

# Shear bond strength of a resin composite to enamel treated with an APF gel

Franklin Garcia-Godoy, DDS, MS

## Abstract

The purpose of this study was to evaluate the effect of an APF gel treatment on the shear bond strength of a resin composite to enamel. A total of 45 noncarious human extracted permanent molars were used. A flat enamel surface was obtained with 600 grit silicon carbide paper and cleansed with a rubber cup and a water slurry of fine flour of pumice. The teeth were randomly distributed into three groups of 15 teeth each: Group 1: Etched for 30 sec with 37% orthophosphoric acid gel (control); Group 2: APF treatment (Topex gel—Sultan Dental Products, Englewood, NJ) for 1 min and then etched as in Group 1; Group 3: APF gel treatment for 4 min and then etched as in Group 1. After etching, rinsing, and drying, an unfilled resin (XR-Bond—Kerr Manufacturing Co., Romulus, MI) was applied thinly with a brush and cured for 20 sec. A nylon ring (internal diameter: 6.69 mm<sup>2</sup>) was placed over the area and filled with a light-cured composite resin (Herculite XR®—Kerr Manufacturing Co., Romulus, MI). The teeth were thermocycled (x500), mounted in plastic cups and plaster, and sheared with a knife-edged blade in an Instron® Testing Machine (Instron Engineering Corp., Canton, MA) running at a crosshead speed of 0.5 mm/min. The results in MPa were as follows: Group 1: 24.88 ± 6.16; Group 2: 23.20 ± 5.11; Group 3: 21.29 ± 8.44. An ANOVA and Student-Newman-Keuls procedure revealed no statistically significant difference among the groups. Enamel fracture (enamel cohesive failure) occurred in all specimens in groups 1 and 2, and in 14 of 15 in Group 3. (*Pediatr Dent* 15:272–74, 1993)

## Introduction

Treating enamel with fluoride for caries prevention has been advocated widely. Fluorides react with the enamel, forming calcium fluoride and fluoroapatite, which act as slow releasing agents, enhancing remineralization of enamel and making it more resistant to acid dissolution.<sup>1</sup> However, the formation of reaction products (mainly calcium fluoride<sup>2,3</sup>) has been reported to reduce resin bond strength to enamel.<sup>4,5</sup>

Topical application of an acidulated phosphate fluoride (APF) gel remains an important strategy for caries prevention, and it has been reported to affect bonding of fissure sealants.<sup>6</sup> However, short exposure of enamel to fluoridated agents such as prophylaxis pastes, dentifrices, and etching gels or solutions had no significant effect on the bonding of sealants, composites, and orthodontic brackets.<sup>7–15</sup>

The results reported almost two decades ago may not necessarily apply to the newer APF gels, etching times and agents, and resin composites.

The purpose of this study was to evaluate the effect of an APF gel treatment on the shear bond strength of a resin composite to enamel.

## Methods and materials

A total of 45 noncarious human extracted permanent molars stored in distilled water were used. A flat enamel surface was obtained on the buccal surface with 600 grit silicon carbide paper. The teeth then were distributed randomly into three groups of 15 teeth each:

Group 1: Rubber cup pumice prophylaxis

Group 2: APF gel treatment (Topex gel—Sultan Dental Products, Englewood, NJ) for 1 min

Group 3: APF gel treatment (Topex gel) for 4 min.

After each tooth was cleansed with pumice, rinsed, and dried, for groups 2 and 3 the APF gel was applied directly over the enamel with a plastic spatula so as to avoid touching the surface. After the required time, the gel was rinsed off with distilled water for 15 sec, and the enamel dried with oil-free compressed air for 20 sec.

In all groups, the enamel surface was etched for 30 sec with 37% phosphoric acid (Kerr Gel Etchant—Kerr Manufacturing Co., Romulus, MI) and then washed thoroughly for 15 sec with water. After drying each specimen with oil-free compressed air for 20 sec, XR-Bond (Kerr Manufacturing Co., Romulus, MI) bonding agent was applied. Immediately, Herculite XR® (Kerr Manufacturing Co., Romulus, MI) (Kerr, light yellow) resin composite was placed in a nylon cylinder and held with finger pressure over the flattened enamel surface. The resin was then cured for three 40-sec intervals, one from the top and two from the sides. After 15 min at room temperature, they were placed in deionized water for 24 hr.

The specimens were thermocycled x500 in deionized water ranging in temperature from 5–55°C at 30-sec intervals, embedded in dental stone, and sheared with a knife-edged blade on the Instron testing machine at a crosshead speed of 0.5 mm/min. The blade was placed at the ring-tooth interface. The force required to break the resin-

**Table 1. Shear bond strength (in MPa) for the different groups**

Group	Number	Mean (MPa)	SD	Range
Pumice prophylaxis	15	24.88	6.16	15.40 – 35.12
APF for 1 min	15	23.20	5.11	15.55 – 30.94
APF for 4 min	15	21.29	8.44	8.23 – 39.34

**Table 2. Debonding failure site for the different groups**

Group	Debonding Failure Site			Total
	Within Composite	Composite-Enamel Interface	Enamel Fracture	
Pumice	0	0	15	15
APF for 1 min	0	0	15	15
APF for 4 min	1	0	14	15

enamel bond was recorded in MPa.

After shearing the specimens, each tooth and composite interface was examined visually and with a stereomicroscope (x40) to record the failure mode, i.e., within the composite (composite cohesive failure), at the composite-enamel interface or within the enamel (enamel cohesive failure). Selected samples also were evaluated with a scanning electron microscope.

The data were statistically analyzed using the analysis of variance (ANOVA) at the 0.01 level of significance and the Student-Newman-Keuls procedure.

## Results

The mean shear bond strength values for the different groups are displayed in Table 1. The statistical analyses revealed that there was no significant difference among the shear bond strength values for the different groups. Table 1 also shows that as the fluoride application time increased, so did the standard deviation. In Group 3 (4-min fluoride application) the lowest and highest bond strengths were recorded.

While debonding, all specimens in groups 1 and 2 fractured the enamel (enamel cohesive failure) while 14 of 15 samples in Group 3 fractured the enamel. The sample not fracturing the enamel in Group 3 displayed a resin fracture (resin cohesive failure) (Table 2).

## Discussion

Fluoride has been negatively associated with resin bonding. This study, however, shows that resin composite shear bond strength to enamel previously treated for 1 or 4 min with the APF gel used in this study was not affected significantly.

Other studies have shown that even incorporating small amounts of fluoride to phosphoric acid agents,<sup>15,16</sup> or applying fluoride after acid etching before placing the resin,<sup>8</sup> did not significantly influence the bond strength of resin composite to enamel. Garcia-Godoy et al.<sup>7</sup> have shown

that the bond strength of orthodontic brackets placed after etching with a sodium fluoride-containing phosphoric acid gel was significantly higher than that of brackets placed after etching with a conventional nonfluoridated phosphoric acid gel. Brannstrom et al.<sup>17</sup> reported that enamel pretreatment with a fluoride varnish had no negative effect on the etching patterns.

The results of this study provide further evidence that the presence of fluoride does not always adversely affect the bonding of resin compos-

ite to enamel. From a clinical standpoint, the APF gel treatment before resin bonding could prove beneficial over time, and because of the potential resistance to solubility provided to enamel. This must be proven clinically.

In this study, all specimens were thermocycled. Temperature cycling was performed to allow water sorption by the resin and to evaluate the effect of disparities in the coefficients of thermal expansion/contraction of the restorative system and tooth structure.<sup>18</sup> The number of cycles during thermal stress is not significant. Crim and Garcia-Godoy<sup>19</sup> have shown no difference in microleakage of resins thermocycled for 100 or 1500 cycles, and Burger et al.<sup>20</sup> reported no significant difference in the shear bond strength of resins thermocycled for 100 or 4000 cycles.

Another interesting variable is etching time. Studies of this parameter have shown no statistically significant difference in bond strength of resin composite to enamel or retention of fissure sealants using a reduced etching time or gel or solutions.<sup>21-28</sup> These results, coupled with those obtained with the 30-sec etch in this study, clearly indicate that a 30-sec etch is as effective as a 60-sec etch. The reduced etching time effectiveness also has been demonstrated in primary teeth.<sup>29</sup> Perhaps the lower etching time accounts for the results obtained. Increasing the enamel etching time produces greater loss of substance.<sup>30</sup> Although this substance loss produces a more pronounced micromorphological etch effect, it could weaken the enamel prisms rendering them more fragile to resist shear forces.

A similar proportion of specimens fractured the enamel (enamel cohesive failure) whether the enamel was treated or not with APF gel for 1 min. However, when the APF gel was applied for 4 min, 14 of 15 specimens fractured the enamel (enamel cohesive failure); the other specimen failed within the resin (resin cohesive failure), which may have been because of some type of testing error. These results reveal that a stronger force than the one actually reported is necessary to debond the resin from the enamel. With advancing resin bonding technology, reporting the bond

strength figure alone does not provide the real value of bond testing—therefore the failure site must be reported. The significance of “bond strength” values would then be superfluous if all the samples display enamel and/or resin cohesive failure, a common finding with the recent resin-adhesive combinations.

Further studies should be conducted to analyze the different APF gels.

Dr. Garcia-Godoy is professor and director, Predoctoral Division, Department of Pediatric Dentistry, and professor, Department of Restorative Dentistry, University of Texas Health Science Center at San Antonio, Texas.

1. Koulourides T, Keller SE, Manson-Hing L, Lilley V: Enhancement of fluoride effectiveness by experimental cariogenic priming of human enamel. *Caries Res* 14: 32–39, 1980.
2. Caslavská V, Moreno EC, Brudevold F: Determination of the calcium fluoride formed from in vitro exposure of human enamel to fluoride solutions. *Arch Oral Biol* 20:333–39, 1975.
3. Larsen MJ, Fejerskov O: Structural studies on calcium fluoride formation and uptake of fluoride in surface enamel in vitro. *Scand J Dent Res* 86:337–45, 1978.
4. Sheykholeslam Z, Buonocore MG, Gwinnett AJ: Effect of fluorides on the bonding of resins to phosphoric acid-etched bovine enamel. *Arch Oral Biol* 17:1037–45, 1972.
5. Gwinnett AJ, Buonocore MG, Sheykholeslam Z: Effect of fluoride on etched human and bovine tooth enamel surfaces as demonstrated by scanning electron microscopy. *Arch Oral Biol* 17:271–78, 1972.
6. Low T, von Fraunhofer JA, Winter GB: The bonding of a polymeric fissure sealant to topical fluoride-treated teeth. *J Oral Rehabil* 2:303–7, 1975.
7. Garcia-Godoy F, Hubbard GW, Storey AT: Effect of a fluoridated etching gel on enamel morphology and shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop* 100:163–70, 1991.
8. Bishara SE, Chan D, Abadir EA: The effect on the bonding strength of orthodontic brackets of fluoride application after etching. *Am J Orthod Dentofacial Orthop* 95:259–60, 1989.
9. Garcia-Godoy F, Perez R, Hubbard GW: Effect on prophylaxis pastes on shear bond strength. *J Clin Orthod* 25:571–73, 1991.
10. Bogert TR, Garcia-Godoy F: Effect of prophylaxis agents on the shear bond strength of a fissure sealant. *Pediatr Dent* 14:50–51, 1992.
11. Garcia-Godoy F, O'Quinn JA, Donohue M: Effect of a triclosan/copolymer/fluoride dentifrice prophylaxis on the shear bond strength of a composite resin to enamel. *Am J Dent* 4:167–69, 1991.
12. Garcia-Godoy F, O'Quinn JA, Donohue M: Effects of prophylaxis agents on shear bond strengths of composite resins to enamel. *Gen Dent* 1993: in press.
13. Garcia-Godoy F, Dodge WW, Donohue M, O'Quinn JA: Effect of a fluoridated etchant on the shear bond strength of a composite resin to enamel. *Int J Paediatr Dent* 2:25–30, 1992.
14. Garcia-Godoy F: Triclosan/copolymer/NaF dentifrice prophylaxis, reduced etching time and shear bond strength of a resin composite to enamel. *Am J Dent* 5: 312–14, 1992.
15. Thornton JB, Retief DH, Bradley EL Jr, Denys FR: The effect of fluoride in phosphoric acid on enamel fluoride uptake and the tensile bond strength of an orthodontic bonding resin. *Am J Orthod Dentofacial Orthop* 90:91–101, 1986.
16. Takahashi Y, Arakawa Y, Matsukubo T, Takeuchi M: The effect of sodium fluoride in acid etching solution on sealant bond and fluoride uptake. *J Dent Res* 59:625–30, 1980.
17. Brännström M, Nordenvall KJ, Malmgren O: The effect of various pretreatment methods of the enamel in bonding procedures. *Am J Orthod* 74:522–30, 1978.
18. Retief DH: Standardizing laboratory adhesion tests. *Am J Dent* 4:231–36, 1991.
19. Crim GA, Garcia-Godoy F: Microleakage: the effect of storage and cycling duration. *J Prosthet Dent* 57:574–76, 1987.
20. Burger KM, Cooley RL, Garcia-Godoy F: Effect of thermocycling times on dentin bond strength. *J Esthet Dent* 4:197–98, 1993.
21. Stephen KW, Kirkwood M, Main C, Gillespie FC, Campbell D: Retention of a filled fissure sealant using reduced etch time: a two year study in 6- to 8-year-old children. *Br Dent J* 153:232–33, 1982.
22. Eidelman E, Shapira J, Houpt M: The retention of fissure sealants using twenty-second etching time. *ASDC J Dent Child* 51:422–24, 1981.
23. Shaffer SE, Barkmeier WW, Kelsey WP III: Effects of reduced acid conditioning time on enamel microleakage. *Gen Dent* 35:278–80, 1987.
24. Barkmeier WW, Gwinnett AJ, Shaffer SE: Effects of enamel etching time on bond strength and morphology. *J Clin Orthod* 19:36–8, 1985.
25. Barkmeier WW, Shaffer SE, Gwinnett AJ: Effects of 15 vs 60 second enamel acid conditioning on adhesion and morphology. *Oper Dent* 11:111–16, 1986.
26. Beech DR, Jalaly T: Bonding of polymers to enamel: influence of deposits formed during etching, etching time and period of water immersion. *J Dent Res* 59:1156–62, 1980.
27. Brännström M, Malmgren O, Nordenvall KJ: Etching of young permanent teeth with an acid gel. *Am J Orthod* 82:379–83, 1982.
28. Garcia-Godoy F, Gwinnett AJ: Penetration of acid solution and gel in occlusal fissures. *J Am Dent Assoc* 114:809–10, 1987.
29. Gwinnett AJ, Garcia-Godoy F: Effect of etching time and acid concentration on resin shear bond strength to primary tooth enamel. *Am J Dent* 5:237–39, 1992.
30. Garcia-Godoy F, Gwinnett AJ: Effect of etching times and mechanical pretreatment on the enamel of primary teeth: an SEM study. *Am J Dent* 4:114–19, 1991.