Scientific Article

Evaluation of a no-rinse enamel conditioning prior to sealant application: an in vitro study of comparison to traditional etching technique

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Abstract

Moisture contamination is a major factor in sealant application, often determining clinical success or failure. A new enamel conditioner using HNO_3 (2.5%) has been introduced that does not require a water rinse after etching. The aim of this study is to compare etching characteristics using sealant retention from shear bond strength tests for traditional etch conditioning using H_3PO_4 (37%) and the HNO_3 (2.5%) conditioner with and without a water rinse. We used 28 crown-intact extracted human teeth. We evaluated eight shear bond strength tests per group, on cylindrical sealant stubs (3.24 mm diameter x 3 mm height) for 12 groups (three etch conditions, two prophylactic methods, and two enamel surface type). The highest mean values of shear bond strength of 22.0 MPa was measured for H_3PO_4, and the lowest of 12.7 MPa for HNO_3 (2.5%) without water wash. No significant differences ($P < 0.05$) were found between water rinse and air blast post-treatment groups after HNO_3 conditioning. (Pediatr Dent 17:301–4, 1995)

The adhesion between sealant materials and tooth enamel depends on the mechanical interlock created by acid etch technique. Buonocore[1] developed this technique, which consisted of etching the enamel surface with orthophosphoric acid, and Cueto[2] presented the first report of a clinical trial using an occlusal sealing technique. Before applying the sealants, 37% H_3PO_4 etchant is recommended to improve sealant material penetration. Due to the acidity, the etchant should be washed away with copious amounts of water for 15 sec to create a debris-free, clean surface and clear, water-soluble reaction product[3] or remove excess acid, which can cause irritation to soft tissues in the oral cavity. High-speed vacuum often is recommended, accompanied with water rinse to remove remnant H_3PO_4. This procedure may contribute to moisture contamination and poor adhesion. Further, the efficiency of treatment in the pediatric dental clinic is affected, where chair time is particularly important while young patients are treated. Sensitivity arising from lack of adhesion and low adhesion strength[4] may lead to clinical failure.

A 2.5% HNO_3 solution has been applied as a dentin conditioner to help remove the smear layer.[5,6] The use of HNO_3 (2.5%) as a conditioning agent for enamel has been evaluated by Berry, who compared bond strength of composite resin to enamel treated with various regimes of HNO_3-NPG (N-phenylglycine) and H_3PO_4.[7] The shear bond strength for 37% H_3PO_4 as an enamel conditioning agent was comparable to that for 2.5% HNO_3 solution when used with a rubbing action during the application. In the case of traditional 37% H_3PO_4 etchant, shear bond strengths of resin restorative materials to enamel have been reported in the range of 16–20 MPa.[2,8] The adhesive strengths of enamel and polyurethane resin used as sealants are reported to be in the range of 6.9–10.3 MPa.[9]

The use of a HNO_3 solution without any water wash has been indicated as sufficient for applying a commercial sealant. The capacity of 2.5% HNO_3 enamel conditioning followed by air blast (no water rinse) to produce: 1) sufficient sealant material penetration on outer enamel and 2) sufficient bond strength estimate of the retention has not been evaluated yet.

The main objective of this study is to evaluate a new enamel conditioner that uses HNO_3 and eliminates the need for a wash-out step prior to sealant application. This study will attempt to achieve the above by comparing three types of enamel conditioning methods: 1) traditional etching with 37% H_3PO_4 followed by water rinse [H_3PO_4 (W)], 2) etching with 2.5% HNO_3 followed by water rinse [HNO_3 (W)], and 3) 2.5% HNO_3 followed by air blast [HNO_3 (A)] in terms of sealant retention characteristics.

Prior studies[10] have noted that shear bond strength of sealants on ground enamel surfaces is not dependent on the prophylaxis agent. Since occlusal enamel surfaces undergo prophylaxis cleaning (pumice or prophy paste) in the clinic prior to sealant placement, we decided to evaluate groups based on the prophylaxis method as well. It is not known whether the sealant retention would compare well for actual clinical situations, namely curved occlusal surfaces rather than flat ground enamel surfaces. Due to the difficulty associated with testing on occlusal surfaces, we decided that buccal or distal enamel surfaces could be used for shear bond strength evaluations. The shear bond strength on ground inner enamel for pumice and prophy paste prophylaxis were used as controls.

Methods and materials

We selected 28 extracted molar and premolar teeth with crowns intact for this in vitro study. Twenty-four teeth were washed with distilled water and mounted with self-cure acrylic resin to permit slicing along the long axis to obtain 48 tooth halves. These were divided into two groups of 24 each and cleaned with prophy paste (Zircate Prophy Paste™, LD Caulk Division/ Dentsply International Inc, Milford, DE) and pumice powder respectively.

Shear bond strength tests

The same tooth specimens were used for shear bond strength evaluations on nonocclusal outer enamel surfaces and ground enamel surfaces. Shear bond strength was evaluated first for outer enamel surface. Subsequently, the tooth samples were ground (> 0.2 mm of surface depth removed) to provide a flat enamel surface for measuring shear bond strength on ground enamel.

We evaluated 24 tooth surfaces each for 1) prophy paste cleaned and 2) pumice slurry cleaned surface pretreatment condition. For each surface pretreatment condition, three etching conditions were used to lead to eight shear bond strength evaluations per etch condition. A 30-layer Scotch tape (Magic Tape 810™, 3M, St. Paul, MN) was punched using a hole puncher to yield a 3.24-mm x 3-mm-height mold. The tape was placed on the etched tooth such that a nearly flat portion of the outer enamel surface was aligned with the hole for sealant application. Sealant stubs were built in two incremental stages and light cured for a total period of 60 sec. After curing, the tooth with the sealant stub was oriented on an acrylic block such that the sealant cylinder was perpendicular to the acrylic surface with 1 mm clearance between the exposed tooth surface and the acrylic block surface. They were then stored for 24 hr at 37°C and ≈ 100% relative humidity. They were tested in shear using a knife edge plunger (1 mm flat tip) on the Instron test machine (Canton, MA), at a displacement rate of 0.5 mm/min. The load to failure and the location of failure were recorded. The distance of the plunger edge from the sealant-tooth interface was maintained at 0.5 mm.

The tooth specimens for the ground enamel surface were polished with #400 and #600 grit SiC emery paper to remove residual sealant (from the previous test) and expose subsurface enamel layer to obtain a flat enamel surface. The enamel pretreatment, sealant preparation, storage conditions, and shear bond testing procedure were the same as for the outer enamel test specimens. The data were evaluated for statistical significance using the group *t*-test and one-way ANOVA, Scheffe test at $P < 0.05$ level.

Results

Shear bond strength tests

The table lists mean values (N = 8) of shear bond strength in Megapascals (MPa), standard deviations (SD), range (minimum and maximum value), and statistical significance (one-way ANOVA, Scheffe test, $P < 0.05$) as a function of enamel surface type and prophylaxis method combinations, etchant type, and postetch cleaning method.

The lowest mean bond strength values are seen for the HNO_3(A) group while the highest values are noted for the H_3PO_4(W) group, for a given type of enamel surface and prophylaxis method (Table). For outer enamel surfaces (OE) the mean values of shear bond strength are highest for surfaces subjected to prophy

TABLE. MEAN SHEAR BOND STRENGTH FOR OUTER AND GROUND ENAMEL SAMPLE GROUPS

*Enamel Type Prophylaxis**	*Group*	*Enamel Treatment*†	N	*Mean (MPa)*	*SD*	*Range (MPa)*	*Significant Difference*‡
OE/PRP	1	H_3PO_4 (W)	8	17.34	3.87	9.17–21.19	NS
	2	HNO_3 (W)	8	15.45	3.10	11.89–21.14	
	3	HNO_3 (A)	8	14.32	2.53	11.40–17.99	
OE/PD	1	H_3PO_4 (W)	8	15.17	4.72	11.52–24.53	NS
	2	HNO_3 (W)	8	14.10	3.54	10.16–20.57	
	3	HNO_3 (A)	8	13.25	3.64	7.81–18.83	
GE/PRP	1	H_3PO_4 (W)	8	17.20	2.99	11.50–20.42	NS
	2	HNO_3 (W)	8	14.96	3.24	11.10–22.05	
	3	HNO_3 (A)	8	15.02	4.34	9.17–21.19	
GE/PD	1	H_3PO_4 (W)	8	22.03	2.69	17.15–25.28	1&2, 1&3
	2	HNO_3 (W)	8	17.06	2.61	13.53–20.20	2&3
	3	HNO_3 (A)	8	12.76	3.81	9.17–18.83	

* OE = Outer enamel; GE = Ground enamel; PRP = Zircate prophy paste; PD = Pumice slurry.

‡ Significant difference between groups; One-way ANOVA, Scheffe test ($P < 0.05$). NS = no significant difference between sample groups. $H3PO_4$ (37%) solution, HNO_3 (2.5%) solution.

† (W) = postetch water rinse for 15 sec followed by air drying; (A) = postetch air drying without water rinse.

paste pretreatment (PRP), whereas the highest values for the ground enamel surfaces (GE) were noted for pumiced surfaces (PD) (Table). The higher shear bond strength for GE surfaces compared with OE surfaces for H_3PO_4 etch condition concurs with similar results reported in a prior study.[11]

The lowest and highest values of bond strength were seen within in the GE/PD group for H_3PO_4(W) and HNO_3(A) conditions, respectively (Table, bottom). The differences in mean values for H_3PO_4(W) and HNO_3(A) conditions are highest for the GE surfaces, when pumice prophylaxis is performed (Table, bottom), and significant differences ($P < 0.05$) are noted only for this enamel type/prophylaxis combination. No statistically significant difference ($P < 0.05$; marked 'NS' in the table) was noted between etch groups for any of the other three enamel/prophylaxis combinations. No significant difference ($P < 0.05$) was seen between HNO_3(W) and HNO_3(A) groups except for the GE/PD, enamel/prophylaxis combination. Further, the bond strength values are roughly the same for GE or OE surfaces subjected to PRP prophylaxis.

Discussion

Shear bond strength

The table shows that the difference between the minimum and maximum values of measured shear bond strength (range) as a percentage of the mean value ranges from ≈ 37 to 87%. This is also reflected in the high values of SD in the table. Such a spread of data is not uncommon in bond strength testing[12] and in some cases, as for the adhesive bond strength testing of dentin bonding agents, the SD values are equal to or greater than that of the mean values.[13] In this study, the calculated mean from eight data values are centered about the midpoint of the range indicating that the values are distributed evenly within the range (Table). Only in the case of the GE/PD subgroup, the mean value differences between the three etch conditions are larger than the population SD, permitting significant differences at $P < 0.05$ level.

The extent of the range and hence the SD value not only depends on experiment and operator errors, but also on material and stress homogeneity of the region under test. In the case of brittle materials such as ceramics or regions containing porosity, the mean value no longer can be considered a true value for the material (i.e., only experimental and operator errors are present) but as a characteristic value for the given material under test.[12, 13] The results of this study indicate that the test method is effective in estimating the differences in etch condition, despite the large SD values in one enamel and prophylaxis combination (GE/PD; Table). We expect that a larger sample size will not produce large differences in the magnitude of the range or the mean value but will reduce the population SD value.

Sealant retention depends on the roughness of an etched surface, wetting ability, and sealant penetration (dependent on the sealant resin viscosity) into the enamel subsurface to fill all available pores or subsurface channels created by etching. Inefficient wetting (dependent on enamel surface energy and cleanliness and/or resin viscosity) and curing contraction associated with resin setting will lead to porosity and gaps at the interface. Insufficient cleaning, air entrapment, and/or extraneous particles associated either with residual reaction products or abrasive particles are bound to interfere with sealant penetration. This is particularly important when the chosen method does not include a water rinse and where water rinsing may not be effective in removing residual debris from recessed surfaces such as pits and fissures.[14] Minimum and maximum values depend on the factors mentioned above.

The effect of prophylaxis at constant enamel surface type can be seen in the significantly higher values noted for GE/PD surfaces compared with GE/PRP surfaces for H_3PO_4(W) condition. Such differences were not noted for OE surfaces. The lower values noted for GE/PRP surfaces may reflect the interference to either etching or sealant permeability from prophy paste prophylaxis. For GE surfaces the significantly higher values ($P < 0.05$) for H_3PO_4(W) group compared with either of the HNO_3 etched groups reflect differences in depth of enamel etching. The differences between the HNO_3(W) and HNO_3(A) groups may be attributed to interference to sealant wetting from the residual reaction products left on the surface for the HNO_3(A) group for pumice cleaned surfaces.

For outer enamel surfaces no statistically significant differences were noted between etch conditions within each of the groups, OE/PRP and OE/PD (Table). Therefore, the mean values of shear bond strength were compared irrespective of prophylaxis for the three etch conditions and were found not to be significantly different ($P < 0.05$) for OE surface. Further, mean values were not significantly different for comparisons within sample groups OE and GE at constant HNO_3 etch condition (W/PRP, W/PD, A/PRP, and A/PD) or within the water-rinsed groups (H_3PO_4/PRP, H_3PO_4/PD, HNO_3/PRP, and HNO_3/PD) for the OE surfaces only. In addition, the mean values associated with HNO_3(W) and HNO_3(A) etch groups were not significantly different for GE and OE surfaces for PRP prophylaxis. This suggests that the nature of outer enamel is not sufficiently altered to lead to significant differences ($P < 0.05$) in mean strength levels. The presence of surface pellicle or films may affect enamel solubility and hence the homogeneity of etch under conditions when the method of prophylaxis is less effective in cleaning the enamel surface. The absence of a water wash does not significantly affect the shear bond strength when HNO_3 is used as an etchant for OE surfaces, irrespective of the nature of prophylaxis.

It is likely that the depth of sealant penetration determines the extent to which the imposed shear at the interface can be accommodated. A larger etch depth would lead to greater depth of penetration but may also leave porosities that may act as stress raisers in the subsurface enamel depending on sealant viscosity. A shorter etch depth would not provide sufficient mechanical interlock retention. Interlocking is improved by the sealant's ability to flow into surface or subsurface cross channels. Evaluation of fracture surfaces shows enamel fracture, sealant fracture, and interface adhesive fracture for surfaces conditioned with phosphoric acid. Shear separation was along the sealant/tooth interface for the majority of the nitric acid conditioned surfaces.

The particle size of the abrasive medium in pumice is larger compared with that in the prophy paste, and a rubber cup was used to clean surfaces. The higher mean values noted for OE surfaces subjected to PRP prophylaxis indicates a greater efficacy of cleaning, while the reverse trend noted for GE surfaces may indicate the incorporation of the additives in prophy paste that are not water soluble. While shear bond strength is important for comparative studies, information on tensile strength values may improve clinical relevance, particularly since the direction of sealant/enamel interface changes continuously outward in a pit or fissure.

Conclusion

The absence of a water rinse step does not significantly affect the bond strength level for the 2.5% HNO_3 enamel etch conditioner on outer enamel surfaces. While significant differences were seen for ground enamel surfaces subjected to pumice prophylaxis, the use of HNO_3 as an enamel conditioner without a postetch water rinse is not contraindicated for outer or occlusal enamel surfaces prior to sealant application.

The applicability of HNO_3 without a water rinse step needs to be evaluated from the point of retention of the etch byproducts and their effect on the interface enamel region, since the effects of NO_3^- impurities in enamel are unknown. Further, HNO_3 conditioning may be applicable to retain orthodontic brackets on labial surfaces considering the small depth of etching and the ease of removal without undue enamel loss.

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