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Effects of Pre-Etching in Class V Cavities Restored with Silorane and Methacrylate-Based Composites

Efekty wstępnego wytrawiania ubytków klasy V wypełnionych materiałami kompozytowymi na bazie siloranów i metakrylanów

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Abstract

Background. Self-etching adhesive (SEA) systems were recently introduced to simplify the bonding procedure and were initially considered a good replacement for the etch-and-rinse adhesive system that uses orthophosphoric acid (PA) for enamel and dentin etching. Siloranes, a new class of ring opening monomers, were synthesized to overcome the problems related to polymerization shrinkage. This new type of monomer is obtained from the reaction of oxirane and siloran molecules with a volumetric shrinkage determined to be 0.99%.

Objectives. To assess the influence of preliminary phosphoric acid etching on the sealing ability of silorane- and methacrylate-based composites.

Material and methods. Standard class V cavities were prepared on the buccal side of 48 extracted, sound, human premolars. The specimens were randomly divided in two groups: A) Silorane System®/Filtek Silorane®; B) Scotchbond Universal®/Filtek Supreme®. Each group was divided in two subgroups. A1) and B1): no pre-etching was performed. A2) and B2): selective, enamel pre-etching was performed. The interfacial sealing ability of the materials was evaluated by scoring the depth of methylene blue penetration through optical microscope observations. The differences in infiltration scores recorded for the tested materials were evaluated for statistical significance (Kruskal-Wallis ANOVA, Mann-Whitney U test, p < 0.05).

Results. No groups showed a “0” score, and group B2 had the lowest individual score, reaching at least a “2” score. In the silorane groups, pre-etching decreased the infiltration score but the result was not statistically significant. No statistically significant differences emerged among the tested materials except for the Bis-GMA composite restored group where the pre-etching significantly reduced the interfacial leakage (p < 0.05).


Key words: adhesion, microleakage, acid-etching, silorane, self-etch.

Słowa kluczowe: adhezja, mikroprzeciek, wytrawianie kwasem, silorany, samowytrawianie.
ite to enamel significantly lower when compared with the etch-and-rinse system, due to its lower etching capability [3].

The better result achieved with the total-etch system seems to be correlated with the particular morphology of the interface obtained using 34–37% PA for enamel etching [4].

Self-etching primers are less aggressive than PA, do not form a proper and defined acid etching pattern and the conditioning effects are also reduced on intact enamel surfaces [5].

Selective etching of enamel with PA is a potential technique to improve the SEA system’s capability to achieve a higher bond strength of composite to enamel. It has been shown that pre-etching could provide a more retentive bonding substrate and more uniform resin-enamel bonding interface [6].

Selective etching of enamel with PA and acidic self-etching monomers could cause a sufficient hybridization of enamel, which results in a reliable bond strength on enamel surface [7].

The combined action of PA and acidic self-etching monomers could cause a sufficient hybridization of enamel, which results in a reliable bond strength on enamel surface [7].

The majority of composites used in restorative dentistry are chemically based on the polymerization reaction of methacrylates. The conversion of methacrylate monomers into a polymer network, through the formation of covalent bonds, leads to a reduction of molecular distances and volume reduction generating polymerization stress [8]. Negative clinical outcomes of composite restorations have often been associated with polymerization shrinkage and contraction stress, which may cause micro cracks, debonding, post-operative sensitivity, secondary caries and marginal gaps [9–12].

Siloranes, a new class of ring opening monomers, were synthesized to overcome the problems related to polymerization shrinkage. This new type of monomer is obtained from the reaction of oxirane and silorane molecules with a volumetric shrinkage determined to be 0.99%. The compensating mechanism for stress in this new system is achieved by opening and extending the oxirane ring, during polymerization, to compensate for volume reduction [13, 14].

In order to ensure the best possible bonding between the silorane composites and hard dental tissues, a special adhesive has been developed. Silorane adhesive compositions include a hydrophilic one-step self-etching primer and a hydrophobic viscous bond coating resin [15]. According to the manufacturer, the silorane primer presents a pH of 2.7, which provides mild etching and demineralization of the tooth structure, as well as a strong and durable bond.

Several laboratory and clinical studies on self-etch adhesives have shown that while adequate bonding could be achieved on dentin, the same approach does not ensure the same results on enamel [16, 17]. Thus, preliminary phosphoric acid etching has been suggested to improve the adhesion to enamel [18–20].

The purpose of this experimental study was to analyze the effects of pre-etching in two different SE systems.

**Material and Methods**

**Sample Preparation**

Two commercially available composites were used in this study. The first composite (Filtek Silorane® 3M, 3M Italy, Milan, Italy) is based on a siloranic resin, while the second composite (Filtek Supreme® XTE 3M, 3M Italy, Milan, Italy) is a Bis-GMA nano-composite.

Forty-eight sound teeth extracted for periodontal reasons were used, stored in a 1% chloramidine solution at room temperature (20°C) for maximum 72 h.

The radicular apexes of the teeth were sealed with epoxy resin and class V cavities were prepared with restoration margins placed in enamel and the bottom of the cavity in dentin. Cavities, with dimensions of 4 × 3 mm and 2 mm of depth were prepared on the buccal surface of each tooth, forming, in section, an axial wall and two perpendicular (cervical and coronal) walls. Variation of ± 1 mm was considered acceptable [21] (Fig. 1).

The 2 mm depth was chosen in order to assure that the resinous materials could be totally...
placed in one single increment. The cervical margin of the cavity was placed about 1.5 mm coronal from the cement-enamel junction. The cavity preparation was carried out by a single operator, using diamond drills (Komet ISO 233012, Komet Italy, Milan, Italy) mounted on a high-speed hand piece (Silent Power Gold CASTELLINI, Castellini, Bologna, Italy) under abundant water cooling [22].

The specimens were randomly divided into two groups (24 teeth each) in relation to the adhesive and the material used for the restorative phase. In the first group (A), a two-step self-etching adhesive (Silorane System 3M ESPE) and a low-shrinking composite (Filtek Silorane 3M ESPE) were used. In the second group (B), a one-step self-etching adhesive (Scotchbond® Universal 3M ESPE, 3M Italy, Milan, Italy) and a nano-filled composite (Filtek Supreme XTE 3M ESPE) were used.

Before the adhesive procedure, each group (A and B) was divided into two subgroups of twelve teeth each (A1–A2 and B1–B2). In the specimens of subgroups A1–B1, no pre-etching was performed. In the subgroups A2–B2, selective enamel pre-etching (15 s) with 37% PA (Scotchbond Universal Etch 3M ESPE) was performed. The cavities were rinsed and dried according to the wet technique [23].

In groups A1–A2, the adhesive used was a two-step Silorane Adhesive System (3M ESPE). According to the manufacturer’s instructions, the primer was applied for 15 s, and cured for 10 s, then bonding was applied and light-cured for 20 s. The restorative composite resin was placed in bulk and light cured for 40 s. Light-polymerization was performed using a LED-curing device (LED Anthos T, CEFLA S.C., Imola, Italy) at 1000 mW/cm².

In groups B1–B2, the self-etching adhesive was applied and rubbed on the cavity walls for 20 s, and then light cured for 10 s. The composite resin used in this group was light cured for 20 s.

All the restorations were finished with flexible polishing disks (Sof-Lex, 3M ESPE, 3M Italy, Milan, Italy), Medium (40 µm), Fine (24 µm), and Super fine (8 µm), mounted on a slow speed hand piece operating at 5000 rev/min for 20 s, according to the manufacturer’s instructions.

To control the marginal adaptation, all operating procedures were carried out using a head-mounted loupe (EyeMag Pro S Zeiss, Carl Zeiss S.p.A., Milan, Italy) at 4× magnification [24].

After 1 week of water storage at 37°C, the teeth were thermo-cycled (1.800 cycles at 5°C and 55°C with 60 s dwell time and 10 s transfer time).

Subsequently, the entire outer surface was covered with a nail varnish within 1 mm of the restoration margins, and, following the manufacturer instructions, stored for 24 h to allow the varnish to dry. Subsequently, the teeth were immersed in a 7% methylene blue solution at room temperature for 3 days. The specimens were then water rinsed and sectioned longitudinally at the center of the restoration, obtaining two sections.

**Optical Microscopy and Microleakage Assessment**

The tracer infiltration was evaluated using an optical microscope (OPMI PROergo 57B Zeiss, Carl Zeiss S.p.A., Milan, Italy) with a magnification of 12.5×.

Infiltration assessment and measurement followed the scheme proposed by Osorio et al. [25], which assigns a progressive score in accordance with the infiltration extent (Table 1) (Fig. 2).

Two different operators separately carried out the evaluation. In case of disagreement, the highest score was used for statistical analysis when the score was assigned.

## Statistical analysis

A Kruskal-Wallis Non-Parametric Analyses of Variance (ANOVA) was applied to assess the significance of the differences in infiltration scores recorded for the tested materials (Table 4). For post hoc comparisons, a series of Mann-Whitney U tests was used (Tab. 5). In all the tests, the level of significance was set at p < 0.05, and calculations were handled by IBM SPSS Statistics software (IBM Corporation, New York, USA).

## Results

The descriptive statistics and score distribution of this experimental in vitro study are shown in Tables 2 and 3.

<table>
<thead>
<tr>
<th>Score</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>No dye penetration</td>
</tr>
<tr>
<td>1</td>
<td>Dye penetration at the interface to 1/2 the depth of the cavity wall</td>
</tr>
<tr>
<td>2</td>
<td>Dye penetration to the full depth of the cavity wall, but not including the axial wall</td>
</tr>
<tr>
<td>3</td>
<td>Penetration to and along the axial wall</td>
</tr>
</tbody>
</table>
The Kruskal-Wallis test showed the presence of statistically significant differences between the tested materials \( (p = 0.028) \) (Table 4). No groups showed a mean overall “0” score. Moreover, the pre-etching technique improved the overall outcome, increasing the mean number of “0” scores found in each group. Both A1 and A2 groups showed a score of “3”. The B2 group had the lowest individual score, reaching at least a “2” (Table 2 and 3).

The Mann-Whitney \( U \) test showed that pre-etching had a significant effect in the Bis-GMA composite restored group (Table 5).

The comparison between the two silorane subgroups (A1, A2) showed that the pre-etching technique decreases the average infiltration value, from a mean value of 1.25 (A1) to 0.58 (A2), however this outcome was not statistically significant (Table 5).

In the evaluation of the pre-etching procedure on the two different restorative materials, silorane and methacrylate, a similar performance was observed, with infiltration mean values respectively of 0.58 and 0.41, which were not significantly different (Table 5). When the pre-etching technique was not performed, in the silorane composite group an average grade of infiltration lower than in the Bis-GMA composite (1.25 vs 1.5) was observed (Table 2). Even this last comparison was not statistically significant (Table 5).

Fig. 2. Score examples: arrows indicate blue colorant solution infiltration. Ce.W. – cervical wall, A.W. – axial wall, Co.W. – coronal wall
Table 2. Descriptive statistics of infiltration score data

<table>
<thead>
<tr>
<th>Specimen</th>
<th>A1 Group (SEA + silorane)</th>
<th>A2 Group (PA + SEA + silorane)</th>
<th>B1 Group (SEA + Meth. Comp.)</th>
<th>B2 Group (PA+ SEA + Meth. Comp.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>3</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>0</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>0</td>
<td>1</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>0</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>1</td>
<td>0</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>7</td>
<td>2</td>
<td>0</td>
<td>2</td>
<td>0</td>
</tr>
<tr>
<td>8</td>
<td>3</td>
<td>0</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>9</td>
<td>0</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>10</td>
<td>1</td>
<td>0</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>11</td>
<td>2</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>12</td>
<td>3</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Mean</td>
<td>1.25</td>
<td>0.58</td>
<td>1.41</td>
<td>0.41</td>
</tr>
</tbody>
</table>

Table 3. Score distribution in the different groups

![Score distribution chart]

Table 4. Kruskal-Wallis analysis

<table>
<thead>
<tr>
<th>Adjusted H</th>
<th>d.f.</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.108</td>
<td>3</td>
<td>0.028</td>
</tr>
</tbody>
</table>

Discussion

Different authors have compared the influence of the pre-etching technique to many other chemical or mechanical techniques on the effectiveness of self-etching adhesives. Fitzgerald et al. [26] demonstrated how a pre-etching step with orthophosphoric acid (37%) for 5 s is preferable to a mechanical preliminary treatment. Nazari et al. [7] recommended the pre-etching prior to application of the adhesive instead of grinding.

Lee et al. [27] showed that the additional acid etching both on enamel and dentin improves bond strength when low acidic one-step self-etching adhesives are used, but adverse effects of additional acid etching, particularly on dentin, were reported.

These conclusions are linked to the possibility of overly-aggressive dentin etching that might cause an excessive removal of the dentinal smear.
layer, hindering a complete monomer infiltration into the collagen network [28].

Moreover, several studies have reported a lower etching capability of self-etching adhesives on enamel, which can be caused by their relatively lower acidity compared to the 37% phosphoric acid used in most of the total-etch systems [29, 30]. This additional acid etching step should work selectively on the enamel and determines increased bond strength values. It could be speculated that the better adhesive performances are linked to a micro-mechanical retention determined by enhanced enamel porosity [28].

This effect is not the only parameter to focus on. Carvalho et al. [12] and Osorio et al. [25] have reported that it is possible that the efficacy of a self-etching primer is not related only to pH level, but also may be caused by the presence, after curing, of unpolymerized acid monomers.

Therefore, in the present study, the authors chose the self-etching adhesive approach because it is clinically most promising, since it eliminates the etching and the rinsing phases, reduces operative application time and greatly decreases the sensitivity of the technique or the possibility of making mistakes [3, 29]. Self-etch adhesive systems create a wider protected collagen fiber area, due to the simultaneous infiltration of monomers that incorporates the smear layer into the hybrid layer [7].

When the self-etching adhesive was used without pre-etching, we observed that siloranes ensured a better seal, while, with pre-etching, the better adhesive effectiveness compensated for the different shrinking degree, and the infiltration score decreased significantly in the methacrylic-resin-restored group, which showed the best performance.

The silorane group without pre-etching achieved the best results and this could be linked to the ring shape polymerization reaction and the consequently low-shrinkage [13, 14].

The results from this study are consistent with those of Krifka et al. [31], who analyzed microleakage in class V comparing methacrylates and siloranes. They observed that silorane-based composites exhibited the best marginal seal.

Furthermore, other studies have underlined the advantages of the use of siloranes compared to other restorative materials. Silorane-based composites were more efficient for high C-factor cavities [8].

The etching patterns of silorane primer are considered mild (pH 2.7) and may cause a reduced demineralization of the intact enamel surfaces. In our study, the limited thickness of the restoration and the low shrinking behavior of the composites may have determined lower infiltration scores [5].

The quality of marginal and internal adaptation of the Filtek Silorane composite was recently evaluated by Gregor et al. [22], who compared different protocols of adhesive application.

The parameter analyzed was the continuous margin, defined as the adaptation of the luting agent to enamel. Selective enamel etching prior to the application of the adhesive showed a significantly higher number of infiltration-free specimens compared to the “non-etched” groups (p > 0.05).

Many authors have investigated the association between a silorane system adhesive and different pre-etching techniques too. Ustunkol et al. [32] compared it to laser etching and to a control group without any preliminary treatment. As with our results, they revealed that PA treatment seems the most promising surface treatment for increasing the enamel bond strength of silorane adhesive systems.

Poureslami et al. [33] analyzed the effect of pre-etching on the marginal adaptation of silorane resins in primary teeth too, concluding that this technique, used with different composite resins, provides the lowest marginal micro-leakage in the primary teeth.

In our research, in the methacrylate resin group, pre-etching significantly improved the quality of the seal compared to the specimens where only self-etching was used.

The results achieved in the Bis-GMA restored group, when PA was applied, showed the best performances, even when compared to the low-shrinking composite group too.

In this study, independently of the restoration material used, the pre-etching groups showed

<table>
<thead>
<tr>
<th>Group</th>
<th>N</th>
<th>Mean (SD)</th>
<th>p-value</th>
<th>Significance (p &lt; 0.05)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>12</td>
<td>1.25 (1.139)</td>
<td>0.15854</td>
<td>A</td>
</tr>
<tr>
<td>A2</td>
<td>12</td>
<td>0.58 (0.90)</td>
<td>0.0164</td>
<td>B</td>
</tr>
<tr>
<td>B1</td>
<td>12</td>
<td>1.41 (0.99)</td>
<td>0.70394</td>
<td>A</td>
</tr>
<tr>
<td>B2</td>
<td>12</td>
<td>0.41 (0.66)</td>
<td>0.74896</td>
<td>A</td>
</tr>
</tbody>
</table>
a better quality of seal than groups without pre-etching. This data is evidenced considering both the average infiltration scores and the number of specimens in each group with no infiltration (Table 3). Furthermore our microscopic reliefs confirmed the low number of specimens with highest infiltration.

The current experimental study demonstrated that the pre-etching of enamel with orthophosphoric acid significantly reduced the degree of marginal leakage in class V cavities restored with Bis-GMA composite and a self-etching adhesive system. In addition, the low-shrinking silorane composite demonstrated the ability to reduce marginal infiltration.

According to this data, it can be concluded that the enamel pre-etching technique guarantees a good seal when self-etching adhesive systems are used, and is especially indicated when Bis-GMA resins are used.

Because of the reduced polymerization shrinkage of the silorane composite compared to the traditional methacrylate composite, the interface is exposed to significantly less stress, so the need for a pre-etching phase is reduced. However, pre-etching is still a step that the dental clinician should take into consideration to obtain better clinical performances even for these new low-shrinking silorane composites.

References


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