Original Article

Marginal Microleakage and Morphological Characteristics of a Solvent-Free One-Step Self-Etch Adhesive (B1SF)

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Abstract

Objective: In recent years, newly developed solvent-free dental adhesives have been introduced. The aim of this study was to evaluate the marginal integrity of a new one-step solvent-free self-etch adhesive and to compare it with a commonly used two-step self-etch adhesive as the gold standard.

Materials and Methods: Class V cavities (2×4×1.5 mm) were prepared on the buccal aspects of 28 human premolars. The cervical margins of the cavity preparations were placed 1 mm apical to the CEJ. Clearfil SE Bond (CSEB) (two-step self-etch adhesive) and Bond 1SF (B1SF) (one-step self-etch adhesive) were applied to the cavities in groups 1 and 2 (n=14), respectively. Then, the specimens were restored with A2 shade of APX composite resin. Each group was evaluated for dye penetration under a stereomicroscope at ×32 after 24 hours and 500 rounds of thermocycling. Statistical analyses were carried out using Mann Whitney test ($\alpha=0.05$). In addition, in each experimental group, two specimens were prepared for analysis under SEM.

Results: There were no significant differences in enamel margin microleakage between the two adhesives used ($P=0.24$); whereas, there were significant differences in dentin margin microleakage between CSEB and B1SF ($P=0.004$). Dentin microleakage of B1SF was higher than that of CSEB.

Conclusion: Results showed that the enamel marginal integrity of B1SF as a newly developed one-step solvent-free self-etch adhesive was similar to that of CSEB as a commonly used two-step self-etch; however, dentinal sealing of CSEB was better than that of B1SF.

Key Words: Composite Resins; Enamel; Dentin; Microleakage; Solvents

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INTRODUCTION

The principal aim of dental adhesives is to provide retention for composite resin-based restorative materials. In addition to provision of resistance against mechanical forces, and in particular polymerization shrinkage stress from the lining composite, a good adhesive should preclude microleakage along the margins of the restoration. From a clinical standpoint, restoration failure occurs more often as a result of inadequate sealing, leading to discoloration of the cavity margins, than due to retention failure [1,2].

In the recent years, the etching step of dental structures has been merged with the priming and bonding steps, giving rise to the self-etch strategy to the satisfaction of dental practitioners worldwide because self-etch adhesives are easy to apply [3,4]. In one-bottle self-etch adhesives, the etchant, the primer and the adhesive resin have been incorporated into one bottle, resulting in single-step application, which allows simultaneous etching and priming of tooth structures with one adhesive component. Previous studies have attributed unfavorable bond defects, including nanoleakage, water trees, bubbles, and phase separation at adhesive resin-tooth interfaces of all-in-one and one-bottle adhesives to their high water content [5,6]. Hydrophilic bonding resins facilitate water absorption, resulting in the replacement of hydrophilic resin monomers even after curing; therefore, hydrolytic degradation takes place in the long run [6-8].

The self-etching ability is a result of incorporation of water into resin monomers, enabling ionization of acidic monomers. In addition to the water in the compounds, the ionizable components of acidic monomers possess hydrophilic properties [8]. Existence of a hydrophilic layer might lead to water sorption and uptake, plasticizing the polymer network [9-11].

Traditionally, it was mandatory to incorporate solvents into the composition of adhesive resins that were to bond to dentin [12]. The wetting properties of the adhesive improve significantly by incorporating hydrophilic monomers and a solvent [13]. Low viscosity of primers and/or adhesive resins is partly attributed to the dissolution of monomers in a solvent, which promotes its diffusion in the microretentive tooth surface. In self-etch adhesive systems, the main function of the solvent, within the combined primer-adhesive resin (one-bottle systems), is to facilitate penetration of monomers into the collagen network of demineralized dentin [14]. When bonding is carried out to air-dried dentin, the solvent should have the capacity to re-expand the collapsed network [15,16].

One-bottle self-etching adhesive systems have the disadvantage of phase separation of the adhesive or micro-size bubbles in the bonding resin layer [5,6]. High water sorption is a characteristic of hydrophilic one-bottle adhesives which have high solvent contents. High water sorption significantly decreases the bond strength in wet-dentin compared to dry bonding procedures [8].

Since such one-bottle adhesives merge the three functions of three-step adhesives – etching, priming and bonding – both hydrophilic and hydrophobic monomers are blended, necessitating a rather high concentration of the solvent to keep them in solution. As a matter of fact, in one-bottle solutions, polar and nonpolar components are mixed together with a solvent of water, acetone and ethanol. In such a bizarre mixture, water is basically essential as an ionization medium to allow self-etching activity. Due to their high hydrophilic properties, one-step self-etch adhesives serve as semi-permeable membranes, through which fluids pass and contribute to bond failure [8,17].

Recently, a typical type of degradation of one-bottle self-etch adhesives was reported at the adhesive-composite resin interface [18]. Large amounts of water and/or solvent decrease viscosity, leading to transport of oxygen to the surface of the cured adhesive layer and the deep uncured layer with the use of one-bottle adhesives, which might be more severe than that with hydrophobic adhesives. In addition, the remaining monomer around the filler adjacent to inadequately polymerized monomers serves as a pathway for the environmental water to penetrate into the bulk polymer, resulting in hydrolysis of the filler-adhesive interface after aging. Inadequate polymerization of this hydrophilic polymer results in rapid deterioration by environmental water; therefore, it is susceptible to interfacial attack by water [8]. Therefore, water is not a proper solvent for organic compounds (such as monomers), because they are usually relatively hydrophobic. The difficulty can be resolved by incorporating a secondary solvent, such as ethanol and acetone. Consequently, in some adhesive systems, ethanol and/or acetone is also incorporated to increase solubility of resin monomers. However, as more co-solvent is incorporated, the number of protons available decreases [3].

In self-etch adhesives, the solvent keeps the ingredients in solution; however, once the adhesive is applied, the solvent evaporates, triggering a phase-separation reaction, as a result of which numerous droplets are formed. If the curing process of the adhesive is accomplished before the separation reaction is terminated, i.e. before the droplets are completely removed, the droplets remain in the adhesive layer [5]. Moreover, if the solvent in the bonding system is entrapped beneath the hybrid layer, the bond will have a poor quality. If dentin demineralization depth increases, more solvent will probably be entrapped. On the other hand, some studies have shown that strength and durability of the bond depend on hybrid layer quality (i.e. on the quality of dentin preparation and conditioning) rather than on the thickness or morphologic characteristics of the hybrid layer/resin tags [19].

Several clinical techniques have been reported to improve infiltration of monomers, to decrease water sorption and to reduce degradation of collagen [20], one of which is to produce adhesives devoid of water, ethanol or acetone as conventional solvents referred to as solvent-free adhesives. Therefore, the purpose of this study was to evaluate marginal sealing and microscopic characteristics of Bond 1SF (B1SF), a recently introduced easy-to-use one-step self-etch adhesive, which is also referred to as a solvent-free adhesive and to compare it with Clearfil SE Bond (CSEB) adhesive, as a control gold standard self-etch adhesive [21]. The hypothesis was that the marginal sealability of B1SF to dentin is similar to that of CSEB.

MATERIALS AND METHODS

Marginal seal evaluation by dye penetration technique

In this laboratory study, 28 sound human premolars, with no carious lesions, restorations, abrasions and cracks were selected. The teeth had been extracted less than 4 months previously. The teeth were cleansed with a brush after all the periodontal fibers and bone remnants were removed and stored in 0.1% thymol solution at 4ºC. The premolar samples were immersed in distilled water at 37ºC for 24 hours. Then, Cl V cavities were prepared on the buccal aspects. The cavities were 1.5 mm deep and 4 and 2 mm in the mesiodistal and occlusogingival dimensions, respectively. The cavities were prepared by using seven diamond fissure burs (D&Z, Hilzingen, Germany), which measured 1 mm in diameter, with one new bur for every four cavities. The occlusal margin of each cavity was placed on the enamel with the gingival margin being placed 1 mm below the CEJ. Two-step self-etch CSEB adhesive were applied for half of the cavities and one-step self-etch solvent-free B1SF adhesive for the other half following the manufacturers' instructions (Table 1). A2 shade of APX composite resin (APX, Kuraray, Tokyo, Japan) was used to fill the cavities with the application of the incremental technique.

After the cavities were restored, the specimens were stored in an incubator (Behdad, Tehran, Iran) for 24 hours in distilled water at 37ºC to decrease the stresses resulting from polymerization. Subsequently, the restorations were polished with the use of flame-shaped polishing burs, polishing disks (3M ESPE, St. Paul, MN, USA) and cup-shaped polishing rubbers from coarse to fine. The specimens in both groups (n=14) were prepared for microleakage evaluation after a thermocycling procedure consisting of 1000 rounds.

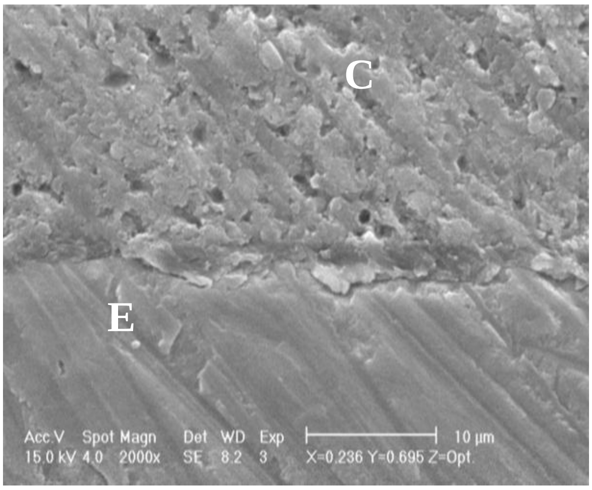

Fig 1. Enamel/composite interfaces using B1SF as bonding agent. C: Composite resin; E: Enamel; Original magnification 2000×

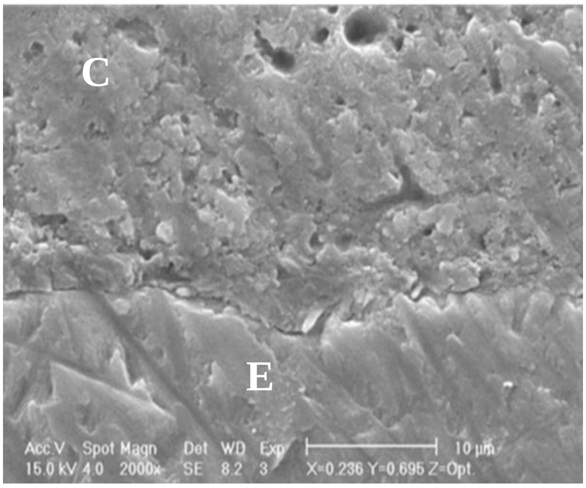

Fig 2. Enamel/composite interfaces using CSEB as bonding agent. C: Composite resin; E: Enamel; Original magnification 2000×

Specimens from all the groups were thermocycled between 5°C and 55°C for 1000 cycles (Mp Based, KARA 1000, Tehran, Iran) with a 30-second dwell time and a 12-second transfer time. The apices of the teeth were sealed with sticky wax and all tooth surfaces were coated with three layers of nail varnish except for a 1-mm zone around the cavities to evaluate microleakage. Then all the specimens were immersed in 2% basic fuschin solution and incubated for 24 hours at 37ºC. In order to facilitate the cutting procedures the specimens were embedded in self-cured acrylic resin. Then a diamond disk (Lemgo, Germany) was used to section the specimens buccolingually parallel to tooth long axis by using a cutting machine. Each specimen was scored for dye penetration under a stereomicroscope (MBC-10, St. Petersburg, Russia) at a magnification of ×16. Dye penetration was scored as follows [22]:

1- Absence of dye penetration
2- Dye penetration up to one-third of the cavity depth
3- Dye penetration up to two-thirds of the cavity depth
4- Dye penetration up to more than two-thirds of the cavity depth toward the pulp

Data were analyzed with Mann-Whitney test at significant level of 0.95 using SPSS 11.5 statistical software.

Scanning electron microscopy (SEM) evaluation

Two additional specimens were prepared for SEM evaluation in each group. After each tooth was prepared in the method described above, the specimens were sectioned. The specimens were placed in ascending concentrations of ethanol (50- 70- 95- 100%) for 1 hour for dehydration, embedded in acrylic resin and polished using 400-, 600-, 800-, 1200-, and 1500-grit abrasive papers (Buehler Ltd., Lake Bluff, IL, USA), respectively, and 0.5-µ diamond paste (Buehler Ltd., Lake Bluff, IL, USA) using a polishing cloth. The specimens were placed in an ultrasonic bath for 15 minutes between polishing steps. The exposed interfaces were subjected to 6N hydrochloric acid for 30 seconds and immersed in 2.5% sodium hypochlorite for 10 minutes. After 10 minutes of ultrasonication, the specimens were dehydrated for 24 hours, mounted on an aluminum stub and sputter-coated with platinum-gold to a thickness of 10 nm in preparation for analysis under SEM. SEM images were provided at a distance of 20 mm under different

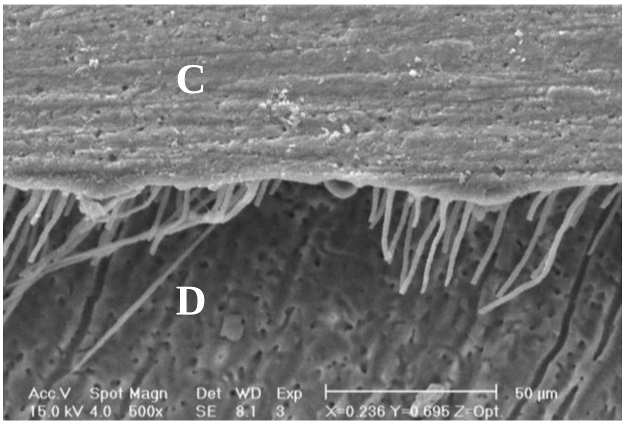

Fig 3. Dentin/composite interfaces using B1SF as bonding agent.C: Composite resin; D: Dentin; Original magnification 500×

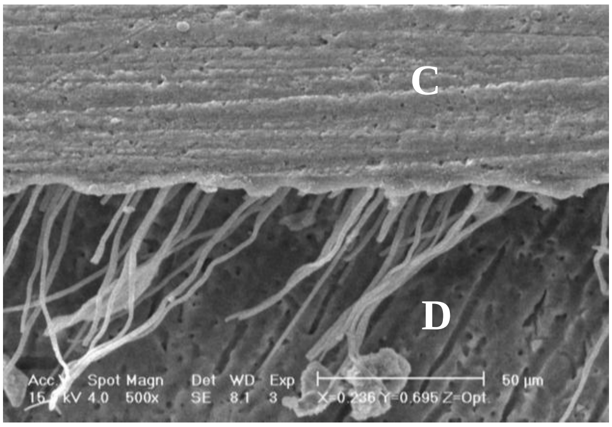

Fig 4. Dentin/composite interfaces using CSEB as-bonding agent. C: Composite resin; D: Dentin; Original magnification 500×

magnifications. An accelerating voltage of 15.0 kV was used for SEM analysis.

RESULT

Tables 2 presents the microleakage scores and mean rank of microleakage for the four experimental groups, respectively. Kruskal-Wallis test did not show any significant differences in the means of enamel microleakage scores between the two adhesives (P=0.24) (Figures 1 and 2). However, significant differences were observed in the microleakage scores of the two adhesives at dentin margins (P=0.004). The CSEB group exhibited significantly lower microleakage scores at dentin margins (Table 2) (Figures 3 and 4) than the other group.

Furthermore, SEM photomicrographs are presented in Figures 1-4 for enamel and dentin margins, respectively. In Figures 1 and 2, good adaptation is observed between the restorative material and enamel for both adhesives.

Table 1. 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, silanated colloidal silica
Bond 1®SF (one-step self-etch adhesive) (Pentron 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: Hydroxyl ethyl methacrylate;10-MDP: 10- Methacryloyloxydodecyl dihydrogen phosphate ; TEGDMA: Triethylen glycol dimethacrylateudma ;4-MET: 4-methacryloxyethyltrimellitic acid,UDMA: Urethane dimethacrylate Bis-GMA: Bisphenol A-Glycidyl methacrylate; CQ: Comphorquinone.

As shown in Figures 3 and 4, the resin tags seem to be longer and more numerous with CSEB; the resin tags seem to be less numerous and shorter with B1SF.

DISCUSSION

In the present study, two self-etch adhesives were evaluated. Self-etch dental adhesives are a complex mixture of ingredients, consisting of reactive monomers, a blend of dissolved hydrophilic and hydrophobic monomers, cross-linkers, initiators and solvents. Dentin consists of 10% water, which is necessary for ionization of functional groups of acid monomers. Dissociation of dental hydroxyapatite involves the release of calcium ions into a water-based formulation in which they are soluble. Decalcification, an ionic process, takes place in a highly polar milieu. Organic molecules with low polarity are present in adhesives and form a homogeneous phase with some other suitable co-solvents, including acetone, ethanol and butanol [23,24].

Presence of solvents causes concerns regarding evaporation of solvent and the effects it might exert on monomer infiltration and polymerization [3]. Incomplete polymerization and permeability of the adhesive are more widespread in simplified adhesives, including both two-step etch-and-rinse and one-step self-etch ones, which might be attributed to the presence of high concentrations of hydrophilic monomers. Since incompletely cured adhesives exhibit greater permeability to fluids [20], they might accelerate water sorption and compromise the longevity of the adhesive-composite resin bond. In contrast, dentin bonding systems consisting of separated non-solvated hydrophobic bonding agents exhibit greater polymerization rates, due to less permeability to water [20]. Various techniques have been suggested to improve monomer infiltration, to decrease water sorption and to decrease degradation of collagen. One of these techniques is to manufacture adhesives that do not contain water, ethanol or acetone as conventional solvents, referred to as solvent-free adhesives.

In the present study, B1SF, a solvent-free adhesive, was evaluated. The most commonly used adhesives in solvents are water, ethanol and acetone. Other polyvalent alcohol solvents have also been evaluated, but are not used commercially [25].

Use of these organic solvents in adhesives is justified by their low cost, availability and biocompatibility. The most important properties of a good solvent are its dipole moment, dielectric constant, boiling point, vapor pressure and H-bonding capacity.

The vapor pressure of a solvent ensures good evaporation of the solvent after the adhesive is applied to tooth structures [26]. Air-drying of the adhesive after it is applied facilitates removal of the remaining solvent from the adhesive [27].

Table 2. Frequency of Microleakage Scores in the Enamel/Dentin Margins for the Study Groups

Groups Numbers and Adhesive Resins Used	Substrate	Scores				
		0	1	2	3	Total
1. CSEB	enamel	7 50%	5 35.8%	1 7.1%	1 7.1%	14 100%
2. B1SF	enamel	3 21.4%	7 50%	3 21.4%	1 7.2%	14 100%
1. CSEB	dentin	10 71.5%	0 0%	1 7.1%	3 21.4%	14 100%
2. B1SF	dentin	0 0%	3 21.4%	4 28.6%	7 50%	14 100%

CSEB*: Clearfil SE Bond;* B1SF*: Bond 1 SF*

In addition, air-drying reduces the thickness of the adhesive layer, which encourages further solvent removal [28]. However, it is difficult to completely evaporate the solvent; it is hampered by the short air-blowing time [3]. Remaining of the solvent in the adhesive might interfere with polymerization because the monomers are diluted, resulting in voids and permeability of the adhesive layer [29]. The aim of air-blowing in solvent-free adhesive resins is not to evaporate the solvent, but rather to produce a uniform adhesive. However, in this study, the authors did not observe any improvement in the dentinal microleakage using the studied solvent-free adhesive.

In this study, B1SF was used as a solvent-free adhesive because it does not contain water, alcohol or acetone as conventional solvent compositions. However, it is noteworthy to know that MMA and HEMA, both small monomer compounds have also been described as diluents for other monomers; as a result, they can be called solvents [3]. B1SF contains 2-hydroxyethyl methacrylate (HEMA), a low molecular-weight monomer, as a solvent. Despite its high allergic potential, HEMA is frequently used in adhesives due to its positive effect on bond strength. In addition, incorporation of HEMA into one-component one-step adhesives can prevent phase separation. It has been reported that 10% HEMA improves the bond strength of a one-step self-etch adhesive. Incorporated in higher concentrations, this positive effect of HEMA on the bond strength is lost due to increased osmosis, production of many droplets, reduced conversion rate and sub-optimal physico-mechanical properties of the resultant poly-HEMA-containing adhesive interface. Its polarity and small dimensions improve the wetting properties of the adhesive solution [5] and the penetration efficacy of the adhesive into demineralized dentin. However, HEMA has a limited H-bonding capacity [5]. It seems HEMA plays the role of a solvent and diluent in B1SF. Therefore, the odds of phase separation and droplet entrapment in the adhesive decrease by elimination of water and the two other conventional solvents. However, it is probable that the disadvantages of HEMA, especially limited H-bonding capacity and limitations in cross-linking, result in less marginal integrity at dentin margins.

Due to its low molecular weight, HEMA has a great role in preventing phase-separation reactions in one-component one-step self-etch adhesives. However, the acidic monomers in the composition of self-etch adhesives might also contribute to the wetting properties of adhesives; there is less need for HEMA in self-etch adhesives. Nevertheless, the results of this study indicate that solvents are beneficial for the dentin margin integrity of self-etch adhesives [5]. In this study, as seen in Figure 4, the numbers and lengths of resin tags in the B1SF group were interestingly high. Contrarily, the resin tags seem to be less numerous and shorter with B1SF (Figure 3), which might be attributed to less opportunity of the adhesive to penetrate into the demineralized dentin.

Some previous studies have shown that the thickness of a hybrid layer is not critically important for a reliable bond [4]. Dentin bond strength might be dependent on the interlocking between resin and collagen, as well as on the quality of the hybrid layer, rather than on its thickness [30]. The hybrid layer is formed concomitant with the penetration of primer into the fluid-filled dentinal tubules, generating rather large resin tags. However, these appear to be of little value to the overall bonding process. The rationale behind this is the fact that this material is generally undercured and behaves as soft flexible tags. If dentin is dehydrated before priming and bonding, these resin tags will probably be quite extensive [30].

In CSEB, a two-step self-etch adhesive, the primer contains water as a solvent to displace water and carry the monomers into the microporosities in the collagen network. During primer application, most of the solvent evaprates quickly. Therefore, several layers should be applied to ensure thorough conditioning.

Recently, some adhesives with new chemical components, such as tert-butanol, have been marketed due to their similar vapor pressure as ethanol, but better stability towards chemical reaction with monomers [31]. These adhesives have yielded acceptable initial results, but further investigations are necessary.

CONCLUSION

Under the limitations of this study, enamel microleakage of B1SF as a one-step solvent-free self-etch adhesive was similar to that of CSEB as a commonly used two-step self-etch adhesive; however, dentinal sealing of CSEB was better than that of B1SF.

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