

# The Effects of Surface Pretreatment on the Microleakage of Resin-Modified Glass-Ionomer Cement Restorations

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**Objective:** To evaluate the effect of different surface pretreatments on the marginal microleakage of Vitremer restorations. **Study Design:** Class V cavities were prepared on the labial and lingual surfaces of extracted human third molar teeth. Cavities were randomly distributed into six groups according to surface treatment, as follows: Group NC (negative control): no surface treatment; Group VP (positive control): Vitremer Primer; Group PA: 32% phosphoric acid; Group PAA: 20% polyacrylic acid; Group PLP: self-etch adhesive (Prompt L Pop); Group PB: etch&rinse adhesive (Prime&Bond NT). All cavities were restored with Vitremer. Teeth were thermocycled, stained with 0.5% basic fuchsin dye and sectioned. Microleakage values were quantitatively assessed by linear measurement of dye penetration using image-analyzing software. Differences between occlusal and gingival microleakage values within groups were evaluated using paired *t*-tests, and differences among groups were analysed using one-way analysis of variance (ANOVA) and Tukey tests. **Results:** There were statistically significant differences between the occlusal and gingival microleakage values for all groups ( $p < 0.05$ ). Microleakage differed significantly among surface pretreatment groups ( $p < 0.001$ ). The negative control and PLP groups showed similar microleakage values, but were significantly higher than other groups for both margins. Although there were no statistically significant differences between positive control and PA, PAA, PB groups, microleakage values of positive control group were smaller than all other experimental groups except for PB group. **Conclusions:** Vitremer restorations require surface pretreatment to prevent excessive microleakage. Pretreatment with etch&rinse adhesives and Vitremer Primer may reduce microleakage of Vitremer restorations.

**Keywords:** Adhesives, Conditioners, Marginal leakage, Resin-modified glass ionomer cement.  
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## INTRODUCTION

Glass ionomer cements (GIC) have undergone many modifications and improvements since their initial development by Wilson-Kent.<sup>1</sup> Resin-modified glass ionomer cements (RMGICs) are a class of hybrid materials created by modifying GICs with the addition of monomer components, typically 2-hydroxyethyl methacrylate (HEMA) and an associated initiator system.<sup>2</sup> RMGICs are

said to overcome the problems of low mechanical strength and moisture sensitivity associated with GICs while retaining their advantages, including adhesion to tooth structure and base metals, biocompatibility, anticariogenicity associated with fluoride release and thermal compatibility with tooth enamel resulting from similar coefficients of thermal expansion.<sup>3-5</sup>

Two types of polymerization are evident in RMGICs: a faster setting promoted by light activation, and a slower acid-base chemical reaction initiated by mixing the powder and liquid components and continuing up to 24 h after mixing.<sup>6</sup>

The exact mechanism by which RMGIC bonds to dentine is not well known. A chemical reaction allows bonding to dental hard tissue to be established through a reaction between the carboxylic components of the cement and the calcium present in the enamel and dentine substrates.<sup>7</sup> In addition to ionic bonding, RMGICs adhere to dentine by micro-mechanical retention.<sup>8,9</sup> Self-adhesiveness of RMGICs may also be attributed to ionic bonding to the hydroxyapatite around collagen.<sup>10</sup>

RMGICs are known to adhere successfully to enamel; however, contraction forces applied to RMGICs during light irradiation may lead to bond failure between RMGIC and

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dentine.<sup>11</sup> The prevalence of cohesive failures of RMGIC restorations to dentine has suggested that the smear layer may interfere with the adhesion mechanism; therefore, surface pretreatment to remove the smear layer has been suggested as a means of improving bonding.<sup>12-15</sup> Studies have examined the affects of numerous commercially available pretreatment agents, such as polyacrylic acid,<sup>15-19</sup> phosphoric acid,<sup>15,17-19</sup> hydrogen peroxide,<sup>15</sup> maleic acid,<sup>15</sup> citric acid<sup>17</sup> and EDTA.<sup>16,20</sup>

Given the presence of resin monomers within the RMGIC matrix, recent studies have examined whether or not pretreatment with resin adhesive agents could increase the bonding ability of RMGIC to dental tissue.<sup>11,16,18,19</sup> It is also possible that surface conditioning of the bonding substrate with self-etch adhesives, most of which are composed of aqueous mixtures of acidic monomers and which were developed to simplify bonding procedures,<sup>21</sup> could help improve bond strength between RMGIC and dentine. This study investigated two hypotheses: (i) pretreatment with a self-etch adhesive reduces the microleakage of Vitremer restorations; and (ii) different surface pretreatments perform differently in terms of preventing microleakage.

**MATERIALS AND METHOD**

Thirty caries-free extracted human third molars were selected and stored in thymol 0.1% saline solution for 1 week following extraction. Residual tissue and calculus were removed with a scalpel and/or scaling instruments, and

teeth were examined under a stereomicroscope for enamel and dentine defects.

Class V cavities were prepared on the buccal and lingual surfaces of each tooth, with the occlusal margins in the enamel and the cervical margins located 1.5 mm apical to the cemento-enamel junction. A cylindrical bur (KG Sorensen Ind Com Ltda, Barueri, SP, Brazil) in a high-speed handpiece and air/water cooling was used, with the bur changed after every five preparations. Standardized cavity dimensions were produced using a template 4.0 mm in width and 3.0 mm in height. Cavity depths were approximately 2 mm, as measured using a periodontal probe.<sup>22</sup> Preparations did not include intentional bevels or undercuts for retention.

Following cavity preparation, specimens were randomly assigned to one of six groups (*n*=10) for different surface treatments, as follows:

- Group NC*: No conditioning (negative control)
- Group VP*: Vitremer primer (positive control)
- Group PA*: 32% phosphoric acid
- Group PAA*: 20% polyacrylic acid
- Group PLP*: Self-etch adhesive (Prompt-L-Pop)
- Group PB*: Etch&rins adhesive (Prime&Bond NT).

Details of the materials and application procedures used in surface pretreatment are given in Table 1. Following surface treatment, all cavities were restored using Vitremer. Vitremer powder and liquid were dispensed at 2.5:1 by weight,

**Table 1.** Materials used in the study.

Material (manufacturer)	Composition	Application Procedures
Vitremer (3M Dental Products Co, St.Paul,MN,USA)	<i>Powder</i> : fluoro-aluminosilicate glass, potassium persulphate, ascorbic acid <i>Liquid</i> : 50% polycarboxylic acid copolymer, 20% HEMA, water, 13% carboxylic acid copolymer	1. Dispense powder and liquid at 2.5:1 by weight. 2. Mix for 45 s. 3. Inject into preparations. 4. Light polymerize 20 s.*
Vitremer primer (3M Dental Products Co, St.Paul,MN,USA)	46% HEMA, 39% ethyl alcohol, 15% vitrebond copolymer	1. Apply with light scrubbing motion for 30 s. 2. Gentle air stream. 3. Light polymerize 20 s.*
UNI-ETCH Phosphoric acid (Bisco, Inc. Schaumburg IL, USA)	32% phosphoric acid, benzalkonium chloride	1. Apply for 15 s. 2. Water rinse for 15 s. 3. Dry gently.
GC Cavity conditioner (GC Corp., Tokyo, Japan)	20% polyacrylic acid, AlCl <sub>3</sub>	1. Apply 10 s. 2. Water rinse for 15 s. 3. Dry gently.
Adper Prompt L Pop (3M ESPE, ST Paul, USA)	Red cushion: Methacrylic phosphates, BisGMA, photo-initiator Yellow cushion: Water, HEMA, polyalkenoic acid polymer	1. Dry surface. 2. Apply adhesive. 3. Gentle air stream. 4. Light polymerize 10 s.*
Prime & Bond NT (Dentsply Ind. Com. Ltda, Petropolis, RJ, Brazil)	BisGMA, PENTA-P, photoinitiator, ethylamine hydrofluoride, acetone	1. Etch with phosphoric acid 15 s. 2. Rinse and blot dry. 3. Apply adhesive. 4. Gentle air stream. 5. Light polymerize 10 s.*

AlCl<sub>3</sub>, aluminum chloride; BisGMA, bis-phenol A diglycidylmethacrylate; HEMA, hydroxyl ethyl methacrylate  
\* Elipar Freelight II, 3M ESPE, USA; light intensity: 1000mW/cm<sup>2</sup>.

loaded into a Centrix syringe, and injected into the preparation. Then the excess material removed by covering the surface with a polyester strip and glass slab and applying slight pressure. Restorations were polymerized, Vitremer finishing gloss was applied to the unfinished restorations and light cured, specimens were then finished with aluminum oxide discs (Sof-Lex, 3 M ESPE, USA), and a second coat of finishing gloss was applied and cured.

Restored specimens were stored for 24 h in deionized water at  $37\pm 1^\circ\text{C}$  and then thermocycled for 1,500 cycles between  $5^\circ\text{C}$  and  $55^\circ\text{C}$  with a dwell time of 10 s and a transfer time of 30 s between baths.<sup>23</sup> Following thermocycling, root apices were sealed with sticky wax, and the entire tooth surface was coated with nail varnish up to 1 mm around the circumference of the restoration margins.

Microleakage was evaluated according to dye penetration. Teeth were soaked in 0.5% basic fuchsin dye for 24 h, removed and rinsed under running water for 5 minutes. The roots were cut from the crown, which was sectioned mesio-distally and buccal-lingually using a slow-speed saw.

Images of the material-tooth interface at the cavity margin were captured at  $\times 30$  magnification using a stereomicroscope (Nikon SMZ-1500, Osaka, Japan) and stored in a digital format. Microleakage, seen as a pink line at the RMGIC-enamel interface, was measured in millimeters using Photoshop software (Adobe Photoshop CS4 expanded, Adobe Systems, USA). Measurements were made by a single operator (NT) who was blinded to the treatment procedure. Both occlusal and gingival measurements were

taken for each section, for a total of 240 measurements (4 measurements  $\times$  30 restorations). For each tooth, the highest measurements were recorded for both occlusal and gingival margins.

Means and standard deviations were calculated from the microleakage data. The difference between the mean occlusal and gingival microleakage measurement in each group was evaluated using paired t-tests ( $p < 0.05$ ). Differences between surface treatment groups for occlusal and gingival margins were separately analyzed by using one-way analysis of variance (ANOVA) and Tukey multiple range tests ( $p < 0.001$ ).

## RESULTS

Mean occlusal and gingival microleakage measurements for each of the six different surface treatments are shown in Table 2.

Mean occlusal and gingival microleakage values did not exceed half the cavity depth in any of the groups (Figures 1, 2). Occlusal microleakage measurements were smaller than gingival microleakage measurements in all groups, and these differences were statistically significant in all groups ( $p < 0.05$ ) (Table 2).

ANOVA revealed statistically significant differences among the surface treatment groups ( $p < 0.001$ ) (Table 2). The negative control and PLP groups showed similar microleakage values, but were significantly higher than other groups for both margins. Although there were no statistically significant differences between positive control



**Figure 1.** Representative stereomicroscopic image of a tooth that without dye penetration in PB group. Original magnification  $\times 30$ .



**Figure 2.** Representative stereomicroscopic image of dye penetration in PLP group. The arrow shows dye penetration. Original magnification  $\times 30$ .

**Table 2.** Mean values and statistically comparisons of microleakage values.

Groups	Occlusal mean (s.d)	Gingival mean (s.d)
Group NC*	0.48(0.17) <sup>c,d</sup>	0.81(0.28) <sup>C,D</sup>
Group VP*	0.05(0.08) <sup>a</sup>	0.51(0.41) <sup>A,B</sup>
Group PA*	0.31(0.29) <sup>a,b,c</sup>	0.77(0.19) <sup>B,C,D</sup>
Group PAA*	0.30(0.28) <sup>a,b,c</sup>	0.53(0.20) <sup>A,B,C,D</sup>
Group PLP*	0.65(0.31) <sup>d</sup>	0.91(0.38) <sup>D</sup>
Group PB*	0.04(0.11) <sup>a</sup>	0.22(0.24) <sup>A</sup>

\*\*\*indicate statistically significant differences between occlusal and gingival measurements within groups ( $p < 0.05$ ).  
\*Differences in superscript letters indicate statistically significant differences in one column ( $p < 0.001$ ).

and PA, PAA, PB groups, microleakage values of positive control group were smaller than all other experimental groups except for PB group.

## DISCUSSION

Resin-modified glass ionomer materials have been widely used and are recommended for restoring cervical lesions that include enamel and dentin margins. RMGICs are specifically recommended for the caries-prone patient because of the possible cariostatic effect of fluoride. Despite recent improvements in RMGIC adhesion to dentin, RMGIC bond strengths are still not comparable to those of dentine bonding adhesives.<sup>11</sup> Recent studies have shown that the use of dentin bonding adhesives in conjunction with RMGICs can improve RMGIC bond strength,<sup>11,16</sup> however, there is little known about the effects of self-etch adhesives on RMGIC bond strengths.<sup>11</sup> The present study examined the effects of different surface pretreatments, including a self-etch adhesive, on the marginal microleakage of RMGIC restorations.

One essential factor in the longevity of a restoration is the marginal sealing ability of the restorative material. Dimensional changes and lack of adaptation of the restoration to the cavity walls can lead to marginal leakage, i.e., fluid and molecular movement and ingress of bacteria or bacterial nutrients. Long-term sealing capacity is required to prevent microleakage and its sequelae – including staining, postoperative sensitivity, pulpal irritation and recurrent caries.<sup>23</sup> Many techniques involving dyes have been used to assess microleakage both *in vivo* and *in vitro*.<sup>24</sup> Dye penetration is a technique to determine the loss of adhesion.<sup>25</sup> Other methods, such as the silver nitrate staining technique, or penetration of radioactive substances, are not commonly used because they are time-consuming and the handling of these

materials is difficult.<sup>26</sup> Although marginal leakage studies usually rely on qualitative criteria,<sup>15,25,27-29</sup> some studies perform linear measurements of dye penetration at the tooth-restoration interface to provide quantitative information.<sup>30</sup> The present study used linear measurements in order to provide an objective evaluation.

Bonding to the tooth substrate in the area of the cemento-enamel junction continues to remain problematic for clinicians. In the present study, none of the treatment groups were able to completely resist microleakage. Microleakage at the gingival margin was statistically higher than at the occlusal margin for all test groups ( $p < 0.05$ ). This finding is in line with previous *in vitro* studies.<sup>27,29</sup>

The application of surface treatment prior to RMGIC restorations is controversial. Some authors consider RMGICs to be adhesives in their own right by virtue of their HEMA content, making surface conditioning unnecessary.<sup>31</sup> However, it is important to note that HEMA, while strongly hydrophilic, constitutes only 5% of the cement content.<sup>32</sup> In the present study, microleakage in the non-conditioned negative control group was higher than in all other groups except the PLP group. This result is in agreement with other studies that have recommended surface pretreatment before RMGIC restorations.<sup>12-15</sup>

RMGIC adhesion has been reported to function through both an ionic exchange between the cement and the hydroxyapatite of the tooth substrate and a mechanical interlocking of the polymer and dentin.<sup>7-9</sup> Acid conditioning of the tooth surface creates irregularities or pores in the substrate surface, thus facilitating material tag formation. However, in view of the role ionic exchange plays in bonding, care must be taken to ensure that the full mineral content of dentine and enamel is maintained.<sup>27</sup> The use of strong acids removes inorganic components, exposing a microporous scaffold of collagen fibrils and depleting the calcium and phosphate ions required for the ion-exchange mechanism to function; the resulting bond is primarily micro-mechanical and may be easily broken.<sup>14</sup> For this reason, some authors recommended not using phosphoric acid, which is stronger than polyacrylic acid, to treat dentin before RMGIC restorations.<sup>14,19</sup> However, Yilmaz *et al*<sup>15</sup> found no significant difference between phosphoric acid and polyacrylic acid pretreatment. Our results are in line with these latter findings. Furthermore, the PA and PAA groups in our study exhibited less microleakage than the NC group, although the differences were not statistically significant for both margins.

The manufacturers of Vitremer recommend the application of Vitremer primer to cavities prior to placement of Vitremer restorative materials. A previous study on permanent dentin found that in line with the manufacturer's claims, Vitremer primer was able to modify the smear layer to permit a closer interaction of the RMGIC and the dentin surface.<sup>33</sup> In the present study, the positive control group (VP group) had lower microleakage values than the PA and PAA groups; however, the differences between them were not statistically



significant ( $p > 0.001$ ). Similarly, Marquezan *et al*<sup>33</sup> suggested that applying a HEMA-rich primer instead of conditioning dentin with polyacrylic acid may compromise the formation of the absorption layer and the continuation of the acid-base reaction taking place within the RMGIC at the dentin interface.

In the present study, the application of an etch&rinse adhesive (group PB) to cavity surfaces prior to the placement of Vitremer reduced the microleakage to levels lower than those of the other materials tested. Pereira *et al*<sup>11</sup> found that the use of adhesive systems associated with RMGICs exhibited the higher bond strength to dentin. It is likely that a properly applied adhesive system is capable of penetrating the demineralized dentin matrix to form a hybrid layer, optimizing the mechanical interlocking between the restorative material and substrate and thus reducing microleakage values.

Surface pretreatment with a self-etch adhesive could represent an interesting alternative to other pretreatment agents used in pediatric dentistry because it would reduce the number of steps and eliminate the need for rinsing. However, the present study, found self-etch adhesives to be significantly less effective in preventing microleakage than the other materials tested. This result is in line with many studies that report self-etch adhesives to have less effective bonding properties than etch&rinse adhesives.<sup>30,34,35</sup> Several factors associated with self-etch adhesives can result in the inadequate formation of a hybrid layer and, consequently, microleakage at the tooth-restoration margins that would decrease long-term bonding effectiveness.<sup>36</sup> Included among these factors are the incomplete alteration and/or removal of smear layer components due to composition (pH, osmolarity) and strength of the acidic primer and inadequate resin film thickness, requiring multiple layering techniques and changes in the monomer-water ratio, resulting in phase separations.<sup>37-41</sup>

## CONCLUSIONS

Although the results of this study may not be directly extrapolated to clinical conditions, they do provide the following information:

- (1) None of the materials tested completely eliminated microleakage, levels of which were higher at the gingival margin than the occlusal margin.
- (2) Surface pretreatment may reduce the microleakage of Vitremer restorations.
- (3) Surface pretreatment with an etch & rinse adhesive and Vitremer Primer resulted in less microleakage than other pretreatment agents. An etch&rinse adhesive could be used for surface pretreatment.
- (4) Surface pretreatment with a self-etch adhesive resulted in more microleakage than other pretreatment agents.

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