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ORIGINAL RESEARCH

A New Solvent-free One-Step Self-Etch Adhesive: Bond Strength to Tooth Structures

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ABSTRACT

Introduction: In recent years some new solvent-free dental adhesives have been marketed. This study evaluated bonding effectiveness of a new one-step solvent-free self-etch adhesive in comparison with a common two-step self-etch adhesive used as gold standard.

Materials and methods: Flat enamel and dentin surfaces were prepared on 60 incisors using silicon carbide papers. Clearfil SE Bond (CSEB) and Bond 1SF (B1SF) adhesives were applied on enamel/dentin surfaces in four groups (n = 15): (1) Enamel surface and CSEB, (2) dentin surface and CSEB, (3) enamel surface and B1SF, (4) dentin surface and B1SF. Composite resin buildups were carried out using Z100 composite resin. All the specimens were stored for 24 hours at 37°C and 100% relative humidity. After 500 rounds of thermocycling, shear bond strength (SBS) test was performed using a universal testing machine at 1 mm/min crosshead speed. Data were analyzed with one-way ANOVA and a post hoc Tukey test ($\alpha = 0.05$). In each experimental group, two additional specimens were prepared for scanning electron microscopy evaluation.

Results: Significant differences were observed between the study groups ($p < 0.001$). The highest enamel/dentin bond strengths were recorded in group 1 (CSEB) ($p < 0.001$). The SBS of the two-step self-etch adhesive to enamel and dentin was significantly higher than that of the one-step self-etch adhesive ($p < 0.001$). There was no significant difference between enamel and dentin SBS with B1SF ($p = 0.559$).

Conclusion: Within the limitations of the present study, when bonded to enamel and dentin the solvent-free adhesive B1SF underperforms as compared to CSEB as the control gold standard.

Keywords: Bond strength, Dentin, Enamel, Self-etch adhesive, Solvent.

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Conflict of interest: None

INTRODUCTION

The weak adhesive layer in composite restorations has resulted in continuous efforts to modify and improve it in order to increase retention.[1] Dental adhesives are classified into two different groups: In the etch-and-rinse group a separate etchant is used and rinsed away; in the self-etch group an acidic monomer is utilized to simultaneously demineralize and infiltrate the tooth structure.[2] From a clinical point of view, self-etch adhesives are more favorable and popular due to the fact that an additional rinse-and-dry step is not required which decreases the risk of surface contamination and clears up over- and under-drying problems. In addition, there is no need to replace cotton rolls when no rubber dam is used.[3]

In the recent decade, a more user-friendly one-step self-etch adhesive system has been marketed, which has resulted in simplification of the clinical procedure, especially proving beneficial when potentially uncooperative patients are involved.[3,4]

Hybridization is the term used to refer to alterations in the chemical composition of the substrate surface with the application of self-etch adhesives; the dentin/enamel surface layer is partially dissolved, with the resin penetrating into the porosities produced.[3,5] Loss of surface minerals is minimal compared to etching with phosphoric acid and the minerals removed are to some extent reincorporated into the bond during the curing process.[3]

Contrary to bonding to enamel which depends on the micromechanical interlocking with the demineralized substrate surface, bonding to dentin is achieved through hybridization with exposed collagen fibrils.[6] It has been reported that some monomers of phosphoric acid ester present in a number of enamel-dentin adhesives bond to the calcium present in hydroxyapatite structure, improving bond strength.[7]

From a theoretical viewpoint these adhesives combine the three functions of three-step adhesives: Etching, priming and bonding; therefore, both hydrophilic and hydrophobic monomers are blended and the solvent should have a relatively high concentration in order to preserve them in solution. Generally, in 'one-bottle' products, polar and nonpolar components have been blended together with water, acetone or ethanol as a solvent. In such a mixture, water is an essential ingredient to function as an ionization medium so that self-etching activity can take place. One-step self-etch adhesives function as semipermeable membranes because they are highly hydrophilic, allowing passage of fluids; as a result, bond longevity is severely jeopardized.[8]

However, water is a poor solvent for organic compounds, such as monomers which are rather hydrophobic. The problem is usually solved by incorporating a complementary solvent, such as ethanol and acetone; nevertheless, the higher the concentration of a cosolvent, the less the formation of protons.[9] As a result, in some adhesive systems, ethanol and/or acetone is also present to increase solubility of resin monomers. The presence of solvents in resins is absolutely necessary in the chemical composition of adhesives so that they can bond to dentin. The wet nature of dentin permits good wetting only when hydrophilic bonding is used.[9-11] Acetone is considered an appropriate solvent in adhesives combining hydrophobic and hydrophilic components. In a manner similar to ethanol, acetone and water produce an azeotrope. Acetone is generally incorporated into wet bonding etch-and-rinse systems to facilitate removal of water.[10,12]

In self-etch adhesives, despite the fact that the solvent preserves the ingredients in solution, after it is dispensed evaporation of the solvent initiates a phase-separation reaction and multiple droplets are formed, which can macroscopically be viewed as an uncured drop of adhesive solution that turns opaque. Microscopically, a large number of droplets are visible in the uncured adhesive. When the adhesive is cured before the separation reaction ends and thus before complete removal of droplets, the droplets are trapped within the adhesive layer.[13] As a result, some manufacturers have formulated some dental adhesives without conventional solvents, referred to as solvent-free adhesives.

In this study the bonding effectiveness and microscopical characteristics of a recently introduced user-friendly one-step self-etch adhesive, that is also a solvent-free adhesive, referred to as Bond 1SF (B1SF), was compared with Clearfil SE Bond (CSEB) adhesive, as control gold standard two-step self-etch adhesive.[3] The hypothesis tested was that the bond strength of B1SF to dental substrates is similar to that of CSEB as a commonly used gold standard adhesive.

MATERIALS AND METHODS

Shear Bond Strength Evaluation

Sixty sound extracted incisors, stored in 0.2% thymol solution in a refrigerator at 4°C for 6 months, were included in this study. The teeth were debrided of any remaining tissue tags and the crowns were cut at the CEJ using a low-speed diamond saw under water spray. The remaining coronal parts were mounted in acrylic molds. Thirty mid-coronal buccal enamel surfaces were ground using 400- and 600-grit papers for 30 seconds just before the bonding procedure. Thirty flat dentin surfaces were prepared by trimming and grinding using silicon carbide papers in the same manner as the enamel surfaces were prepared. The dentin surfaces were examined for the absence of enamel and/or pulp tissue exposure under a stereomicroscope. The specimens were equally divided into four groups (n = 15).

Groups 1 and 2: The teeth underwent a composite resin adhesion procedure on enamel surfaces based on manufacturer's instructions using a two-step self-etch adhesive (CSEB, Kuraray, Osaka, Japan) and a one-step self-etch adhesive (Bond 1SF, Pentron Clinical, Wallingford, USA), respectively (Table 1).

Groups 3 and 4: The same procedures were used as those in groups 1 and 2, but the composite resin adhesion procedures were carried out on dentin surfaces.

Z100 composite resin (3M ESPE, St Paul, MN, USA) was bonded to the surfaces using cylindrical molds with a height of 1 mm and a surface diameter of 2 mm and light-cured for 40 seconds with a conventional quarts halogen light-curing unit at a light intensity of 500 mW/cm^2. All the specimens were stored in distilled water for 24 hours at 37°C and then thermocycled for 500 cycles at 5/55°C. Shear bond strength (SBS) values were measured using a universal testing machine (DARTEC, HC10, Stourbridge, England) at a crosshead speed of 1 mm/min.

Data were analyzed using one-way ANOVA and a post hoc Tukey test ($\alpha = 0.05$). Fracture modes of RMGI cylinders on enamel and dentin surfaces were classified under a light microscope at ×16 as follows:

I. *Cohesive fracture:* Fracture in the RMGI or dental tissue
II. *Adhesive fracture:* Fracture in the adhesive interface
III. *Mixed fracture:* Adhesive/cohesive fracture (Table 3).

Interface Evaluation by Scanning Electron Microscopy

In each experimental group, two additional specimens were prepared for scanning electron microscopy (SEM) evaluation. After each tooth was prepared according to the method described above, the specimens were prepared by

Table 1: The adhesive resins used in the study, their compositions and manufacturers' instructions for use

Material	*Manufacturers' instructions*	*Composition*
Clearfil SE Bond (two-step, self-etch adhesive) Kuraray Co Ltd, Osaka, Japan pH: 1.9	Apply primer gently on the surface and leave undisturbed for 20 seconds. Gently air blow. Apply bond. Air thin and light cure for 10 seconds	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, N,N-diethanol-p-toluidine, water Bonding resin: 10-MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, CQ, N,N-diethanol p-toluidine, silanized colloidal silica
Bond 1®SF (one-step self-etch adhesive)(Penetron Clinical, USA) pH: 3-4	Scrub a layer of the bonding agent on the tooth's surface for 20 seconds Light cure for 10 seconds	UDMA, TEGDMA, HEMA, 4-MET, silane treated barium glass, silica (amorphous), photo curing system*

HEMA: Hydroxyethyl methacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA: Triethylene glycol dimethacrylate udma; 4-MET: 4-methacryloxyethyl trimellitic acid; UDMA: Urethane dimethacrylate Bis-GMA: Bisphenol A-glycidyl methacrylate; *All information is from manufacturer

Table 2: Bond strength of the specimens in the enamel/dentin groups in MPa ($p < 0.001$)

Bonding agent	*Substrate*	*Mean ± SD*	*95% CI*		*Minimum*	*Maximum*
			LB	*UB*		
CSEB	Enamel	42.52 ± 6.4^{a}	38.97	46.06	31.84	52.54
B1SF	Enamel	10.99 ± 1.58^{b}	10.11	11.86	8.59	14.33
CSEB	Dentin	27.15 ± 11.34^{a}	20.86	33.43	10.19	46.17
B1SF	Dentin	14.15 ± 1.53^{b}	13.30	15.00	11.78	16.58

CSEB: Clearfil SE Bond; B1SF: Bond 1 SF; CI: Confidence interval; LB: Lower bound; UB: Upper bound; Groups with the same superscript are not statistically different ($p > 0.05$); ^{a,b}Significant differences exist between groups.

Table 3: Different fracture modes in the study groups N (%)

Mode of fracture / *Groups*	*Adhesive*	*Cohesive*	*Mixed*	*Total*
(CSEB on enamel)^{a}	4 (26.5%)	4 (26.5%)	7 (47%)	15 (100%)
(B1SF on enamel)^{b}	7 (46.2%)	6 (40%)	2 (13.2%)	15 (100%)
(CSEB on dentin)^{a}	6 (40%)	3 (20%)	6 (40%)	15 (100%)
(B1SF on dentin)^{b}	9 (59.4%)	5 (34%)	1 (6.6%)	15 (100%)

CSEB: Clearfil SE Bond; B1SF: Bond 1 SF; Different superscript letters denote significant differences between similar groups; ^{a,b}Significant differences exist between groups

sectioning. The specimens were dehydrated in ascending concentrations of ethanol (50, 70, 95 and 100%) for 1 hour, embedded in acrylic resin molds and polished using abrasive papers of 400-, 600-, 800-, 1,200-, and 1,500-grit, respectively (Buehler Ltd. Lake Bluff, IL, USA) and 0.5 µ diamond paste (Buehler Ltd., Lake Bluff, IL, USA) using a polishing cloth. Between each polishing step, the specimens were placed in an ultrasonic device for 10 minutes. The exposed interfaces were treated with 6 N hydrochloric acid for 30 seconds followed by a 10-minute immersion in 2.5% sodium hypochlorite solution. Subsequent to a 10-minute ultrasonication procedure, the specimens were dehydrated for 24 hours, affixed to an aluminum mounting stub, and sputter coated with platinum-gold to a thickness of 10 nm for analysis under SEM. Different magnifications were used to prepare SEM images at a distance of 20 mm. An accelerating voltage of 15.0 kVp was used during the analysis.

RESULTS

Descriptive statistics of SBS values in MPa for the four groups under study are presented in Table 2. Bond strength of B1SF to enamel/dentin was significantly lower than that of CSEB ($p < 0.001$). Bond strength of CSEB to dentin was significantly higher than that to enamel ($p < 0.001$). There were no significant differences between bond strengths of B1SF to dentin and enamel surfaces ($p = 0.559$).

The fracture modes are summarized in Table 3. According to the results, the majority of adhesive fractures were observed in dentin groups with both adhesives.

SEM photomicrographs are shown in Figures 1 to 4 for enamel and dentin groups, respectively. As shown in Figures 3 and 4, the resin tag numbers and lengths seem to be much higher with CSEB. The resin tags seem to be less numerous and shorter with B1SF, which might be attributed to less opportunity of the adhesive to penetrate into the demineralized dentin. In Figures 1 and 2, proper adaptation is observed between the restorative material and enamel with both adhesives.

DISCUSSION

Bond strength tests are the most frequently used techniques to evaluate adhesive systems. The rationale behind this

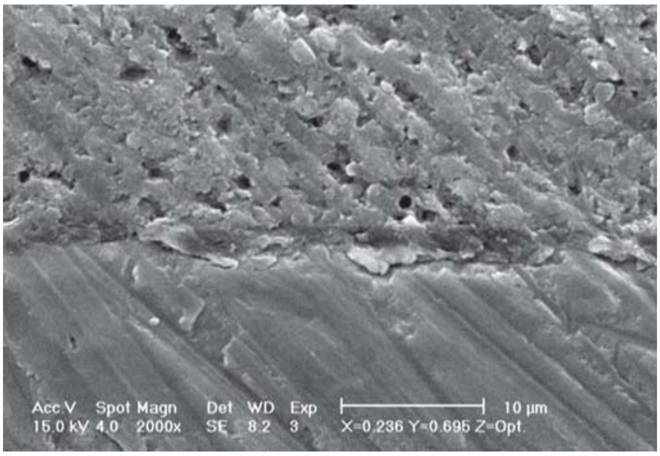

Fig. 1: Enamel/composite interfaces using B1SF as bonding agent

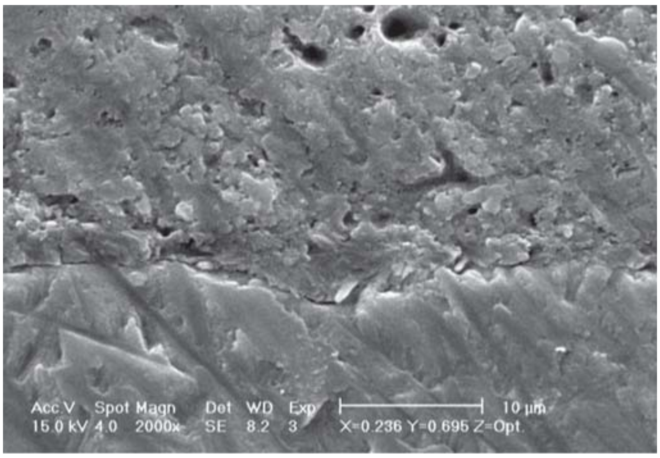

Fig. 2: Enamel/composite interfaces using CSEB as bonding agent

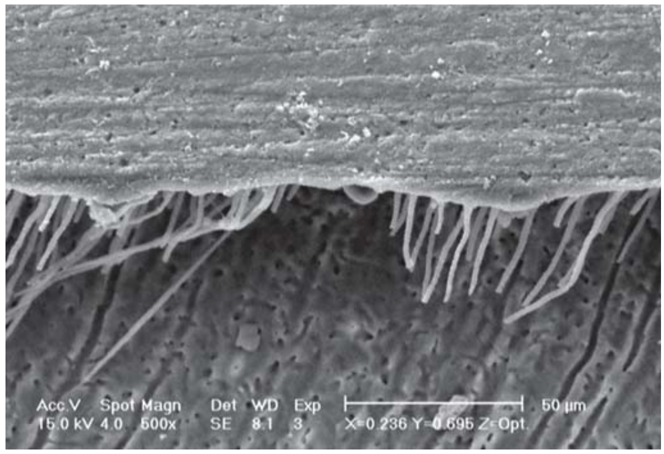

Fig. 3: Dentin/composite interfaces using B1SF as bonding agent

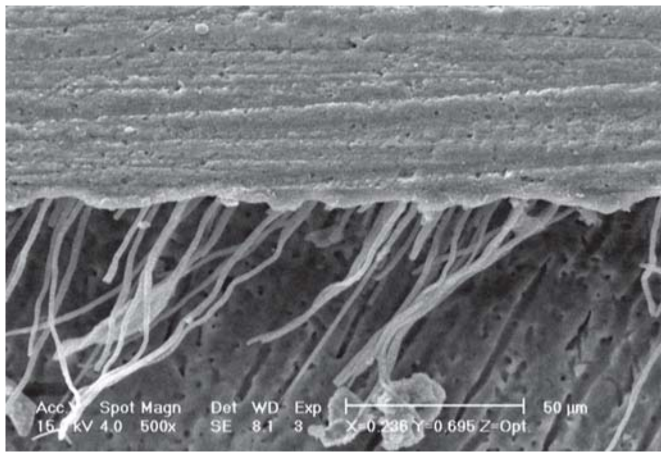

Fig. 4: Dentin/composite interfaces using CSEB as bonding agent

testing technique is that the stronger the adhesion between the tooth structure and the biomaterial is, the better it can resist stresses resulting from resin polymerization and oral functions.[8]

In this study, considering the adhesive system used, the SBS to enamel was lower than that to dentin for B1SF, which is consistent with the results of some previous studies.[13-15]

Generally, lower bond strength of dental adhesives to dentin might be attributed to various factors: First, dentin is less mineralized and has higher water content compared to enamel. Second, presence of the smear layer makes it very difficult to make dentin wet. Third, fluid-filled channels in dentin that are under slight, but constant, outward pressure from the pulp decrease the stability and durability of the composite resin-dentin bond.[16]

Solvent-free adhesive evaluated in this study might has etched the enamel less effectively compared with dentin, resulting in incomplete penetration of the adhesive resin, creating a more heterogeneous interdiffusion zone and reducing bond strengths. Another hypothesis is that the reprecipitation of calcium phosphates might take place in partially demineralized interfibrillar spaces, interfering with the penetration of the adhesive.[13] The self-etch systems do not create a deep etching pattern and form resin tags that are probably not adequately strong to resist thermal stresses. This hypothesis is supported by SEM evaluation of resin-enamel and adhesive substrates that were not present before the thermocycling procedure.[8,15]

The results of the present study showed a better bonding performance of CSEB compared to that of B1SF. The superior performance of two-step self-etch systems in comparison to one-step self-etch systems might be attributed to the lower pH value of organic solvents, low concentration of the solvent and its low hydrophilicity. Limited etching and demineralization of the underlying dentin over longer periods of time and greater degree of polymerization and thickness of the solvent are the reasons for these outcomes.[16,17] Several previous studies have shown similar results in terms of better performance of two-step self-etching adhesives bonded to enamel/dentin compared to that of one-step self-etching adhesives,[2,3,8,11] confirming the results of the present study.

A clear correlation between the pH value of self-etch primers and the depth of interaction with dentin has been reported. In this study, CSEB and B1SF were used as two self-etch adhesives with pH values of 1.9 and ~3 to 4, respectively. Four categories can be distinguished for self-etch adhesives: First, strong self-etch adhesives with a pH value lower than 1 and interfacial micromorphologies (3-4 µm deep fully demineralized hybrid layers) very similar to those with etch-and-rinse adhesives; second, intermediately strong self-etch adhesives with a pH value of around 1.5. These adhesives have a hybrid layer of about 1 to 2 µm, with some hydroxyapatite preserved at the bottom part; third, mild self-etch adhesives with a pH value around 2. The hybrid layer is less than 1 µm thick and is only partially demineralized; fourth, recently, a category of ultra-mild self-etch adhesives have been incorporated into self-etch adhesive that are available with a primer pH value higher than 2.5. These adhesive do not eliminate the smear layer and interact with the smear layer-covered dentin only up to a few hundredths of a nanometer. The pH of CSEB used in this study was 1.9;[8] therefore, it belonged to the category of mild self-etch adhesives. The pH of B1SF used in this study was 3 to 4, belonging to the category of ultra-mild adhesives. B1SF from this viewpoint is in the category 4. Although the SEM photomicrographs of this adhesive show a penetration of almost 5 to 30 micrometers in the dentin, the length and numbers of resin tags are noticeable. Clinically, an adhesive that can bond to any surface is preferred, as can mild self-etch adhesives.[17]

B1SF is a solvent-free adhesive but water is present in CSEB as a solvent. As a result of the one-component nature of the all-in-one adhesives, they usually give rise to very thin hybrid and bonding layers. From an esthetic viewpoint this can be advantageous because only the composite resin will be optically detectable, which might be attributed to oxygen inhibition that can compromise polymerization of a thin layer.[18] On the other hand, CSEB contains the functional monomer of 10-MDP, which enables an intensive and stable chemical bond with hydroxyapatite, which is left around the collagen fibrils within the hybrid layer. Such a primary chemical interaction improves the hydrolytic stability; therefore, clinically the restoration margins remain sealed for a longer period.[3] B1SF does not have conventional solvents in its chemical composition, which results in a thicker adhesive layer. The authors assumed that this increases the hydrophilic content in B1SF in comparison to CSEB, and as a result of their high hydrophilicity, these adhesives behave as semipermeable membranes, allowing more fluids to pass through in comparison to CSEB, which seems to leads to lower bond strength by one-step self-etch adhesives.[16] Studies have shown that this water acts as a major factor interfering with polymerization, leading to unpolymerized acidic and aggressive monomers to continue etching the dentin, thereby resulting in a detrimental effect on the bond.[8,16] Similar results with two-step self-etch systems have shown a superior *in vitro* performance in relation to µSBS compared to one-step self-etch systems in recent studies;[3,7] in contrast, one study has shown no difference in clinical performance after a year between water-based, ethanol and solvent-free adhesives, nor between two-bottle and one-bottle adhesives, all of which are etch-and-rinse systems,[1] necessitating more investigations.

In this study, B1SF was used as a solvent-free adhesive because it does not contain water, alcohol or acetone as a conventional solvent in its chemical composition. However, the adhesive contains 2-hydroxyethyl methacrylate (HEMA), a low molecular weight monomer, as a solvent. Despite its high allergenic potential, HEMA is frequently used in adhesives due to its positive influence on the bond strength. Furthermore, presence of HEMA in one-component one-step adhesives can prevent phase separation. A small amount of HEMA (10%) improved the bond strength of a one-step self-etch adhesive.[9,19] Its polar properties and small dimensions enhance the wetting properties of the adhesive solution.[13,20,21] When HEMA is added in higher concentrations, its beneficial effect on bond strength is compromised due to increased osmosis, resulting in the production of a large number of droplets, reduced polymerization conversion rate, and suboptimal physicomechanical properties of the resultant poly-HEMA containing adhesive interface.[13] It appears HEMA plays the role of a solvent in B1SF. The authors assumed that high concentrations of the material in the adhesive result in lower conversion rate and consequently, lower bond strength; however, further investigations are necessary.

The results of this study, in relation to the failure of two adhesives in the groups under study, showed a higher rate of adhesive failures in the two enamel and dentin substrates in groups 2 and 4 (B1SF groups), consistent with the numerical bond strength values. In addition, in the present study, given the limitations of SEM, the photomicrographs prepared in the groups under study were not appropriate to extend the results to bond strength results. Meticulous TEM evaluations will be useful in this respect and are highly recommended.

CONCLUSION

Under the limitations of the present study it was concluded that the solvent-free one-step self-etch adhesive B1SF when use for bonding composite resin to enamel and dentin, underperforms as compared to two-step self-etch CSEB as the control gold standard.

CLINICAL SIGNIFICANCE

When bonded to enamel and dentin the solvent-free adhesive B1SF underperforms as compared to CSEB as the control gold standard.

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