

Enamel bond strengths of "one-bottle" adhesives

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Abstract

Purpose: Several "one-bottle" dental adhesives recently have been introduced. These contain hydrophilic resin monomers that should readily wet tooth surfaces. Most also contain solvents that could increase enamel bond strengths by driving out residual moisture from enamel and increasing resin penetration. The purpose of this study was to evaluate bond strengths obtained by six one-bottle bonding agents and one conventional unfilled resin (control).

Methods: Seventy bovine teeth were randomly assigned to seven groups of 10. Enamel was etched for 15 s with 35% phosphoric acid. Following application of the adhesive, composite resin was bonded using a gelatin capsule technique. Shear bond strengths to enamel were determined.

Results: Mean bond strengths ranged from 14.2 MPa for Syntac Single-Component to 27.8 MPa for Single Bond. The mean for Syntac Single-Component was significantly less than that of all other systems tested.

Conclusion: The results of this study indicate that one-bottle bonding agents, with the exception of the Syntac material, provide enamel bond strengths at least equal to that of a conventional unfilled resin. (*Pediatr Dent* 20:259-62, 1998)

Several "one-bottle" dental adhesives have been introduced recently. These combine the functions of the primer and adhesive components of conventional three-step (conditioner, primer, bonding agent) adhesive systems. Each system still requires a separate conditioning step and some require multiple applications of the adhesive.

Although these materials are relatively new, a number of dentin bond strength studies have already been reported.¹⁻⁷ Fewer studies of enamel bonding are available, but very high enamel bond strengths (up to 42 MPa) have been reported.²⁻⁴ In one study, enamel bond strengths for five commercial one-bottle systems were 8-24 MPa higher than corresponding dentin bond strengths.² Differences of that magnitude have rarely been seen since the development of effective dentin bonding agents. Although exaggerated somewhat by relatively low dentin bond strengths found for two agents, these differences underscore the magnitude of the enamel bond strengths. It is possible that solvents in the one-bottle adhesives such as acetone or ethanol remove residual moisture and enhance resin wetting of etched enamel.²⁻⁴

The purpose of this study, therefore, was to evaluate the enamel bond strength of several one-bottle adhesives containing different solvents. The specific hypothesis was that one-bottle adhesives containing acetone or ethanol provide enamel bond strengths equal to or greater than that of a conventional unfilled bonding resin.

Methods

The compositions and batch numbers of the adhesives used in this study are listed in Table 1. Seventy defect-free bovine incisors were collected, débrided, disinfected in aqueous chloramine solution, and stored in tap water until use. (Bovine teeth are considered an acceptable substitute for human teeth in bond strength testing.⁸) Each tooth was mounted by placing it in a 1-in. diameter phenolic ring (Buehler, Ltd., Lake Bluff, IL) and filling the ring with self-cure acrylic resin (Trayresin, Dentsply York, York, PA). Embedded specimens were ground with 120-grit abrasive on a water-cooled Ecomet (Buehler) grinder to flatten the labial enamel. Enamel was hand polished to 600-grit on a series of wet silicon carbide abrasive papers. Ten specimens were randomly assigned to each of seven groups.

Enamel was etched for 15 s with a 35% phosphoric acid gel (3M Dental Products Division, St. Paul, MN). The etchant was rinsed for approximately 10 s under running tap water. The enamel was dried with compressed air so that all visible moisture was removed and the surface had the classic "ground-glass" appearance. Bonding agents were applied and cured according to manufacturers' directions, specifically:

1. Scotchbond Multi-Purpose adhesive (control) (3M) was applied and thinned with a brush, and light cured for 10 s.
2. Two consecutive coats of One-Step (Bisco, Inc., Itasca, IL) were applied using a saturated brush with slight agitation. A gentle stream of compressed air was used to evaporate the solvent, and the resin was light cured for 10 s.
3. A single coat of OptiBond Solo (Kerr Corporation, Orange, CA) was applied using a light brushing motion for 15 s to help evaporate the solvent. The material was light cured for 20 s.
4. Prime & Bond 2.1 (Dentsply Caulk, Milford, DE) was applied to the surface, and was reapplied as

necessary to ensure that the surface remained wet with the bonding agent for 20 s. Excess solvent was removed with gentle air drying, and the material was light cured for 10 s. A second coat was applied and immediately air dried, but was not light cured.

5. Syntac Single-Component (Ivoclar Vivadent, Amherst, NY) was brushed on the enamel for 10 s and was then left undisturbed for 20 s. The material was air dried, beginning very gently and gradually increasing the pressure. The resin was light cured for 20 s. A second application was made and cured in the same manner.
6. Two consecutive coats of Single Bond (3M) were applied using a saturated brush tip. After gently air drying for 5 s, the material was light cured for 10 s.
7. Three consecutive coats of Tenure Quik with Fluoride (Den-Mat Corporation, Santa Maria, CA) were applied using a saturated brush tip. The material was allowed to remain on the enamel undisturbed for 15 s. Excess solvent was removed by air drying for 10 s, and the resin was light cured for 15 s.

#5 gelatin capsules (Torpac, Inc., Fairfield, NJ) were preloaded with composite (Restorative Z100 shade A3, 3M) to about two-thirds of their length, and the composite was cured in a Triad light-curing unit (Dentsply York). A final increment of composite was added to fill each capsule. The capsule was applied to the treated enamel surface and excess composite was carefully removed with an explorer. The composite was cured for 40 s from two opposing sides of the capsule using an Optilux 401 visible light-activation unit (Demetron/Kerr, Danbury, CT). Light intensity was checked periodically with a dental radiometer and remained in excess of 600 mW/cm².

Specimens were stored for 24 h in tap water at 37°C. Shear bond testing was conducted with a blunt-edge shearing chisel in a model 4411 universal testing machine (Instron Corporation, Canton, MA). The crosshead speed was 5 mm/min and the chisel was located at the enamel/composite interface. Shear bond strength (MPa) was calculated by dividing the failure load (N) by the cross-sectional area of the bonded composite post (14.65 mm²). Data were subjected to one-way ANOVA using the Systat for Windows 5.0 statistical software package (Systat, Inc., Evanston, IL).

Results

Mean shear bond strengths ranged from 14.2 MPa for Syntac Single-Component to 27.8 MPa for Single Bond (Table 2). ANOVA revealed a significant difference between means ($P < 0.0001$) (Table 3). Tukey's multiple comparison procedure showed that the mean shear bond strength of Syntac Single-Component was significantly less than that of all other systems tested ($P < 0.05$). Differences between other means were not statistically significant.

Discussion

Shear bond strengths of composite resin to phosphoric acid-etched enamel are typically in the range of 20 MPa.^{9, 10} For example, a recent study found that Scotchbond Multi-Purpose Adhesive had a bond strength to bovine enamel of 23–25 MPa.⁷ Bond strengths of 17–24 MPa are required to effectively resist the polymerization contraction forces of composite resin.^{11, 12}

Unfortunately, bonding between composite resin and enamel may be compromised by moisture contamination. Prevention of moisture contamination is particularly important for pit and fissure sealants, where retention and seal are solely dependent on the quality of the resin/enamel bond.¹³

One method to reduce the effects of moisture on bonding of sealants is the use of drying agents. A clinical trial of a proprietary drying agent (unknown composition) found that it enhanced sealant retention.¹⁴ Another potential method for reducing the effects of moisture on sealant bonding is the use of dentin adhesives. An in-vitro study showed that use of a phosphonated resin-bonding agent (original Scotchbond, 3M) dramatically improved the bond strengths of resin to enamel that was contaminated by saliva or made moist by brief storage in a humidity chamber.¹⁵ A more recent study demonstrated

TABLE 1. COMPONENTS AND BATCH NUMBERS OF ADHESIVE SYSTEMS

<i>System</i>	<i>Composition</i>	<i>Batch Number*</i>
One-Step	Bis-GMA, BPDm, HEMA, acetone	019207
OptiBond Solo	HEMA, Bis-GMA, GPDM, SiO ₂ , barium glass, fluoride, ethanol	611378
Prime & Bond 2.1	UDMA, PENTA, resin oligomers, fluoride, acetone	961025
Single Bond	Bis-GMA, HEMA, dimethacrylates, polyalkenoic copolymer, ethanol, water	19970207
Syntac Single-Component	methacrylate-modified polyacrylic acid, HEMA, maleic acid, fluoride, water	808658
Tenure Quik with Fluoride	dimethacrylate resins, HEMA, PMDM, fluoride, acetone	227012
Scotchbond MPP (control)	Bis-GMA, HEMA	19970303

* Z100 batch number 19970217 (shade A3).

that resin penetration of enamel fissures was enhanced by the use of the All-Bond 2 (Bisco) and Scotchbond Multi-Purpose adhesive systems.¹⁶ The authors concluded that the use of such systems might be particularly beneficial in deep fissures that are not completely dry. However, the literature provides no consensus on whether dentin primers are beneficial for enamel bonding. Studies have shown that bond strengths either increase, decrease, or do not change when a primer is applied to etched enamel.¹⁷⁻²¹

Clinical studies of dentin bonding agents for enamel bonding are also inconclusive. A clinical trial using the phosphonated resin Scotchbond on saliva-contaminated etched enamel showed that the 2-year retention rate was similar to that of sealants applied to dry etched enamel.²² However, a later study of another phosphonate-ester adhesive, Prisma Universal Bond (Dentsply Caulk), found that its use did not improve sealant retention rate.²³

None of these laboratory or clinical studies evaluated the new one-bottle adhesives, which typically contain polymerizable resin monomers dissolved in a solvent such as acetone or ethanol. The composition of these new materials could be particularly well suited for enamel bonding, as the solvents can "chase" any residual moisture from the etched enamel, carrying the resin monomers into close adaptation with the surface.

In fact, some recent work²⁻⁴ supports the hypothesis that these materials are excellent materials for bonding to enamel, and that work is supported by the results of this study. el-Kalla and Garcia-Godoy recently reported particularly dramatic evidence of their bonding effectiveness.⁴ In that study, etched enamel and dentin were

contaminated for 20 s with fresh saliva. The saliva was either rinsed off with water or allowed to remain, simulating a clinical situation in which saliva contamination went unnoticed. Excess moisture (either water or saliva) was removed with a cotton pellet or, in the case of Syntac Single-Component, with compressed air. One-bottle bonding agents were applied in the usual manner. With one exception, saliva contamination did not adversely affect the bond of these agents to either dentin or enamel. The one exception was Syntac Single-Component on dried contaminated enamel. In all other groups, moisture remained on the surface and probably prevented salivary proteins from adsorbing and blocking resin penetration. The agents were able to displace that moisture and penetrate enamel effectively, resulting in very high bond strengths.

Although each of these one-bottle adhesives contains hydrophilic monomers that should help to wet surfaces and enhance resin penetration, the type of solvent may have some effect on enamel bonding capability. In our study, those materials containing ethanol or acetone as a solvent provided bond strengths to enamel equal to or exceeding those provided by a conventional unfilled resin. Acetone is a particularly effective "water chaser", displacing water from the tooth surface. It has been suggested that acetone is the best solvent for carrying resin into conditioned tooth surfaces, and this suggestion is supported by experimental evidence.²⁴⁻²⁶ The results of this study suggest that ethanol might have a similar effect. The shear bond strength of one ethanol-containing agent was the highest of any group, and both the ethanol- and acetone-containing agents wet the etched-enamel surfaces remarkably well. Previous reports have shown that ethanol 1) improves resin penetration of etched enamel²⁷ and 2) is as effective as acetone for reversing the adverse effects of bleaching solutions on enamel bond strengths.²⁸

Previous studies have shown that Scotchbond Multi-Purpose, which includes an aqueous primer, has similar bond strengths to dry and moist enamel and dentin.^{29,30} However, the only water-based system tested in the present study, Syntac Single-Component, had significantly lower enamel bond strengths than the other systems tested. It is unclear how much of this difference is related to the use of a water solvent.

Of the three acetone-based adhesives evaluated in this study, One-Step had a lower mean bond strength than either Prime & Bond 2.1 or Tenure Quik with Fluoride, although the difference was not statistically significant. For One-Step, two brief consecutive applications were made and the solvent was evaporated immediately with compressed air. In contrast, for both Tenure Quik and Prime & Bond, a large quantity of the adhesive remained on the dentin surface for 15-20 s. This extended contact time probably provides both more acetone to chase residual wa-

TABLE 2. RESULTS OF SHEAR BOND STRENGTH TESTING (N = 10)

Adhesive	Solvent	Shear Bond Strength (MPa)
		Mean ± SD
Single Bond	ethanol/water	27.8 ± 4.3
Prime & Bond 2.1	acetone	26.4 ± 4.0
Tenure Quik w/F	acetone	24.5 ± 5.7
Scotchbond MPP	n/a (control)	22.9 ± 6.2
OptiBond Solo	ethanol	21.8 ± 4.0
One-Step	acetone	21.7 ± 4.1
Syntac SCC	water	14.2* ± 5.0

* Significantly different from other means ($P < 0.05$).

TABLE 3. ANOVA

Source	Sum-of-Squares	DF	Mean-Square	F-Ratio	P
Adhesive	250890.258	6	41815.043	8.335	0.0001
Error	316041.643	63	5016.534		

ter from the surface and more resin to infiltrate the etched enamel. In other words, the difference between One-Step and the other two materials may be related more to application technique than to any inherent difference in hydrophilic or other properties of the materials themselves.

It should be noted that the data reported in this study were obtained by bonding to enamel that was dried with compressed air and appeared to be free of moisture (although moisture may have remained microscopically). Currently, dry enamel is encountered in "enamel-only" bonding situations, such as pit and fissure sealants, veneers, and orthodontic bonding. In most restorative situations, resin is bonded to both dentin and enamel, and for most current adhesive systems, the surface is left somewhat moist. Planned future research will compare the effectiveness of the one-bottle systems for bonding to visibly moist enamel and to enamel contaminated with oil from the handpiece.

Conclusions

In summary, all but one of the systems tested in this study achieved a mean shear bond strength to enamel approaching or exceeding 20 MPa. Prime & Bond 2.1 and Single Bond systems had the highest mean bond strengths, while Syntac Single-Component had the lowest. Its bond strength was significantly less than that of the control (conventional bonding resin) and the other one-bottle adhesives.

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