



Microleakage of a Microhybrid Composite Resin Using Three Different Adhesive Placement Techniques

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Purpose: To evaluate the efficacy of two adhesive systems in reducing microleakage when applied with three different adhesive placement techniques.

Materials and Methods: Sixty freshly extracted caries-free human premolars and molars were used. MO/DO Class II standardized preparations were performed with the gingival margin placed 1 mm above the CEJ. Teeth were randomly divided into 2 groups (group I: Prime& Bond NT, Dentsply/Caulk; group II: Single Bond, 3M Espe). Each group was divided into 3 subgroups: (A) application of 2 coats and one cure: IA-IIA; (B) 2 coats and 2 cures of each adhesive system: IB-IIB; and (C) one coat of each adhesive along with the manufacturers' B1 flowable resin (0.5-mm thick layer) cured together at once: IC-IIC. Each coat was cured for 20 s at 800 mW/cm² using a quartz-tungsten halogen light (Elipar Trilight, 3M ESPE). Teeth were then restored using 2-mm increments of an A2 microhybrid composite (Esthet-X, Dentsply/Caulk). All teeth were stored in distilled water at 37°C for 24 h, thermocycled (500x, 5° to 55°C, 30 s dwell) and then placed in a 0.5% methylene blue dye solution for 24 h at 37°C. Samples were sectioned longitudinally and evaluated for microleakage at the gingival margin under a stereomicroscope at 20X magnification. Dye penetration was scored using an ordinal scoring system, where 0: no penetration; 1: enamel penetration; 2: gingival dentin penetration; 3: axial dentin penetration. Kruskal-Wallis and Mann-Whitney tests were used.

Results: A Mann-Whitney U-Test revealed no statistically significant difference between subgroups. Although not statistically significant, P&B NT (two coats and one cure) revealed the lowest microleakage scores.

Conclusion: In the experimental model adopted for this study, microleakage was not affected either by the adhesive or its placement technique.

Key words: adhesive system, Class II restoration, composite resin, microleakage.

J Adhes Dent 2004; 6: xx-xx.

Submitted for publication: 06.05.03; accepted for publication: 28.07.03.

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The use of composite resin may be associated with marginal discoloration, recurrent decay, and postoperative sensitivity. This is the result of composite resin polymerization shrinkage, which may be responsible for the formation of a gap between composite resin and the cavity walls. This gap may vary from 1.67% to 5.68% of the total volume of the restoration, and it may be filled with oral fluids and bacteria.²

Various studies^{3,7,8} pointed out that the marginal adaptation of composite resin is not related only to enamel and dentin bond strength, but is influenced by several other factors. At present, clinicians are aware that cavity configuration or C-factor,⁷ the modulus of elasticity of resin,^{14,15,31} the restorative technique,^{5,17} and the rate of polymerization^{4,10,13} can play important roles in the clinical performance of composite resin restorations.



Table 1 Groups according to adhesive and procedure

Group I (Prime & Bond NT)	Group II (Single Bond)
A) two coats and one cure	A) two coats and one cure
B) two coats and two cures	B) two coats and two cures
C) one coat + B1 Dyract	C) one coat + B1 Filtek

Presently, the use of low-viscosity composite resins and filled adhesives as liner materials is increasing in popularity.^{6,16,26} Even though these new materials have been introduced onto the market, the problem of adequate composite resin marginal adaptation – and microleakage elimination – is far from being solved. An important role in this area may be played by enamel-dentin adhesive systems and application technique. In the last five years, fourth generation three-step total-etch adhesive systems have been progressively replaced by fifth generation two-step total-etch adhesive systems. In spite of a simplified application technique, fifth generation adhesive systems are more technique sensitive than their precursors.^{30,32} This phenomenon may be related to a higher solvent-to-monomer ratio, which may be responsible for the formation of an uneven hybrid layer.

Platt et al²⁴ reported improved dentin bond strength for 2 coats vs one coat of Prime & Bond NT. Van Meerbeek et al³³ recommended “brushing thinning rather than air thinning” to avoid reducing the adhesive film thickness to an extent that the air-inhibited layer corresponds to a great part of the resin layer, reducing the bond effectiveness. According to Rueggeberg and Margeson,²⁵ the top 15 μm of the adhesive resin cannot polymerize because of oxygen inhibition. Any layer thinner than this cannot be polymerized, thereby preventing establishment of a bond.

To overcome this problem, Unterbrink and Liebenberg²⁹ proposed the use of flowable composites as filled adhesives; they combined the use of a single component adhesive as a dentin primer and a thin layer of flowable composite as a filled adhesive, which were cured together to avoid oxygen inhibition of very thin adhesive layers. Conversely, the importance of precuring direct composite restorations was previously reported.¹⁸

The purpose of this study was to evaluate the ability of two different fifth generation adhesive systems to reduce microleakage when applied with three different placement techniques: (A) two coats and one cure, (B) two coats and two cures, and (C) one coat of each adhesive along with the manufacturers' B₁ flowable resin cured together at once.

The null hypotheses tested were: (1) the application of two coats and one cure and two coats and two cures of fifth generation adhesive systems will result in a similar mean microleakage score; (2) the application of one coat along with flowable resin will result in increased microleakage scores because of incomplete resin polymerization.

MATERIALS AND METHODS

Sixty freshly extracted caries-free human premolars and molars were kept in distilled water at 4°C for 24 h. The dimensions of the preparation were standardized: 4 mm long, 3 mm wide, and 5 mm deep. MO/DO Class II cavity preparations were prepared with the gingival margin 1 mm above the CEJ using a 245 carbide bur (Brasseler, Savannah, GA, USA) with a high-speed handpiece and copious amounts of water. The teeth were divided into two groups, which were subdivided into three subgroups.

Each prepared tooth was etched with 34% H₃PO₄ (Tooth Conditioner Gel, Dentsply/Caulk Mildford, DE, USA) for 15 s, rinsed for 20 s, and then gently blown to remove excess water, being careful to maintain a moist surface. A nanofilled acetone-based adhesive system (Prime& Bond NT, Dentsply/Caulk) was used in group I, and an unfilled ethanol/water-based adhesive system (Single Bond, 3M Espe, St. Paul, MN, USA) was used in group II.

Each group was divided into 3 subgroups: (A) application of two coats of each adhesive system with the two coats cured together for 20 s at 800 mW/cm²; (B) two coats of each adhesive system and two cures with each coat cured for 20 s at 800 mW/cm²; and (C) one coat of each adhesive system along with the manufacturers' B₁ flowable resin (even, 0.5-mm thick layer) cured together at once. Dyract flowable compomer resin (Dentsply/Caulk) was used with Prime& Bond NT and Filtek flowable composite resin (3M Espe) was used with Single Bond (Table 1). Each coat of adhesive and flowable resin was cured for 20 s at 800 mW/cm² using a quartz-tungsten halogen light (Elipar Trilight, 3M ESPE). The light intensity of the curing light was evaluated using a light meter (curing radiometer, Kerr/Demetron, Orange, CA, USA) before starting each restoration.

A 0.0015-inch (0.038 mm) Toffelmire metal matrix band was used to reconstruct the proximal surface and simulate clinical conditions. The restorations were completely filled with an A2 microhybrid composite (Esthet-X, Dentsply/Caulk) using an apico-occlusal vertical layering technique with each layer not exceeding 2 mm. A conventional continuous mode of polymerization (800 mW/cm² continuous energy output for 20 s) was used to cure each composite resin layer. Restorations were finished using carbide burs, and polished using Enhance (Dentsply/Caulk) points and cups and Prisma Gloss (Dentsply/Caulk) polishing paste. All teeth were stored in distilled water at 37°C for 24 h.

The restored teeth were thermocycled 500x in a 5° – 55°C water bath with a dwell time of 30 s in each bath. The samples were then blotted dry with a paper towel. A clear nail polish was applied on the entire tooth surface with the exception of the restoration and the area surrounding the cavosurface margins and then air dried. All specimens were then immersed in 0.5 methylene blue dye solution for 24 h. The dye solution was buffered to pH 7 in order to avoid demineralization of the tooth structure, which would have given false readings. Teeth were rinsed

Table 2 Group I microleakage scores

Subgroup	Scores				Samples
	0	1	2	3	
IA	6	0	1	3	10
IB	5	0	1	4	10
IC	1	2	0	7	10
Total	12	2	2	14	30
Kruskal-Wallis Anova revealed no statistically significant difference between IA vs IB vs IC (p = 0.104)					

Table 3 Group II microleakage scores

Subgroup	Scores				Samples
	0	1	2	3	
IIA	1	1	0	8	10
IIB	3	0	1	6	10
IIC	2	3	0	5	10
Total	6	4	1	19	30
Kruskal-Wallis Anova revealed no statistically significant difference between IIA vs IIB vs IIC (p = 0.421)					

in running water, blotted dry, and embedded in an acrylic resin block (Orthodontic Resin, Dentsply/Caulk). The teeth were sectioned longitudinally from mesial to distal with a water-cooled diamond-wheel saw (Isomet-Buehler, Lake Bluff, IL, USA). Dye penetration at the gingival margin was examined using a stereomicroscope at 20X by two independent evaluators precalibrated at 85% reliability. If any disagreements in score between the two evaluators were reported, the higher score was taken. Samples were scored utilizing an ordinal scoring system, where 0: no penetration; 1: enamel penetration; 2: gingival dentin penetration; 3: axial dentin penetration. Statistical analysis was performed using Kruskal-Wallis Anova at $p < 0.05$ and the Mann-Whitney U-Test.

RESULTS

None of the adhesive placement techniques tested in this study completely eliminated microleakage. The data showing the extent of leakage in groups I and II are summarized in Tables 2 and 3. In group I, subgroup C exhibited a higher microleakage score with a prevalence of axial dentin penetration; dentin penetration was reduced for subgroups B and especially A. The latter had a higher number of samples with no gap formation. Dentin penetration of the dye solution was also higher in the three subgroups of group II; subgroup C showed a lower level of axial dentin penetration in this group.

The Kruskal-Wallis ANOVA test revealed no statistically significant difference between the two groups ($p = 0.1378$). Further analysis by the Mann-Whitney U-test was undertaken to compare the six different subgroups; no statistically significant difference was found between these subgroups.

DISCUSSION

Maintaining a moist dentinal surface is of crucial importance in enhancing bond strength *in vitro*.^{12,27} Moisture prevents collagen fibrils from collapsing into demineralized dentinal surfaces after acid etching, thus allowing

the adhesive monomers to penetrate into dentin, forming a hybrid layer.¹⁹ This technique is commonly known as the wet bonding technique and has been considered a paramount mechanism in adhesive dentistry. Quantifying the amount of moisture that should be left after acid etching is, however, problematic. Moreover, differing amounts of moisture may be required with specific solvents contained in each adhesive system (eg, acetone and ethanol vs water); overwetting and overdrying phenomena may occur and lead to the formation of an altered hybrid layer known as a hybridoid layer.^{28,33}

In this study, particular attention was paid to preserving a moist dentin surface prior to the start of bonding procedures, both with Prime & Bond NT and Single Bond. The excess water was removed using an air syringe; this method is particularly technique sensitive. The formation of totally or partially collapsed collagen layers may be responsible for differences in microleakage scores within groups IA and IB. The use of damp cotton pellets to remove excess water may help to create an ideal moist dentinal surface without the risk of dentin desiccation.²³ Perdigão et al²¹ reported a dramatic decrease in bond strength *in vitro* after air drying dentin for 15 s. This was a common result with acetone, ethanol, and ethanol/water-based adhesive systems, but not with a water-based adhesive system, which demonstrated an intrinsic rewetting capability. A rewetting time twice as long as the time spent on drying was required to restore bond strength in water-free adhesive systems. In this context, one could expect better performance of Single Bond, which contains water. Conversely, there is a trend for Single Bond to show higher microleakage scores. This was particularly relevant for groups IIA and IIB, in which the adhesive system was applied in multiple coats avoiding the formation of a too-thin hybrid layer and assuring its complete polymerization. Although Single Bond is an unfilled adhesive system, it contains polyalkenoic acid, contributing to the thick resin layer even when using a single coat.

Moreover, doubling the number of coats recommended by the manufacturer could affect the performance of this adhesive system. Conversely, a more uniform hybrid and resin layer can result from doubling the number of coats of Prime & Bond NT; this procedure can promote the for-

mation of a stress-absorbing resin layer which is able to reduce microleakage.^{9,11,31} The use of multiple coats along with a flowable resin on the uncured adhesive system were attempts at excluding the formation of a too-thin unpolymerized adhesive resin layer, created by oxygen inhibition. In both cases, no improvement in microleakage score was observed. The amount of dentin moisture may have played a more important role than the effect of the oxygen inhibited layer over the hybrid layer. Notable is the fact that this study contradicts the theory by Unterbrink and Liebenberg²⁹ that the use of flowable composites as filled adhesives reduces leakage. Laboratory or clinical data have never been presented in support of this theory. This is the first study to scientifically analyze this technique in vitro.

The level of moisture may be less important clinically than under laboratory conditions. A recent study reported similar results for Prime & Bond NT and Single Bond applied either on moist or dried dentin after six months of clinical service.²² The discrepancy between laboratory and clinical studies may be related to the different level of dentin's intrinsic moisture in vitro and in vivo. Vital dentin and its fluid filled tubules may be a critical factor. It can be hypothesized that dentin possesses a self-rewetting capacity in vivo; this means that when slightly dried, dentin can recover some degree of moisture, leading to a reduction in technique sensitivity.

A recent in vitro study⁶ conducted in our laboratory by the same clinician reported a lower microleakage score for Prime & Bond NT when used with the two coats/two cure technique. In the previous study, the packable composite resin Pyramid (Bisco, Schaumburg, IL, USA) was utilized instead of the microhybrid composite resin Esthet-X. We hypothesized that the different composite resin was not the only factor responsible for such a difference; indeed, the light intensity could have played a very important role (600 vs 800 mW/cm²). The higher light intensity used in the current study may be responsible for a faster polymerization reaction of the adhesive system and microhybrid composite resin, thus leading to the development of higher interfacial stress and increased gap formation. The use of self-cured and dual-cured bonding agents should be encouraged. They can contribute to a reduction in microleakage. The polymerization reaction progresses at a slower rate than in light-cured adhesive systems; additionally, the porosities incorporated during mixing may be responsible for a stress relaxation mechanism.^{1,20,34}

CONCLUSION

The results of this study confirmed the high degree of technique sensitivity associated with the use of fifth generation adhesive systems and composite resin restorations. The null hypotheses were only confirmed in part. The attempt to establish a thick resin layer on the top of the hybrid layer through the application of multiple coats resulted in reduced microleakage scores for the nano-

filled acetone-based adhesive system Prime & Bond NT, but not for the unfilled ethanol/water-based adhesive system Single Bond. However, this was not statistically significant. The association of Prime & Bond NT with Dyract flowable compomer resin and Single Bond with Filtek flowable composite resin did not prevent microleakage. Further research should be conducted to provide clinicians simpler and faster adhesive systems that can guarantee a long-term clinical seal at the cavosurface margins.

ACKNOWLEDGMENTS

The authors would like to express their gratitude to Dentsply/Caulk and 3M ESPE North America for providing materials.

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Deliperi et al

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Clinical relevance: Composite resin microleakage was not affected either by the adhesive system or application technique. Precuring the adhesive system is recommended to assure complete resin polymerization.