**ABSTRACT**

This study aimed to evaluate the influence of the association of different adhesive systems with a silorane-based resin composite on the microleakage of class V cavities. Fifteen premolars were freshly extracted and randomly divided into three experimental groups, according to the adhesive system used, as follows: P-90: Filtek P-90 adhesive; SE PLUS: Adper SE Bond adhesive; and SBMP: Adper Scotchbond Multi-Purpose adhesive. Class V cavities were constructed on the buccal and lingual surface of each premolar exhibiting enamel margin at the occlusal surface and dentine margin at the cervical surface. The cavities were filled with a silorane-based resin composite (Filtrek™ P90 Silorane Low Shrink Restorative, 3M ESPE). After 24 hours of water storage at 37 °C, the restoration was finished and polished. The teeth were water sealed, immersed in 50% silver nitrate for 24 hours, followed by immersion in a radiographic developing solution for 2 hours. The samples were cut and the degree of dye penetration was assessed with the aid of stereoscopic loupe at x20 magnification, through a 4-point score system (0 to 3). The mean microleakage values were: P-90 = 23.48; SE PLUS = 31.13; and SBMP = 36.90. The results were submitted Kruskal-Wallis and Mann-Whitney tests, showing statistically significant differences among groups (p ≤ 0.05). P-90 showed significant difference when compared to SBMP, exhibiting the smallest scores of dye penetration. The association of different adhesive systems with the silorane-based resin composite (Filtrek P-90) influenced on the microleakage degree.

**KEYWORDS**

Adhesion; dental leakage; resin.

**INTRODUCTION**

The use of direct restorative materials, such as resin composites, has been increasingly accepted in Dentistry. Most of the resin composites currently in the market are methacrylate-based whose setting is achieved through double-carbon bonding of the organic matrix, from a source of free radicals [1], consequently reducing the intermolecular spaces around 0.3-0.4 nm and resulting in the decrease of the volume of the restorative material [2]. The main disadvantage of these materials is the polymerization shrinkage which results in stress forces at the adhesive interface [3]. Therefore, formulas of these materials have been continuously modified to improve their clinical performance because the shrinkage generated during the polymerization reaction jeopardized the marginal integrity of the restoration, leading to marginal leakage, post-operative sensibility, microleakage and adhesive failures [4,5]. Microleakage can be defined as the passage of fluids, bacteria or
molecules between a cavity wall and the restorative material because of the presence of micrometric spaces [1].

A new category of resin monomers has been developed as an alternative to reduce the polymerization shrinkage below to 1% of its volume, and it is based on the combination of siloxane and oxirane molecules, so-called silorane [1]. The opening of the cationic rings of the oxirane radicals accounts for the low shrinkage and stress generation, while the silorane gives the hydrophobic nature to the material [6].

Because of the combination of its chemical components, the silorane-based composite has revealed reduced polymerization shrinkage [1], biocompatibility, hydrophobia [7] and physical-mechanical properties comparable with those presented by the methacrylate-based composites [7-9]. The two-step self-etch silorane-based adhesive system is specific and comprised: 1) a one-step self-etch hydrophilic primer which is light-cured and contained mono- and bifunctional methacrylate phosphate; and 2) an adhesive agent composed of a viscous hydrophobic resin based on a chemical composition with bifunctional methacrylate [10], which could suggest a certain compatibility with the methacrylate-based composites. However, the degree of microleakage of silorane-based resin composites associated with either its adhesive system or methacrylate-based adhesive systems is little known. Although this new composite may show a strong bonding to similar materials, its bonding capacity to materials displaying different chemical compositions is still unknown. Thus, the hypothesis of this present study was to evaluate in vitro the effect of different adhesive systems associated with a silorane-based composite resin on the microleakage degree of class V cavities.

The null hypothesis was that there were no differences in the association of different adhesive systems with the silorane-based resin composite regarding to the microleakage of class V cavities.

**MATERIAL AND METHODS**

Fifteen sound human premolars freshly extracted at the Clinic of Dental Surgery of the School of Dentistry of the Federal University of Rio de Janeiro (UFRJ) were selected. These teeth had their extraction indicated because of either periodontal disease or orthodontic reason. This research was approved by the Ethical Committee in Research of both the Study Institute of Collective Health (short IESC - Rio de Janeiro – BR) and UFRJ - under protocol number 69/2010. All patients or their parents signed a Free and Clarified Consent Form to agree in participating of the research and the Consent Form of Permanent Tooth Donation to donate the teeth.

On each tooth, two class V standardized cavity preparations were performed: one at the buccal surface and one at the lingual surface. The cavity preparations were constructed with the following measurements: 2.0 mm depth, 3.0 mm of mesial-distal extension and 3.0 mm of occlusal-cervical extension, with a size 330 carbide bur (KG Sorensen, Cotia, São Paulo, Brazil), at high speed, under copious irrigation. The margins exhibited enamel at the occlusal surface and dentine at the cervical surface.

Then, the teeth were randomly assigned into three experimental groups according to the adhesive systems: P-90: Filtek P-90 (3M ESPE); SE PLUS: Adper SE Bond (3M ESPE); SBMP: Adper Scotchbond Multi-Purpose (3M ESPE). Table 1 described the composition of all materials used in the study.

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Type</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>P-90</td>
<td>3M ESPE, St. Paul, MN, USA (batch: 9BL / 9BH)</td>
<td>2-step self-etch system</td>
<td>Self-etch Primer methacrylate phosphate, copolymer of Vitrebond, Bis-GMA, HEMA, water, ethanol, silica particles treated with silane, initiators, stabilizers Bond Hydrophobic dimethacrylate, methacrylate phosphate, TEGDMA, silica particles treated with silane, initiators, stabilizers</td>
</tr>
<tr>
<td>Adper SE Plus</td>
<td>3M ESPE, St. Paul, MN, USA (batch: 8BA / 8BB)</td>
<td>2-step self-etch system</td>
<td>Flask A water, 2-hydroxyethyl methacrylate and rose bengal sodium Flask B surface treated with zirconia, TEG-DMA, di-HEMA phosphate, mono-HEMA phosphate, methacrylate pyrophosphate, tri-HEMA phosphate, 6-phosphoric acid-methacryloxy phosphoric ester methacrylate hexyl esters, dimethacrylate 1,6-hexanediol, urethane dimethacrylate, trimethylpropane trimethacrylate, ethyl 4-aminobenzoate methyl and DL-camphorquinone</td>
</tr>
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</table>
The cavity preparations were submitted to the different restorative treatments, according to the experimental groups. Each material was applied according to the manufacturer’s instructions. In P-90 group, the self-etch primer was applied for 15 s with the aid of an applicator brush, followed by a gentle air jet to evaporate the solvent and to obtain a uniform film and light-cured for 10 s. The bonding agent was applied for 15 s with a new applicator brush followed by a gentle air jet to obtain a uniform film and light-cured for 10 s.

In the SE PLUS group, the Flask A liquid was applied with the aid of an applicator brush to achieve a continuous red layer. By using another brush the Flask B liquid was applied by rubbing it onto all moistened surface until the red color disappeared, which indicated that all self-etching components were activated. The brush was rubbed for more 20 s to assure a proper etching. Next, a gentle air jet was applied to evaporate the water and a second layer of the Flask B liquid was applied followed by another air jet and light-cured for 10 s.

In the SBMP group, a previous conditioning was performed with 37% phosphoric acid (Super etch - SDI, St. Brunsdon, Australia (batch: 070741)) for 30 s onto the enamel and 15 s onto the dentine, washed with water for 15 s, and followed by the removal of the water excess with a piece of absorbent paper without excessively drying the dentine, followed by the application of the primer and a gentle air jet for 5 s, and the bonding agent application for 10 s.

All teeth were restored with the same resin (Filtek P-90, 3M ESPE, St. Paul, MN, USA), A3 shade, inserted in three increments: the first two were placed sideward towards the occlusal and cervical walls; the last increment was placed to fill the cavity. Each increment was light-cured for 40s with a LED light-curing unit (G-Light, GC Corporation, Tokyo, Japan). The light intensity of the device was periodically monitored at 600 mW/cm² in average. The teeth were kept for 24h in distilled water at 37°C. Then, the restorations were finished and polishing with the aid of Soflex discs in decreasing order (3M ESPE, St. Paul, MN, USA) and the teeth were again stored in distilled water for seven days. The teeth were then waterproofed with two thin layers of red nail polish, leaving a 2.0-mm free margin around the restorations. The root apexes received a complementary sealing with epoxy resin (Loctite, Henkel Ltda, Itapevi, São Paulo, Brazil). The specimens were immersed in an aqueous solution of 50% silver nitrate (B. Herzog Varejo Produtos Químicos Ltda., Rio de Janeiro, RJ, Brazil) for 24h, under light, washed for 20 min and placed into a radiographic developer solution (Kodak Dental, Carestream Health, Inc. Rochester, New York, USA), for 2h [11,12]. All nail polish layer were removed with the aid of a Lecron spatula (SS White Duflex – SS White – Rio de Janeiro – Brazil) and the specimens were included into acrylic resin (Clássico, São Paulo, SP, Brazil). The specimens were sectioned at the buccal-lingual direction at the center of the restoration with the aid of double-side diamond discs. The surfaces were polished with the aid of 600-grit sandpaper (ERIOS, Ipiranga, São Paulo, Brazil), by using a circular polisher (DP-10, Panambra, São Paulo, SP, Brazil), under copious irrigation. The specimens were evaluated by three double-blind examiners calibrated (inter- and intra-examiner) regarding to the degree of microleakage at tooth-restoration interface at the occlusal and cervical margins with the aid of a stereoscopic loupe (XTD 217, LabKlass, Buenos Aires, Argentina) at x20 magnification, through a 4-point score system (table 2), modified from Dalli et al., 2010 [13].

| Adper Scotchbond Multi-Purpose Plus | 3M ESPE, St. Paul, MN, USA (batch: 8BU) | 3-step conventional adhesive system | Acid 35% phosphoric acid and thickener
| Filtek P-90 | 3M ESPE, St. Paul, MN, USA (batch: 9ER) | Microhybrid resin composite | Silorane resin, initiator system (camphorquinone, iodonium salt, electron donor), particles of quartz, yttrium fluoride, stabilizers and pigments
| Super etch | SDI, St. Brunsdon, Australia (batch: 070741) | Phosphoric acid etching gel | 37% phosphoric acid and thickener
**Table 2 - Dye infiltration depth scores**

<table>
<thead>
<tr>
<th>SCORE</th>
<th>MEANING</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Total lack of dye penetration into the tooth/restoration interface</td>
</tr>
<tr>
<td>1</td>
<td>Dye penetration into either the occlusal or cervical wall without reaching the axial wall</td>
</tr>
<tr>
<td>2</td>
<td>Dye penetration into either the occlusal or cervical wall reaching the axial wall</td>
</tr>
<tr>
<td>3</td>
<td>Dye penetration further than the axial wall towards the pulp</td>
</tr>
</tbody>
</table>

The results obtained were tabulated and submitted to the nonparametric statistical analysis through Kruskal-Wallis and Mann-Whitney tests for multiple comparisons with level of significance of 5%.

**Results**

To verify the differences existing among the three groups studied, the nonparametric Kruskal-Wallis test was used, which compare the type of the adhesive system used during the restoration in relation to the lack of microleakage, adopting a level of significance of 5% (α= 0.05).

Figure 1 shows the mean values for the microleakage test in the different groups.

![Figure 1 - Mean values for the microleakage test in the different groups (n= 5); kruskal-wallis test (p ≤ 0.05).](image)

There were no statistically significant differences between P-90 and SE PLUS groups, and between SE PLUS and SBMP groups. SBMP exhibited the highest degree of microleakage when compared to P-90. Concerning to the degree of microleakage related to the restoration margins, the data submitted to Mann-Whitney test showed statistically significant differences between the occlusal (22.30) and cervical (38.70) regions with p ≤ 0.05. Generally, the cervical margins showed the highest degree of microleakage.

**Discussion**

Silorane-based composites were launched into dental Market as an alternative to methacrylate-based composites because of their lowest polymerization shrinkage. As their matrix significantly differs from that of methacrylate-based composites, a new adhesive system was developed to enable the bonding of this new composite to the dental tissues. However, this adhesive system still has the characteristics similar to the conventional adhesive systems, especially regarding to its mechanism of bonding to the enamel and dentine. The adhesive system of the silorane-based composites little differs from a common 2-step self-etch adhesive system, including the application of two resin solutions: an essentially hydrophobic primer to bond to the dental tissue; and a hydrophobic adhesive to bond appropriately the dental substrate previously conditioned by the primer to the hydrophobic composite [14].

The microleakage assessment is the most common method to evaluate the efficiency of the sealing of several restorative materials [15-18]. It is commonly evaluated in vitro through studies of dye penetration to detect the adhesive failures in the enamel-resin adhesive interface [19-21]. Notwithstanding, there is not a gold-standard dye, therefore in this study, 50% silver nitrate solution was employed for 24h, similarly to other studies [22,23].

Class V cavities were used because of their higher C-factor, relatively easier to restore, consequently leading to less inter-examiner variability. Moreover, this cavity type exhibited margins both in enamel and dentine and lack of any mechanical retention [24].

When the degree of microleakage of Filtek P-90 resin composite associated with its own adhesive system is compared with methacrylate composite resins, it exhibited a smaller degree of microleakage [25], because of its polymerization shrinkage [1]. However, little is known regarding to the association of other adhesive systems with this resin composite.

A study compared the degree of conversion of Filtek P-90 resin composite associated with different conventional and self-etch adhesive systems and it showed that the association of the conventional adhesive systems (Scotchbond Multi-Purpose Plus and Adper Single Bond 2) could be used as bonding agents together with Filtek P-90 composite, because
they has a highest degree of conversion. P-90 adhesive system showed an intermediate degree of conversion, and the self-etch adhesive systems (Adper SE Bond and Easy One Bond) exhibited the lowest values [26]. However, in this present study, the self-etch adhesive system (SE Plus) did not exhibit statistically significant difference compared with P-90 adhesive system regarding to the degree of microleakage, as seen in graph I. This would indicate that the association of other adhesive systems with Filtek P-90 composite resin seems to be a viable clinical alternative.

In this present study, a higher degree of microleakage was observed at the cervical area because of the lack of enamel, regardless of the restorative protocol used. According to Hashimoto et al [18], the presence of a enamel margin at the tooth/restoration interface would assure a more stable bonding, therefore reducing the degree of microleakage and the long-term restoration failure.

According to Duarte Jr. et al [27], the adhesive system of the silorane-based composite can be considered as a “soft” self-etch adhesive system because its pH is around 2.7, resulting in an intense intertubular microporosity with presence of residual smear layer on the dentinal surface and the maintenance of the smear plug. This showed that the silver capitation found all over the hybrid layer would indicate the presence of porosity inside this layer, consequently with microleakage at the adhesive interface. The authors also reported that the cationic polymerization systems are susceptible to the moist presence and when the contact of the silorane-based composite with moisture occurs, the water will compete with the monomers from the oxonium ions and the cationic reaction will be inactivated, therefore affecting the polymerization. Considering that the water within the self-etch P-90 adhesive system reacts with the methacrylate phosphorylate and generates the hydrogen ions necessary to demineralize the dental substrates, if this water remained within either the collagen net or the adhesive interface, either areas of incomplete polymerization or hydrogels would be formed [28]. According to Tay et al [29], the non-polymerized resin monomers of the adhesive system would initiate an osmotic pressure gradient within the dentinal tubules, with hybrid layer degradation after the restoration aging, which would result in microleakage.

The conventional adhesive systems, such as Adper Scotchbond Multi-Purpose, demand a previous step of phosphoric acid conditioning, both on enamel and dentine. Therefore, the technique becomes more sensible when compared with the self-etch adhesive systems, which do not require a control of the etching time and of the dentinal moist by the presence of an acidic primer in a single flask. In the conventional adhesive systems, a failure in the control of the acid time period may cause adhesive failures [5], collapse of the collagen fibers, with consequent gaps formation and microleakage [4]. In an study with class V restorations, Rosales Leal et al [30] showed that the simulation of the intrapulpar pressure induced a higher micropermeability in conventional adhesive than in one-step self-etch adhesive systems. This would confirm the fact that SBMP showed the highest degree of microleakage in this present study. P-90 and SE PLUS groups did not show significant statistical differences regarding to the dye penetration. Because both agents are self-etch adhesive systems, their action mechanisms are similar. Although P-90 composite does not have in its chemical composition methacrylate monomers, its adhesive system still contains Bis-GMA monomers. This would result in polymerization shrinkage of the adhesive system, leading to microleakage at the adhesive layer of P-90 similar to that of SE PLUS.

Despite the fact that the P-90 adhesive system (specific for the silorane-based resin composite) exhibited the smallest dye penetration values, it was not capable of perfectly sealing the tooth/restoration interface. Therefore, since the degree of microleakage showed by P-90 and SE PLUS groups did not exhibit statistically significant differences, further studies are necessary to determine the real necessity of using a specific adhesive system prior to the restoration with Filtek P-90 composite. Even with the studies reporting that the silorane-based composite showed the smallest polymerization shrinkage than methacrylate-based ones, microleakage is still a problem to be solved [7,28].

Thus, further studies are necessary to evaluate the association of different adhesive systems with Filtek P-90 resin composite.

**Conclusion**

The association of different adhesive systems with Filtek P-90 silorane-based resin composite influenced on the degree of microleakage. The specific adhesive system of Filtek P-90 composite showed the smallest degree of microleakage than SBMP.

The use of different adhesive protocols with the Filtek P-90 composite, which is not recommended by the manufacturer, should not be performed until more studies are conducted, especially in vivo.
Resumo

O objetivo deste estudo foi avaliar a influência da associação de diferentes sistemas adesivos a um compósito à base de silorano na microinfiltração de cavidades Classe V. Quinze pré-molares recém-extraídos e divididos aleatoriamente em três grupos experimentais, de acordo com o sistema adesivo empregado, sendo: P-90: Adesivo Filtek P-90; SE PLUS: Adesivo Adper SE Bond; e SBMP: Adesivo Adper Scotchbond Multi-Uso. As cavidades de classe V foram confeccionadas nas faces vestibular e lingual de cada pré-molar com margem occlusal em esmalte e cervical em dentina. As cavidades foram restauradas com um compósito à base de silorano (Filtek™ P90 Silorane Low Shrink Restorative, 3M ESPE). Após 24 horas de armazenamento em água a 37 °C, as restaurações receberam acabamento e polimento. Os dentes foram impermeabilizados, imersos em nitrato de prata a 50% por 24 horas e, posteriormente, em solução reveladora de radiografias por 2 horas. As amostras foram secionadas e o grau de penetração do corante foi avaliado por meio de uma lupa estereoscópica com 20 vezes de aumento, utilizando-se um sistema de escores de 0 a 3. Os postos médios foram: P-90 = 23.48; SE PLUS = 31.13; e SBMP = 36.90. Os resultados foram tratados pelos testes de Kruskal-Wallis e Mann-Whitney, que revelaram diferença estatisticamente significante entre os grupos (p ≤ 0.05). P-90 apresentou diferença significante se comparado ao SBMP, exibindo menores escores de penetração do corante. A associação de diferentes sistemas adesivos ao compósito a base de silorano Filtek P-90 influenciou no grau de microinfiltração.

Palavras-chave:
Adesão; infiltração dentária; resina.

Referências


