

Microleakage of "All-in-One" Adhesive Systems on Enamel and Dentinal Margins: An *In Vitro* Study

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Abstract:

Objective: The use of presently available dentin bonding systems has greatly reduced microleakage; however, the ideal situation where the adhesive resin completely penetrates the demineralized dentin is not yet achieved. The purpose was to compare the microleakage of fifth and sixth generation bonding agents at enamel and dentinal margins.

Materials and Methods: Class V cavities were prepared at the CEJ of thirty extracted human premolars. The teeth were divided into three groups (n=10). In group I, cavities were treated with Prime&Bond NT; in group II, UniFil Bond; and in group III, Prompt L-Pop bonding agents were used following which composite resin (Z100) was placed incrementally. The specimens were stored in an environment of 100% humidity, immersed in a fresh solution of 50% Silver Nitrate each for 24 hours and then placed in a developing solution for 8 hours. After rinsing and being sectioned buccolingually through the center of the restoration, the samples were evaluated under a stereomicroscope at x50 magnification for microleakage along occlusal and gingival margins. The data were analyzed using Kruskal-Wallis and Multiple Comparison tests.

Results: There was a significant difference between the three groups. The fifth generation dentin bonding agent (Prime&Bond NT) showed the least amount of microleakage, while the sixth generation ones (UniFil Bond and Prompt L-Pop) showed higher amounts at enamel and dentinal margins.

Conclusion: Fifth generation bonding agents seem to generate better results than those of sixth generation.

Key Words: Dentin-Bonding Agents; Dental Leakage; Prompt L-Pop; Prime and Bond NT; Unifil Bond; Z100 composite resin

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INTRODUCTION

Adhesive dentistry has been progressing with a rapid pace over the past decade. A large part of this success is attributed to the significant advances in dentin bonding technology. From the early generation systems in the seventies that yielded weak and unpredictable bonds, to the contemporary hydrophilic systems that produce significant improvements in bond strength to normal dentin; it seems fair to say

that the progress in the development of dentin adhesives has been nothing short of phenomenal [1]. Efficient adhesion between cavity walls and restorative materials is desired, producing well-sealed and long durable restorations. The major shortcoming of visible light cured composite is the polymerization contraction, resulting in gap formation, particularly at dentin interface and making way for microleakage with ingress of bacteria, their toxins,

fluids, molecules, or ions between the cavity walls and the restorative materials [2]. The use of presently available dentin bonding systems with their micromechanical adhesion to the tooth structure has greatly reduced the mentioned disadvantage, however, the ideal situation where the adhesive resin completely penetrates into the demineralized dentin is not yet achieved. Current generation of dentin bonding agents is designated as fifth and sixth generations [2-4]. The fifth generation bonding agent is available as one-bottle adhesives. One bottle system combines the primer and adhesive in one solution to be applied after enamel and dentin are etched simultaneously, the so-called "Total-etch technique". In this system, the dentin surface should remain in a moist state to prevent collapse of unsupported collagen and promote primer-resin infiltration. Some adhesives combine the etching and priming steps, resulting in a so-called, "self-etching primer", that simultaneously etches and infiltrates both enamel and dentin (Sixth generation bonding agents). Self etching primers enable resin monomers to penetrate into the underlying dentinal substrate through the smear layer needless of separate etching, rinsing and drying procedures [5]. The combination of these two steps reduces the working time, eliminates the need to rinse the acidic gel, and eliminates the risks of over etching and over drying [4,6,7].

More recently, following the trend of simplification, "All - in- one", self-etching adhesive systems that combine etching, priming and adhesive in one solution has been developed. These also have been categorized as the sixth generation dentin bonding agents. In these systems methacrylated phosphoric esters function as an etching agent in the primer, so that separate acid etching of enamel and dentin is not required [4]. These current dentin adhesives employ two different means to achieve the goal of micro-mechanical retention between resin and dentin. The first method removes the

smear layer completely and demineralizes the intact underlying dentin via acid etching with chelating agents or mineral acids. Following rinsing, a multi- step application of a primer and an adhesive, or a simplified self-priming adhesive (fourth and fifth generation), is applied to the conditioned substrate to complete the bonding protocol (Total-etch technique). The second method uses the smear layer as a bonding substrate. Known as "Self-etching primers/adhesives", (sixth generation), they are applied to the smear layer-covered dentin for a designated period of time, with no further rinsing, a layer of adhesive resin is then applied to the treated dentin and light cured [4,6,8]. In this system the goal is to incorporate the smear layer into the hybrid layer. There are several studies which done to evaluate the efficiency of different generations of dentin bonding agents, their sealing properties and bond strength [2,3,9].

The purpose of this *in vitro* study was to evaluate the microleakage of current generation dentin bonding systems (Prime&Bond NT, UniFil Bond and Prompt L-Pop) at the enamel and dentinal margins around class V composite restorations.

MATERIALS AND METHODS

In this experimental study 30 extracted non-carious human premolars were selected. The teeth were immersed in 0.1% thymol solution for disinfection. Cylindrical carbide burs were used in with a high-speed water-irrigated hand piece to prepare Class V cavities, (1.5 mm deep, -2 mm high occlusogingivally, -3 mm wide mesiodistally) on the buccal surface of the teeth at the C.E.J, so that the upper margins were in enamel and lower margins were in dentin/cementum. A 45 degree, 0.5 mm wide bevel was performed at the enamel margin using a flame shaped bur.

After the preparations, the teeth were randomly divided into three groups of 10. The cavities were treated as follows: in group I the

cavities were etched with 37% phosphoric acid gel (Ivoclar, Vivadent, AG, Schaan, Liechtenstein). Etching time was 30 seconds for enamel and 15 seconds for dentin. Cavities were rinsed with an air-water spray for 10 seconds. Excess water was blot dried using cotton pellet, leaving a visibly moist surface. Prime&Bond NT bonding agent (Dentsply, Dentre, Str. 1,78467 Konstanz, Germany) was applied in an even layer to enamel and dentin surfaces of the cavity using microbrush applicator for 20 seconds, left undisturbed for 10 seconds and gently air dried for 3 seconds before it was light cured (Kulzer halogen curing light, Heraeus, Germany, 400 mw/cm²) for 10 seconds. In group II self-etching primer bonding agent UniFil Bond (GC Corporation, Tokyo, Japan) was used to treat the cavities. The primer was applied to the cavities using microbrush applicator for 20 seconds and left undisturbed for 10 seconds. After the primer was air dried with a mild air flow for 3 seconds, the adhesive resin was applied with another microbrush applicator to the entire cavity wall for another 10 seconds (Acc. to manufacture instruction) before being light cured for 10 seconds. In group III the cavities were treated with a self-etching adhesive bonding agent Prompt L-Pop (ESPE, AG, Seefeld, Germany). The upper container (Red color) was squeezed and folded over the lower container (green

color), the content of two containers got mixed together and saturated the microbrush applicator. Afterwards, the applicator was pulled out of the container and applied to the entire cavity by rubbing action for 20 seconds. A gentle stream of air was blown for 3 seconds to disperse the material to a thin film thickness and light cured for 20 seconds. For all the three groups the cavities were restored with resin composite, Z-100 (3M, ESPE, Dental Product, USA) B2 Shade using incremental technique. All the materials used in the study and their composition were carefully recorded (Table 1). After the specimens were stored in 100% humidity for 24 hours, final finishing and polishing was done using fine diamond burs and Soflex polishing disks (3M ESPE, USA). The restorations were subsequently thermally stressed for 500 cycles with an exposure time of two seconds at 5 °C and 55 °C and a dwell time of 10 seconds in a resting bath of 34 °C. All the surfaces of the restored teeth were sealed with two layers of nail varnish, except for the restored part and a 1.0 mm wide zone adjacent to the margins of the restoration. After the teeth were thoroughly dried, they were immersed in a fresh solution of 50% Silver Nitrate for 24 hours and then placed in a developing solution for 8 hours. After being rinsed, they were exposed to flood light in order to reduce the silver ions to metallic silver

Table 1. Materials used in the study and their composition

Material	Characteristic	Main Components	Manufacturer	Lot Number
Total Etch	Etchant gel	37% Phosphoric acid	Ivoclar , Vivadent, Liechtenstein	H17816
Prime&Bond NT	One-bottle total-etch adhesive	PENTA, T.Resin, D.Resin, UDMA, Butylated hydroxitoluence, 4 Ethyl dimethylamino benzoate, Silica, Acetone	Dentsply, DenTrey, Konstanz, Germany	0611001781
UniFil Bond	Two-step self-etch adhesive	Primer: HEMA, 4MET, UDMA, Photoinitiator ;Adhesive: UDMA, TEGDMA, HEMA, Silanated Colloidal Silica, Water, Ethanol	GC International, Tokyo, Japan	Primer:0302281 Adhesive: 0302281
Prompt L-Pop	One-step self-etch adhesive	Liquid-1 :Methacrylic phosphate, Stabilizer, Phosphin Oxide; Liquid-2: Water, Fluoride complex, Methyl and Propyl Prabene	3M , ESPE, Germany	FW 62210
Z100, B2	Resin composite	Bis-GMA, TEGDMA, Zirconia/Silica	3M , ESPE, Dental Product, USA	20021212

for better visualization. Following that, the teeth were kept in running water for 5 minutes for the developing solution to be thoroughly removed. The specimens were then cleaned and sectioned both vertically and buccolingually through the center of the restoration with a diamond disk and observed under a stereomicroscope (Olympus model BX-50, Tokyo, Japan) at a magnification of x50 for microleakage along the occlusal and gingival margins. The following scores were used to assess the extent of dye penetration at the mentioned margins:

0-No dye penetration

1-Dye penetrating up to 1.0 mm in depth

2-Dye penetrating beyond 1.0 mm in depth

3-Dye penetrating beyond the axial wall

The mean amount of leakage was recorded for each group together with standard deviation. Statistical analysis was carried out using Kruskal-Wallis and multiple comparison (Dunn test) tests separately for enamel and dentinal margin.

RESULTS

The results of the present study showed that the amount of microleakage at dentinal margins was higher than that at enamel margins in all the three groups. The sixth generation dentin bonding agent (Prompt L-Pop) showed the highest amount of microleakage, while the fifth generation (Prime&Bond NT) showed the least amount.

As for enamel margins a microleakage score of 0 was assigned to ten specimens in group I, eight specimens in group II, and eight specimens in group III (Table 2) and Dunn test re-

vealed no significant difference between the specimens in groups II & III, however, there was a significant difference between the groups I & II, and also between the groups I & III ($P<0.001$) (Table 3).

Regarding to dentinal margins, a microleakage score of 0 was assigned to four specimens in group I, four specimens in group II, and three specimens in group III (Table 2). Dunn Test illustrated no significant difference between the specimens in groups I & II; yet, there was a significant difference between the groups I & III, and also between the groups II & III ($P<0.001$). The specimens in group I (Fifth generation dentin bonding system, Prime&Bond NT) had the least amount of microleakage among the groups, and specimens in group III (Sixth generation dentin bonding agent, Prompt L-Pop) had the highest amount of microleakage at enamel and dentinal margins. Also it was noted that the value of microleakage in dentin was higher than in enamel in all the groups regardless of type of the bonding agent used.

DISCUSSION

The results of the present study showed that amount of microleakage was greater at dentinal margin than enamel margins in all the groups regardless of the type of the bonding agent used.

Bonding to enamel is a relatively simple process, with no major clinical requirements or difficulties. Bonding to dentin, on the other hand, seems to present a much greater challenge [2,10]. Three different types of bonding agents were used in the present study, Prime&Bond

Table 2. Microleakage as at enamel and dentinal margins.

Microleakage	Group I		Group II		Group III	
	Enamel	Dentin	Enamel	Dentin	Enamel	Dentin
No dye penetration	100%	40%	80%	40%	80%	30%
Dye reaching 1.0 mm in depth	0%	50%	10%	50%	10%	50%
Dye reaching beyond 1mm in depth	0%	10%	10%	10%	10%	20%
Dye reaching or beyond the axial wall	0%	0%	0%	0%	0%	0%

NT (Fifth generation dentin bonding agent), UniFil Bond and Prompt L-Pop (Sixth generation dentin bonding agent). The samples in group I were treated with Prime&Bond NT bonding agent. The result showed that this bonding agent had the least amount of leakage at enamel and dentinal margins. Prime&Bond NT is a fifth generation bonding agent that contains primer and resin adhesive both in one bottle. It consists of PENTA, T. Resin (Cross linking agent) and D. Resin (small hydrophilic molecules) as primer, UDMA, Butylated hydroxitoluence, 4 ethyl dimethylamino benzoate as adhesive, and silica as nanofillers. The solvent used in this bonding agent is acetone [11,12]. Nano-technology gets adhesion closer to nature. Nanofillers have the perfect size to penetrate the typical micro-sized key-hole etch pattern of enamel as well as the smallest dentin channels. The nanofillers in this bonding agent may help to establish a film thickness of uniform resin that establishes the hybrid layer. The nanofillers in Prime&Bond NT are 0.007 micrometer silica particles of, which a film thickness of up to five micrometer can be provided. These tiny particles support the natural components of dentin while building the foundation for a perfect link between the tooth structure and the restorative materials. They can provide a great ability to flex, and help dissipate the stress of composite polymerization resulting in a lesser amount of microleakage acting as a shock absorber [6,13,14].

Group II specimens were treated with UniFil Bond bonding agent, an advanced, easy-to-use, light cured resin bonding system which utilizes both chemical and micromechanical adhesion

principles. It is a two-step smear layer-dissolving adhesive, which is supplied in two bottles. Both the etchant and the primer are there in one bottle with the resin adhesive in another one. The first bottle contains HEMA, 4 MET, UDMA, photo initiator, and the second bottle contains UDMA, TEGDMA, HEMA, Silanated colloidal silica. The solvents are water and ethanol. 4-MET monomer provides the acid etching effect on tooth surfaces penetrating and diffusing throughout the dentin while decalcifying tooth structure, polymerizing together with the bonding agent to form a hybrid layer (approximately 1 micrometer thick). Carboxylic acid ions chemically bond to calcium ions in the tooth structure. Monomer component of the bonding agent blends with that of the self-etching primer. Once polymerized, resin tags are formed in enamel and a hybrid layer is generated in dentin. In this technique the cavity was not rinsed one of the advantages when using this system is that the cavity is not rinsed which makes it less time consuming. The specimens treated with this bonding agent showed relatively good sealing. There was no significant difference in microleakage at dentin margins between the samples in this group and those in group I. The main advantage of these bonding agents, apart from being less time consuming, is that unlike so-called the total etch technique, the depth of resin penetration into dentin is the same as the depth of demineralization. Thus, there will be no unsupported and unfilled collagen fibers left behind through acid etching [15,16]. Penetration of the resin into partially demineralized dentin,

Table 3. Multiple Comparison Dunn Test For Enamel and Dentin Margins.

Comparison	Rank Difference		Standard Error		P Value	
	Enamel	Dentin	Enamel	Dentin	Enamel	Dentin
3 VS 2	0	22.5	2.325	3.58	P>0.05	P<0.001
3 VS 1	30	22.5	2.325	3.58	P<0.001*	P<0.001
1 VS 2	30	0	2.325	3.58	P<0.001*	P>0.05

There is a statistically significant difference between group I and II; and between group I and III at enamel margin (P<0.001); and between group II and III; and between group I and III at dentin margin (P<0.001).

engaging both chemical bond between the resin and hydroxyapatite crystals and micro-mechanical bond, is the key to a successful bond to the tooth structure. HEMA is included as a component in most priming resins and due to its wetting behavior and affinity towards dentin, makes it acid resistant after impregnation. HEMA may also generate hydrogen bonds (or a new bond to ester group) inside the microporosities of demineralized dentin mechanically interlocking into the substrate by undergoing hygroscopic expansion after polymerization [17,18].

The specimens in group III were treated with Prompt L-Pop self etching adhesive bonding agent. This single dose packaging bonding agent with an integrated applicator, allows the operator to etch, prime, and bond, all in one-step, in seconds. Its simplest mechanism of "squeeze-fold over-squeeze" provides an uncomplicated hygienic process resulting in reduced sources of error and contamination. It contains two containers; the upper container (red color) carries methacrylated phosphoric ester, (DiHEMA phosphate), initiator (Phosphin Oxide) and stabilizer. The lower container (yellow color) houses fluoride complex, Butyl hydroxy toluene), preservative (Methyl and Propyl parabene) and water as a solvent. The green part carries the applicator tip. This adhesive system is an aqueous phosphoric ester solution that can dissolve the surface of enamel and dentin because of its low pH of 1 or less and interact more profoundly with dentin [12,19]. As mentioned previously, the rationale behind the action of these self-etching agents is the formation of a continuum between tooth surfaces and the adhesive material accomplished through the concurrent demineralization and penetration of resin in enamel and dentin surfaces. Nevertheless, omitting the conventional etching step by phosphoric acid may eliminate the characteristic demineralization of enamel and dentin [11]. With Prompt L-Pop bonding agent, the most extensive pene-

tration of silver nitrate was observed. There is a possibility that the lack of a separate primer may reduce the infiltration depth or the wettability of dentin adhesives, thereby, reducing adhesion and sealing capacities [15,20]. The result of this study is in agreement with the one obtained by Yazici et al [3] who compared and evaluated the microleakage of Prompt L-Pop bonding agent with fifth generation bonding agents. They concluded that the greatest amount of microleakage was seen when using Prompt L-Pop bonding agent. Several authors have reported superior results when Prompt L-Pop is used with poly acid-modified resin composites (Compomers) [9,17,18]. This might be due to the fact that compomers have lower viscosity and are hydrophilic restorative materials; therefore, they are chemically more compatible with Prompt L-Pop being a water-based material. On the other hand, resin composites are hydrophobic restoratives, and this may explain the severe microleakage observed in this group [9,11,12]. Another explanation could be that this self-etching adhesive bonding agent was not strong enough to be able to dissolve the smear layer efficiently and subsequently penetrate through it [21].

In the present study, the specimens in group III (Prompt L-Pop) showed the highest amount of microleakage both at enamel and dentinal margins. Although there was no significant difference between group I and group II, there were significant ones between the groups III & II and the groups III & I at dentin margin.

These results represent *in vitro* data and definite conclusions should not be drawn until *in vivo* studies are completed, however, they do provide some guideline for the performance of new dentin bonding systems.

CONCLUSION

From the results of the present study it can be concluded that none of the latest generations of adhesive resin bonding systems can create gap-free margins in class V composite restora-

tions. The amount of microleakage at dentinal margins was greater than enamel ones and the fifth generation dentin bonding agent showed less amounts of microleakage compared to the sixth generation.

According to the results of the present study, the sealing abilities of the three bonding systems can be put in order as follows:

Prompt L-Pop < UniFil Bond < Prime & Bond NT

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