

Effect of EDTA Conditioning on Microleakage of Four Adhesive Systems in Composite Restorations

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Abstract:

Objective: Evaluating the effect of dentin conditioning with EDTA on microleakage of composite resin restorations, using two etch and rinse and two self-etch adhesives.

Materials and Methods: One hundred and sixty extracted molars received class V cavity preparations right under the CEJ and were randomly divided into eight groups of 20, using four different adhesive systems. These adhesives included Adper Scotchbond Multipurpose (SBMP), Adper Single Bond (SB), Clearfil SE Bond (CSEB), and Adper Prompt L-Pop (PLP). In the SBMP and SB experimental groups, EDTA was applied instead of phosphoric acid. In the CSEB and PLP experimental groups, EDTA conditioning was added to the bonding process. After thermocycling, the amount of dye penetration was evaluated using stereomicroscope. The data were analyzed using the Kruskal-Wallis and Dunn tests.

Results: Two etch and rinse adhesives (SBMP, SB) showed a significantly lower microleakage than the two self-etch adhesives, CSEB and PLP, ($P < 0.05$). No significant difference was observed among the experimental groups. PLP and CSEB showed significantly less microleakage using EDTA conditioning ($P < 0.05$). There was no significant difference for SBMP and SB when applying either phosphoric acid or EDTA.

Conclusion: In the cases of SBMP and SB, EDTA conditioning is as effective as phosphoric acid in preventing microleakage. In cases of CSEB and PLP, EDTA conditioning can significantly improve the sealing ability.

Key Words: Dental Leakage; Adhesives; Phosphoric Acids; ethylenediaminetetraacetic acid bis-(ethyl phenylalaninate)

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INTRODUCTION

Although enamel bonding is an established issue in restorative dentistry, a reliable and predictable bond to dentin is yet difficult to achieve which is partly due to structural characteristics of dentin. In addition, a layer of smear is created immediately after cavity preparation [1,2]; while, its complete/partial removal or maintaining of is a controversial issue.

In etch and rinse adhesives, phosphoric acid etching completely removes the smear layer,

opens the dentinal tubules and also exposes a demineralized collagen network that will subsequently be filled with monomers [1-4]. Incomplete resin infiltration leaves an unprotected mineral-deprived collagen layer at the base of the hybrid layer leading to hydrolytic degradation and bonding failure [3-5].

In self-etching adhesives, acidic monomers either dissolve the smear layer or incorporate it into the bonding interface. Because of simultaneous dentin demineralization and resin infiltration, formation of the unprotected colla-

gen network is prevented; however, resin-reinforced smear layer can result in bonding defects and may be unstable [1,6-8].

Ethylenediaminetetraacetic acid (EDTA) is a gentler chelating agent at neutral pH that removes the smear layer and mildly demineralizes the dentin [9,10].

Takarda [9] reported that the hybridized dentin, conditioned with EDTA 3-2 (0.2M ferric ions in 0.5M EDTA solution), was continuous with the underlying intact bovine dentin and improved bonding durability. This was due to incomplete demineralization of the hydroxyapatite especially at the deeper portion of dentin. High-quality hybridization and proper bond strength have been reported when combining the EDTA conditioner and phenyl-p/HEMA primer [11,12]. Using EDTA 3-2 instead of acid phosphoric etching can prevent collapse of the collagen network [9,13].

Little is known about the effect of EDTA conditioning on the microleakage of dentin adhesive systems. Thus, the purpose of this study was to compare EDTA conditioning and phosphoric acid etching in two etch and rinse adhesives and also to evaluate the prior additional EDTA conditioning in two self-etching adhesives on their sealing ability in Class V composite restorations.

MATERIALS AND METHODS

Intact caries-free extracted human molars (n=160) were stored in 1% thymol solution at room temperature for two weeks and then in distilled water. The teeth were used within

three months after extraction. Class V cavities (1.5 mm in depth, 2 mm in height and 3 mm in width) were prepared on the buccal surface with a fissure diamond bur just under the CEJ. The bur was replaced after every four preparations. The teeth were randomly divided into eight groups of 20 each (n=20).

Four adhesives: Adper Scotchbond Multipurpose (SBMP; 3M, Dental Products, Germany); Adper Single Bond (SB; 3M, Dental Products, Germany); Clearfil SE Bond (CSEB; Kuraray Inc, Japan); and Adper Prompt L-Pop, (PLP; 3M, Dental Products, Germany) were used according to the manufacturers' instructions (Table1) as control groups.

In the four experimental groups, the same adhesives were used but, in SBMP and SB groups, 0.5 mol EDTA at pH=7.4 (MERCK Co, Germany) was applied for 60 seconds instead of phosphoric acid. In CSEB and PLP groups, EDTA conditioning was added to the bonding process.

The teeth were restored with hybrid resin composites, Z100 (3M, Dental Products, Germany) and Clearfil-APX (Kuraray Inc, Japan) with respect to the manufacturer of the adhesive used in each group. Curing was accomplished using a light curing unit (Coltolux, Coltene, Switzerland, 400 mW/cm²). After storage in water at room temperature for 24 hours, final finishing and polishing were done with Sof-Lex disks (3M, Dental Products, Germany).

The specimens were then thermocycled (500 cycles between 5°C and 55°C, 1min in each

Table 1. Adhesive Application Technique for Control Groups.

Adhesive	Manufacture	Technique
Adper Scotchbond Multipurpose (SBMP)	3M Dental product, USA	Apply 37% phosphoric acid for 15 sec, rinse, air dry. Apply Primer, air dry Apply Adhesive, light cure.
Adper Single Bond (SB)	3M Dental product, USA	Apply 37% phosphoric acid for 15 sec, rinse, air dry, leaving to dentin moist. Apply 2 consecutive coats of the adhesive. Dry gently for 5 sec. Light cure.
Clearfil SE Bond (CSEB)	Kuraray Inc, Japan	Apply primer for 20 sec. Mild air dry. Apply Bond. Gentle air dry. Light cure.
Adper Prompt L. Pop (PLP)	3M Dental product, USA	Mix using unit-dosed blister pack. Apply with agitation for 15 sec. Air dry, light cure.

bath with a 10-second transfer time).

The root apices were sealed with sticky wax and all the teeth surfaces, except for a 1-mm wide zone around the margins the restorations, were sealed with two layers of nail polish. All the teeth were then immersed in 0.5% basic fuchsin dye solution for 24 hours before being rinsed and sectioned facio-lingually across the middle of the restorations using a diamond saw (Letiz, 1600, Germany) with continuous water irrigation. The sectioned teeth were examined under a stereomicroscope (Ziess, Germany) at $\times 20$ magnification and scored for dye penetration on a scale of 0 to 4: 0=no microleakage; 1=dye penetration up to 1/3 of the cavity wall; 2=dye penetration between 1/3 and 2/3 of the cavity wall; 3=dye penetration extending more than 2/3 the cavity wall; 4=dye penetration spreading along the axial wall.

The nonparametric data were analyzed using Kruskal-Wallis and complementary Dunn tests at a 0.05 level of significance ($P < 0.05$).

RESULTS

The Kruskal-Wallis test revealed significant differences among the four adhesives in the control groups ($P < 0.001$). However, there was no significant difference among the four adhesives in experimental groups ($P > 0.05$) (Table 2 and 3).

Multiple comparison tests (Dunn) found a significant difference between SB and CSEB; SBMP and CSEB; SB and PLP; and SBMP and PLP in the control groups ($P < 0.05$). SBMP and SB showed a significantly lower microleakage than CSEB and PLP.

In addition, Mann-Whitney test showed that there was a significant difference between the control and EDTA experimental groups for PLP and CSEB ($P < 0.001$); while, there was no such difference for in cases of SBMP and SB ($P > 0.05$).

EDTA conditioning significantly decreased dentinal microleakage in PLP and CSEB groups. EDTA conditioning provided the same sealing ability of phosphoric acid etching in SBMP and SB groups.

DISCUSSION

In this study, two categories of adhesives were compared with respect to the effect of dentin conditioning on microleakage: etch and rinse (SBMP, SB) and self-etch (CSEB, PLP) adhesives.

In general, the two etch and rinse adhesives showed a significant less amount of microleakage in comparison with the two self-etch adhesives. This was consistent with previously published literature [14-15].

It seems that etch and rinse adhesives are still the most effective approach to achieving stable and efficient dentin adhesion [6,14-17]. However, some other authors have reported that self-etch adhesives show equal or less microleakage compared to etch and rinse adhesives [18-21].

In self-etch adhesives, incorporation of the smear layer into the hybrid layer is considered a potential disadvantage. Therefore, its removal by acid etching prior to the application of these adhesives may be necessary [7,22]. On the other hand, some studies have previously demonstrated that separate phosphoric

Table 2. Frequency of Microleakage Scores for four Adhesives Control Groups.

Adhesive	Microleakage Scores					Total
	0	1	2	3	4	
SBMP	16	3	1	0	0	20
SB	18	1	1	0	0	20
CSEB	2	10	5	3	0	20
PLP	6	10	3	1	0	20

SBMP: Adper Scotchbond Multi- purpose, SB: Adper Single Bond, CSEB: Clearfil SE Bond, PLP: Adper Prompt L-Pop

acid etching could decrease the bond strength and durability [23,24].

EDTA contains carboxylic acid groups giving it the ability to remove hydroxyapatite selectively. Since most of the intrafibrillar minerals remain, the structural support by the minerals is preserved, and resin infiltration is facilitated; whereas, phosphoric acid etching of dentin leads to dissolving both the extra and the intrafibrillar minerals resulting in recession and collapse of the collagen matrix. This may interfere with hybrid layer formation [25,26].

The results of the present study indicate that EDTA conditioning decreases microleakage of the two self-etch adhesives (PLP, especially CSEB) significantly.

Torri et al [27] reported that EDTA conditioning was effective in improving dentin bonding for all-in-one adhesives but offered no improvement for CSEB and SB. The possible reasons for these findings were probably due to the insufficient removal of the smear layer in the all-in-one adhesives leading to the disturbance of monomer infiltration into the underlying demineralized dentin. The smear layer residues may weaken the physical properties of the cured adhesive and adversely affect the bonding durability [8,27]. In the present study, removal of the smear layer using EDTA conditioner prior to PLP may prevent this interference.

In another study, pre-treatment with EDTA improved the dentin bond strength of CSEB and produced a stronger and more homogeneous hybrid layer [28]. It was considered that bonding between the dentin and the adhesive is established by the chemical reaction be-

tween the calcium and the functional monomer such as 10-MDP [29]. This may help prevent or retard microleakage [5].

In our study, removing the smear layer by EDTA conditioning and permitting the direct contact of CSEB with the dentin may provide a more intimate chemical interaction with 10-MDP on a molecular level and help to decrease the amount of microleakage.

According to the results of this study, there was no significant difference in microleakage for SBMP and SB with the application of either phosphoric acid or EDTA. In fact, EDTA can almost prevent dentinal microleakage of SBMP and SB effectively. This result is in accordance to previously bond strength studies of other etch and rinse products [10,30,31].

In a recent study, using EDTA instead of phosphoric acid was seen to improve the bonding durability of Single Bond [32].

In a study on contraction gap measurement, when the dentin was conditioned with EDTA, gap formation was completely prevented when using a multi-step adhesive while as for an acetone-based one-bottle adhesives, it was clearly inferior to the multi-step adhesive and also in case of two ethanol-based adhesives, no difference was reported when compared to the multi-step one [33]. It has been suggested that the contraction gap width increases in conjunction with either reduction in dentin hardness or decalcification through conditioning, especially in the absence of functional monomer in the adhesive [34,35]. Using EDTA instead of phosphoric acid can improve marginal integrity, as more than 90% of the dentin hardness remains [33]. This effect may be important in

Table 3. Frequency of microleakage scores for four adhesives experimental groups.

Adhesive	Microleakage Scores					Total
	0	1	2	3	4	
SBMP	18	2	0	0	0	20
SB	19	1	0	0	0	20
CSEB	19	1	0	0	0	20
PLP	16	3	1	0	0	20

SBMP: Adper Scotchbond Multi- purpose, SB: Adper Single Bond, CSEB: Clearfil SE Bond, PLP: Adper Prompt L-Pop

bonding durability as well. However, long-term in vitro and in vivo studies are still required to confirm these findings.

CONCLUSION

EDTA conditioning is significantly effective in decreasing dentinal microleakage when using the two self-etch adhesives of PLP and CSEB. Whereas, microleakage in etch and rinse adhesives (SBMP and SB) using EDTA conditioning instead of phosphoric acid had no effect on the results. The sealing ability of the two etch and resin adhesives was acceptable using either phosphoric acid or EDTA.

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