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Effects of Potassium Oxalate on Knoop Hardness of Etch-and-Rinse Adhesives

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Clinical Relevance

Application of potassium oxalate to acid-etched dentin may interfere with the properties of adhesives that are subsequently applied to dentin.

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SUMMARY

The objective of this study was to determine whether the hardness of etch-and-rinse adhesives may be affected by the pretreatment of acid-etched dentin with potassium oxalate desensitizer. Unerupted human third molars were cut into crown segments by removing the occlusal enamel and roots. The pulp chamber of these crown segments was connected to a syringe barrel filled with phosphate-buffered saline so that the moisture of dentin was maintained during the bonding procedures. Three etch-and-rinse adhesives—two two-step systems (Adper Single Bond 2 [SB], One-Step [OS]) and one three-step system (Adper Scotchbond Multi-Purpose [MP])—were applied to acid-etched dentin that had been treated (experimental groups) or not (control groups) with potassium oxalate (BisBlock). The Knoop hardness (KHN) of adhesives was taken at different sites of the outer surface of the adhesive-bonded dentin. The KHN of the three tested adhesives applied to acid-etched dentin

treated with potassium oxalate was significantly lower than that exhibited by the respective controls (not treated with oxalate; $p<0.05$). Regardless of the adhesive, the treatment with potassium oxalate reduced the adhesives' KHN ($p<0.05$), with the OS system exhibiting the lowest KHN compared with the MP and SB systems.

INTRODUCTION

In contemporary dental adhesives, high concentrations of relatively hydrophilic methacrylate monomers (ie, HEMA, BPDM, PENTA) are blended with relatively hydrophobic monomers (ie, Bis-GMA, UDMA) and solvents (ie, ethanol, acetone) to enhance bonding to intrinsically water-wet dentin. The presence of hydrophilic monomers and volatile solvents improves the wetting performance of dental adhesives when applied to acid-etched dentin that is intentionally saturated with water. Volatile solvents facilitate the displacement of water from the acid-etched dentin matrix,[1] ensuring better penetration of resin monomers into the micro- and nanoporosities left between the collagen fibrils,[2] which in turn improve the microretention between the restorative material and tooth substrates.[3,4]

Nevertheless, while the water-wet bonding technique may facilitate the infiltration of hydrophilic resin monomers into demineralized dentin, the presence of residual solvent/water before the photoactivation of adhesives has been thought to be responsible for producing areas of incomplete monomer conversion,[5,6] which correspond to the porosities that are revealed by silver deposition in nanoleakage studies.[7] These porous interfaces/polymers are prone to permeation by water[7] and, to a certain extent, by small solutes.[8,9] Polymers containing a mixture of hydrophilic and/or ionic domains become swollen due to water sorption,[10] allowing fluid transport in and out across the cross-linked polymer network.[6,7] The hydrophilic adhesives, therefore, behave as permeable membranes[11] that cannot achieve the requirement for perfect sealing of dentin.[12,13]

During bonding procedures, most of the water that is trapped within the adhesive layer or accumulated on its surface originates from the underlying hydrated dentin.[14] Studies have suggested that even the solvent evaporation procedures (ie, air blast) or the hypertonicity of solvated adhesive may be responsible for creating an osmotic gradient that induces outward water movement from the underlying hydrated dentin into the adhesive.[6,12]

The application of oxalate desensitizers to dentin prior to the bonding procedures has been considered as an alternative to block the transit of fluid across the resin-bonded interface and adhesive layer.[15,16] Oxalate solutions or gels react with ionized calcium in dentin to form insoluble crystals of calcium oxalate.[17,18] Because of their ability to occlude the dentinal tubules, oxalate-based desensitizers are considered potent agents in the treatment of dentin sensitivity.[18] The calcium oxalate crystals formed into the dentinal tubules were shown to reduce the fluid conductance of dentin,[19,20] reducing the pain sensation.[21] As a side effect, the obstruction of dentinal tubules with oxalate crystals may help clinicians gain better control of the moisture that is present on the surface of acid-etched dentin during bonding procedures. In theory, the presence of oxalate crystals reduces the free fluid conductance of dentin, creating environmental conditions for adhesives to polymerize more suitably, without any or limited presence of water.

When applied to acid-etched dentin, the calcium oxalate crystals are formed beyond the acid-etched surface, and in theory, they should not interfere with the subsequent bonding procedures.[15] However, the data on the effect of oxalate desensitizers on the bonding performance of adhesives are scant and unclear. While the oxalates can constitute an alternative to prolong the longevity of resin-dentin bonds, we have recently observed that the application of potassium oxalate to the acid-etched dentin affected the baseline bond strength of two- and three-step etch-and-rinse adhesive systems.[22] We speculated that even though the oxalate solution has been thoroughly rinsed before the application of adhesives to dentin, residual oxalic acid or any other of its by-products (ie, thickening agent) could have remained on the dentin surface, interfering with the proper polymerization of the adhesives and, consequently, compromising their bonding performance.

The aim of this study was to determine whether the polymerization of adhesives can be affected by the treatment of acid-etched dentin with a potassium oxalate desensitizer. Since hardness measurements have been accepted as a good predictor of the degree of polymerization of dental resins,[23-25] we tested the hardness of three etch-and-rinse adhesives after their application to acid-etched dentin that was treated or not with potassium oxalate gel. The hypothesis of this study was that the application of potassium oxalate to acid-etched dentin interferes with the hardness of etch-and-rinse dental adhesives.

MATERIALS AND METHODS

Tooth Preparation

Thirty noncarious human third molars extracted for orthodontic reasons were collected after patients' informed consent had been obtained under a protocol reviewed and approved by the Ethics Committee of the University of Campinas. Teeth were stored in saline containing 0.02% sodium azide and used within no longer than six months after extraction.

Crown segments were prepared by removing the occlusal enamel and root of these teeth using a slow-speed diamond saw under water cooling (Labcut 1010, Extec Corp, Enfield, CT, USA). The pulp tissue was carefully removed with a pair of small forceps. Care was taken to avoid touching the pulp chamber walls in order not to crush the predentine toward the dentinal tubules, which could alter the final permeability of dentin (D. Pashley, personal communication). The dentin surface was further abraded with 600-grit silicon carbide paper, until a remaining dentin thickness of 1.5 ± 0.2 mm was achieved from the ground surface to the highest pulp horn. The resulting crown segments were glued to Plexiglass slabs (1.8×1.8×0.7 cm) using viscous cyanoacrylate (Zapit, Dental Ventures of American, Corona, CA, USA), which also covered the entire peripheral cementum. Each Plexiglass slab was penetrated by a short length of 18-gauge stainless-steel tubing, permitting the pulp chamber to be filled with phosphate-buffered saline (pH 7) supplemented with 0.02% sodium azide to keep dentin hydrated during bonding procedures.

Bonding Procedure

The exposed dentin surfaces were polished with a 600-grit SiC paper during 30 seconds and then were acid-etched with 35% phosphoric acid gel (3M ESPE, St Paul, MN, USA) for 15 seconds and rinsed thoroughly with distilled water for 30 seconds. The specimens were divided into two groups: 1) control: the bonding procedures were performed as recommended by manufacturers; 2) experimental: the bonding procedures were performed after the treatment of acid-etched dentin with potassium oxalate. For the experimental group, the potassium oxalate gel, BisBlock (BISCO Inc, Schaumburg, IL, USA), was applied on the surface for 30 seconds and rinsed off with distilled water for 60 seconds. The enamel margins were re-etched for 15 seconds and rinsed thoroughly with water as recommended by the manufacturer (BisBlock, Technical Profile).

Three etch-and-rinse adhesive systems were selected for this study: the two-step systems Adper Single Bond ([SB] 3M ESPE) and One-Step ([OS] BISCO) and the three-step system Adper Scotchbond Multi-Purpose ([MP] 3M ESPE). In principle, these adhesives were applied to the acid-etched dentin surfaces of control and experimental groups while dentin was visibly moist with water as recommended by manufacturers. Nevertheless, this condition created such a soft bonded surface that the hardness could not be recorded even after storing the specimens in dry conditions for 48 hours after the adhesives' polymerization. Thus, adhesives were applied vigorously to the acid-etched dentin of control and experimental groups after the dentin surface was air dried for 30 seconds with oil-free compressed air.[26] This dry bonding technique ensured that the only possible source of water that could potentially interfere with adhesives hardness was that present in the pulp chamber.

Surfaces were checked to ensure complete covering with adhesives, and a glass coverslip was placed on the top of the adhesive to create a flat surface, avoiding excessive contact with the atmospheric oxygen during light activation.[27] The adhesives were light cured for 20 seconds using a halogen-tungsten unit (Degulux, DEGUSSA HÜLS, Frankfurt, Germany) operated at 500 mW/cm^2. Once polymerized, the specimens were stored in dry conditions at 37°C until the hardness measurement was taken.

Hardness Measurement

Twenty-four hours after the bonding procedures were completed, the specimens' hardness was determined with a Shimadzu HMV-2 hardness tester (Shimadzu Corporation, Kyoto, Japan), equipped with a Knoop indenter at 25 g of load and 6 seconds of dwell time. Six indentations were performed on the top of the adhesive-bonded dentin surfaces, over the sites that correspond to the pulp horns, where the pulp chamber wetness could impose a challenge to dental adhesive systems. At least three indentations per crown segment were performed in bonded enamel, which represents a condition in which the adhesives hardness should not be affected by the surface wetness (negative control).

The Knoop hardness was determined by examining the surface with an optical microscope (40×) and expressed as the Knoop hardness number (KHN).

Statistical Analysis

The KHN determined for control and experimental groups was analyzed by two-way analysis of variance tests, having as main factors the adhesives (ie, SB,

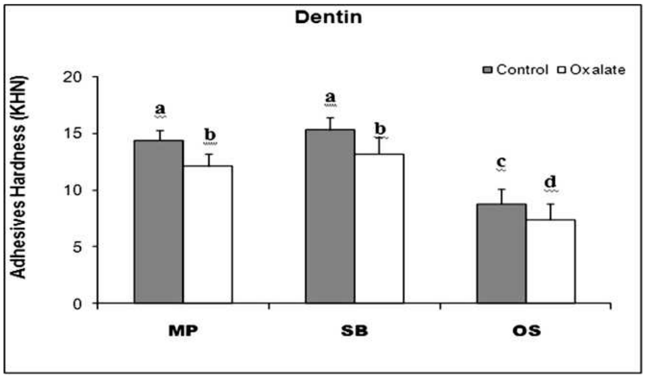

Figure 1. *The mean KHN and standard deviation (in KHN) of adhesives applied to dentin (n=60).* ***MP*** *= Adper Scotchbond Multi-purpose system;* ***SB*** *= Adper Single Bond 2 system;* ***OS*** *= One-step system. The height of the bars is KHN mean; half-brackets indicate plus one standard deviation. Groups identified with the same case letters did not differ statistically (p>0.05).*

OS, and MP) and the substrate treatments (ie, control vs oxalate treated), with the data derived from dentin being analyzed separately from those derived from enamel. Post hoc multiple comparisons were performed using Tukey's tests. Statistical significance was preset at α=0.05.

RESULTS

The mean KHN of the adhesive-bonded dentin and adhesive-bonded enamel are seen Figures 1 and 2, respectively. The treatment of dentin or enamel with potassium oxalate was shown to affect significantly the KHN of the three adhesives when compared with respective controls ($p<0.05$; Figures 1 and 2).

For both tested substrates (ie, dentin and enamel), the OS system exhibited the lowest KHN when compared with the SB and MP systems ($p<0.05$), regardless of the surface treatment (control or oxalate treated). The mean KHN for OS adhesive applied to dentin varied between 8.6 (control) and 7.6 (oxalate-treated dentin). For the MP adhesive system, the mean KHN tested on dentin ranged from 14.2 (control) to 12.4 (oxalate-treated dentin). These values did not differ significantly ($p>0.05$) from those observed for the SB adhesive (for comparisons between correspondent groups, ie, MP control vs SB control; MP experimental vs SB experimental), for which the mean KHN varied between 15.0 (control) and 13.2 (oxalate-treated dentin).

For enamel substrate, the differences in the KHN were significant only for the factor substrate treatment ($p<0.05$), while statistical significance for the main factor adhesives and the interaction between

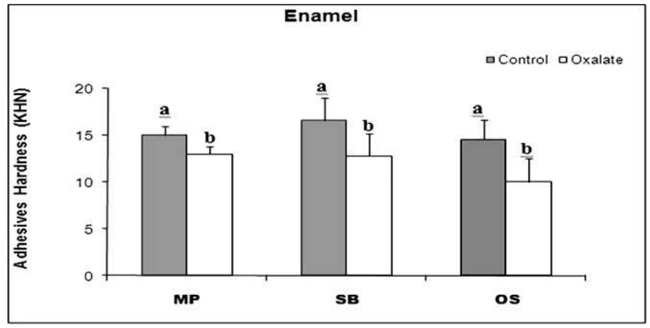

Figure 2. *The mean KHN and standard deviation (in KHN) of adhesives applied to enamel substrate (n=60).* ***MP*** *= Adper Scotchbond Multi-purpose system;* ***SB*** *= Adper Single Bond 2 system;* ***OS*** *= One-step system. The height of the bars is KHN mean; half-brackets indicate plus one standard deviation. Groups identified with the same case letters did not differ statistically (p>0.05).*

the two main factors (adhesives × substrate treatment) was not observed ($p>0.05$; Figure 2).

DISCUSSION

All tested etch-and-rinse adhesives (OS, SB, and MP) had their Knoop hardness significantly compromised, which led us to accept the anticipated hypothesis.

Several studies have found a positive correlation between hardness and degree of conversion.[23-25] While this may not be a full consensus,[28,29] as hardness may reflect the degree of cross-linking between polymer chains,[30] it has been conveniently used to compare the extent of polymerization exhibited by different dental resins under numerous testing conditions (ie, varying the light-curing time, the amount of energy delivered during photoactivation, the type of light source, the distance between the light-curing unit and the sample, etc).[23,29,30] The density and distribution of cross-links between polymer chains[31,32] play an important role in the final cohesion of polymers.[33] Polymer networks with homogeneous packing density (ie, restricted free volume and high level of polymer's chain cross-linking) tend to exhibit higher mechanical properties.[34] According to Rueggeberg and Craig,[27] hardness is sensitive in detecting small changes in polymer cross-linking so that reductions in KHN may suggest the existence of a less densely cross-linked polymer.[30] If this assumption was extrapolated to explain the present results, we could presume that the application of potassium oxalate interfered with adhesives' polymerization and mechanical properties by favoring the formation of poorly cross-linked adhesives.

Previous studies that evaluated the effectiveness of oxalate desensitizers to reduce the permeability of the resin-bonded dentin also indicated that these oxalates may have a varying effect on the bond strength of resin-bonded dentin,[15,16,35] depending on with which adhesive they were combined.[35] Yiu and coworkers[35] showed that fluoride ions released from fluoride-containing adhesives with relatively low pH may potentially interact with calcium-oxalate crystals to form spherical loosely bound calcium fluoride crystals that could interfere with the resin monomers' infiltration/polymerization, thereby compromising the hybrid layer formation. According to that study,[35] the pH of the adhesives SB, OS, and MP would not be low enough to cause the solubility of calcium oxalates. For this reason, we decided to test adhesives with low fluoride content and relatively high pH (3.3 for MP, 3.6 for SB, and for 4.5 for OS; 3M ESPE, Technical Product Profile, respectively). We believe, therefore, that the low values of hardness observed for adhesives applied on oxalate-treated dentin were not related to their pH and/or fluoride content.

In principle, it might be speculated that despite the oxalate solution having been thoroughly rinsed before the adhesives' application, residual oxalic acid may have remained to react with calcium, causing crystal precipitation on the dentin surface, which could compromise the adhesives' hardness. However, since the dentin surface was probably deprived of calcium phosphate due to its previous demineralization with phosphoric acid,[36] we are induced to consider that the decrease in the adhesives' hardness was probably not caused by the crystals' precipitation. We also do not believe that instantaneous dissolution of calcium oxalate crystals present in the dentinal tubules may potentially provide free calcium for reacting with residual oxalic acid to cause precipitation of calcium oxalate on the acid-etched dentin surface. Indeed, it seems more likely that other by-products of BisBlock had not been completely removed from the surface after the rinsing, thereby interfering with the proper polymerization of adhesives. This speculation may also explain the decrease in KHN observed for enamel specimens that were acid etched with phosphoric acid after potassium oxalate application (Figure 2) as recommended by the manufacturer.

An unexpected finding of this study was that the three-step adhesive (ie, MP) did not show a higher KHN value when compared with SB, a two-step adhesive. Supposedly, the inclusion of solvent and hydrophilic components in two-step etch-and-rinse adhesives should make these materials softer than those nonsolvated systems.[5,37] This is because residual solvent, which cannot be completely eliminated from the adhesive before light curing,[38] may plasticize the polymer network and reduce its mechanical strength. Thus, the adhesive MP, which requires a separate application of a relatively hydrophobic, nonsolvated resin over the acid-etched primed dentin, was supposed to exhibit the highest KHN values. However, by assuming that a BisBlock by-product has remained in tested specimens, one may speculate that these by-products could have also contaminated the bonding agent of MP adhesive as this was partially mixed with the unpolymerized primer solution that was set on the oxalate-treated dentin.

Although the results of the present study showed that potassium oxalate applied on acid-etched dentin affected the baseline hardness of the tested adhesives, a parallel study that we recently concluded showed that potassium oxalate played an important role in decelerating the long-term degradation of the resin-dentin bonds created using the same adhesives.[22] Most likely, the presence of calcium oxalate crystals partially blocking the fluid transudation across the dentinal tubules may have prevented the adhesives to prematurely absorb water, decelerating their mechanical disruption by the plasticizing effects of water.

The use of potassium oxalate on acid-etched dentin could also be useful in the ethanol wet-bonding technique.[39] The objective of this technique is to use more hydrophobic resin blends for dentin bonding to reduce adhesive permeability and dentin-bond degradation. As hydrophobic monomers do not bond well to the water-saturated dentin, ethanol is used to replace rinse water from acid-etched matrices.[40,41] However, as the ethanol wet-bonding protocol was found to be very technique sensitive in the presence of water, the dentinal tubules could be patently blocked with calcium oxalate crystals to prevent fluid contamination during the application of hydrophobic adhesives.[42]

Recently, studies have shown that infiltration of relatively hydrophobic monomers in ethanol-filled interfibrillar spaces produced similar or higher initial bond strengths to dentin in comparison to those achieved using the wet-bonding technique.[40,41] If the concept of ethanol wet bonding with hydrophobic monomers was proven to be effective in prolonging the durability of resin-dentin bonds, the application of oxalates to acid-etched dentin saturated with ethanol might be a way to avoid the contamination of hydrophobic monomers with dentinal water, thereby favoring monomer conversion. However, before the clinical implementation of this approach, additional and conclusive studies are necessary to access the influence of potassium oxalate gels on the performance of hydrophobic and hydrophilic adhesives applied to ethanol-saturated dentin.

CONCLUSIONS

Although potassium oxalate has previously shown to produce resin-dentin bonds that are more stable over time, it may adversely affect the baseline hardness of etch-and-rinse adhesives.

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