

Shear Bond Strength of Two Self-Etch Adhesives to Permanent and Deciduous Dental Enamels: Effect of Acid Preconditioning

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Abstract

Background and Aim: There are some studies that strongly recommend acid conditioning before applying the self-etch adhesives. The aim of this in vitro study was to compare the shear bond strength (SBS) of two self-etch adhesives to the enamel of primary and permanent teeth with acid preconditioning.

Materials and Methods: The buccal surfaces of 48 permanent molars and 48 primary molar teeth were prepared for bonding of the adhesives to the enamel. Then the samples were randomly divided into eight groups, each containing 12. According to the manufacturers' instructions, Clearfil SE Bond (CSEB) and ACE All Bond SE (ABSE) self-etch adhesives were applied on the enamel and the etched enamel of both permanent and primary teeth and bonded with composite resin. After 24 hours of storage in 37°C and 500 rounds of thermocycling, the specimens were tested in a shear at a crosshead speed of 1.0 mm/min. The results were statistically analysed using SPSS 11.5 with Kruskal-Wallis and t-test at a significance level of 0.05.

Results: The mean±SD in permanent enamels using CSEB and ABSE were 16.10±7.96 and 8.49±2.34 MPa, respectively. The same data for the deciduous enamels were 16.08±3.91 and 9.23±3.20 MPa, respectively. After acid etching, SBS for permanent enamel using CSEB and ABSE were 32.05±7.13 and 31.39±6.51 MPa, respectively. The same data for deciduous enamels were 24.73±10.74 and 21.70±6.18 MPa, respectively.

Conclusion: The bond strength of CSEB was significantly higher than ABSE adhesive in both primary and permanent teeth. In addition, acid etching leads to increase in the enamel bond strength of both studied adhesives.

Key Words: Bond strength, Self-etch adhesive, Acid etching, Enamel, Permanent teeth, Primary teeth

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Introduction

Nowadays in pediatric dentistry there has been an increase in the demand for tooth-colored restorations [1]. Composite restorations are more technique-sensitive and a high degree of fracture

has been reported in deciduous teeth [2-4], which may be related to lack of cooperation in the child leading to loss of isolation or decrease in bond continuity and increase in marginal microleakage of the restoration [5]. Adhesive systems have

been constructed to produce effective adhesion to the dental tissue. Recently dental adhesives have been demonstrated in various systems for composite resin adhesion [6]. Self-etch adhesive systems combine etching and priming into one step; therefore, these systems result in simplification of clinical stages, decrease of technical sensitivity and increase in work speed [7]. In addition, usage of rubber dam for children, especially infants, is not always possible and the unpleasant taste of the etching material at the time of rinsing provokes the gag reflex subsequently leading to contamination of the prepared tooth surface with saliva. As a result, most of clinicians prefer applying of self-etch adhesives [8].

Clearfil SE Bond (CSEB) is a type of two-step self-etch adhesive which is used in esthetic prosthesis and has had satisfactory laboratory and clinical results. ACE all bond SE (ABSE) is a type of one-step two-component self-etch adhesive which has been introduced by Bisco manufacturer in 2009 [9]. This adhesive is an ethanol/water based and water that bonds to the etched dentin and enamel and according to the manufacturer's claim, is compatible with resin based cements, light-cure, self-cure and dual-cure composites. Studies have stated that self-etch adhesives that consist of acid monomers, especially one bottle self-etch adhesives, lead to separation phase. ABSE is a type of one-step, but two-component self-etch adhesive which according to the manufacturer's claim is more stable in comparison to one-component adhesives. In a study conducted by the manufacturer, the shear bond strength of this adhesive was higher than the other sixth and seventh generation self-etch adhesives [9].

In a three-year clinical study, Ermis *et al.* have evaluated the effect of acid phosphoric etching on the marginal enamel of permanent teeth using a two-step self-etch adhesive Clearfil SE Bond. They found no significant difference regarding adhesion and integrity of the restoration. Therefore, they stated that etching of the enamel

margins leads to marginal quality improvement [10]. Erickson *et al.* compared the strength of four self-etch adhesive systems; namely, Clearfil SE Bond, Xeno IV, Adper Prompt L-Pop and Clearfil SE Bond with an etch adhesive system and Adper Single Bond Plus rinsing on acid-etched enamel and polished enamel. They noted that acid etching of the enamel before usage of self-etch adhesive may increase the bond strength to the extent of etch- and- rinse systems and may also cause improve in clinical outcomes [11].

Stalin *et al.* (2005) compared the bond strength of fifth and sixth generation adhesives in deciduous teeth. The results showed that self-etch adhesives are more appropriate for deciduous teeth [12]. On the contrary, Frankenberger *et al.* (2008) reported a study in which acid phosphoric bond etching to the enamel is more efficient and also bonding to the enamel by two-step self-etching using acid phosphoric improves the situation [13].

However, there has been different points of view regarding the bond strength of self-etch adhesive systems to the enamel of deciduous and permanent teeth and also the use of acid preconditioning. There is not much information about the bond strength of newer self-etch adhesive systems in the literature. In addition, manufacturers do not represent separate instructions for deciduous and permanent teeth. Therefore, the aim of this study was to compare the use of two kind of one- and two-step self-etch adhesives on the shear bond strength of resin composites to permanent and deciduous teeth in condition of acid phosphoric preconditioning.

Materials and Methods

This experimental study was an *in vitro* study performed on 48 normal permanent human third molars and 48 normal deciduous human molars. The samples were extracted maximum 6 months prior to the study and were embedded in normal saline. 24 hours before the study, the samples were cleaned and embedded in 4°C thymol 0.2%

in the refrigerator. In both deciduous and permanent teeth, the buccal surface was used as the bond surface. After separating the root, all the samples were mounted in self-cure acrylic resin (Acropars, Marlic Medical, Tehran, Iran). For preparation of the enamel surfaces for bonding, the enamel of the buccal surface was freshend by a high speed diamond bur (D&Z, Diamate, Germany). Then all the enamel surfaces were freshend by 400 and 600 grit silicon carbide papers (Soft-flex, Germany). The samples were randomly divided into four separate groups. (Table 1)

Table1. Application of Two Evaluated Adhesives on the Enamel Surface of Deciduous and Permanent Teeth

Groups	Deciduous and Permanent Teeth
Group 1	CSBE application on enamel
Group 2	CSEB application on etched enamel
Group 3	ABSE application on enamel
Group 4	ABSE application on etched enamel

Subsequently, every studied self-etch adhesive (Table 2) according to the manufacturer's instructions was placed on the prepared enamel surface in groups 1 and 3 and also etched surfaces with acid phosphoric in groups 2 and 4 and were finally cured (Tables 1 and 2). For placement of the one-colored APX composite (A3 Kuraray Co., Osaka, Japan) plastic tubes with an internal diameter of 2 mm were used and the samples were hardened for 60 seconds by a light cure unit (LED Unifive Co, China). Before the curing and throughout the study as the light intensity of the machine should be set on at least 600 mw/sec, the power of the LED machine was measured by a radiometer (Kerr, Orange, CA, USA). All the samples were placed in 37°C in sterile water for 24 hours in the incubator (Behdad, Tehran, Iran) and subsequently thermocycled (MP Based, Kara 1000 CO, Tehran, Iran) for 500 cycles of 5-55°C in a way that before shear bond strength evaluation they

were placed in cold water for 30 seconds and then warm water for 30 seconds and transfer time was 10 seconds. The shear bond strength of the samples was measured by a universal testing machine (DARTEC, HC10, England) with a speed of 1 mm/min and a blade parallel to the surface between the adhesive and the tooth. Data analysis was carried out by SPSS 11.5. with Kruskal-Wallis and t-tests. The fractures mode were evaluated by stereomicroscope (MBC-10, St. Petersburg, Russia) and the fractures were classified as adhesive fracture, cohesive fracture and mixed fracture.

Results

The mean and standard deviation obtained in permanent teeth for CSEB and ABSE adhesives were 16.10 ± 7.96 MPa and 8.49 ± 2.34 MPa, respectively and for deciduous teeth these figures were 16.08 ± 3.91 MPa and 9.23 ± 3.20 MPa, respectively. After applying acid phosphoric, these figures were 32.05 ± 7.13 MPa for CSEB and 31.39 ± 6.51 MPa for ABSE in permanent teeth and 24.73 ± 10.74 MPa for CSEB and 21.70 ± 6.18 MPa for ABSE in deciduous teeth (Table 3). The CSEB two-step self-etch adhesive has showed a higher primary bond strength in comparison to ABSE one-step self-etch in permanent ($p=0.007$) and deciduous ($p=0.001$) teeth. The studied adhesives did not produce a higher bond strength in permanent teeth in comparison to deciduous teeth ($p>0.05$). Acid preconditioning increased the bond strength of the studied adhesives in both permanent and deciduous teeth significantly. The bond strength in ABSE showed a higher than acceptable level in the enamel by using acid phosphoric. The results of the bond strength test have been demonstrated in Table 4. In both studied adhesives in the control groups, adhesive failures were the most failures detected. After acid preconditioning, there was an increase in cohesive and mixed enamel failures (Table 4).

Discussion

Table 2. The Materials Used in the Study, the Properties and the Manufacturers

The Material Used and the Manufacturer	Application Method
Clearfil SE Bond Two-step self-etch adhesive Kurary Medical Inc Osaka, Japan Lot S 1452	1-Primer application on the surface of the tooth for 20 seconds 2-Air flow on the surface after prime application 3-Use of adhesive (bond) on the surface 4- Air flow on the surface after adhesive application 5-Light cure for 10 seconds
ACE ALL Bond SE Two-component one-step self-etch adhesive Bisco Inc. IL. USA Lot G90000&□ 26	1-Putting one drop of primer and adhesive in a special pit 2-Mixing the substance to reach a uniform pink mixture 3-Placing 1-2 layers of All-Bond SE adhesive on the surface and agitation of each layer for 5-10 seconds 4-Air flow at a 5 cm distance from the surface for 5 seconds till there is no movement of the substance mild airflow in the cavosurface for 5 seconds (the surface should be completely shiny) 5-Light curing for 10 seconds
ACE All Bond Liner BiscoInc, IL, USA	Application of a layer of less than 1 mm thick All Bond SE layer and light cure for 10 seconds for radio-opacity of the adhesive
APX Composite Kurary medical INC Japan Lot 125 GAC	As one layer and cure for 20 seconds
Phosphoric Acid gel 32% BiscoInc , IL, USA	Etching for 20 seconds and rinsing

Table3. Mean and Standard Deviation and P Value in Pairwise Comparison of Self-Etch Adhesive Shear Bond Strength in Eight Groups

Adhesive Groups	Permanent Teeth		DeciduousTeeth	
	Control	Etched	Control	Etched
CSEB	16.10±7.96	32.05±7.13	16.08±3.91	24.73±10.74
ABSE	8.49±2.34	31.39±6.51	9.23±3.20	21.70±6.18
	0.007	0.8	0.001	0.4
CSEB: Clearfill SE Bond	ABSE: ACE All Bond SE			P.V

Table4. Types of Failure in the Studied Groups

Type of Failure	Adhesive		Mixed		Cohesive	
Adhesive Groups	CSEB	ABSE	CSEB	ABSE	CSEB	ABSE
Permanent Enamel	9 (75%)	12 (100%)	3 (25%)	-	-	-
Etched Permanent Enamel	3 (25%)	3 (25%)	4 (33.3%)	5 (41.6%)	5 (41.6%)	4 (33.3%)
Deciduous Enamel	10 (83.3%)	11 (91.6%)	2 (16.6%)	1 (8.3%)	-	-
Etched Deciduous Enamel	4 (33.3%)	5 (41.6%)	2 (16.6%)	6 (50%)	6 (50%)	1 (8.3%)
	CSEB: Clearfil SE Bond			ABSE: ACE All Bond SE		

In this study, the shear bond strength of two CSEB and ABSE self-etch adhesives to the with a mild pH are such adhesives. CSEB is a two-step self-etch adhesive with a mild pH of 1.8.

This adhesive has been introduced as the “gold standard” self-etch adhesive [14] and this is the reason why it has been used as the control adhesive in this study. In addition, in two-step self-etch adhesives such as CSEB, because the resin layer is separate from the primer its hydrophobic property is higher; therefore, it is used in another step and in contrast to one-step self etch adhesives may lead to a higher bond strength. In one-step self-etch adhesives such as ABSE, it is stated that presence of a solvent, especially water, prevents sufficient polymerization of the adhesive layer and causes decrease in primary bond strength [15]. ABSE is a one-step two-component self-etch adhesive with a pH of 2.2 which has been introduced by Bisco manufacturing company (USA). In a study conducted by the manufacturer, using a special liner, the shear bond strength of this adhesive to the dentin was reported as 38 MPa which was higher than the reported shear bond strength of CSEB (32 MPa) to the dentin. Lately, Van Landuyt et al. [16] have compared the microshear bond strength of some one-step self etch adhesives to the enamel with CSEB and an etch-and-rinse adhesive (Optibond FL). In their study, Adper Prompt L– Pop, Xeno III, One up F Bond Plus were one- step and two-step self-etch adhesives which are similar to ABSE. These researchers compared the microshear bond strength of these adhesives to the enamel resulting in a higher-microshear bond strength in CSEB showing similar results to the present study. In our study, there was a significant difference between CSEB and ABSE regarding their shear bond strength to the non-etched enamel in permanent teeth. The bond strength of ABSE was much less than necessary to produce an appropriate bond to the enamel (17-20 MPa). In the enamel, using acid etch before the

application of ABSE adhesive, led to an increase in the bond strength causing no significant difference between the bond strength of ABSE and CSEB. CSEB primer consisting 10-MDP which is a functional monomer solved in HEMA and water with an approximate pH of 2 [17]. The laboratory and clinical results for this adhesive may be related to the two way bonding mechanism. The mild pH of this adhesive leads to formation of the micromechanical bonding through configuration of the thin and uniform hybrid layer making it resistant against immediate debonding forces such as the forces of shear bond strength testing. In addition, this adhesive consists of 10-MDP functional monomer providing a stable chemical bond with hydroxyapatite. This bond increases the resistance against hydraulic pressure and seals the restoration margins clinically for a long time [18].

There are many reasons for the lower bond strength of one-step self etch adhesives; including, permeability and presence of droplets in the adhesive layer as a result of osmosis phenomenon, phase separation, low conversion rate and less mechanical strength of the adhesive resin [18]. Miranda et al. [19] and also Osorio et al. [17] in separate studies compared the shear bond strength of some adhesive systems such as CSEB with one-step self-etch adhesives to deciduous teeth and the results are congruent to our study.

In our study, in the CSEB groups the shear bond strength to the non etched enamel of permanent and deciduous teeth has not showed a significant differences. In samples with etched enamels the bond strength were increased, although the bond strength was higher in permanent teeth with no significant difference ($p=0.06$). Of course taking the p value into consideration, there is a possibility that an increase in the sample volume may lead to a significant difference. In a study performed by Shimada et al. [20] who compared the bond strength of CSEB and Single Bond in the enamels of permanent and deciduous teeth,

the results showed that there were no significant differences between the bond strength of utilized adhesive systems, and between permanent and deciduous teeth, showing agreement with the results of our study.

The results of our study express a higher bond strength in permanent teeth in comparison to deciduous teeth and these results are in accordance to previous studies [21-23]. Previously, the higher density and the more organized composition of the enamel of the permanent teeth, the direction and number of the enamel prisms, and the higher crystal condensation of the permanent teeth in comparison to the deciduous teeth are reasons why bond strength is reported higher in permanent teeth. Regarding the enamel, it has been observed that in all parts of deciduous teeth enamel prisms do not reach the surface; therefore, enamels without rods or prisms are produced [19]. In the present study, the effect of acid preconditioning before application of the self-etch adhesive was evaluated and compared between the groups. Based on the results in all groups, application of acid etching led to increase in bond strength and the difference was significant. Overall, the results showed that regarding the structure of new self-etch adhesives, which are mostly mild adhesives concerning pH and the degree of acidity, acid preconditioning may improve bond strength remarkably. The positive effect of acid preconditioning of the enamel in self-etch adhesives has been mentioned in some studies [23-25]. Despite most study results, Hanning *et al.* [26] did not find a difference in the bond strength of self-etch adhesives in comparison to etch-and-rinse systems to the etched enamel. Anyway the need for separate etching of the enamel with acid phosphoric for self-etch adhesives and even CSEB that is the most common adhesive in previous studies has been controversial among researchers. The results of this study showed a significant difference in the application of this adhesive to the enamel of etched and non-etched permanent and deciduous

teeth. It seems that simplification of dental adhesives towards decreasing utilization steps and elimination of etch-and-rinse stages although facilitating the bonding process, especially in pediatric dentistry, does not indicate clinical improvement and further evaluation is recommended before clinical application.

In this study, the type of failure was assessed in each of the adhesives in the studied groups. In both studied adhesives, particularly ABSE in the control groups, adhesive failures were the prominent failures. After acid phosphoric preconditioning, cohesive failures and mixed failures increased. Taking into consideration the higher degree of bond strength in CSEB and the acid etch subgroups, the resulted failures are almost in agreement with the bond strength outcomes.

Conclusion

- 1-The two step self-etch adhesive CSBE leaded to a higher bond strength compared to one-step self etch adhesive ABSE.
- 2-The studied adhesives in deciduous teeth in comparison with the permanent teeth showed a higher bond strength.
- 3-Acid preconditioning increased the bond strength in the studied adhesives in a way that the bond strength of ABSE adhesive with the application of acid reached the acceptable degree in the enamel.

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