

# EFFECT OF ACID ETCHING OF GLASS IONOMER CEMENT SURFACE ON THE MICROLEAKAGE OF SANDWICH RESTORATIONS

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## ABSTRACT

The purposes of this study were to evaluate the sealing ability of different glass ionomer cements (GICs) used for sandwich restorations and to assess the effect of acid etching of GIC on microleakage at GIC-resin composite interface. Forty cavities were prepared on the proximal surfaces of 20 permanent human premolars (2 cavities *per* tooth), assigned to 4 groups (n=10) and restored as follows: *Group CIE* – conventional GIC (CI) was applied onto the axial and cervical cavity walls, allowed setting for 5 min and acid etched (E) along the cavity margins with 35% phosphoric acid for 15 s, washed for 30 s and water was blotted; the adhesive system was applied and light cured for 10 s, completing the restoration with composite resin light cured for 40 s; *Group CIN* – same as Group CIE, except for acid etching of the CI surface; *Group RME* – same as CIE, but using a resin modified GIC (RMGIC); *Group RMN* – same as Group RME, except for acid etching of the RMGIC surface. Specimens were soaked in 1% methylene blue dye solution at 24°C for 24 h, rinsed under running water for 1 h, bisected longitudinally and dye penetration was measured following the ISO/TS 11405-2003 standard. Results were statistically analyzed by Kruskal-Wallis and chi-square tests ( $\alpha=0.05$ ). Dye penetration scores were as follow: CIE – 2.5; CIN – 2.5; RME – 0.9; and RMN – 0.6. The results suggest that phosphoric acid etching of GIC prior to the placement of composite resin does not improve the sealing ability of sandwich restorations. The RMGIC was more effective in preventing dye penetration at the GIC-resin composite-dentin interfaces than CI.

**Uniterms:** Glass ionomer cements; Composite resins; Dental leakage.

## INTRODUCTION

The glass-ionomer cement (GIC) was introduced by Smith<sup>26</sup> in the late 1960's, resulting from the replacement of phosphoric acid by polyacrylic acid in zinc phosphate cements. The original idea was to unite properties of the silica glass powder, such as high strength, hardness and the capacity to release fluoride, with the biocompatibility and adhesion ability of the polyacrylic acid liquid<sup>26</sup>. The anticariogenic property resulting from fluoride release turned out to be the most attractive aspect of this dental material. In addition, GIC adhesion mechanism to tooth structure, thermal compatibility with tooth enamel, biocompatibility and low cytotoxicity render to GIC an interesting clinical option for restorative treatments<sup>30</sup>.

Since the first commercial appearance of GIC (ASPA, Dentsply De Trey Ltd, Weybridge, UK) in 1976, this material has undergone important modifications in composition to

improve its tensile and fracture strengths, working time, chemical solubility, and polishing appearance<sup>4,27,28</sup>. Therefore, new types of GICs have been developed, such as the light-cured resin modified glass ionomer cement (RMGIC)<sup>1,27</sup>. This material is obtained by adding a resin, usually the water-soluble polymerizable 2-hydroxyethyl methacrylate (HEMA), to the liquid and its bonding process to tooth structure takes place by micromechanical retention, like in resin composites<sup>27</sup>. The setting reaction of RMGIC follows two distinct mechanisms: resin polymerization and acid-base reaction. The former can be initiated by either light exposure or redox reaction (self-curing) and is responsible for the immediate setting. The acid-base reaction takes place slowly and continues after clinical setting. The advantages of this restorative material include longer working time and operator control over the setting reaction by light activation of the resin component and, consequently, an earlier development of higher bond

strength, reduced brittleness, increase of tensile and flexural strengths, resistance to desiccation and acid attack, lower moisture sensitivity and solubility<sup>2,9,11,16,27,28</sup>. Yet, the mechanical properties (Table 1) and esthetic appearance still limit its clinical use. Thus, the so-called sandwich restoration or “composite-laminated GIC” technique has been used by clinicians to preserve the fluoride release mechanism and the chemical bond to tooth structure provided by the GIC and RMGIC, and to improve the esthetic and mechanical properties using a resin composite laminate.

Leakage has long been recognized as a problem in restorative dentistry<sup>17</sup>. Microleakage studies are used to estimate the resistance of tooth-restoration interface to the passage of bacteria, fluids, chemical substances, molecules and ions<sup>3</sup>. The absence of a seal at restoration margins promotes tooth discoloration, adverse pulp response, postoperative sensitivity, and recurrent caries<sup>8</sup>. Thus, some *in vitro* studies developed to predict the clinical marginal sealing ability of several restorative techniques found lesser microleakage when GIC was used as a filling material beneath the composite resin<sup>10,14,23</sup>. Despite these enthusiastic results, the clinical performance of sandwich restorations does not seem to be very effective and some failures have been reported, mainly concerning the GIC portion<sup>29</sup>. Some authors have stated that this laminate restoration is an interesting alternative to amalgam, especially in high-caries risk patients<sup>7</sup>. Other authors have suggested that the failures could be related to an inadequate selection of the GIC-composite system, which should provide high tensile strength, a bonding agent with high wettability and a composite resin with small setting shrinkage<sup>17,18</sup>.

The bonding mechanisms of restorative materials to tooth tissues are often explained in the literature<sup>7</sup>. Yet, few studies have addressed aspects regarding the restorative materials used in sandwich techniques<sup>18,20</sup>. Although enamel and dentin pre-treatment before the application of bonding systems and restorative materials is well established in the literature<sup>6</sup>, the need for GIC surface treatment before the placement of composite resin in sandwich restorations still

remains debatable. Although acid etching of GIC’s surface increases the tensile bond strength to resin composite<sup>12</sup>, the influence of this conditioning step on microleakage has not been reported. The purposes of this study were to evaluate the sealing ability of different GIC materials used for the sandwich restoration technique, and to assess the effect of acid etching of GIC surface on microleakage between the ionomer and composite materials. The tested hypothesis was that acid etching of GIC before placement of the composite resin does not increase the sealing between both restorative materials.

## MATERIAL AND METHOD

Twenty extracted non-carious human permanent premolars with fully developed roots were selected for this study, which was approved by the local Ethics in Research Committee. The teeth were cleaned of calculus, soft tissue and other debris, and stored in 2% chloramine solution at 5°C. Two window-like cavities were prepared on both proximal surfaces of each tooth using diamond burs (no. 3145 and FG58L; KG Sorensen, São Paulo, SP, Brazil) at high speed, as shown in Figure 1.

The tested materials included a conventional GIC (CI) (Ketac Fill Plus, 3M-ESPE, St. Paul, MN, USA; lot 178566), a light-cured RMGIC (Vitremar; 3M-ESPE, St. Paul, MN, USA; lot 4JP 2007-12), an adhesive system (AS) (Adper Single Bond; 3M-ESPE, St. Paul, MN, USA; lot 4BU 2007-11) and a composite resin (CR) (Filtek Z250; 3M-ESPE, St. Paul, MN, USA; lot 2LR 2006-05). All materials were handled at room temperature (23°C) following the manufacturers’ instructions. The 40 cavities were randomly assigned to 4 groups (n=10) and restored according to the sandwich technique<sup>18</sup>. The experimental groups were as follows:

*Group CIE* - CI was applied onto the axial and cervical cavity walls, allowed setting for 5 min and acid etched (E) along the cavity margins with 35% phosphoric acid for 15 s, washed with air-water spray for 30 s, and blotted with an

**TABLE 1-** Literature-based data on representative properties of GIC and RMGIC

Property	Conventional GIC	RMGIC
Working time <sup>28</sup>	2 min	3 min 45 s
Setting time <sup>28</sup>	4 min	20 s
Young’s Modulus <sup>2</sup>	20.5 GPa	55.9 GPa
Compressive strength <sup>30</sup>	265.3-306.2 MPa	196.5-301.3 MPa
Diametral tensile strength <sup>28</sup>	16 MPa	37 MPa
Flexural strength <sup>30</sup>	71-82 MPa	21.2-31.4 MPa
Knoop hardness <sup>30</sup>	66.4-84.5 KHN	29.7-176.8 KHN
Tensile bond strength to human enamel <sup>21</sup>	4.9 MPa	11.36 MPa
Tensile bond strength to human dentin <sup>21</sup>	2.52 MPa	5.55 MPa
Shear bond strength to bovine enamel <sup>28</sup>	4.6 MPa	11.3 MPa
Shear bond strength to bovine dentin <sup>28</sup>	4.3 MPa	8.2 MPa

absorbent sponge. The AS was applied and light cured (XL 3000, 3M-ESPE, St. Paul, MN, USA) for 10 s, completing the restoration with a layer of CR that was light cured for 40 s; *Group CIN* - same procedures described for *Group CIE*, except for acid etching of the CI surface; *Group RME* - same procedures described for *Group CIE*, but using the RMGIC. *Group RMN* - same procedures described for *Group RME*, except for acid etching of the RMGIC surface.

All restorations were polished with Sof-Lex discs (Polishing Discs, 3M-ESPE, St. Paul, MN, USA). Teeth were coated with 3 layers of nail varnish (Risqué, Niasi S/A, Taboão da Serra, SP, Brazil; lot 331526), except for a window area that included the restoration and 1 mm around it, and soaked in 1% methylene blue dye solution at 24°C for 24 h, then rinsed under running water for 1 h.

The teeth were adapted to an automatic cutting machine (Struers Minitom, Struers A/S, Denmark) with a water-cooled low-speed (250 rpm) diamond wheel saw, and were bisected longitudinally to separate the mesial from the distal surfaces. Then, at least 2 approximately 1-mm-thick slices were obtained within the restored area. Whenever more than 2 slices were obtained *per* restoration, only 2 of them were used in the study, being one slice above the cemento-enamel junction (CEJ) and another below the CEJ. Therefore, 4 surfaces *per* restoration were examined in an optical microscope (Meiji EMZ-TR, Meiji Techno Co LTD., Tokyo, Japan; magnification 10x-65x) for marginal sealing and leakage (40 surfaces *per* group). Representative images were recorded from all specimens and maximum degree of dye penetration was registered according to the following scores (ISO/TS 11405-2003)<sup>13</sup>: 0 = no dye penetration; 1 = dye

penetration into the enamel portion of the cavity wall; 2 = dye penetration into the dentin portion of the cavity wall but not including the pulpal floor of the cavity; 3 = dye penetration including the pulpal floor of the cavity.

Results were statistically analyzed by Kruskal-Wallis at 5% significance level ( $\alpha=0.05$ ) and chi-square test for the two types of GIC and for the acid-etching effect on both GICs ( $\alpha=0.05$ ).

## RESULTS

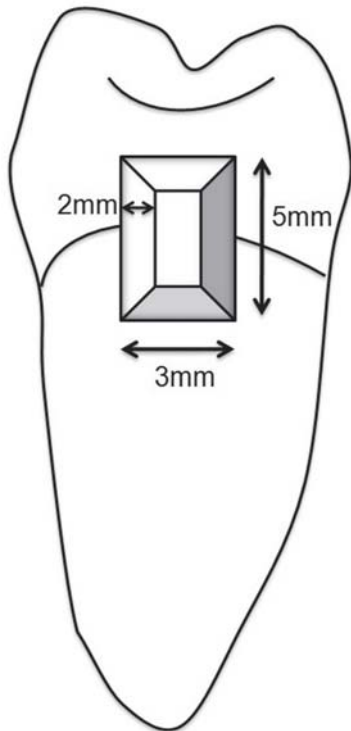
The sample size, dye penetration scores, dye penetration score means and statistical grouping are summarized in Table 2.

No significant differences were found between Groups CIE and CIN ( $p>0.05$ ), and between Groups RME and RMN ( $p>0.05$ ) regarding dye penetration means. When both surface treatments (E and N) were considered, irrespectively of the materials (CI and RMGIC), no statistically significant differences were found ( $p>0.05$ ). However, significant differences were observed between the GICs (CI and RMGIC), regardless the surface treatments, CI showing a significantly greater dye penetration score mean than that of RMGIC ( $p<0.05$ ).

Dye penetration at the GIC-CR interface occurred in 4 teeth, being 3 in *Group CIE* and 1 in *Group CIN*. This finding was statistically insignificant ( $p>0.05$ ). Whenever microleakage occurred, the maximum degree of dye penetration was always found within the slice obtained below the CEJ.

## DISCUSSION

The polymerization shrinkage developed during the conversion of the monomer molecules into a polymer network is an undesirable resin composite phenomenon and can be considered one of the factors responsible by the lack of adaptation of the restoration to the cavity walls, increasing the susceptibility to caries especially in deep regions of the proximal box of Class II cavities<sup>7</sup>. The use of GIC as an underfilling material in conventional sandwich restoration reduces considerably the bulk resin composite used, thus,



**FIGURE 1-** Schematic image of the window-like cavity prepared in each proximal tooth surface and its dimensions

**TABLE 2-** Sample size, dye penetration scores, dye penetration score means and statistical grouping

Group	n	0	1	2	3	Mean*
CIE	10	1	0	2	7	2.5 a
CIN	10	0	0	5	5	2.5 a
RME	10	6	1	1	2	0.9 b
RMN	10	7	0	3	0	0.6 b

\*Group means followed by the same letter are not statistically different at  $p=0.05$

the amount of polymerization shrinkage of the composite resin is decreased and the marginal adaptation may be improved. A further advantage of the sandwich technique is the fluoride-release property of GICs, which is considered to have some inhibitory effect on caries formation and progression around the restoration<sup>27</sup>.

The GIC is still considered the only material with self-adherence to dental tissue<sup>6</sup> and it has been previously shown that GIC and composite resin can adhere effectively to each other<sup>9,12,15,17</sup>, regardless of the limitations concerning this system<sup>18</sup>. The bond strength between these materials is influenced by, at least, four factors: 1) the tensile strength of GIC, which is mostly dependent on the powder/liquid ratio; 2) the viscosity of the bonding agent and its ability to wet the GIC's surface; 3) the volumetric change in the composite resin during polymerization and; 4) the difficulties in packing and adaptation of the composite resin to the GIC without incorporation of voids<sup>18</sup>. It has been suggested that the acid etching of GIC would allow a cleaned mildly roughened surface with high surface energy<sup>17</sup>. It has been assumed that this procedure would fulfill the requirements to a closer contact and a greater interlocked interface between GIC and composite resin<sup>12,15</sup>. In spite of these considerations, the results of the present study indicate that acid etching of CI and RMGIC surfaces did not improve the sealing ability of sandwich restorations.

Considering the tested GIC materials, the sandwich restorations using RMGIC showed significantly less dye penetration than those using CI, which is in agreement with the results of a previous report<sup>11</sup>. In addition, no significant differences were found between the surface treatments (E and N) on same material. These findings suggest that the accomplishment of acid etching for GIC is not significantly relevant compared to the type of GIC selected, which should be a material with improved mechanical properties and chemical composition (Table 1).

Previous studies have shown that the inability of conventional GICs to produce an effective seal depends on two factors: 1) the material's sensitivity to moisture during placement and early set; and 2) the dehydration after setting, resulting in crazing and cracking<sup>5,16</sup>. Yet, it is assumed that the better sealing produced by RMGIC is a result of the formation of resin tags into the dentinal tubules allied to the ion exchange process present in the interface between dentin and RMGIC, as previously reported<sup>19,20,22</sup>. Although some studies do not testify the presence of these resin tags or even the formation of a hybrid layer<sup>24,25</sup>, this assumption stands to be the reason for the superior performance of the RMGIC. In addition, the presence of HEMA in the RMGIC is responsible for the increased bond strengths to resin composite<sup>9</sup> and should contribute to prevent dye penetration through the interface of these materials, as demonstrated by the results of the present study.

## CONCLUSION

Within the limitations of this study, the results suggest

that phosphoric acid etching of GIC prior to the placement of composite resin does not improve the sealing ability of sandwich restorations. The resistance to dye penetration at the interfaces (GIC-composite resin-dentin) seems to be primarily controlled by the type of GIC, RMGIC producing significantly less dye penetration than the conventional GIC.

## REFERENCES

- 1- Antonucci JM, McKinney JE, Stansbury JW. Resin-modified glass ionomer cement. USA Patent Application 160856. 1988.
- 2- Attin T, Vataschki M, Hellwig E. Properties of resin-modified glass-ionomer restorative materials and two polyacid-modified resin composite materials. *Quintessence Int.* 1996;27:203-9.
- 3- Bauer JF, Henson JL. Microleakage around dental restorations: a summarizing review. *J Am Dent Assoc.* 1972;87:1349-57.
- 4- Bowen RL, Marjenhoff WA. Dental composites/glass ionomers: the materials. *Adv Dent Res.* 1992;6:44-9.
- 5- Charbeneau GT. Principles and practice of operative dentistry. Philadelphia: Lea & Febiger; 1988.
- 6- De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res.* 2005;84:118-32.
- 7- Dijken JWV, Kieri C, Carlen M. Longevity of extensive class II open-sandwich restorations with a resin-modified glass-ionomer cement. *J Dent Res.* 1999;78:1319-25.
- 8- Eriksen HM, Pears G. In vitro caries related to marginal leakage around composite resin restorations. *J Oral Rehabil.* 1978;5:15-20.
- 9- Fortin D, Vargas MA, Swift EJ Jr. Bonding of resin composites to resin-modified glass ionomers. *Am J Dent.* 1995;8:201-4.
- 10- Gupta S, Khinda VI, Grewal N. A comparative study of microleakage below cemento-enamel junction using light cure and chemically cured glass ionomer cement liners. *J Indian Soc Pedod Prev Dent.* 2002;20:158-64.
- 11- Hallett KB, Garcia-Godoy F. Microleakage of resin-modified glass ionomer cement restorations: an in vitro study. *Dent Mater.* 1993;9:306-11.
- 12- Hinoura K, Moore BK, Phillips RW. Tensile bond strength between glass ionomer cement and composite resin. *J Am Dent Assoc.* 1987;114:167-72.
- 13- International Standards Organization. ISO Standard 11405:2003: dental materials-testing of adhesion to tooth structure. Geneva: The Organization; 2003.
- 14- Loguercio AD, Alessandra R, Mazzocco KC, Dias AL, Busato AL, Singer Jda M, et al. Microleakage in class II composite resin restorations: total bonding and open sandwich technique. *J Adhes Dent.* 2002;4:137-44.
- 15- McLean JW, Powis DR, Prosser HJ. The use of glass-ionomer cements in bonding composite resins to dentine. *Br Dent J.* 1985;158:410-4.
- 16- Mount GJ. Restoration with glass-ionomer cement: requirements for clinical success. *Oper Dent.* 1981;6:59-65.

- 17- Mount GJ. Clinical requirements for a successful 'sandwich' - dentine to glass ionomer cement to composite resin. *Aust Dent J.* 1989;34:259-65.
- 18- Mount GJ. The tensile strength of the union between various glass ionomer cements and various composite resins. *Aust Dent J.* 1989;34:136-46.
- 19- Mount GJ. Adhesion of glass-ionomer cement in the clinical environment. *Oper Dent.* 1991;16:141-8.
- 20- Nezu T, Winnik FM. Interaction of water-soluble collagen with poly(acrylic acid). *Biomaterials.* 2000;21:415-9.
- 21- Pereira LC, Nunes MC, Dibb RG, Powers JM, Roulet JF, Navarro MF. Mechanical properties and bond strength of glass-ionomer cements. *J Adhes Dent.* 2002;4:73-80.
- 22- Pereira PNR, Yamada T, Tei R, Tagami J. Bond strength and interface micromorphology of an improved resin-modified glass ionomer cement. *Am J Dent.* 1997;10:128-32.
- 23- Platt JA, Rhodes B. Microleakage of high-strength glass ionomer: resin composite restorations in minimally invasive treatment. *J Indiana Dent Assoc.* 2001;80:20-2.
- 24- Ramos JC, Perdigo J. Bond strength and SEM morphology of dentin-amalgam adhesives. *Am J Dent.* 1997;10:152-8.
- 25- Sidhu SK, Schmalz G. The biocompatibility of glass-ionomer cement materials: a status report for the American Journal of Dentistry. *Am J Dent.* 2001;14:387-96.
- 26- Smith DC. A new dental cement. *Br Dent J.* 1968;124:381-4.
- 27- Tyas MJ. Milestones in adhesion: glass-ionomer cements. *J Adhes Dent.* 2003;5:259-66.
- 28- van Noort R. Introduction to dental materials. Saint Louis: Mosby; 1994.
- 29- Welbury RR, Murray JJ. A clinical trial of the glass-ionomer cement-composite resin "sandwich" technique in class II cavities in permanent premolar and molar teeth. *Quintessence Int.* 1990;21:507-12.
- 30- Xie D, Brantley WA, Culbertson BM, Wang G. Mechanical properties and microstructures of glass-ionomer cements. *Dent Mater.* 2000;16:129-38.