

# Microleakage comparison of three types of adhesive systems versus GIC-based adhesive in class V composite restorations

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Received 25 November 2015 and Accepted 5 February 2016

## Abstract

**Background and aims:** New dentin bonding agents and techniques have been developed to reduce microleakage and create higher bond strength. This *in-vitro* study compared the microleakage of three resin-based adhesives versus a GIC-based adhesive on class V composite restorations. **Materials and Methods:** Class V cavities were prepared on the buccal surfaces of 72 sound premolars, randomly assigned to six groups (n=12) and treated as follows: without any treatment (negative control group); total-etch (OptiBond Solo Plus); two-step self-etch (OptiBond XTR); one-step self-etch (OptiBond All-in-One) and GIC-based adhesive (Fuji bond LC) with pre-cure and co-cure techniques. The treated cavities were filled with a micro-hybrid resin composite (Point 4, Kerr). Following finishing and polishing procedures, the specimens were placed in 100% humidity, stored in distilled water, thermocycled and then immersed in a methylene blue, sectioned, evaluated for microleakage and scored on a 0 to 3 ordinal scale. **Results:** None of the adhesives tested were capable of completely eliminating marginal microleakage. There were statistically significant differences among the test groups at occlusal margins; but at cervical margins were not. The Fuji Bond LC with co-cure and control groups had significantly greater microleakage scores at the occlusal margins. At the cervical margins, the bonded restorations with OptiBond XTR and OptiBond All-in-One adhesives presented significantly lower microleakage scores. Also, there were no significant differences between the resin adhesive groups both at occlusal and cervical margins. The microleakage scores at the cervical margins were markedly higher than the occlusal margins in the groups bonded with OptiBond Solo Plus and Fuji Bond LC with pre-cure. The differences between Fuji Bond LC adhesive with pre-cure and co-cure techniques were significant. **Conclusion:** This study encourages

application of the Fuji bond LC adhesive with pre-cure technique instead of the resin adhesives for marginal seal in class V composite restorations.

**Key words:** composite resins, dental adhesives, dental leakage, glass ionomer cement.

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Sadghi M. Microleakage comparison of three types of adhesive systems versus GIC-based adhesive in class V composite restorations. J Dent Mater Tech 2016; 5(2): 86-93.

## Introduction

In spite of numerous advantages of resin composites, polymerization shrinkage has been identified as a major drawback of the composite material, because of marginal gap formation between cavity wall and the restoration (1-3) to achieve clinical success with composite restorations, efforts have been made to develop dentin bonding agents that can withstand the stresses induced by polymerization shrinkage and finally create higher bond strengths and reduce microleakage (1, 4, 5).

The cervical cavities usually have little or no enamel for bonding to adhesive materials. The heterogeneous nature of the dentin and the difficulties of moisture control cause complications for perfect sealing in this area.4, 6 Recently, new bonding agents have been developed, which produce a more reliable bonding to the dentin. These adhesives with monomers in primers can infiltrate into moist demineralized dentin surfaces and polymerize within the collagen network and produce a resin-reinforced or hybrid layer (3, 7).

In two-step total-etch systems, a hydrophilic primer and hydrophobic resin are combined into one application (4, 8, 9). These systems utilizing 30-40% phosphoric acid are efficient in the removal of smear

layer, causing demineralization of the inorganic enamel surface, thus creating micro-porosities for a micro-mechanical bond(3,10,11). During dentin etching, over-conditioning can occur, causing a collapse and shrinking of the collagenous fibular network due to loss of structural, inorganic support. As a result, exposed collagen fibrils and lack of support by partially infiltrated resin monomers result in a significant reduction in material-tooth structure adhesion (3, 7, 9).

Two-step self-etch adhesives are able to partially remove the smear layer and penetrate the dentinal surface, creating a less pronounced resin tag formation and hybrid layers that are thinner than those of total-etch systems (4,11,12). Very high dentin bond strengths comparable to total-etch adhesives are reported (6, 13); conversely, a common concern is inability commercial self-etching adhesives to etch enamel to the same depth as phosphoric acid (14). Single-step self-etch adhesives were also introduced to simplify bonding procedures, which compose of aqueous mixtures of phosphoric acid esters, primer and hydrophobic resin monomers into one container (9, 10, 15).

In 1995, a modern RMGIC was developed for direct bonding of resin composites to tooth enamel and dentin. After pretreatment of the cavity with a weak polyalkenoic acid, self-adhesion of the adhesive is obtained by both a micromechanical interlocking by a submicron hybrid layer (0.5–1 mm) and a chemical bond through ionic bonds between the carboxyl groups of the glass ionomer and calcium of hydroxyapatite that remains around the collagen (16-18).

However, from the best of our knowledge, there are few studies that assess the microleakage pattern around class V composite restorations bonded with different resin-based and GIC-based adhesives to date. Hence, the purpose of this *in-vitro* study was to compare the microleakage of three different kinds of generations of resin adhesives: total-etch (OptiBond Solo Plus), two-step self-etch (OptiBond XTR) and one-step self-etch (OptiBond All-in-One) adhesives versus a GIC-based adhesive (GC Fuji bond LC) with the pre-cure and co-cure techniques at the occlusal and cervical margins of class V resin composite restorations. The null hypothesis was that GIC-based adhesive doesn't perform better than resin-based adhesives in reducing microleakage of class V composite restorations.

## Materials and Methods

In this study, 72 sound human maxillary premolars extracted for orthodontic reasons were collected; then scaled and cleaned with pumice and stored in an aqueous buffered solution of formaldehyde (Yekta Chem Co., Tehran, Iran) for two hours for infection control. The teeth were randomly assigned into six

groups (6 groups × 12 teeth) according to perform the cavity treatment.

Primarily, box shaped class V cavities (3.0 mm in height, 3.0 mm in mesiodistal direction, and 1.5 mm in depth) were prepared on the buccal surface with a fissure diamond bur (Diatech Dental AG, Heerbrugg, Switzerland) mounted with a high-speed handpiece, under copious water spray. The occlusal margins were located 2.0 mm above the cemento-enamel junction (CEJ) level in enamel and the cervical margins were located 1.0 mm apically to the CEJ level in dentin/cementum. A 1.0 mm, 45° bevel was placed on the occlusal margins using a flame-shaped diamond bur (Diatech Dental AG), although the cavosurface wall at cervical margin was finished to a butt joint. Cavity preparations were rinsed with an air/water spray and gently air dried and the manufacturers' instructions were followed for the all materials precisely (Table 1).

Group I (Negative control group): No acid etch and bonding agent were applied on the cavity walls.

Group II (OptiBond Solo Plus): The cavity walls was etched with a 37.5% phosphoric acid gel (Gel Etchant, Kerr Italia S.p.A., Salerno, Italy) for 15s, then thoroughly rinsed with water for 20s and gently air dried for 5s to remove excess moisture without desiccation of dentin. The two-step total-etch adhesive (OptiBond Solo Plus, Kerr Italia S.p.A. Salerno, Italy) was applied on the cavity walls and rub for 15s; and dried for 5s, a second layer of adhesive was also used and then light cured for 10s.

Group III (OptiBond XRT): The self-etch primer of two-step self-etch adhesive (OptiBond XTR, Kerr Italia S.p.A., Salerno, Italy) was applied on the cavity walls using a micro brush with a scrubbing motion for 20s. The adhesive was shaken briefly and then applied using a light brushing motion for 15s; air thinned for at least 5s and finally light cured for 10s.

Group IV (OptiBond All-in-One): The bottle of one-step self-etch adhesive (OptiBond All-in-One, Kerr Italia S.p.A., Salerno, Italy) was shaken for 10s and rubbed on the cavity walls for 20s; a second layer of adhesive was applied in the same fashion, air thinned lightly for 5s and then light cured for 10s.

Group V (Fuji Bond LC with pre-cure technique): The cavity walls were conditioned with GC cavity conditioner (GC Corporation, Tokyo, Japan) for 10s, rinsed thoroughly with water and then air dried but do not desiccate, keeping the dentin surface glistening. The standard of powder/liquid ratio (0.7g/1.0g) of Fuji Bond LC (GC Corporation, Tokyo, Japan); on the other hand, one level spoonful of powder and two drops of liquid were mixed for 10s. The mixture then was applied in a thin layer to the conditioned enamel and dentin surfaces using a disposable brush and light cured for 20s (the pre-cure technique).

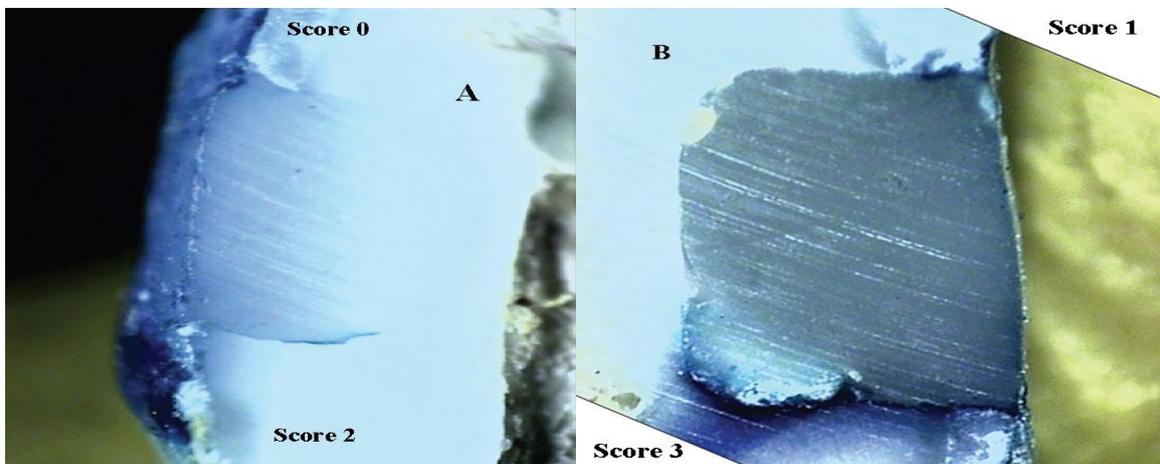
Group VI (Fuji Bond LC with co-cure): Same as group five, a thin layer of the Fuji Bond LC adhesive (GC Corporation, Tokyo, Japan) was applied to the conditioned enamel and dentin surfaces, but it was not cured, the GIC-based adhesive and resin composite was co-cured for 20s (the co-cure technique).

The all light cure materials were polymerized with light-emitting diode (LED) curing unit (Demetron A.2, Kerr Italia, S.p.A.) with a light intensity of 1000 mW/cm<sup>2</sup>; and the tip of light cure unit was placed 1-mm away from the surface of the restoration materials. Immediately following cavity treatment in each specimen, the preparation was filled with a micro-hybrid resin composite (Point 4, Kerr Italia, S.p.A., Salerno, Italy, A2 Body Shade) in a two-layer increment technique, curing each increment for 20s.

The restorations were finished with finishing diamond burs (Diatech Dental AG) and polished with aluminum oxide disk (Kerr Hawe, Bioggio, Switzerland) from course to fine under constant air/water coolant, one disk for each five restorations. Subsequently, the specimens were placed in 100% humidity at room temperature for one month, then stored in distilled water at 37 ± 1°C for two weeks and finally thermocycled 1500 cycles between 5°C to 55°C to simulate the clinic situation with a dwell time of one minute in each bath and transfer time five second. The apices of the teeth were sealed with sticky wax. All the external surfaces of the teeth, except for a 1.0-mm

margin around the restorations were covered with two layers of nail varnish. The teeth were then immersed in a 2% methylene blue solution (Merck KGaA, Darmstadt, Germany) for 24 hours at room temperature. The specimens were rinsed under tap water, air dried and sectioned into two halves mesiodistally in an occlusocervical direction through the middle of restoration with a water-cooled diamond disk (D&Z Diamant GmbH, Lemgo, Germany). Dye penetration was assessed in the two halves under a stereomicroscope (Nikon Eclips E600, Tokyo, Japan) at an ×20 magnification at the occlusal and cervical margins; if the microleakage score on the two halves was different, the half that showed more leakage was selected for assessment. Two independent pre-calibrated investigators blindly scored all interfaces and the consensus was forced when disagreements occurred. Dye penetration was scored on a scale from 0 to 3: 0=absence of dye penetration; 1=dye penetration less than half of cavity wall; 2=dye penetration more than half of cavity wall without reaching the axial wall and 3=dye penetration spreading along the axial wall. (Fig. 1) (1, 4, 8, 19).

Data were analyzed using SPSS-18 software (SPSS Inc, Chicago, IL, U.S.A.). The microleakage scores were analyzed using the non-parametric Kruskal-Wallis H test and Mann-Whitney U-test. The occlusal and cervical margins were compared with each other with Wilcoxon signed rank test ( $\alpha=0.05$ ).



**Figure 1.** Scoring of microleakage based on dye penetration: (A) score 0 at occlusal margin and 2 at cervical margin; (B) score 1 at occlusal margin and 3 at cervical margin.

## Results

Descriptive statistics including the frequency, means and standard deviations of the microleakage scores in the experimental groups are summarized in table 2 and table 3. Based on the results, none of the adhesives tested were capable of completely eliminating marginal microleakage. The findings from the Kruskal-Wallis H

test showed that there were statistically significant differences among the test groups at occlusal margins ( $P=0.002$ ); but at cervical margins were not ( $P=0.1$ ).

The results of the inter-comparison of the groups by the Mann-Whitney U-test revealed that only the Fuji Bond LC with co-cure had not significantly difference on microleakage scores, compared to the control group at the occlusal margins ( $P>0.05$ ). On the other hand, this

group (Fuji Bond LC with co-cure) and control group presented the highest microleakage; the other groups had no significant differences at the occlusal margins with together. Also the results of the inter-comparison of the groups by the Mann-Whitney U-test showed that at the cervical margins, the OptiBond XTR and OptiBond All-in-One had statistically significant lower microleakage scores, compared to the other groups ( $P=0.02$ ); whereas, the other groups had no significantly different from one another ( $P>0.05$ ). Also the results of this study demonstrated that there were no significant

differences between the resin adhesive groups both at occlusal and cervical margins ( $P>0.05$ ). At the cervical margins, the Fuji Bond LC with co-cure technique had only significantly higher microleakage scores than the groups that were bonded with OptiBond XTR and OptiBond All-in-One ( $P=0.023$ ).

The results of the Wilcoxon signed rank test showed that the microleakage scores at the cervical margins were markedly higher than the occlusal margins only in groups that were bonded with OptiBond Solo Plus or Fuji Bond LC with the pre-cure technique ( $P<0.05$ ).

**Table 1.** Various restorative materials and their compositions used in the study and mode of their applications according to the manufacturers' instructions.

Material	Composition	Manufactures' Instructions
OptiBond Solo Plus (two-step etch-and-rinse)	Bis-GMA, GPDM, GDMA, HEMA, ethanol, mono and di-functional methacrylate monomers, CQ, fumed Silica, barium aluminum borosilicate glass, sodium hexafluorosilicate	<ol style="list-style-type: none"> <li>1. Etch cavity walls with a 37.5% phosphoric acid gel for 15s.</li> <li>2. Rinse thoroughly for 20s and dry for 5s.</li> <li>3. Apply the adhesive and rub for 15s and dry for 3s.</li> <li>4. Light cure for 20s</li> <li>5. Place composite and light cure for 20s.</li> </ol>
OptiBond XTR (two-step self-etch)	Primer: GPDM, HEMA, Dimethacrylate, CQ, water, ethanol, acetone. Adhesive: Bis-GMA, HEMA, Tri-functional monomer, ethanol, CQ, barium glass filler, fluoride-containing filler, nano-filler.	<ol style="list-style-type: none"> <li>1. Apply the self-etch primer using a micro brush with a scrubbing motion for 20s</li> <li>2. Air thinning for 5s.</li> <li>3. Shake the adhesive briefly.</li> <li>4. Apply the adhesive using a light brushing motion for 15s and air thinning for 5s.</li> <li>5. Light cure for 10s.</li> <li>6. Place composite and light cure for 20s.</li> </ol>
OptiBond All-in-One (one-step self-etch)	GPDM, self-etching adhesive monomer, mono and di-functional methacrylate monomers, water, acetone, ethanol, CQ, nano-sized fluoride-releasing fillers.	<ol style="list-style-type: none"> <li>1. Shake the bottle for 10s.</li> <li>2. Apply the adhesive and rub for 20s.</li> <li>3. Apply a second layer of adhesive in the same fashion.</li> <li>4. Air thinning lightly for 5s.</li> <li>5. Light cure for 10s.</li> <li>6. Place composite and light cure for 20s.</li> </ol>
Fuji Bond LC (GC-based adhesive)	Powder: fluoroaluminosilicate glass. Liquid: copolymer of acrylic and maleic acids, HEMA, tartaric acid, water, chemical initiators	<ol style="list-style-type: none"> <li>1. Condition cavity walls with a GC cavity conditioner for 10s.</li> <li>2. Rinse thoroughly with water and dry without desiccate.</li> <li>3. Dispensing powder and liquid with one level spoonful of powder and two drops of liquid.</li> <li>4. Mixing powder and liquid for 10s.</li> <li>5. Apply Fuji Bond LC in a thin layer to the conditioned enamel and dentin surfaces.</li> <li>6. Light cure for 20s in the pre-cure technique, then place composite and light cure for 20s.</li> <li>7. Co-cure the adhesive and resin composite for 20s in co-cure technique.</li> </ol>

Bis-GMA: Bisphenol A diglycidyl methacrylate; CQ: Camphorquinone; GDMA: Glycerol dimethacrylate; GPDM: Glycerol phosphate dimethacrylate; HEMA: 2-hydroxyethyl methacrylate.

**Table 2.** Frequency of microleakage scores and p value of Wilcoxon signed rank test of the three different resin adhesives versus a glass ionomer-based adhesive (n=12).

Groups	Occlusal Margins				Cervical Margins				P value*
	0	1	2	3	0	1	2	3	
Control	4	4	1	3	3	1	4	4	0.222 <sup>‡</sup>
OptiBond Solo Plus	10	1	1	0	3	6	3	0	0.021 <sup>†</sup>
OptiBond XTR	10	2	0	0	8	2	1	1	0.102 <sup>‡</sup>
OptiBond All-in-One	10	1	1	0	8	2	1	1	0.334 <sup>‡</sup>
Fuji Bond LC as Pre-cure	9	3	0	0	6	1	4	1	0.047 <sup>†</sup>
Fuji Bond LC as Co-cure	4	3	3	2	5	1	5	1	0.914 <sup>‡</sup>

\*Wilcoxon signed rank test; <sup>†</sup>significant; <sup>‡</sup>non-significant

**Table 3.** The mean±SD of microleakage scores of the three different resin adhesives versus a glass ionomer-based adhesive (n=12).

Groups	Occlusal Margins	Cervical Margins
Control	1.25±1.22	1.75±1.22
OptiBond Solo Plus	0.25±0.62	1.00±0.74
OptiBond XTR	0.17±0.39	0.58±1.00
OptiBond All-in-One	0.25±0.62	0.58±1.00
Fuji Bond LC as Pre-cure	0.25±0.45	1.00±1.13
Fuji Bond LC as Co-cure	1.25±1.14	1.17±1.12

### Discussion

Despite vast advancements in restorative techniques and adhesive materials, microleakage has not been fully resolved due to the situation of oral environment. However, attempts to produce an ideal restorative material should be continued by manufactures that a permanent and perfect seal is created between the restoration margin and the tooth structure (9, 18). The investigators also should be with laboratory tests and clinical trials predict the clinical performance of new dental materials (20).

The results performing of the present study demonstrate that neither the resin adhesives nor the glass ionomer-based adhesive were capable of eliminating marginal microleakage completely in class V composite restorations. This finding is in agreement with the other previous studies (1, 2, 5, 19). In addition, this study revealed significant differences among the test groups in microleakage scores at occlusal margins; but no significant differences were found at cervical margins. Adhesion to enamel is a relatively simple process and durability of adhesive systems to enamel has proven to be effective to enhance bond strength and decrease leakage at the enamel-restoration interface

(3,9). In contrast to enamel, adhering composite resin to dentin is still a universal challenge, because dentin is a vital tissue with a heterogeneous nature which bonding to it is always problematic and difficult (3, 21).

The present study demonstrated that the microleakage scores obtained from the resin adhesives and the Fuji Bond LC with the pre-cure technique were significantly lower than the Fuji Bond LC adhesive with the co-cure technique and the control group at occlusal margins; on the other hand, the Fuji Bond LC with the pre-cure and resin adhesive groups had not significant differences with together at the occlusal margins. These findings support the effectiveness of these adhesive systems to reduce microleakage at the enamel margins. The results of the inter-comparison of the groups revealed that at cervical margins, only the groups that were bonded with OptiBond XTR and OptiBond All-in-One had significantly lesser microleakage scores than the other test groups. This implies that the new generations of resin adhesive systems are promising materials for bonding to dentin as well as to enamel and provided excellent bond strengths to both dentin and enamel; consequently, the microleakage of the restorations is reduced (22) hence, the null hypothesis

was accepted. OptiBond XTR self-etch is a 2-bottle light-cure universal dental adhesive with fluoride release; it employs GPDM technology, ternary solvent system, filled adhesive and optimized formulation to produce outstanding adhesion. OptiBond All-In-One is a single-component light-cure self-etch adhesive that greatly simplifies the restorative procedure by providing superior adhesion to all surfaces and substrates; it utilizes GPDM dental adhesive monomers and filler technology, delivering excellent penetration into dentinal tubules, for extraordinary bond strength and protection against microleakage and post-operative sensitivity and creates a deeper etched surface for higher mechanical retention (3, 7, 23).

The inter-comparison of the pre and co-cure techniques of Fuji Bond LC showed pre-curing technique provided better marginal sealing at occlusal margins. In agreement with this study, Satish *et al.* reported that the Fuji bond LC with pre-cure technique was most effective in reducing microleakage, and it exhibited the least microleakage than Prompt L-Pop and Single Bond, but statistically there is no difference between Fuji bond LC and Single Bond and among all the groups tested the Fuji bond LC performed better (24). Another study also states that the Fuji Bond LC with pre-cure technique exhibited lesser microleakage than Syntac single component adhesive and, it may be effective in reducing gingival microleakage in class V situations with gingival margins in cementum and dentin (25). In this study, combinations of the various materials were selected based on the same manufacturer's products (Kerr Italia S.p.A.). The only exception was Fuji Bond LC bonding agent (GC Corporation, Tokyo, Japan). Point 4 resin composite contains approximately 76% by weight (57% by volume) inorganic filler with an average particle size of 0.4 microns (3, 7, 23).

Fuji Bond LC is a diluted version of the restorative RMGIC that has been introduced as an alternative to traditional resin adhesives. It produces a hybrid layer upon the partial demineralization achieved through the use of 20% polyalkenoic acid conditioner. The Fuji Bond LC also contains Hydroxy Ethyl Methacrylate in its composition, similar to the fifth-generation and sixth-generation bonding agents, which provide for good wetting of dentin surface (16, 17).

In this study, the Fuji Bond LC with co-cure and control group presented greater microleakage than other groups. It is seem that the GIC-based adhesive is removed from some part of the dentin surface during resin composite placement and cause of adhesive detachment from the underlying tooth structure. However, this state could be the reasons for having the highest leakage similar to the control group. Tulunoglu *et al.* also resulted that the Fuji Bond LC with pre-cure

technique exhibited significantly greater microleakage scores than Scotchbond Multi-Purpose and Clearfil Liner Bond adhesives. In addition, there were no statistically significant differences between the pre-cure and co-cure techniques of the Fuji Bond LC(17). In addition, Yap *et al.* concluded that the marginal seal of one-step (Prime & Bond 2.0 and Bisco One-step) and RMGIC bonding systems appear to be as effective as two-step systems like Scotchbond Multi-purpose (26).

It is expected that new generations of resin bonding systems have better sealing ability, but the results of this study demonstrated that there were no significant differences between the different generations of experimental resin adhesives both at occlusal and cervical margins. A systematic review concluded that three-step etch-and-rinse adhesives and two-step self-etch adhesives have a clinically reliable and predictably good clinical performance. The clinical effectiveness of two-step etch-and-rinse adhesives was less favorable, while an inefficient clinical performance was noted for the one-step self-etch adhesives. Although there is a tendency towards adhesives with simplified application procedures, simplification so far appears to induce loss of effectiveness (11).

In this study, the OptiBond XTR and OptiBond All-in-One performed similarly microleakage at occlusal and cervical margins, while OptiBond Solo Plus and Fuji Bond LC with the pre-cure technique demonstrated better seal to enamel than to dentin. However, there are also controversial studies regarding differences in microleakage at occlusal and cervical margins. (1, 2, 4, 8, 19) The microleakage observed at the cervical margins may be related to the mainly organic nature of the dentin substrate. When polymerized, the composite resin shrinks toward the superior bond at the enamel margins and away from the weaker bond at the dentinal/cementum margins. Therefore, leakage occur at the less strongly bonded dentinal and cemental margins (27). OptiBond Solo Plus is a single-component adhesive, which is 15% filled with the same 0.4 micron filler found in the Kerr Point 4 composite. The filler not only reinforces the hybrid zone but also penetrates the dentin tubules, creating a true "structured bond" and provides the highest level of protection against microleakage (3, 7, 23).

These findings of this study encourage the use of Fuji bond LC with the pre-cure technique as a bonding agent for marginal seal in class V composite restorations. Nevertheless, the performance of the Fuji Bond LC with pre and co-cure techniques should be evaluated further with long-term storage in the laboratory and with clinical trials. This study encourage application of the Fuji bond LC adhesive with pre-cure technique instead of the resin adhesives for marginal seal in class V composite restorations.

## Conclusion

Within the limitations of this *in-vitro* study, the following conclusions were drawn:

- 1- None of the tested adhesives were capable of completely eliminating marginal microleakage.
- 2- At the occlusal margins, the Fuji Bond LC with co-cure technique and at the cervical margins, this group and the control group had the highest microleakage scores.
- 3- The Fuji Bond LC with pre-cure technique provided better microleakage properties than the co-cure.
- 4- There were no significant differences between the resin adhesives both at occlusal and cervical margins.
- 5- The microleakage scores at the cervical margins were markedly higher than the occlusal margins in the specimens that were bonded with OptiBond Solo Plus and Fuji Bond LC with pre-cure technique.

Therefore, according to advantages of RMGIC, this study encourage application of the Fuji bond LC adhesive with pre-cure technique instead of the resin adhesives for marginal seal in class V composite restorations.

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