Study of microleakage in dental enamel using nanofillers composite resin restoration with total-etch and self-etch adhesive

Iin SUNDARI, Viona DIANSARI, Niska DARLIANTI

ABSTRACT Microleakage is a poor marginal adaptation of the restoration, often found between cavity and restoration material. One of the reasons is the shrinkage during the composite resin during polymerization, causing the restoration’s adaption disturbed. Application of adhesive material on the cavity affects microleakage at the margin restoration and increases the adaptation between cavity and restoration material. This research aims to determine microleakage in dental enamel using nanofiller composite resin restoration with fifth-generation total-etch adhesive and eight-generation self-etch adhesive. This research used 16 specimens premolars, which were prepared cavity Class I G.V. Black and divided into two groups (group A and B). Group A used nanofiller composite resin FiltekTM Z350 + fifth-generation total-etch Adper Single Bond 2 (n=8), group B used nanofiller composite resin Filtek TM Z350 + eight-generation self-etch Universal Single Bond Adper (n=8). The specimen isolated using nail polish except in the work area, then immersed in methylene blue 1% at 25°C (for 24 hours). After that, all specimens were washed and cut longitudinally. The results were observed using a Stereomicroscope and Scanning Electron Microscope (SEM). The observation’s results showed that the amount number of microleakage in group A (75%) less than in group B (100%), while the non-parametric statistic test using the Mann Whitney showed no significant differences (p>0.143). Based on SEM images, the average distance between enamel and composite resin with total-etch adhesive was 1.40 ± 0.007µm, and 1.84 ± 0.509 µm for resin composite with self-etch adhesive. This research concluded that microleakage using nanofiller composite with the fifth-generation total-etch adhesive was smaller than nanofiller composite resin with the eighth -generation self-etch adhesive.

KEYWORDS: Adhesive system, nanofillers composite resin, microleakage, enamel

INTRODUCTION

Composite resin is increasingly used as a restorative material because it has an a restorative material because it has an aesthetic value used to bind enamel and dentin. The most recent composite resin material is known as nanofiller composite resin. Micromechanical retention can be formed from the adhesive material, creating a significant chemical and interlocking interaction between the composite resin and the dental structure.

Adhesive material interaction is based on two systems, namely a total-etch and self-etch adhesive system. The fifth-generation adhesive is known as total-etch material simplified into two stages of an application by combining primary and bonding in one application. The total-etch in fifth-generation used in clinical procedures by adhesive application to minimize working time. While the eighth generation adhesive is used as a self-etch adhesive in one single bottle. Self-etch adhesive systems are increasingly in demand because they provide more advantages than total-etch straightforward applications and shorter time. Self-etch adhesive contains organic solvents, water, and acid resin monomer. The monomer of self-etch adhesive material produces chemical bonds such as 10-Methacryloyloxydecyl Dihydrogen Phosphate (MDP) with calcium ions in hydroxyapatite in enamel.

Enamel is an outermost layer that covers dentine on the tooth crown. The enamel's mineral composition consists of an inorganic material 96% of hydroxyapatite, namely calcium and phosphorus, and the remaining components are water and organic material 1-2%. The use of etching in enamel
can lead to demineralization by losing calcium and forming microporous. Monomer resin from adhesive material infiltrates into microporous and forms a resin tag.\textsuperscript{5,7} Resin-tag is classified as a macro tag and micro tag. The macro tag is marked if the resin surrounds the enamel prism, while the micro tag if the resin infiltrates porosity in the enamel prism. The micro tag is considered the basis of resin-enamel retention.\textsuperscript{7}

One of the disadvantages of the composite resin material is that it can occur shrinkage during polymerization.\textsuperscript{9} Shrinkage on composite resin can produce traction force during polymerization, leading to poor restoration boundary adaptation characterized by microleakage.\textsuperscript{2,10}

### RESEARCH METHODS

This research was an experimental type of laboratory research with the Post-test Only Design Method. The microleakage image in dental enamel using nanofiller composite resin restoration with fifth-generation total-etch adhesive and eighth-generation self-etch adhesive was observed using Stereomicroscope and Scanning Electron Microscope (SEM).

#### Specimen Selection and Preparation

The specimens used were 16 maxillary premolars without the presence of caries, fractures, and restoration. Preprepared dental specimens formed a Class I G.V buccolingual width of 2 mm, mesiodistal length 4 mm, a depth of 3 mm without bevel, and a flat cavitation base. Dental specimens were divided into two groups, group A nanofiller composite resin with fifth-generation total-etch adhesive (n=8) and group B nanofiller composite resin with eight-generation total-etch adhesive (n=8).

#### Application of nanofillers composite resin restoration with total-etch and self etch adhesive on the specimens

In Group A, the cavity was applied with phosphoric acid 37% by using a micro brush for 15s, rinsed with water for 10s, and cleaned the remaining water with cotton pellet.\textsuperscript{2,5,12} After that, the total-etch adhesive was applied using a micro brush for 15s as much as three layers continuously, then curing with Light Curing Unit (LCU) for 10s and restoration nanofiller composite resin applied incrementally.\textsuperscript{2,12,14}

In Group B, the cavity was applied with the self-etch adhesive using a micro brush for 20s, then sprayed with air for 5s gently and curing with LCU for 10s, then nanofiller composite resin applied incrementally.\textsuperscript{2,15,16}

#### Specimen immersion

All specimens that had applied with adhesive and nanofiller composite resin, then immersed in aquadest at 37°C (stored in an incubator) for 24 hours, aimed to simulate the oral cavity state.\textsuperscript{9,10,16} After that, the tooth surface covered with two nail polish layers, except 1 mm from the restoration area. Using nail polish was to prevent penetration of the substances entering other areas other than the restoration area, after dried and immersed in a 1% methylene blue solution of 20cc in a plastic vial for 24 hours at 25°C. After 24 hours, the specimens were rinsed under the water flows.\textsuperscript{2,16}

#### Longitudinal Cutting of the specimens

Specimens are divided into two parts with buccolingual direction on the tooth axis using carborundum discs.\textsuperscript{10,16} Microleakage was observed using Stereomicroscope and Scanning Electron Microscope (SEM).

#### Specimen observation using a Stereomicroscope

Observed each group of specimens by using a stereomicroscope with 10x magnification to see if there was 1% methylene blue dye penetration. The specimens were measured and graded based on the scale proposed by Santosh et al.\textsuperscript{13} with score 0: No dye penetration; score 1: dye penetration up to 1/3 from the depth of cavities; score 2: dye penetration > 1/3 but < 2/3 of cavities depth; score 3: dye penetration > 2/3 from the depth of the cavities;
score 4: dye penetration up to the base of the cavity and involving tubules dentin.

**Specimen examination using a Scanning Electron Microscope (SEM)**

Specimen images were observed using SEM with the magnification of 800x and 1500x on each specimen group for the distance between restoration materials and dental structure due to poor marginal adaptation. Before observation, the specimens were vacuumed for three minutes. ¹⁷

**RESULTS**

Microleakage was measured using a Stereomicroscope with 10x magnification of nanofiller composite resin restoration (Z350 XT 3M ESPE) with fifth-generation total-etch adhesive and eight-generation self-etch adhesive was defined by modus scoring number of 0 to 4.

The observation of microleakage using a stereomicroscope marked the dye penetration of methylene blue 1%. There was dye penetration on the specimen using methylene blue 1% had a tiny molecular size of 0.5-0.7nm, smaller than bacteria (0.5-1μm), so that the dye substance of methylene blue 1% could penetrate further at a distance existed on the surface of the restoration.²⁰ SEM image shows the distance formed, the average distance formed in this TE5 specimen was 1.40μm larger than the size of methylene blue molecule 1% (0.5-0.7nm).

Dye penetration with the blue line between the enamel and composite resin was observed using Stereomicroscope after immersion in methylene blue showed in Figure 1.

**Figure 1. Stereomicroscope overview.**
(a) specimens (TE3) contained microleakage characterized by the absence of a blue line between composite resin and enamel (white line); (b) specimens (TE6) with no microleakage, not marked by a blue line between composite resin and enamel (white line).

**Table 1. Microleakage Score of Nanofiller Composite Resin with Total-etch and Self-etch Adhesive**

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Microleakage Score</th>
<th>Group A (Total-etch)</th>
<th>Group B (Self-etch)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

Based on the measurement results in the modus of the microleakage score is shown in Table 1. There were score 0 for two specimens and a microleakage score 1 for six specimens in group A (75%) in Table 2. In comparison, group B was a microleakage score 1 in all specimens (100%) in
Table 2. Microleakage occurred in all specimens (n=8) in composite resins with self-etch adhesives (100%), while in composite resins with total-etch adhesive, microleakage only occurred in six specimens (75%). This result shows that the microleakage in self-etch adhesive more often occurs than total-etch adhesive material. The measurement of microleakage could be analyzed using a non-parametric statistical test of the Mann Whitney. The difference in microleakage score occurred between the total-etch adhesive and the self-etch was not significant $p = 0.143$ ($p > 0.05$), as seen in Table 3.

Table 2. The modus and percentage of microleakage score

<table>
<thead>
<tr>
<th>Adhesive Material</th>
<th>Microleakage Score</th>
<th>n</th>
<th>Microleakage Percentage (%)</th>
<th>Modus</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A (Total-Etch)</td>
<td>2 6 0 0 0</td>
<td>8</td>
<td>75</td>
<td>Score 1</td>
</tr>
<tr>
<td>Group B (Self-Etch)</td>
<td>0 8 0 0 0</td>
<td>8</td>
<td>100</td>
<td>Score 1</td>
</tr>
</tbody>
</table>

This result of the microleakage in self-etch adhesive more often occurs than total-etch adhesive material. The measurement of microleakage could be analyzed using a non-parametric statistical test of the Mann Whitney.

Table 3. Results of the non-parametric statistical test of Mann Whitney on two systems adhesive material

<table>
<thead>
<tr>
<th>Adhesive Material</th>
<th>n</th>
<th>Amount of specimen had microleakage</th>
<th>$p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A (Total-Etch)</td>
<td>8</td>
<td>6</td>
<td>0.143</td>
</tr>
<tr>
<td>Group B (Self-Etch)</td>
<td>8</td>
<td>8</td>
<td></td>
</tr>
</tbody>
</table>

*Significance value of Mann Whitney test ($p < 0.05$)

Microleakage’s observation used the Scanning Electron Microscope (SEM) with 800x and 1500x magnification of nanofiller composite resin restoration with the fifth-generation total-etch adhesive and the eight-generation self-etch adhesive. The observation was conducted to see the nanofiller composite resin material (Z350 XT 3M ESPE) with the enamel, as shown in Figure 2 below.

Figure 2. Overview of microleakage under the observation of SEM with 1500x magnification. (a) Microleakage in the specimen (TE5) with a distance of 1.40μm; (b) Microleakage in the specimen (TE7) with a distance of 1.41μm; (c) Microleakage in the specimen (SE1) with a distance of 2.20μm; (d) Microleakage in the specimen (SE3) with a distance of 1.48μm. Blue Arrow: Enamel; Red Arrow: Composite Resin
The observation using SEM showed the distance between the composite resin and enamel, as seen in Figure 2. These observations measured the average distance between the enamel surface and the nanofiller composite resin material in the μm unit.\(^{17}\)

Table 4 showed that the specimen in group A were found better attachment than the specimen in group B. The result showed that the microleakage score was higher occurred in group B than group A.

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Adhesive Material</th>
<th>Group A (Total-etch) (μm)</th>
<th>Group B (Self-etch) (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>1.40</td>
<td>2.20</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>1.41</td>
<td>1.48</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td></td>
<td><strong>2.81</strong></td>
<td><strong>3.68</strong></td>
</tr>
<tr>
<td><em>(x ±SD)</em></td>
<td></td>
<td><strong>1.40 ± 0.007</strong></td>
<td><strong>1.84 ± 0.509</strong></td>
</tr>
</tbody>
</table>

**DISCUSSION**

Based on statistical analysis, the difference between group A and group B (p>0.05) is insignificant, as shown in Table 3. The insignificant difference was expected due to total-etch and self-etch adhesive materials that had an acidic composition that was almost the same as the presence of phosphoric acid in the total-etch adhesive and 10-Methacryloyloxydecyl Dihydrogen Phosphate (MDP) in the self-etch adhesive.

Both adhesive systems had acidic compositions. In total-etch adhesive consists of primary (pH 4.7) and bonding agent/etching (pH 0.6) to acidic content.\(^{12}\) In comparison, self-etch adhesive contains acidic content (pH>2.5), lower or mild than total-etch adhesive.\(^{11}\) It was suspected that it could affect the total microleakage score in group A was smaller than in group B, as shown in Table 1. According to Sayed et al.\(^{12}\) suggested that the use of the total fifth-generation total-etch with Primary content (pH 4.7) and etching (pH 0.6) showed less microleakage in the occlusal margin and the gingival margin than the use of the seventh-generation self-etch adhesive (pH 2-2.2).

The microleakage occurred in this study with a total-etch adhesive containing phosphoric acid 37%. Application of phosphoric acid to the enamel was capable of changing enamel surface into irregular surfaces and increasing free energy on the tooth surface, thus producing a micromechanical attachment by forming more microporosity on the tooth surface. All of these caused the resin monomer to wet the surface quickly and penetrated the microporous (10-20 μm), which would be polymerized and also produced mechanical retention that made a resin tags form and quite large chemical interactions that could reduce the chance of microleakage between the surface of the restoration and enamel.\(^{19,20}\)

The self-etch adhesive contains 10-Methacryloyloxydecyl Dihydrogen Phosphate (MDP), which releases H\(^+\) ions to bind with calcium from hydroxyapatite in enamel through ionic bonds of electrostatic interactions that can rival the ability of phosphoric acid from adhesive total-etch. The methacrylate groups in MDP are polymerized with Bis-GMA and TEGDMA monomers in nanofiller composite resins capable of forming a good attachment between enamel, adhesive, and composite resin. However, the microleakage still occurred.\(^2\) It is supported by Patrascu et al.\(^{21}\) that the use of self-etch adhesive is less infiltrated into microporous in enamel. While other content of self-etch material HEMA (2-hydroxyethyl methacrylate) is a bifunctional material that has two clusters, i.e., hydroxyl groups (hydrophilic) that are able to penetrate enamel rods meanwhile the hydrophobic methacrylate groups, after the polymerization process occurs will bond with Bis-GMA and TEGDMA which are hydrophobic in composite resins.\(^{22}\)

Min et al.\(^{23}\) reported that nanofiller composite resin underwent a polymerization shrinkage that was 4.12% larger than the hybrid composite resin of 2.31%. This nanofiller composite resin was due to the lower inorganic filler content, while the matrix content was high. Some other factors related to the composite resin’s polymerization process are the beam intensity and beam distance to the composite resin.\(^{17}\) According to Fitriyani,\(^{24}\) the LED (Light Emitting Diode) for the proper polymerization of nanofiller composite resin with intensity light was 800 mW/cm\(^2\). The restoration thickness was 2-3mm, presumably because the content of nanooagglomerate...
and nanocluster in composite resin caused the intensity light to penetrate optimally. In this study, the source of the light used LED with intensity light was 800 mW/cm², and a restoration thickness of 3mm was able to minimize the microleakage. In comparison, Powers et al. Cit. Diansari, et al.² suggested that the maximum distance between sources with the restoration was 1 mm with a thickness of 2-2.5mm to obtain optimal polymerization.

In this research, the illumination distance was 2 mm with a 2 mm restoration thickness in each composite resin material and dental enamel in self-etch higher than total-etch specimens.

CONCLUSION

The microleakage’s number of the total-etch specimens less than self-etch. Scanning Electron Microscope also confirmed a gap between nanofiller group. The microleakage was formed less in group A than group B because it contained stronger acid in the total-etch adhesive than self-etch adhesive. Based on Diansari,² the study result stated that the distance of light-curing of 2 mm could still produce optimal polymerization by using total-etch adhesive because it contained phosphate acid etch than the use of self-etch adhesive. Thus, the amount of microleakage with total-etch adhesive is lower than the use of self-etch adhesive.²

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Conflict of Interest: The authors declared no conflict of interest.

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