal build-up was done using restorative composite resin. Sectioning was done parallel to the long axis using a carborundum disc. To observe resin tag formation in the RDIZ, one section of each root was stored in 30% HCl for 30 seconds and studied at three levels under 2000x magnification using a scanning electron microscope.

## RESULTS

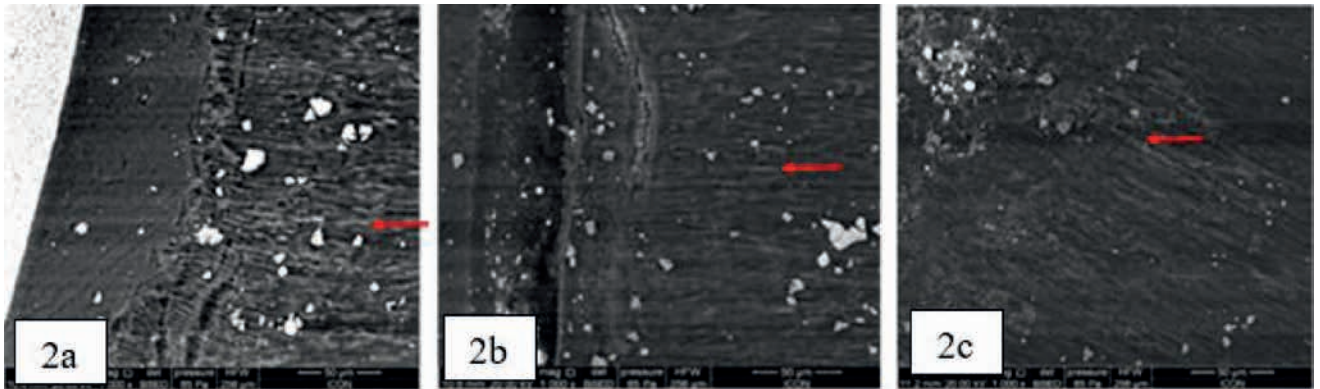
The study found that etching with PA and bonding with light cure adhesive resin had 80% uniform resin tag density without lateral branches in the coronal third and 40% in the middle third. When etching radicular dentin using PA, there were 20% fewer and shorter resin tags and 80% uniform resin tag density without lateral branches in the coronal third, 60% fewer and shorter and 40% uniform resin tags without lateral branches in the middle third, and 80% few and short resin tags in the apical third (Figures 1, 2). Etching with GA and bonding with light cure adhesive resin had 60% few and short resin tags and 40% uniform resin tags without lateral branches in the coronal third, 90% few and short resin tags in the middle third, and 60% few and short resin tags in the apical third. Scanning Electron Microscopy (SEM) images at the fiber post/resin cement interface showed no voids, suggesting a good bond (Figures 3, 4). The inter-group comparison revealed that Group 1 had significantly higher scores as compared to Group 2 at all locations ( $P < 0.05$ ).

Following were the observations from SEM images: In both groups, the resin tag density was found to be the most in the coronal one-third followed by the middle one-third, and least in the apical one-third of the radicular dentin. ( $P < 0.05$ ) (Figures 5-7). Resin tag density was similar for light-cure and self-etch adhesives in the coronal and middle thirds of the radicular dentin. In the apical third lesser number of resin tags was observed for both the adhesive systems.

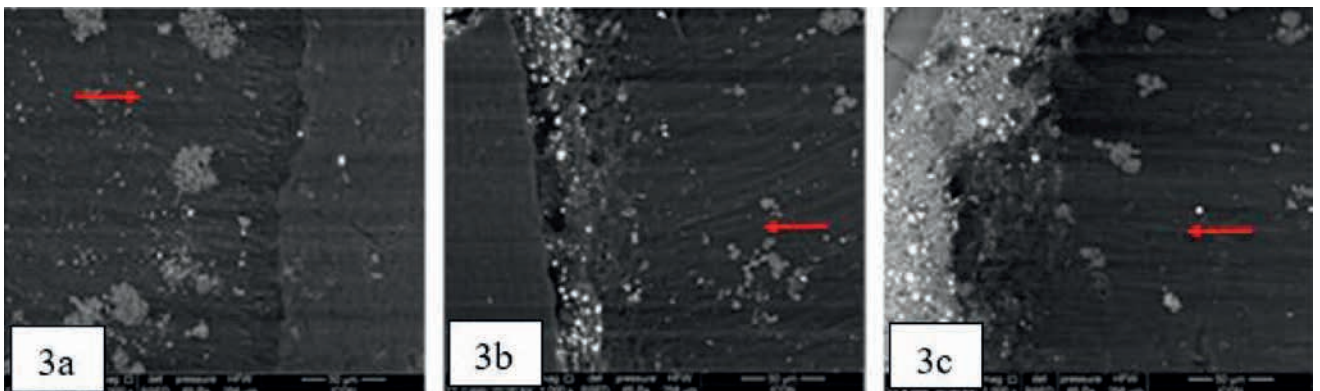




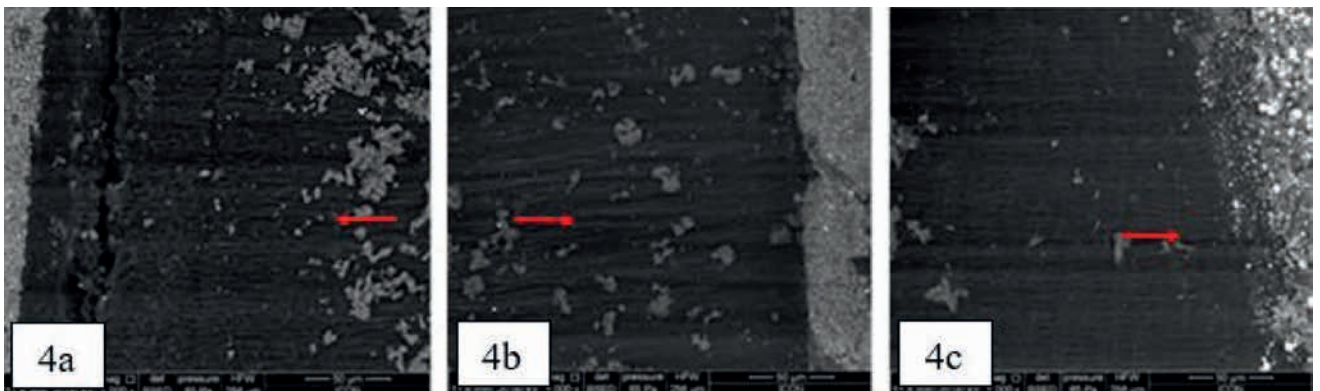
**FIGURE 1.** Shows the SEM images of Group 1A (Etching with PA and Bonding with Light Cure adhesives). a – Coronal, b – Middle third, c – Apical images are shown.



**FIGURE 2.** Shows the SEM images of Group 1B (Etching with PA and Bonding with Self Etch adhesives). a – Coronal, b – Middle third, c – Apical images are shown



**FIGURE 3.** Shows the SEM images of Group 2A (Etching with GA and Bonding with Light Cure adhesives). a – Coronal, b – Middle third, c – Apical images are shown



**FIGURE 4.** Shows the SEM images of Group 2B (Etching with GA and Bonding with Self Etch adhesives). a – Coronal, b – Middle third, c – Apical images are shown

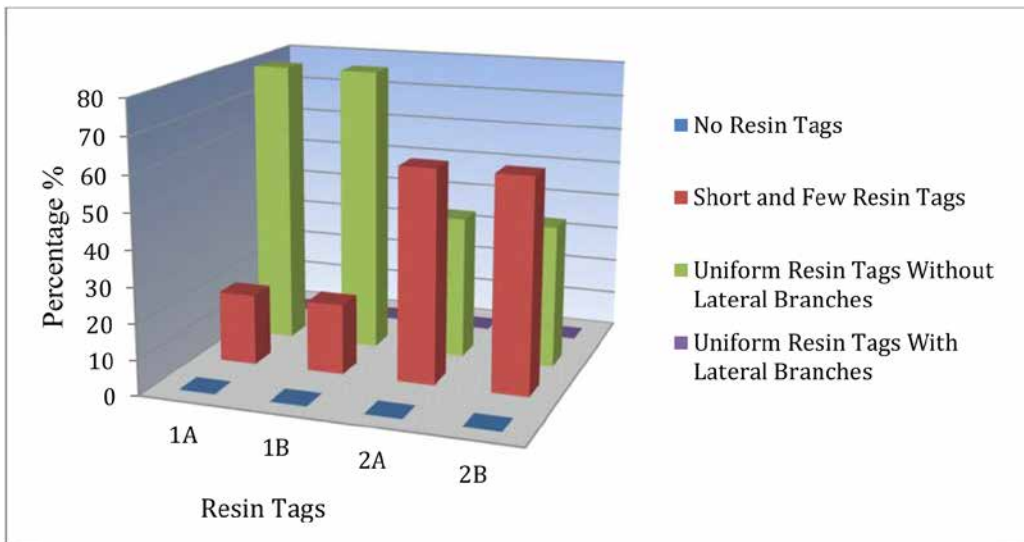


FIGURE 5. Resin tag density in the coronal third of radicular dentin of all the groups

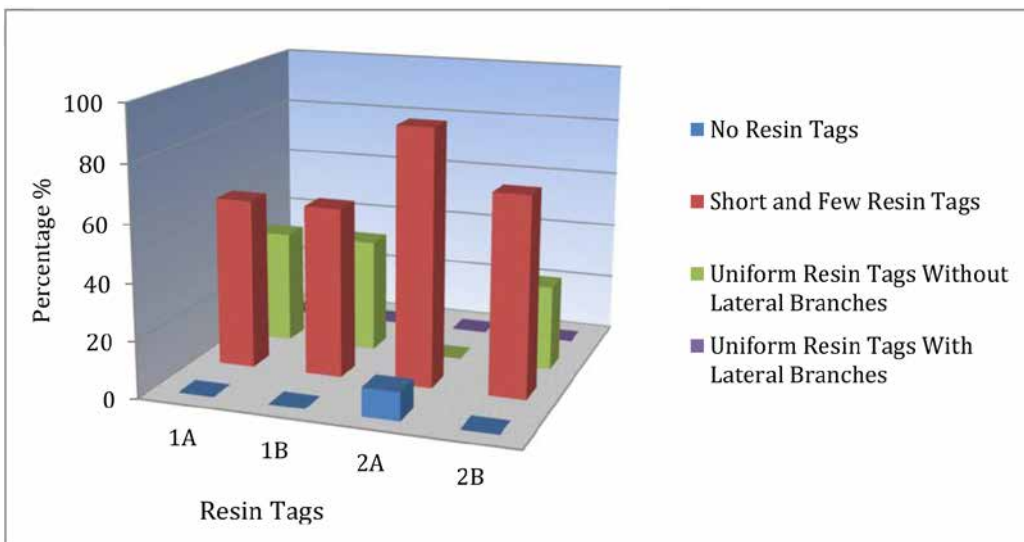


FIGURE 6. Resin tag density in middle third of radicular dentin of all the groups

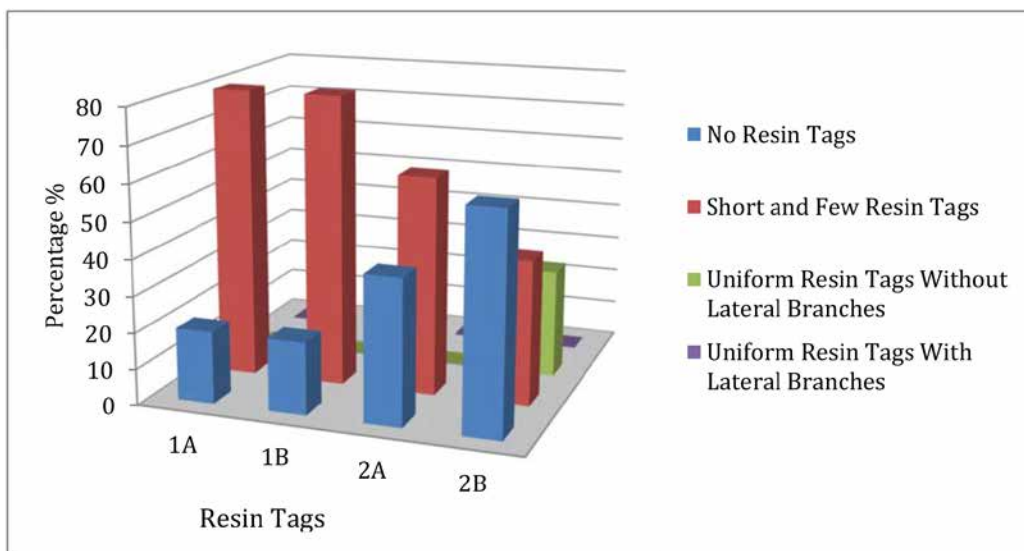


FIGURE 7. Resin tag density in apical third of radicular dentin of all the groups



## DISCUSSION

Dentin is a vital component in adhesive dentistry [11]. Etching patterns in peritubular and intertubular dentin create a micro-retentive network for resin infiltration [12]. Cleaning and shaping produce a smear layer composed of hydroxyapatite and collagen debris. The importance of removing the smear layer was a topic of discussion in the early 21st century [13]. Bonding between adhesive systems and radicular dentin is micromechanical in nature, based on the infiltration of etched dentin surfaces, formation of an RDIZ, resin tags, and adhesive lateral branches. The RDIZ formed with each variable was analyzed and compared using SEM [14,15].

The resin tag density was measured at the coronal, middle, and apical one-thirds of roots, using a four-step scale. The highest density was found in the coronal one-third, followed by the middle third, and the least in the apical third. This result was in concurrence with a study by Giachetti et al wherein it states that the traditional SEM techniques used to identify resin tags found filamentous organic structures, similar to the lamina limitans found in dentinal tubules. This is due to glycosaminoglycans being resistant to typical specimen preparation methods, and the degree of penetration of resin tags has become unclear due to over-reliance on SEM morphology [16]. PA is an excellent tooth substrate etchant but has its own set of flaws. It has a low pH of 0.12, which is harmful to vital tissue, and interferes with organic components in dentin. Etching with PA accelerates the degradation of the RDIZ by activating gelatinolytic or collagenolytic enzymes, such as matrix metalloproteinases and cysteine cathepsins. PA does not have self-limiting action, making it difficult to control the depth of demineralization [8]. The effect of depth of dentin demineralization on bonding effectiveness has been studied, and it has been concluded that a lower concentra-

tion of acid or short application time does not affect bonding efficacy [17]. This observation led to the search for alternative approaches to etching radicular dentin with etching patterns similar to PA [18].

SEM images showed similar etching patterns, but resin tag density varied among the groups and radicular dentin locations [19]. GA is a naturally occurring organic acid in the  $\alpha$ -hydroxy acids (AHA) group, commonly used in dermatology for chemical peeling of skin. It has several advantages over polyacrylamide (PAA) in terms of pH, collagen synthesis, demineralization depth, lower molecular weight, and reduced gelatinolytic activity. The adhesion-decalcification concept, developed by Yoshida et al., explains the mechanism of tooth demineralization for AHA acids. GA is an attractive etchant in adhesive dentistry due to its advantages [18]. The present study results coincided with the findings by Darze et al which showed GA exhibiting microhardness and a similar etching pattern as that of PA. [20]. The main drawback or limitation of this study is the presence of voids within resin cement used for luting that is associated with factors such as viscosity, root anatomy, and inadequacy between matrix and filler content.

## CONCLUSION

The study found that PA and GA dissolved intratubular dentin, exposing collagen fibers, and allowing adhesive resin penetration in the RDIZ. PA had a deeper penetration of resin tags than GA. The resin tag density was highest in the coronal, middle, and apical regions. Light cure and self-etch adhesives had similar resin tag density, while the apical third had a lesser number of tags. The study also recommends that GA could be a potential etching agent for radicular dentin on considering the various properties exhibited by the same.

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