Original Article

Bonding Durability of Four Adhesive Systems

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Abstract:

Objectives: This study aimed to compare the durability of four adhesive systems by assessing their microtensile bond strength (MTBS) and microleakage during six months of water storage.

Materials and Methods: A total of 128 human third molars were used. The adhesives tested were Scotch Bond Multipurpose (SBMP), Single Bond (SB), Clearfil-SE bond (CSEB), and All-Bond SE (ABSE). After sample preparation for MTBS testing, the microspecimens were subjected to microtensile tester after one day and six months of water storage. For microleakage evaluation, facial and lingual class V cavities were prepared and restored with composite. After thermocycling, microleakage was evaluated. Bond strength values were subjected to one-way ANOVA and Tamhane's test, and the microleakage data were analyzed by the Kruskal-Wallis, Dunn, Mann Whitney and Wilcoxon tests ($P<0.05$).

Results: Single Bond yielded the highest and ABSE yielded the lowest bond strength at one day and six months. Short-term bond strength of SBMP and CSEB was similar. After six months, a significant decrease in bond strength was observed in ABSE and SBMP groups. At one day, ABSE showed the highest microleakage at the occlusal margin; however, at the gingival margin, there was no significant difference among groups. Long-term microleakage of all groups at the occlusal margins was similar, whilst gingival margins of SBMP and SB showed significantly higher microleakage.

Conclusion: The highest MTBS and favorable sealability were obtained by Clearfil SE bond. Water storage had no effect on microleakage of self-etch adhesives at the gingival margin or MTBS of CSEB and SB.

Keywords: Dental Leakage; Adhesives; Tensile Strength.

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INTRODUCTION

In the late 1990s, two-step etch and rinse and self-etch adhesives were introduced to the market [1]. Technique sensitivity has been reduced in self-etch adhesives by the use of non-rinse acidic primers [2]. In the two-step self-etch adhesives, a self-etch primer and hydrophobic resin are applied in separate steps; a mild self-etch primer is commonly used (pH of approximately 2)[3]. This primer is able to partially remove the smear layer and penetrate into the dentinal surface, resulting in less distinct resin tag formation; thus, the hybrid layers produced are thinner than those in the etch and rinse systems [3]. Formation of a resin-reinforced hybrid layer has been generally accepted as the mechanism for improving bond strength of adhesives to dentin [4]. Higher bond strength and improved sealing ability with an increase in

the adhesive layer thickness were explained by the improvement of stress distribution within the bonding interface and strain-induced relief of contraction stress of the composite resin, respectively [5,6]. One-step self-etch adhesives include an etchant, primer and adhesive in one or two bottles and are marketed as "all-in-one" adhesives. In vitro microleakage studies indicated that self-etch adhesive systems at the dentinal margins were as effective as adhesives with a separate etching phase but the former group were less effective at the enamel margins [3,7,8].

Mild acidity of the self-etch primers may be the reason for their weakness in enamel adhesion, while strong self-etchants can produce a more effective enamel etch than mild agents [3,9]. High dentin bond strength for self-etch adhesives, comparable to that of etch and rinse adhesives, has also been reported [10-12].

Water storage and thermocycling are two common artificial aging techniques used to predict the durability and clinical performance of dental materials [13]. Significant decrease in bond strength has been reported even after short storage periods (i.e. three months)[12-15].

The current study was designed to evaluate the effect of water storage on microleakage and MTBS of two self-etch and two etch and rinse adhesives.

A total of 128 extracted caries- and defect-free human third molars were selected and stored in distilled water containing sodium hypochlorite (10:1) at room temperature for no longer than three months after the extraction.

MATERIALS AND METHODS

Microtensile bond strength testing:

A total of 32 teeth where sectioned approximately 5 mm below the cemento-enamel junction (CEJ), at the furcation level, using number 2 round bur with high speed handpiece under cooling water. Pulp tissue remnants were removed by scaling curettes. Occlusal enamel was ground flat under running water with number 2 round bur and long cylindrical bur to provide a flat dentine surface. Then, they were polished with number 3 Sof-Lex disc (Sof-Lex, 3M ESPE, St. Paul, MN, USA) at low-speed under cooling water for 15 seconds to create a uniform smear layer. Prepared teeth were randomly divided into four adhesive groups listed in Table 1.

All adhesives were applied according to the manufacturers' instructions. Composite core was built with incremental layers of Z250 light-cure composite resin (Filtek^{TM} Z250, 3M ESPE St. Paul, MN, USA) to a 6 mm height. After 24 hours of storage at room temperature, they were embedded in self-cure acrylic resin and were sectioned in buccolingual and mesiodistal directions with a water-cooled low-speed diamond saw (M5-ISOMET diamond saw, Buehler, Esslingen, Germany) to create four to six sticks from each tooth. The cross-section of each stick was about 1×1 (±0.2) mm^2. The specimens were stored in distilled water containing 0.5% chloramine T at 37°C, to provide a wet environment preventing bacterial growth. Each group was divided into two subgroups:

Table 1. Adhesive materials

Materials	Adhesive Type	Company
Scotchbond Multi-Purpose (SBMP)	3-step etch & rinse	3M ESPE, Seefeld, Germany
Single Bond (SB)	2-step etch & rinse	3M ESPE, Seefeld, Germany
Clearfil SE Bond CSEB)	2-step self-etch	Kuraray, Sakazu, Kurashiki, Okayama, Japan
All-Bond SE (A-B SE)	1-step (2-component) self-etch	BISCO, Schaumburg, Irving Park Rd., IL, USA

one was tested at one day and the other was tested after six months of storage. The specimens were attached to the microtensile tester (Microtensile Tester; Bisco, Schaumburg, IL, USA) with cyanoacrylate glue (Mitreapel, Beta Chemical Ind. & Trade Inc. Co., Istanbul, Turkey). The tensile load was applied at a crosshead speed of 1 mm/min until fracture occurred. Then, the interface cross sections were measured by a digital micrometer (Mitutoyo Corp., Tokyo, Japan). The bond strength values were expressed in MPa.

Microleakage evaluation:

Class V cavities were prepared in 96 teeth (3mm occlusogingivally, 4mm mesiodistally and 2mm depth on the buccal and lingual surfaces of each tooth). The gingival margin was placed 1.5 mm below the CEJ and the coronal margin was 1.5 mm above it. The cavities were prepared with a 008 diamond bur and high speed handpiece under copious water. A new bur was used for every 10 cavities. Enamel margins were beveled with a flame diamond bur (0.5 mm width).

The teeth were randomly assigned to four groups (n=24), and the adhesives were applied according to the manufactures' instructions. Before applying self-etch adhesives, the enamel margins were etched separately. Then, cavities were restored with wedge-shaped light-cure composite increments (Filtek^{TM}Z250, 3M ESPE, St. Paul, MN, USA). Restorations were finished using composite finishing burs and polished by Sof-Lex discs (3M-ESPE, St. Paul, MN, USA). The teeth were stored in 0.5% Chloramine T solution (Chloramine T Trihydrate, Merck Corp., Darmstadt, Germany) in distilled water and incubated (ShimiFann, Tehran, Iran) at 37°C. Microleakage assessment was performed for half the samples at one day and the other half was assessed after six months. For the six-month samples, the solution was changed monthly.

The samples were first thermocycled for 1000 cycles in separate water baths at 5°C and 55°C with a dwell time of 30 seconds and three seconds of transfer time. The apical foramen and pits and fissures were sealed with composite resin and fissure sealants, respectively. Then, two layers of nail varnish were applied to the entire tooth surface except for one millimeter margin around the restoration. After 24 hours of immersion in 0.5% fuchsin, the teeth were embedded in auto-polymerizing acrylic resin and sectioned longitudinally at the mid-buccal and mid-lingual surfaces using a water-cooled low speed diamond saw (Vafaei Industrial Factory, Tehran, Iran) to obtain two sections from each tooth [1,16].

Each section (48 sections per group) was examined under a stereomicroscope at ×40 magnification (Carton Optical Industries, Pathumthani, Thailand). The degree of leakage was determined as follows: 0 = no dye penetration; 1 = partial dye penetration along the occlusal or gingival wall; 2 = dye penetration up to the full length of the cavity walls not including the axial walls; 3 = dye penetration to the full length of the cavity walls including the axial walls [1,16].

Statistical analysis:

The effect of time and type of adhesive on bond strength were analyzed by two-way ANOVA and since the interaction effect was significant at each time point, the comparison between the adhesive groups was made by one-way ANOVA. The Tamhane's post hoc test was used because the equality of variances was not assumed.

For evaluating the differences between microleakage groups, the non-parametric Kruskal-Wallis test was applied and followed by a Dunn's test. The differences in dye penetration between the occlusal and gingival margins in each group were analyzed by the Wilcoxon Signed Rank test and the difference

between the one day and six-month samples was analyzed by the Mann Whitney U test. All statistical analyses were performed by SPSS version 16 (Microsoft, IL, USA) (P<0.05).

RESULTS

The overall bond strength values are listed in Table 2. Single Bond achieved the highest values at one day (27.42±3.67 MPa) and six months (27.28±3.25 MPa). The lowest values at one day and six months belonged to the ABSE group. There was no significant difference between SBMP and CSEB at one day (P>0.980). The effect of time on bond strength varied depending on the type of adhesive. After six months of water storage, significant changes in bond strength values were observed in SBMP (P<0.0350) and ABSE (P<0.040) groups but not for CSEB and SB groups.

Dye penetration scores for occlusal and gingival walls are presented in Tables 3 and 4. At one day, there was a significant difference between ABSE and other adhesive groups. At the occlusal margin and at the gingival margin, there were no statistically significant differences between groups (P>0.05). At six months, there was no statistically significant difference among groups at the occlusal margin (P>0.05), but at the gingival margin there was a statistically significant difference between self-etch adhesives (CSEB and ABSE) and etch and rinse adhesives (SBMP and SB) (P<0.001). At the occlusal margin, significant differences were found between one day and six months groups except for SBMP (P= 0.054). At the gingival margin, there was no statistically significant difference between one day and six months groups except for SB (P<0.001).

The Wilcoxon test indicated statistically significant differences between the occlusal and gingival scores of each group after one day and six months of storage (P<0.001), except for one day ABSE and six-month CSEB (P=0.4 and P=0.08, respectively).

DISCUSSION

High bond strength and low microleakage are the two main goals of composite adhesion. The correlation between bond strength and microleakage in laboratory studies has not been significant [17].

However both of these mechanical properties are important clinically; bond strength is required for restoration retention and microleakage must be minimized to prevent secondary caries and pulpal reactions and maintain marginal integrity [17]. The MTBS test is a relatively new method for evaluating bond strength in dental research [18] and has shown lower test variance compared to macrotensile test [19]. Aging by water storage and/or thermocycling are two common methods applied to simulate the bonding degradation overtime in the oral cavity [13].

In the current study, among one day groups, ABSE showed the highest microleakage at the occlusal margin; there was no significant difference at the gingival margin. Several studies revealed more leakage in the occlusal margin of self-etch adhesives compared to etch and rinse adhesives; however, no significant difference was reported for gingival margins [8,20,21].

Table 2. The mean microtensile bond strength values. Standard deviations (SD) are shown in parentheses. Within the same column or row, identical letters or numbers indicate no differences (P>0.05).

Time / Adhesive systems	Mean (SD) at 6 months	Mean (SD) at 1 day
SBMP	14.39(2.25) ^{c2}	21.48(3.07) b1
SB	27.28(3.25) ^{a1}	27.42(3.67) a1
CSEB	18.76(3.44) ^{b1}	20.59(2.10) b1
ABSE	9.33(1.71) ^{d2}	11.77(2.81) c1

High microleakage of self-etch adhesives may be due to incomplete etching of the surface by acidic monomers, allowing higher microleakage values than etch and rinse adhesives (which use a separate phosphoric acid etchant). Scanning electron microscopic (SEM) studies have shown that applying phosphoric acid as an enamel etchant improves monomer penetration and the subsequent attachment of self-etch adhesives [22,23].

In our study, enamel margins were etched by 37% phosphoric acid before applying self-etch adhesives.

However, ABSE did not cause a proper seal at the occlusal margin, which might be related to its formulation. Since, two components of ABSE were mixed together immediately before use, water which is an indispensable component, creates permeable membranes, and hydrophobic resin layer formation will be compromised [24].

After six months, sealing ability of self-etch groups at the occlusal margin was similar to that of etch and rinse groups.

However, at the gingival margin, etch and rinse groups showed higher microleakage values. This was in agreement with a study by Rosales-Leal et al [25]. The depth of dentinal penetration is different between phosphoric acid and self-etch primers. In self-etch systems as the adhesive is applied, the dentinal surface is sealed causing a reduction in dentin permeability [25]. By comparing the occlusal and gingival microleakage values, all groups leaked more at the gingival margin rather than the occlusal margin, as reported previously [1,16,21,25]. The reason is related to the hydrated nature of dentin. In presence of water, creation of polymers with cross links will not occur which leads to weak polymerization of adhesive material; however, this situation will not occur in enamel bonding [26].

Table 3. One-day microleakage scores

Scores	Occlusal				Mean rank	Gingival				Mean rank
Group	0	1	2	3		0	1	2	3	
SBMP	32	7	1	8	95.09	3	0	21	24	127.78
SB	32	7	6	3	93.38	4	4	23	17	112.33
CSEB	23	20	2	3	105.91	22	4	15	7	69.85
ABSE	33	5	9	1	91.63	16	3	25	4	76.03

Table 4. Six-month microleakage

Scores	Occlusal				Mean rank	Gingival				Mean rank
Group	0	1	2	3		0	1	2	3	
SBMP	41	1	0	6	93.07	17	2	7	22	109.98
SB	44	0	0	4	87.08	23	6	7	12	89.48
CSEB	46	0	0	2	82.79	25	0	4	19	96.13
ABSE	25	6	7	10	123.05	21	4	14	9	90.42

Moreover, additional acid etching of enamel margins for self-etch groups may improve their sealing ability in occlusal margins [22,23] except for the short-term results of ABSE group. The reason for this was explained earlier. The CSEB and SB groups demonstrated the greatest bond strength values and durability overtime. However, CSEB (two-step self-etch adhesive) had poorer short-term results than SB. Moreover, these two adhesives remained stable over time. Generally, SB (containing ethanol/water solvent) functions well in laboratory studies [27-29].

Hosaka et al. demonstrated that ethanol in adhesive systems was responsible for higher bond strength values and durability overtime [30].

Ethanol is usually used to replace water in the collagen fibrils and is a better solvent for comonomers than water. It can also maintain the collagen fibrils in an expanded position after solvent evaporation [31] to allow better resin infiltration [29,30]. The presence of multiple acidic carboxylic groups may further enhance this process [32]. Finally, a distinct surface layer of hydrophobic resin will be formed resulting in high bond strength values [27]. After six months, the SBMP group showed a great reduction in bond strength, which is in agreement with the findings of Shinohara et al [33].

The CSEB was the only self-etch adhesive with comparable bond strength durability to SB, which is in accordance with the results of Armstrong et al, [15] and implicates their high resistance against degradation. Favorable durability of CSEB has also been previously reported in several studies [25,34,35]. Despite less distinct enamel etching patterns on SEM micrographs, there was no separation between the adhesive and enamel in CSEB group [36]. The high amount of comphorquinone in this adhesive may improve the degree of polymerization [34].

In addition, the presence of 10-methacryloxydecy dihydrogen phosphate (10-MDP) may also have contributed to the higher bonding durability [35]. This functional monomer has a high chemical bonding potential to hydroxy- apatite and may cause a stable molecular adhesion [37].

The ABSE had the lowest bond strength and durability over time in our study. It also acquired a low score for sealing the occlusal margin at one day. There are some reasons for suboptimal performance of one-step self-etch adhesive systems: (I) the more aggressive etching process, which may destabilize the collagen matrix [38], (II) weak cohesive strength [39] and (III) low degree of polymerization [27].

The applied method in this study for bond strength and microleakage evaluation had some limitations, such as lack of pulpal pressure upon the bonding procedure and aging, lack of mechanical loading during aging and absence of saliva. Studies using fatigue tests and pulpal pressure simulation in the saliva media are recomm- ended for better clinical generaliz-ability of results.

CONCLUSION

Under the conditions of this in vitro study, CSEB had a relatively comparable performance to etch and rinse adhesive systems, regarding both microleakage and MTBS. However, ABSE showed the lowest values for MTBS.

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