One-Year Tensile Bond Strengths of Two Self-Etching Primers to Bovine Enamel

PATRICIA A. MIGUEZ, DDS, MS*
PATRICIA N.R. PEREIRA, DDS, PHD†
EDWARD J. SWIFT JR, DMD, MS‡

ABSTRACT

Purpose: The purpose of this study was to evaluate the enamel bond strengths of two self-etching primer systems and a total-etch self-priming adhesive system at 24 hours and after a 1-year period of storage in water at 37°C.

Materials and Methods: An experimental self-etching priming system (ABF, Kuraray Medical, Tokyo, Japan), Clearfil™ SE Bond (Kuraray), and One-Step® (Bisco, Schaumburg, IL, USA) were used as the adhesive systems. Eleven bovine incisors stored frozen were ground with 600-grit SiC paper, bonded as recommended by the manufacturers, and restored with Clearfil™ AP-X (Kuraray) or Renew® (Bisco). After 24 hours in water at 37°C, the teeth were sectioned into 0.7 mm–thick slabs that were trimmed and tested for microtensile bond strength using a tabletop tester (EZ test, Shimadzu, Kyoto, Japan) at a crosshead speed of 1 mm/min. Alternate slices were either tested at 24 hours or kept in an incubator at 37°C for 1 year prior to testing. The data were analyzed using paired *t*-test ($p < .05$).

Results: Differences in bond strengths were not significant at 24 hours. After 1 year, ABF and Clearfil™ SE Bond had significantly higher mean bond strengths than at 24 hours ($p < .001$).

Conclusions: Enamel bond strengths of the total-etch two-step adhesive remained stable, and the bond strengths of the two self-etching primer systems increased with time.

CLINICAL SIGNIFICANCE

Although self-etching primers create a milder etching pattern than does phosphoric acid, the results of this study suggest that the ones evaluated in this study produce stable bond strengths over a 1-year period.

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Bonding to enamel using the acid-etch technique has proven to be a reliable method of adhesion to tooth structure since 1955, when Buonocore established the foundation for resin-based adhesive restorative and preventive dentistry.[1] Using 32 to 40% phosphoric acid, resin bonding to enamel has been predictably successful.[2] Resin bond strengths to enamel are reliable even when hydrophobic resins are used, as long as the operator carefully dries the enamel to ensure that water is totally removed to avoid displacement of the bonding material.[3] Since 1979, when Fusayama and colleagues introduced the "total-etch" philosophy,[4] phosphoric acid has been used routinely to etch dentin. Although the total-etch technique has been demonstrated to be successful, technique and post-operative sensitivities are concerns

*Graduate student, Department of Operative Dentistry, University of North Carolina at Chapel Hill, Chapel Hill, NC, USA
†Assistant professor, Department of Operative Dentistry, University of North Carolina at Chapel Hill, Chapel Hill, NC, USA
‡Professor, Department of Operative Dentistry, University of North Carolina at Chapel Hill, Chapel Hill, NC, USA

for many clinicians. To overcome these problems, two-step self-etching primers have been developed, combining etching and priming into a single step with a mild acidic solution. This solution demineralizes and primes the dentin simultaneously and is followed by the application of the bonding agent. Therefore, the potential problem of a deeply demineralized dentin that is not fully saturated by a primer and a bonding agent is avoided, and postoperative sensitivity is diminished.[5,6]

The literature has shown that self-etching primers generally do not etch enamel as well as phosphoric acid.[7,8] An acidic primer solution dissolves the smear layer and demineralizes the underlying substrate while simultaneously being buffered by the substrate, creating a milder demineralization pattern in the tooth substrate than does the total-etch technique.[9,10] Various self-etching primers have different etching patterns, but those are not necessarily correlated with variances in bond strengths.[3,9] It has been reported that the self-etching primers produce bond strengths on ground enamel in the range of 14 to 20 MPa, which is less than the values typically achieved using phosphoric acid.[5,10–13]

Although several short-term in vitro studies have shown that certain self-etching primers can be reliable adhesive systems for enamel and dentin,[6,8–12] the longevity of the bonds created by these agents still needs to be evaluated. Recent studies have shown that bond strengths of self-etching primers to enamel significantly decrease after thermocycling when compared with phosphoric acid etching, which raises concerns about the longevity of the bond.[14]

Therefore, the purpose of this study was to evaluate bond strengths of a commercially available and an experimental two-step self-etching primer system, using a total-etch two-step adhesive system as a control at 24 hours and after a 1-year period of storage in water at 37°C. The hypothesis tested was that bond strengths of the two self-etching systems to enamel would decline significantly over time.

MATERIALS AND METHODS

Eleven frozen bovine incisors were used for this study. The labial surface of the enamel was ground with wet 600-grit abrasive paper under running water until a flat surface of enamel was obtained. Each tooth was carefully examined to be sure that no dentin was exposed. If exposed dentin was detected at any part of the ground enamel, the tooth was discarded from the experiment. The two-step self-etching primers used in this study were Clearfil™ SE Bond (Kuraray Medical, Tokyo, Japan) and ABF experimental (Kuraray). The "total-etch two-step" adhesive system was One-Step® (Bisco, Schaumburg, IL, USA), which was used as the control adhesive system. All adhesives were applied according to the manufacturers' instructions. The composite resins, Clearfil™ AP-X (Kuraray) and Renew® (Bisco), were placed according to the adhesive system (Table 1). All light curing was performed with an Optilux™ 501 (Demetron Kerr, Danbury, CT, USA) at 570 mW/cm^2. Light output was checked before and after the light-curing procedure to confirm intensity of the light source.

Specimens were stored in tap water at 37°C. Clearfil™ SE Bond and ABF groups were composed of four teeth each and One-Step® by three teeth. After 24 hours' storage, the specimens were sectioned into 0.7 mm–thick slabs using a diamond saw (Isomet®, Buehler Ltd., Lake Bluff, IL, USA) under water cooling and were further trimmed to an hourglass shape for the microtensile bond test (area of 1 mm^2).[15] Alternate sections were either tested at 1 day or kept in water at 37°C for 1 year before testing. The storage solution was changed weekly.[16] The slabs were glued with Zapit (DVA, Corona, CA, USA) to a Ciucchi jig, a device that consists of two stainless-steel components that slide away from each other, thus pulling the specimen apart. The jig was attached to an EZ test tabletop universal tester (Shimadzu, Kyoto, Japan), subjected to tensile forces at a crosshead speed of 1 mm/min; bond strengths were calculated in megapascals by dividing the values (in newtons) by the area (1 mm^2) (Figure 1). Because the study was

TABLE 1. MATERIALS USED AND APPLICATION.

Materials	Lot No.	Components	Application
Clearfil™ SE Bond	61137	Primer: MDP, HEMA, hydrophilic dimethacrylate, camphorquinone, N,N-diethanol *p*-toluidine, water Adhesive: MDP, BIS-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, N,N-diethanol *p*-toluidine, colloidal SiO_2	Applied to enamel for 20 s and air dried. The bonding agent was applied, gently air thinned, and light cured for 10 s over the bovine enamel.
ABF	Primer (ABP): 000411 Bond (KBF): 991130	Primer: MDPB, MDP, HEMA, water, initiator Adhesive: MDP, HEMA, hydrophobic dimethacrylate, camphorquinone, surface-treated NaF, colloidal SiO_2, initiator	Primer agent was applied to enamel for 20 s and gently air dried. The bonding agent was applied, air thinned, and light cured for 10 s.
One-Step®	0000007052	BIS-GMA, HEMA, BPDM, acetone	After acid etching, two coats of the adhesive system were applied to enamel, thoroughly dried for 10 s, and light cured for 10 s.
Clearfil™ AP-X	631AA	BIS-GMA, camphorquinone, TGDMA, silanated barium glass, silica	Incremental layers up to 4 mm were placed, and each increment was light cured for 40 s.
Renew®	990009737	BIS-GMA, BPDM, TGDMA, glass, silica	Incremental layers up to 4 mm were placed, and each increment was light cured for 40 s.

ABP = antibacterial self-etching primer; BIS-GMA = bisphenol diglycidyl metacrylate; BPDM = biphenyl dimethacrylate; HEMA = 2-hydroxyethyl methacrylate; KBF = Kuraray bonding agent with fluoride; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; MDPB = 12-methacryloyloxydodecylpyridinium bromide; TGDMA = thiethylene glycol dimethacrylate

not intended to compare the adhesive systems but to evaluate the bond strengths over time, data were analyzed using paired *t*-test ($p < .05$) (Statview® Software Version 5.0.1, SAS, Cary, NC, USA).

For observation of the fracture modes, the debonded specimens were fixed in 10% neutral buffered formalin for at least 8 hours.[17] The enamel and resin sides of the fractured specimens were trimmed, placed on stubs and subjected to room desiccation, and gold sputter coated in a 5100 sputter-coater (Polaron Equipment Ltd., Watford, England). Fracture modes were examined using a JSM 6300 scanning electron microscope (JEOL USA Inc., Peabody, MA, USA).

RESULTS

Microtensile bond strengths of all adhesive systems to enamel are summarized in Table 2. The mean bond strength of each adhesive

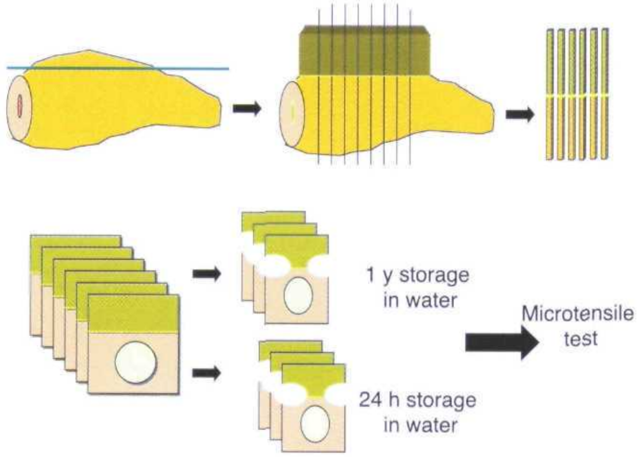

Figure 1. Method used to prepare the teeth for microtensile testing (see text for details).

TABLE 2. MICROTENSILE BOND STRENGTHS AND STANDARD DEVIATIONS (MPa).

	Clearfil™ SE Bond	ABF	One-Step®
1 d	20.4 ± 3.9 (n = 15)	22.8 ± 5.8 (n = 14)	18.1 ± 4.7 (n = 12)
1 yr	24.3 ± 5.9 (n = 16)	30.5 ± 5.1 (n = 14)	20.7 ± 7.2 (n = 12)

was higher at 1 year. The difference was significant for both Clearfil™ SE (p = .0081) and ABF (p = .0004) but not for One-Step® (p = .4300).

Scanning electron microscope (SEM) analysis showed that ABF groups tested at 24 h and 1 year failed cohesively in resin and adhesive. At 24 hours Clearfil™ SE had approximately 50% of the failures in resin and adhesive. When tested at 1 year, the pattern of failure was mostly cohesively in resin. For One-Step®, the fracture pattern changed mainly from a mixed failure in enamel, adhesive, and resin to mostly cohesive in enamel or adhesive. The percentage distribution of failures is illustrated in Figure 2.

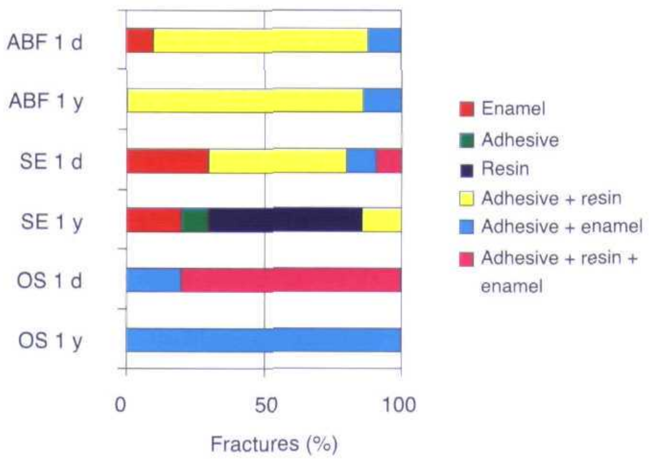

Figure 2. Fracture modes of all groups in percentages. OS = One-Step®; SE = Clearfil™ SE Bond.

DISCUSSION

Adhesion to enamel has been studied extensively over the years. Bond strengths produced by using phosphoric acid as an etchant have proven to provide adequate retention and prevention of microleakage around restorations. However, with the increasing popularity of self-etching primers, research on enamel bonding has again become essential. The pattern of demineralization created by two-step self-etching primer systems is milder than that with phosphoric acid etchant and therefore subject to questions concerning immediate bond strengths and long-term stability.[3,9,10]

This study evaluated the bond strengths of two self-etching primer systems and one total-etch, two-step system at 24 hours and 1 year. The bond strengths of the two self-etch systems were significantly higher at 1 year. The bond strengths are similar to those reported after 24 hours in other studies of self-etching primers.[3,9,10,12] Sano and colleagues also have reported that bond strengths of a two-step self-etching primer to dentin continued to be stable during 1 year in vivo.[15] Although it is true that in vitro durability studies do not simulate in vivo conditions, they might, within limitations, have some predictive value for clinical performance.

Interestingly, mean bond strengths in the present study increased over the 1-year period. Presumably, the post-curing of the adhesive resin and more specifically of the oxygen-inhibited layer may have occurred owing to the 37°C heat storage condition during the long period of time. Because this increase was significant for ABF and Clearfil™ SE Bond, it is believed that the thickness of the oxygen-inhibited layer—approximately 25 µm and 22 µm, respectively (data provided by manufacturer)—produced by these adhesive resins as compared with approximately 5 µm produced by One-Step® (data provided by manufacturer) plays an important role in the greater increase of bond strength. The enhanced polymerization of a thicker oxygen-inhibited layer might be responsible for the significant increase in bond strengths for ABF and Clearfil™ SE Bond. Unfortunately, because of the limitations of this study, there is a lack of interim bond strength data (eg, 3- and 6-month intervals) to reveal when bond strengths started increasing. Further studies are necessary to elucidate the clinical importance of post-curing of adhesive resins intraorally owing to the relatively warm body temperature.

Bond strengths were evaluated on enamel that had been ground with 600-grit SiC paper. There are concerns regarding the use of self-etching primers in the presence of thick smear layers as well as in nonprepared enamel. In a study done in dentin, Watanabe and colleagues showed that a thick smear layer would reduce the ability of the priming agent to achieve good bond strength in dentin, and they suggested the removal of the smear layer with mild acid etching.[18] A thick smear layer may interfere with the diffusion of the self-etching primer in enamel as well, limiting the etch and resin penetration. On the other hand, intact enamel shows a much more resistant structure because the outer layer of the tooth structure is constituted of more disoriented enamel prisms, which makes it more difficult for acids to penetrate and dissolve them. Hence, intact enamel treated with the acidic monomers of the self-etching primer show lower bond strengths than does ground enamel.[8] Further studies are also necessary to evaluate long-term bond strengths of self-etching primers to intact enamel.

The SEM observations of fractured sites showed similar images of adhesive and resin failures at 24 hours and 1 year for ABF. Clearfil™ SE presented mostly cohesive failures in adhesive and resin, which changed to mostly fractures within the resin composite at 1 year. The integrity of the bonded interface may be affected by hydrolysis of partially cured monomers over time. No sign of degradation or water trapping was found in the SEM observations, which suggests a good interaction between those two self-etching systems with ground enamel.

The hypothesis was rejected because the bond strengths of the self-etching primers to enamel did not decrease over time. Further studies are necessary to examine the mechanical and morphologic durability of restorations bonded to enamel with self-etching primers either under simulated occlusal stresses or in vivo.

CONCLUSIONS

Enamel bond strengths of the total-etch two-step adhesive remained stable with time, and the bond strengths of the two self-etching primer systems increased with time.

DISCLOSURE

The authors do not have any financial interest in the companies whose materials are discussed in this article.

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Reprint requests: Patricia A. Miguez, DDS, MS, Department of Operative Dentistry, 302 Brauer Hall, CB #7450, University of North Carolina at Chapel Hill, Chapel Hill, NC, USA 27599-7450; e-mail: miguezp@dentistry.unc.edu