In Vitro Comparison of the Effects of Two Different Acid Etchants on the Microleakage of Composite Restorations

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Abstract

Objective: Etching of enamel and dentin is an important step in restoration of teeth with composite resin ensuring the retention of restoration and decreasing microleakage. This in vitro study sought to compare the effects of Kimiya and Etch-Rite acid etchants on the microleakage of composite restorations.

Methods: This experimental study was conducted on 30 sound human molar teeth. Class V cavities were prepared on the buccal surface of teeth and the teeth were randomly divided into 2 groups of 15. Iranian acid etchant (Kimiya, Iran) was used in group 1 and Etch-Rite (Pulpdent, Watertown, MA, USA) in group 2. The teeth were restored with Valux Plus (3M, ESPE, St. Paul, MN, USA) composite resin and polished by disc. The canal apices were sealed with sticky wax and the entire tooth surface was coated with 2 layers of nail varnish except for 1 mm around the restoration margins. Specimens were immersed in 2% Fuchsin (Fuchsin Dye, Merck, Germany) for 24h and then buccolingually sectioned. Specimens were evaluated under a stereomicroscope (SF-100B, Lomo, Russia) at 40X magnification and the degree of microleakage was determined using a 0-4 scale. The degree of microleakage was statistically analyzed using Mann Whitney U test.

Results: No significant difference was found between the two groups of Kimiya and Etch-Rite in the degree of microleakage at the occlusal wall (p=0.1). The degree of microleakage at the gingival wall was not significantly different between the two groups either (p=0.68).

Conclusion: Based on the results, Kimiya Iranian acid etchant has an efficacy equal to that of Etch-Rite.

Key words: Acid etchant, Microleakage, Thickening agent.

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Introduction:

Esthetics is always important for patients. Thanks to the advances in tooth colored restorative materials, dentists are now capable of conservatively reconstruct the tooth structure while maintaining esthetics (1). Despite the extensive use of composite resins and advances in adhesive systems, microleakage is still one of the most important problems of these restorations. This issue is attributed to several factors such as the inefficacy of the available bonding agents, inadequate etching, poor physical properties of composites or a combination of the mentioned factors (2, 3). Microleakage is an important shortcoming of resin restorations compromising their survival. Due to microleakage, a gap forms at the tooth-restoration interface allowing the entry of microorganisms and food particles and subsequently leading to secondary caries and pulpitis. Adequate marginal adaptation ensures less marginal discoloration and reduced incidence of secondary caries, post-operative hypersensitivity and pulp injury (1, 4). The resin-enamel bond by the use of acid etchants
has a simple mechanism. The enamel surface, due to high mineral content and a homogenous structure, provides adequate micromechanical bond to resin materials. Application of etchant to enamel surfaces leads to minimum marginal microleakage (1,5). Etching is defined as cutting into the enamel surface and roughening it by using acids. Enamel surface etching with phosphoric acid was first described by Buonocore in 1955 (6). In the process of etching, hydroxyapatite crystals at the interface are dissolved and fluids penetrate into the newly formed porosities. Moreover, abrasion also causes surface roughness and leads to the formation of a smear layer of hydroxyapatite crystals and deformed collagen with 1-3 μ thickness. This layer should be removed or the adhesive has to penetrate into it. Etching with acids or conditioners dissolves this layer and provides a surface free from the smear layer and with negative retentive areas enhancing micromechanical interlocking. By the application of a resin-based flowable material on the etched porous surfaces, resin penetrates into the surface. This process is also enhanced by the capillarity action. Resin monomers are then polymerized and the material is locked into the enamel surface. Formation of microscopic resin tags into the enamel surface is the basis of resin-enamel bond (7,8). Type and concentration of acid etchant, its percentage, thickness and consistency (gel, semi-gel, aqueous solution), duration of etching, duration of rinsing, chemical composition and condition of enamel, type of tooth (permanent or primary) and protecting the etched surface from the saliva and moisture contamination can all affect the efficacy of etching (9).

Dentin with its high organic content, the odontoblast processes and intratubular fluid has a non-homogenous moist structure making the adhesion of resins difficult. Several theories have been suggested for the resin bond to dentin; the most acceptable one is the hybrid layer formation mechanism proposed by Nakabayashi in 1982. Based on this theory, acid demineralizes the dentin surface and forms a network of collagen fibers along with microscopic porosities. These porosities are covered by the dentin bonding systems. Monomers are then polymerized forming a layer comprising of resin, collagen fibers and mineral crystals called the hybrid layer that enhances the micromechanical retention between the restorative material and the dentin surface (10).

Dentists have been searching for ways to achieve an optimal dentin-resin bond. At present, various acid etchants with different properties are available in the market. All manufacturers claim that their products are ideal for achieving a favorable outcome. Acid etchants are produced by both Iranian and foreign manufacturers. Considering the more reasonable price and easier accessibility of the Iranian products, comparison of their efficacy with the foreign made products seems necessary. This study aimed to compare the effects of Kimiya Iranian acid etchant and Etch-Rite (Pulpdent) on microleakage at the composite-dentin interface under in vitro conditions.

**Methods:**

This in vitro experimental study was conducted on 30 extracted intact human third molars. The teeth were disinfected by immersing in 0.5% chloramine T solution for 24h and were stored in distilled water at room temperature until the experiment. All teeth were cleaned using a rubber cup and pumice paste. Class V cavities measuring 4mm in length, 3mm in width and 2mm in depth were prepared on the buccal surface of teeth using a 008 fissure diamond bur and high speed hand piece (NSK, Japan) under water and air spray. The bur was changed after preparing 10 cavities. The gingival margin of cavities was prepared 1mm below the cementoenamel junction (CEJ) and the occlusal
margin was in the enamel. Specimens were randomly divided into 2 groups of 15. Group 1 were subjected to acid etching with Kimiya gel (Kimiya, Iran) containing 37% phosphoric acid and group 2 cavities were etched with Etch-Rite (Pulpdent, Watertown, MA, USA) containing 38% phosphoric acid. Specimens in both groups were etched for 15s, rinsed with water spray for 20s and gently air dried with air spray for 5s. Two layers of Single Bond adhesive (3M ESPE, St., Paul, MN, USA) were applied to the cavity walls, gently air dried with air spray and light cured for 20s using the Bonart (4F11, Taiwan) light curing unit. The A3 shade of microhybrid Valux Plus composite resin (3M ESPE, St., Paul, MN, USA) was applied in two increments of gingival first and occlusal second and light cured for 40s. All restorations were then polished using Sof-Lex (3M ESPE, St., Paul, MN, USA) polishing discs under similar conditions (from coarse to fine) and all specimens were stored in distilled water at 37°C for 24h.

In order to assess the degree of microleakage, the entire tooth surface was sealed with 2 layers of nail varnish except for 1mm around the restoration margins. The varnish was allowed to dry and the root apices were sealed with sticky wax. The teeth were immersed in 2% basic Fuchsin (Fuchsin Dye, Merck, Germany) for 24h and rinsed under running water. Each restoration was sectioned in half buccolingually using a double blade 0.3 mm thick diamond disc and a cutting machine (Dentarapid, Krupp Dental, 759 DR 2, Hilzingen, Germany). Cutting was done under running water to wash out debris. Sections were evaluated under a stereomicroscope at 40X magnification (SF-100B, Lomo, Russia) and the depth of dye penetration in the occlusal and gingival walls was scored using the following scoring system:

- 0: No dye penetration
- 1: Dye penetration less than ½ of the gingival floor
- 2: Dye penetration more than ½ of the gingival floor
- 3: Dye penetration extending to the gingivo-axial line angle
- 4: Dye penetration to the axial wall

To compare the degree of microleakage, data were analyzed using non-parametric Mann Whitney U test.

### Results:

In this study, the degree of microleakage was evaluated in 30 intact molars in two acid etch systems using a stereomicroscope. The degree of microleakage in the occlusal and gingival walls in the two groups of Kimiya and Etch-Rite is shown in Table 1.

<table>
<thead>
<tr>
<th>Acid etch-surface/Scale</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Etch-Rite occlusal</td>
<td>3</td>
<td>8</td>
<td>1</td>
<td>1</td>
<td>2</td>
<td>15</td>
</tr>
<tr>
<td>2 Etch-Rite gingival</td>
<td>0</td>
<td>4</td>
<td>2</td>
<td>2</td>
<td>7</td>
<td>15</td>
</tr>
<tr>
<td>3 Kimiya occlusal</td>
<td>4</td>
<td>1</td>
<td>1</td>
<td>0</td>
<td>9</td>
<td>15</td>
</tr>
<tr>
<td>4 Kimiya gingival</td>
<td>1</td>
<td>3</td>
<td>1</td>
<td>1</td>
<td>9</td>
<td>15</td>
</tr>
</tbody>
</table>

As seen in Table 1, of group 1 specimens, 3 samples had degree zero, 8 samples had degree 1, one sample had degree 2, one sample had degree 3 and 2 samples had degree 4 microleakage in the occlusal wall. Of group 2 specimens, 4 samples had degree zero, one sample had degree 1, one sample had degree 2, 9 samples had degree 4 and no sample had degree 3 microleakage in the occlusal wall. Of group 1 specimens, 4 samples had degree 1, 2 samples had degree 2, 2 samples had degree 2, 2 samples had degree 3, 7 samples had degree 4 and no sample had degree zero microleakage in the gingival wall. Of group 2 specimens, 1 sample...
had degree zero, 3 samples had degree 1, one sample had degree 2, one sample had degree 3 and 9 samples had degree 4 microleakage in the gingival wall. No significant difference was found in the occlusal wall microleakage between the two groups of Etch-Rite and Kimiya ($p=0.1$). Gingival wall microleakage was not significantly different between the two groups either ($p=0.68$). Overall, no significant difference was found in either the occlusal or gingival microleakage between the two groups.

**Discussion:**

Direct composite restorations are among the popular dental treatments due to advantages like conservative cavity preparation, no need for laboratory phases, conduction of all steps by the clinician, higher precision, fewer patient visits and higher patient and dentist comfort (11). Adequate retention and minimal microleakage are the main criteria for a successful resin restoration. Post-operative hypersensitivity, discoloration of restoration margins and secondary caries are among the consequences of microleakage of oral fluids into the tooth-restoration interface (12, 13). Retention of composite restorations mainly depends on the micro-structural changes in the enamel surface created by the application of an appropriate acid for an appropriate time duration called enamel acid etching. Phosphoric acid is most commonly used for this purpose. However, other acids and methods have also been proposed (14). Chemically changing the enamel surface by acid application in order to modify the surface texture is a suitable method to enhance the resin-enamel bond. In this method, resin tags are formed leading to micromechanical interlocking (15, 16). The concentration of acid and duration of etching are among the important factors affecting the depth of etching or the amount of removed surface enamel (14).

In our study, 37% (Kimiya) and 38% (Etch-Rite) phosphoric acids were used. It has been reported that 30-40% concentration of phosphoric acid has the best efficacy in terms of etching depth, bond strength and degree of microleakage (17). Also, it has been demonstrated that etching for 15s is sufficient (18, 19). The cavosurface angle of cavities in the present study was 90°. Kerejci and Lutz in 1991 showed that the marginal adaptation was higher in conventional cavity walls with 90° cavosurface angle than those with 130° cavosurface angles (20). Considering the results of Bagheri and Ghawamnasiri (2008) (2008) and Ameri, et al. (2010) studies reporting the insignificant effects of enamel margin bevel on decreasing the degree of microleakage, in our study no enamel bevel was done (21, 22). Similar to their results, we found no significant difference in the occlusal and gingival margin microleakage. Several methods have been used to assess the degree of microleakage and the sealability of restorations namely the radioisotopes, dyes, air pressure, neutron activation analysis, bacterial penetration, pH changes and SEM (23). Dye penetration and radioisotopes are among the most commonly used techniques for this purpose. Comparison of these two methods for the assessment of microleakage has demonstrated that direct microscopic observation of dye penetration is more reliable than the radioisotope technique due to greater flexibility, less complexity, easier performance and providing more information (24). The dye penetration technique is more applicable since it is easily accessible and affordable. However, due to the high affinity of Fuchsin to tooth structure and the restorative materials, the size and depth of gaps are usually over-estimated in this technique. In order to prevent this error, we used 2% Fuchsin dye for determination of the degree of microleakage (25, 26).

Etching the tooth surface is the first step in the preparation of tooth for bonded restorations. Acid etchants are used for cleaning and
demineralization of enamel and dentin surfaces to increase the bond strength to restorative materials. At present, different etchants are used and 10-40% phosphoric acid is among the most commonly used agents available in the form of gel and aqueous solution. Aqueous solutions have high wetability and are easily washed out. However, their main drawback is their high flowability causing the undesired etching of healthy tooth surfaces. To overcome this drawback, the gel form of phosphoric acid was introduced containing hydrocarbon polymers or fumed silica as the thickening agents. This product was not flowable but had another drawback. It was sticky and bulky and by quick drying clogged the dispenser. The newer etchants were aqueous acid gels containing acid, pearl-like or elongated-form colloidal silica sol and a selective organic thickening agent (7). Colloidal silica sol as the thickening agent confers thixotropic properties to the compound; as the result, the compound is neither sticky nor flowable with adequate wettability. It does not clog the dispenser either. Colloidal silica sols are comprised of silicon dioxide particles in water. This colloid is made of negatively charged amorphous silicon dioxide particles in water. This compound contains pearl-like sols with 10-60 nm diameter and elongated-form particles with 10 nm diameter and 50-100 nm length. It also contains phosphoric acid by 10-50 weight%. Organic thickening agents include carboxymethyl cellulose, polyethylene oxide and polyacrylic acid salts. The thickening agent is usually added by 0.1-3 weight%. The etching gel may also include fluoride, dyes (methylene blue) or antimicrobial agents (27).

In this study, Kimiya Iranian etching gel containing 37% phosphoric acid and carboxymethyl cellulose thickening agent and Etch-Rite foreign-made etching gel containing 38% phosphoric acid and amorphous fumed-silica thickening agent were used. Comparison of these two etchants revealed that the degree of microleakage was not significantly different between the two groups. Thus, it seems that type of acid does not make any difference in the degree of microleakage and the two acids were not significantly different in this regard.

In this study, both products were etching gels with similar viscosity. Perdigo et al., in 1996 showed that similar concentrations of phosphoric acid with different thickeners caused different demineralization depths and morphologic patterns in dentin. The depth of dentin demineralization caused by the silica-thickened etchant was less than the polymer-thickened etchant. Submicron hiatuses were observed in the lower zone of demineralized dentin in all specimens etched with polymer-thickened phosphoric acid. The least amount of hiatuses was seen in specimens etched with silica-thickened phosphoric acid (27). The presence of polymer (carboxymethyl cellulose) thickening agent in Kimiya etching gel, with the reportedly highest rate of hiatuses in the lower zone of demineralized dentin, justifies the greater microleakage in Kimiya etching gel group compared to Etch-Rite.

Limited data available regarding the Kimiya etching gel all belong to the previous products of the company reporting that the etching gel is not easily and adequately washed off the surface and leaves deposits that lead to higher microleakage. In our study, after the application of Kimiya etching gel and rinsing, no acid deposit was left on the surface; this may indicate the changed composition of the newly manufactured products and explain the difference in this respect between our study results and the previous ones.

**Conclusion:**

Within the limitations of this study, it may be concluded that the degree of microleakage following the use of etchants containing polymer thickening agent (Kimiya etching gel) is comparable to that following the application of
etchants with fumed silica thickening agent (Etch-Rite etching gel).

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Conflict of Interest: “None Declared”

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372.