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The Effect of Oxalate Desensitizers on the Microleakage of Resin Composite Restorations Bonded by Etch and Rinse Adhesive Systems

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Clinical Relevance

The effect of oxalate desensitizer application after acid etching on the dentinal marginal sealing ability of resin composite restorations may be adhesive-specific.

SUMMARY

This *in vitro* study evaluated the effect of an oxalate desensitizer (OX) on the marginal microleakage of resin composite restorations bonded by two three-step and two two-step etch and rinse adhesives. Class V cavities were prepared on the buccal surfaces of 126 extracted premolars at the cementoenamel junction and randomly divided into nine groups of 14 each. In the control groups (1-4), four adhesives were applied, respectively, including Adper Scotchbond Multi-Purpose (SBMP), Optibond FL (OBFL), One-Step Plus (OS) and Excite (EX). In the experimental groups (5-8), the same adhesives, in combination with OX (BisBlock), were applied. And, in one group, OX was applied without any adhesive, as the negative control group (9). All the groups were restored with a resin composite.

After 24 hours of storage in distilled water and thermocycling, the samples were placed in 1% methylene blue dye solution. The dye penetration was evaluated using a stereomicroscope. The data were analyzed using non-parametric

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tests. The OX application, in combination with OBFL and EX, resulted in significantly increasing microleakage at the gingival margins (*p*<0.05), while it had no effect on OS and SBMP (*p*>0.05). At the occlusal margins, no significant difference in microleakage was observed after OX application for each of four adhesives (*p*>0.05).

INTRODUCTION

Dentin is living tissue with a tubular structure filled with cell processes that contain dentinal fluid. The fluid is under a slight and constant positive pressure.[1-2] The removal of tooth structure by cavity preparation results in increasing permeability of the remaining dentin.[1] Reproducing the lost seal of dentin is one of the most important goals of restorative dentistry. Achieving a perfect seal is still problematic with commercially available simplified etch and rinse adhesives. As the smear layer and smear plugs are formed during cavity preparation, dentin permeability drops by 86%. Acid etching by removing the smear layer facilitates fluid flow onto the exposed dentin surface.[1-5]

On the other hand, these adhesives act as permeable membranes due to the incorporation of solvated hydrophilic monomers for improving compatibility with intrinsic hydration of the dentin.[6-9] Air drying the adhesive and solvated ionic co-monomer mixture of the adhesive induce outward evaporative and osmotic water transudation, respectively, before polymerization of the adhesive.[10-11] This water movement may result in the entrapment of water blisters along the adhesive interface, creating a rapid fluid flow across the tubules during chewing, which causes postoperative sensitivity.[8,10-12]

Also, postoperative hypersensitivity rises as the extent of microleakage or marginal permeability to fluid, bacteria and their enzymes increases. This movement within the microgap formed at the dentin/restoration interface is the result of adhesive failure of the interface.[1,12-13] Considering the adhesive permeability, formation of the marginal microgap could facilitate the exogenous fluid penetration into dentinal tubules, thus, aggravating the negative effects of the two phenomena.

In an attempt to decrease dentin and adhesive permeability, the adjunctive use of a potassium oxalate desensitizer on acid-etched dentin for occluding tubules prior to application of the adhesive has been suggested.[14-16] Simultaneous formation of hybridized resin tags created a secondary seal during this bonding procedure[16] and completely blocked the permeability of dentin.[14] The beneficial effects of oxalate solutions in reducing fluid flow across dentinal tubules alone[14,17-22] on acid-etched dentin or with polymerized simplified adhesives with low acidity and compatible with oxalate have been shown.[14,16,22]

However, the penetrability of dentinal tubules in adhesive-bonded restorations depends not only on the sealing ability of the adhesive, but also on bonding of the restorative material to the adhesive, as shrinkage of resin tags from the tubular walls during composite polymerization may result in incomplete dentinal sealing.[14-15]

The marginal penetrability of composite restorations bonded by various adhesive systems may have a different influence as result of this bonding procedure. Thus, the null hypothesis tested was that the application of oxalate solution on acid-etched dentin would have no effect on the microleakage of resin composite restorations bonded by two two-step and two three-step etch and rinse adhesive systems.

METHODS AND MATERIALS

In this *in vitro* study, 126 extracted human premolars without caries, cracks or previous restorations, were selected and cleaned of calculus and other debris. The teeth were stored in a 0.1% thymol solution for two weeks.

Standard Class V cavities (3 mm wide, 3 mm high, 1.5 mm deep) with the gingival margin 1 mm below the cementoenamel junction were prepared using straight diamond burs (#878/d2, Teeskavan, Iran) with a high-speed handpiece on the facial surface of each tooth. The occlusal margins were located in enamel and the gingival margins were located in dentin. Each bur was discarded following the preparation of each group.

The prepared teeth were randomly divided into nine equal groups of 14 teeth each. In the first four groups, which were the control groups, four different adhesives were used. In the remaining four experimental groups, the same adhesives, with a dentin desensitizer (BisBlock, BISCO, Inc, Schaumburg, IL, USA), were used. In the negative control group (9), BisBlock was applied similarly, without any adhesive. All the materials were applied according to the manufacturers' instructions (Table 1).

In the four control groups (1-4), each prepared tooth was etched with 32% phosphoric acid gel (Uni-Etch, BISCO, Inc) for 15 seconds, rinsed for 20 seconds, then gently blown to remove excess water, while care was taken to maintain a moist surface. Then, two three-step total etch (Adper Scotchbond Multi-Purpose [SBMP], 3M ESPE, St Paul, MN, USA; Optibond FL [OBFL], Kerr Corporation, Orange, CA, USA) and two two-step total etch adhesives (One-Step Plus [OS], BISCO, Inc, Schaumburg, IL, USA) and Excite [EX], Ivoclar Vivadent, Schaan, Liechtenstein) were applied

Table 1: *Materials Used in This Study*

Adhesive	Composition/Batch #	Application Mode
Adper Scotchbond Multi-Purpose (3M ESPE, St Paul, MN, USA)	Primer: HEMA, polyalkenoic acid copolymer, water/6BE Adhesive: bis-GMA, HEMA, CQ, amine/6PM	Primer applied, dried with gentle air stream, adhesive applied, light-cured for 10 seconds
Optibond FL (Kerr, Orange, CA, USA)	Primer: HEMA, GPDM, PAMM, CQ, ethanol, water/3093079 Adhesive: TEGDMA, UDMA, bis-GMA, HEMA, GPDM, filler, CQ/3096500	Primer applied with light brushing motion for 15 seconds, air-dried for five seconds, adhesive applied with light brushing motion for 15 seconds, air-thinned for three seconds, light-cured for 20 seconds
One-Step Plus (BISCO, Inc, Schaumburg, IL, USA)	Bis-GMA, BPDM, HEMA, dental glass, acetone/0800004236	Adhesive applied two coats, agitated slightly for 10-15 seconds, gently air dried, light-cured for 10 seconds
Excite (Ivoclar Vivadent, Schaan, Liechtenstein)	Bis-GMA, HEMA, GDMA, phosphonic acid acrylate, silica, ethanol, catalysts, stabilizers/k29948	Adhesive applied, agitated slightly for at least 10 seconds, gently air thinned for one-to-three seconds, light cure 10 seconds
BisBlock (BISCO, Inc, Schaumburg, IL, USA)	Oxalic Acid <%5/0800011529	Etched for 15 seconds, rinsed and gently air-dried for two-to-three seconds, BisBlock applied and dwelled for 30 seconds, rinsed, Re-etched enamel margins for 15 seconds, rinsed, adhesive applied

Bis-GMA, bisphenyl-glycidyl-methacrylate; GDMA glycerol dimethacrylate; GPDM glycerol phosphate dimethacrylate HEMA, 2-hydroxyethyl methacrylate; BPDM, biphenyl dimethacrylate; UDMA, urethane dimethacrylate; PAMM phthalic acid monoethyl methacrylate and CQ, camphoroquinone.

Table 2: *pH Values and F Concentrations of the Adhesives Used in This Study*

Adhesive	pH Value	F Concentration (ppm)
Adper Scotchbond Multi-Purpose	Primer = 3.31	3.10
Optibond FL	Primer = 1.78	3400.93
One-Step Plus	4.61	806.82
Excite	2.25	60.55

in groups 1-4, respectively. The method of application for the adhesives/oxalate is provided in Table 1.

In four experimental groups (5-8), each prepared tooth was etched with 32% phosphoric acid gel for 15 seconds, rinsed for 20 seconds, then gently blown to remove excess water. The dentin desensitizer was applied for 30 seconds and rinsed for 60 seconds. The enamel margins were then re-etched for 15 seconds,[15] rinsed thoroughly and blot-dried. The same four adhesives were applied according to the manufacturers' instructions.

pH and F Concentration Measurements of Adhesives

The pH and F concentration measurements of the adhesives were determined in order to evaluate the effects of these factors on sealing ability of the adhesives in combination with oxalate desensitizer. The pH value of four of the adhesives was measured using a digital pH meter (WTW, 523, Wissenschaftlich-Technische Werkstätten GmbH, Weilheim, Germany) at room temperature in a dark room using a special red light. The measurements were performed on approximately 10 drops of each adhesive. Water-free adhesives that dissolved in the polar solvents did not usually dissociate into ionic species that are necessary for pH measurement. Thus, a solution of each adhesive in 70% ethanol and 30% distilled water was prepared in a clean glass vial. The values were read after 15 seconds, when the pH reading was stable. Also, the fluoride concentrations were determined at room temperature by an ion-selective electrode (Jenway, Bibby Scientific Limited, Staffordshire, UK). The pH values and F-concentrations in ppm are presented in Table 2.

All the cavities were restored with a micro-hybrid resin composite, Z250 (3M ESPE), in three increments and each increment was polymerized for 40 seconds using a conventional light-curing unit (Coltolux 75, Coltène/Whaldent AG, Alstätten, Switzerland, 500mW/cm^2). The restorations were finished and polished with Opti-Disk (Kerr Corporation).

The teeth were stored in distilled water at room temperature for 24 hours before being subjected to 500 thermal cycles between 5°C and 55°C water baths with a 30-second dwell time and a 15-second transfer time. The root apices were sealed with utility wax, and all the surfaces, except for the restorations and 1 mm from the margins, were coated with two layers of nail varnish. The teeth were immersed in a 0.5% methylene blue dye solution for 24 hours. They were then rinsed in running water, blot-dried and sectioned longitudinally through the center of the restorations from the facial to lingual surface with a water-cooled diamond wheel saw (Leitz 1600, Wetzlar, Germany).

The sections were blindly assessed for dye penetration by two independent evaluators using a stereomi-

Table 3: *Microleakage Scores Obtained from Control Groups (Adhesives Alone)*

Group	Adhesive System	Occlusal Margins				Gingival Margins			
		0	1	2	3	0	1	2	3
1	Adper Scotchbond Multi-Purpose	12	2	0	0	11	2	1	0
2	Optibond FL	13	1	0	0	11	3	0	0
3	One-Step Plus	13	1	0	0	6	7	1	0
4	Excite	9	5	0	0	6	5	3	0

Table 4: *Microleakage Scores Obtained from Experimental Groups (Adhesives + Oxalate Desensitizer)*

Group	Adhesive System	Occlusal Margins				Gingival Margins			
		0	1	2	3	0	1	2	3
5	Adper Scotchbond Multi-Purpose	10	3	1	0	11	1	2	0
6	Optibond FL	13	1	0	0	0	7	7	0
7	One-Step Plus	13	1	0	0	7	7	0	0
8	Excite	11	3	0	0	2	3	8	1
9	No adhesive	0	0	1	13	0	0	0	14

Table 5: *Mann-Whitney U-test Results of the Comparison of the Effect of Oxalate at the Occlusal and Gingival Margins (n=14)*

Occlusal Margin			
Adhesive System	**Mean Rank Control**	**Mean Rank Experimental**	***p*-Value**
Adper Scotchbond Multi-Purpose	13.43	15.57	0.51
Optibond FL	14.50	14.50	1.00
One-Step Plus	14.50	14.50	1.00
Excite	15.50	13.50	0.54
Gingival Margin			
Adper Scotchbond Multi-Purpose	14.39	14.61	0.94
Optibond FL	8.25	20.75	0.00*
One-Step Plus	15.25	13.75	0.63
Excite	11.11	17.89	0.02*

$p<0.05$ = significant difference

croscope (Carl Ziess Inc, Oberkochen, Germany) at 20x magnification. Dye penetration at the composite/tooth interface was scored for both the occlusal and gingival margins on a non-parametric scale from 0 to 3: 0 = no dye penetration; 1 = dye penetration of less than half of the cavity depth; 2 = dye penetration more than half of the cavity depth; 3 = dye penetration spreading along the axial wall.

A statistical analysis was performed using the Kruskal-Wallis and Mann-Whitney U-tests. A comparison of the occlusal and gingival margins of the groups was performed using the Wilcoxon Signed Rank test ($\alpha=0.05$).

RESULTS

Dye penetration scores for the occlusal and gingival walls are presented in Tables 3 and 4. In order to assess the effect of oxalate desensitizers (BisBlock) on microleakage of the four different adhesives, the Mann-Whitney test was performed between the control and experimental groups of each adhesive for both the occlusal and gingival margins. A significantly higher dye penetration at the gingival margins was observed in the experimental groups for which OBFL ($p<0.001$) and EX ($p<0.05$) was applied, but there was no significant difference for OS and SBMP ($p>0.05$). Moreover, there was no significant difference between the control and experimental groups at the occlusal margins ($p>0.05$) (Table 5).

Furthermore, the Kruskal-Wallis test was used for comparison among the four control groups, revealing no significant difference at the occlusal ($p=0.133$) and gingival margins ($p=0.056$). In addition, the Kruskal-Wallis test indicated a significant difference among the four experimental groups at the gingival margins ($p<0.001$) but not at the occlusal margins ($p>0.05$). Pairwise multiple comparisons of the four experimental groups at the gingival margins was performed using the Mann-Whitney U-test, which showed a significant difference between groups 5 and 6, 8 and 5, 7 and 6 and 7 and 8 ($p<0.05$).

Comparing the occlusal and gingival scores for each group showed a significant difference (more gingival than occlusal leakage) only for OS among the control groups and for OS, EX and OBFL among the experimental groups ($p<0.05$) by the Wilcoxon Signed Rank test.

DISCUSSION

Despite some reports of a decrease in dentin permeability following application of an oxalate solution by means of quantitative measurement of the fluid flow across the polymerized adhesive,[14,16,22] this fluid transport setup is not capable of detecting any microgap formed at the dentin or enamel margin/restoration interface.

The dye penetration method used in the current study is a gross assessment of the quality of the interface. Although microleakage increases as dentin permeability rises,[12] a reduction in dentin permeability does not necessarily result in a drop in microleakage, because dentinal sealing and coupling resin composite to adhesive-lined dentin involves two very different mechanisms. Any interference with hybrid layer formation and adaptation/bonding to the composite would adversely affect marginal microleakage. A significant role of hybridization on marginal leakage and bond strength has previously been reported.[23] On this base, in the current study, high scores of dye penetration were observed in the negative control group with oxalate treatment without any adhesive application.

According to the results of the current study, no significant difference was observed among the four control groups of adhesives at the occlusal margins. Three-step compared to two-step etch and rinse adhesives showed a better, but not statistically significant, sealing ability at the gingival margins. The better performance of three-step etch and rinse adhesives has been observed in other studies.[24-25] This could be attributed to more complete hybridization of the demineralized dentin, with a separate primer and adhesive resin application when compared to a combination of two of these components in one bottle.[24-25]

Some studies indicated that the adhesive systems are not capable of bonding well to oxalate-treated dentin, because the dentin surface and tubular orifices were covered with calcium oxalate crystals.[15,26-27] This acid-resistant coverage could neutralize the acid etching and might interfere with formation of a sufficient hybrid layer,[28] contributing to gap formation between oxalate-treated dentin and the restoration.[27] To solve this problem, in the current study, the dentin surface was etched before oxalate application, as recommended by Tay and others.[14-15] The depletion of calcium ions from the surface dentin resulted in the formation of subsurface calcium oxalate crystals and tubular occlusion. Therefore, they should not compromise the formation of a typical hybrid layer on top of dentin surfaces and dentin bond strength.[14-15,27]

The results of the current study showed that the microleakage of dentinal margins bonded with OS and SBMP was not affected by oxalate application, while the other two adhesives, OBFL and EX, showed significantly higher microleakage with the application of oxalate. Thus, the null hypothesis was accepted in the cases of OS and SBMP and rejected in the cases of OBFL and EX. The difference might be attributed to a different pH value of these adhesives. This was probably due to the fact that the solubility of calcium oxalates is affected by pH, as the anion is the conjugate base of a weak acid.[22,29] The low pH of EX and the primer of OBFL dissolved the calcium oxalate crystals formed in the dentinal tubules. A TEM study showed the incompatibility of low pH adhesives with oxalate-treated dentin by forming spherical globules.[29] These surface globules may have interfered with the hybridization of demineralized dentin. This adverse effect could have decreased dentin bond strength.[29] Additionally, the precipitation of globules of CaF2-like material formed following interaction with the free fluoride ions present in the adhesives with calcium and phosphate of the dentinal surface was reported.[29] Furthermore, the tubular blocking capacity of oxalate treatment on acid-etched dentin deteriorated following application of the adhesives with high acidity.[22] It was concluded that incompatibility between the etch and rinse adhesives and oxalate desensitizer may be associated with the acidity and fluoride content in these adhesives.[22,29]

However, in the current study, OS with weaker acidity containing fluoride (806 ppm) showed no incompatibility with oxalate with respect to microleakage results. This finding was in accordance with the manufacturer's instructions that recommended the application of BisBlock in combination with One-Step or One-Step Plus. OBFL and OS had fluoride, but SBMP and EX had no fluoride or minimum amounts of fluoride, respectively. The gingival marginal sealing of these two adhesives (OBFL and EX) with high acidity might be compromised following the oxalate application. Therefore, it seems that the pH value compared to the fluoride content of the adhesives might be more important in determining the effect of oxalate on marginal sealing of the adhesives.

The findings of the current study indicate that the oxalate application had no effect on enamel microleakage. After application of the oxalate desensitizer, the enamel margins re-etched for 15 seconds, following the manufacturer's instructions. Tay and others[15] proposed that the formation of calcium oxalate crystals on etched enamel can interfere with resin-enamel adhesion. These surface crystals can be removed by a second, brief etching that dissolves the apatite crystals of enamel beneath the calcium oxalate crystals that then fall off, resulting in better resin penetration into etched enamel. This explanation could be true in the current study, as oxalate treatment had no adverse effect on enamel microleakage. Additionally, on the basis of Tay and others' observations,[15] which reported decreased water tree formation and nanoleakage in the adhesive layer following oxalate application on etched dentin, it may be concluded that this bonding technique has a beneficial effect on the bonding durability of etch and rinse adhesives with low acidity.

Moreover, after acid etching, since the tubular fluid flow can interfere with resin infiltration under *in vivo* conditions, the blocking potential of oxalate may facilitate better formation of a homogenous hybrid layer in the whole depth of demineralized dentin. This function would inhibit the hydrolysis degradation of the bonding interface. Further *in vitro* and clinical studies should be performed to confirm the findings of the current study and the possible beneficial effects of oxalate or other blocking agents on the long-term sealing ability of etch and rinse adhesives in resin composite restorations.

CONCLUSIONS

Within the limits of the current study, it may be concluded that the effective adhesion of resin composite to oxalate desensitizer-treated etched dentin may be adhesive-specific. This depended on the pH value of the primer/adhesive.

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