

Effect of Different Dentin Conditions and Different Co-Solvent Ingredients of One Step Self-Etch Adhesives on Shear Bond Strength

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Abstract

Objective: To evaluate Effect of different dentin conditions and different co-solvent ingredients of one step self-etch adhesives on shear bond strength.

Materials and Methods: 60 extracted teeth were divided according to the adhesive systems and dentin conditions into 6 groups of 10 teeth each [Xeno III – dry dentin, Xeno III – moist dentin, Adper Prompt L-Pop – dry dentin, Adper Prompt L-Pop – moist dentin, iBond – dry dentin, and iBond – moist dentin]. Resin composite cylinder was built up on each specimen, and then thermocycled. A shear load was applied to the specimens using universal testing (Instron machine) at a cross-head speed of 0.5 mm/min until failure occurred. Data were statistically analyzed by one-way ANOVA and Bonferroni multiple comparison test at 95% confidence level.

Results and Conclusion: The highest mean shear bond strength to dry dentin was seen when Xeno III containing ethanol co-solvent ingredient was used. The highest mean shear bond strength to moist dentin was seen when iBond which contains acetone co-solvent ingredient was used. In the absence of a co-solvent ingredient in self-etch adhesive (Adper Prompt L-Pop), the mean shear bond strengths to dry and moist dentin were low with no significant difference between them.

Keywords: Adhesives; Dental Etching; Dentin; Shear Strength

Introduction

The durability of direct esthetic restorative materials depends for large extent on the bond strength between the adhesive and the tooth substrate. This goal can be achieved through different approaches; etch and rinse (total etching), self-etching, or glass ionomer approach [1]. Bonding to enamel does not constitute a challenge unlike dentin. This is due to its simple composition; mainly hydroxyl appetite crystals. Unfortunately, the largest portion of the prepared cavities composed of dentin. Bonding to dentin constitutes a real challenge to the dentist. The difficulty of bonding to dentin is related to its high organic contents, inherit wetness, and permeability. Also, the presence of smear layer may decrease the bond strength [2,3].

Total acid etching technique when used for bonding to dentin utilizes many advantages. The advantages of this technique include complete removal of smear layer, demineralization of the superficial dentin, and exposure of the collagen fibers. The exposed collagen

fibers will be then infiltrated with the adhesive monomer forming the hybrid zone. Hybrid zone plays a significant role in the bonding mechanism [4-6]. On the other hand, this technique is quite sensitive and multi-stepped. Careless use of this technique may result in over etching, over wetting, or over drying of the dentin. To overcome these drawbacks, two-steps self-etching adhesive systems have been developed [6,7].

To simplify the technique, in self-etching approach, the adhesive monomer was added to the acidic conditioner in one bottle. Also, co-solvents are added to decrease the viscosity of the adhesive that permitting proper wetting required for good adhesion. These co-solvents may be acetone, ethanol, or even water [8,9]. Water is added to enable dissociation of the acidic monomer, while acetone or and ethanol accelerates water elimination [8].

This study was carried out to evaluate the bond strength of three self-etching systems with different solvent contents to both moist and dry dentin substrate.

Materials and Methods

A total number of 60 sound human molars were used in this study. The selected teeth were free of caries, hypocalcification, or other apparent defects. The teeth were then randomly divided into two groups (n = 30) according to dentin condition; group 1 dry dentin, and group 2 moist dentin. Each group was subdivided into three subgroups (n = 10) according to the used adhesive system; Xeno III subgroup A, Adeper Prompt L-Pop subgroup B, and iBond subgroup C. All materials used in this study are listed in table 1.

Materials	Manufacturers	Co-solvent	Composition	Application procedure
Xeno III	Dentsply	Ethanol	Bottle A: HEMA, ethanol, water, BHT, Highly dispersed silicon dioxide. Bottle B: Pyro-EMA, PEM-E, UDMA, CQ.	Mix equal amount of bottle A and B, apply on dentin substrate surface, leave undisturbed for at least 20 s, brief air-dry for 2 s, light polymerization for 10 seconds.
Adper Prompt L-Pop	3M ESPE	None	Red blister\Liquid 1: Methacrylate phosphoric acid, bis-GMA, initiators, stabilizers. Yellow blister\Liquid 2: Water, HEMA, polyalkenoic acid, stabilizers	Mix liquid 1 and 2, apply and agitate on dentin substrate surface for 15 s, gentle air-dry, apply second coat, light polymerization for 10 s.
iBond	Heraeus Kulzer	Acetone	Single bottle: 4-META, UDMA, glutraldehyde, acetone, water, photoinitiators, stabilizers	Apply three consecutive coats on dentin substrate surface, leave undisturbed for at least 30 s, gently air-dry, light polymerization for 20 s.

Table 1: Self-etch adhesives used in this study with their co-solvents, compositions, and application procedures.

HEMA: Hydroxyethyl methacrylate; BHT: Butylhydroxytoluene; Pyro-EMA: Tetra-methacryloyloxyethyl -pyrophosphate; PEM-F: Penta-methacryloyloxyethyl cycloexaphosphazene monofluoride; UDMA: Urethane Dimethacrylate; CQ: Camphorquinone; E-4-DMAB: ethyl-4-dimethyleaminobenzoate; Bis-GMA: bisphenol-glycidyl methacrylate; 4-META: 4-methacryloxyethyl trimellitic anhydride.

Before samples preparation, all teeth wear cleaned and manually scaled to remove any calculus or soft tissue might be adhered to them. Then the selected teeth were stored in distilled water till the time of the experiment (maximum one month).

Sample Preparation

At first, a 2 mm hole was done at the central pit on the occlusal surface using bur no. 330 (Midwest No.330 DENTSPLY). Then, the occlusal surface was ground till the level of the hole bottom using 180 grit silicon carbide paper mounted on a water-cooled wheel to create a flat dentin surface. The occlusal surface was then examined under stereomicroscope at 25X to assure there is no enamel remnant.

Each tooth was then embedded in a cylinder 15 mm diameter and 20 mm height filled with self-cured acrylic resin (Pattern Bright: Yamaha chi Dental MFG., CO. Japan) till the level of the cervical line. The cut dentin surface was then finished using 600, 400,320, and 240 and -grit silicon carbide papers (Shofu SF 201 Ra): Shofu Inc, Kyoto, Japan) at low speed. The prepared samples were then placed in distilled water for one day before adhesion of resin composite (Figure 1).



Figure 1: Prepared specimens.

Restorative Procedures

Before adhesive application the samples were rinsed under tapping water. For group 1 (dry dentin), the samples were then dried for 20 seconds using oil-free compressed air. While for group 2, the samples were kept moist using a blot technique with gauze to absorb the excess water. Manufacturer's instructions were followed for the application of each adhesive. For Xeno III (subgroup A), equal amounts of bottle A and B was mixed over a glass slab, then applied to the dentin substrate using microbrush. The adhesive is left undisturbed for 20 seconds then air dried for 2 seconds and light polymerized for 10 seconds. For Adper Prompt L-Pop (subgroup B), liquid 1 and 2 was mixed then applied on dentin substrate for 15 seconds. After air drying for 2 seconds, a second coat is applied. Then the adhesive was light polymerized for 10 seconds. For iBond (subgroup C), three coats of the adhesive were applied to the dentin surface, left undisturbed for 30 seconds, air dried for 2 seconds then light polymerized for 20 seconds. All samples were cured using Elipar Highlight at 400 mW/cm²-verified with a Model 100 Curing Radiometer.

After curing of the adhesive, a copper mold (2 mm long and 4 mm internal diameter) was placed onto the center of the dentin surface and filled with resin composite (Filtek Z250-shade A2) as seen in Figure 2 and as seen in table 2. The composite resin was then cured with

the same device for 40 seconds to form a composite cylinder. The prepared samples were then thermocycled between 5 C and 55 C water baths for 2500 cycles with a 30-s dwell time, and a 10-s transfer time prior to bond strength testing.



Figure 2: Copper mold.

Material trade Name	Filler size\content	Matrix Composition	Manufacturer	Batch No.
Filtek Z250 A2	0.01 - 3.5 µm 60 vol.% zirconia\ silica	Bis-GMA, UDMA and Bis- EMA	3M\ESPEE Dental Products, St. Paul, MN, USA	#1464A2

Table 2: Composite resin in the study.

Testing Procedures

The samples were mounted on a universal testing machine (Model 8500 Plus Dynamic Testing System-1341 Instron– Instron Corporation) as seen in Figure 3 A and B for testing with the long axis of the specimen being perpendicular to the direction of the applied force. The circular knife-edge was located at the interface between the composite cylinder and the dentin surface. Bond strength was measured in the shear mode at a cross-head speed of 0.5 mm/min until failure occurred.



Figure 3: Universal instrument testing machine (A, B).

Statistical Analysis

All statistical analyses were carried out using SPSS statistical software (V. 20, SPSS, Chicago, IL, USA). Data were submitted to a one-way ANOVA and Bonferroni multiple comparison test. The significant level was set at P = 0.05.

Results and Discussion

The mean shear bond strengths of three different one-step self-etch adhesives [Xeno III, Adper Prompt L-Pop, and I Bond] to different dentin conditions [dry and moist] are presented in Table 3 and Figure 4.

Material	Mean shear bond strength ± SD in MPa		One-way ANOVA	Bonferroni - t test		
	Dentin condition	Value	p-Value	Group	Group	p- Value
Xeno III	Dry	16.87± 4.14	0.001	Xeno III Dry	Adper Dry	0.01
	Moist	11.07± 2.44		Xeno III Moist	Adper Moist	1.000
Adper Prompt L-Pop	Dry	12.65± 2.19	0.073	Adper Dry	iBond Dry	0.599
	Moist	10.73± 2.31		Adper Moist	iBond Moist	0.001
iBond	Dry	10.92± 2.01	0.001	iBond Dry	Xeno III Dry	0.0001
	Moist	16.05± 3.4		iBond Moist	Xeno III Moist	0.001

Table 3: Statistical analysis summary.
The mean difference is significant at $p \leq 0.05$.

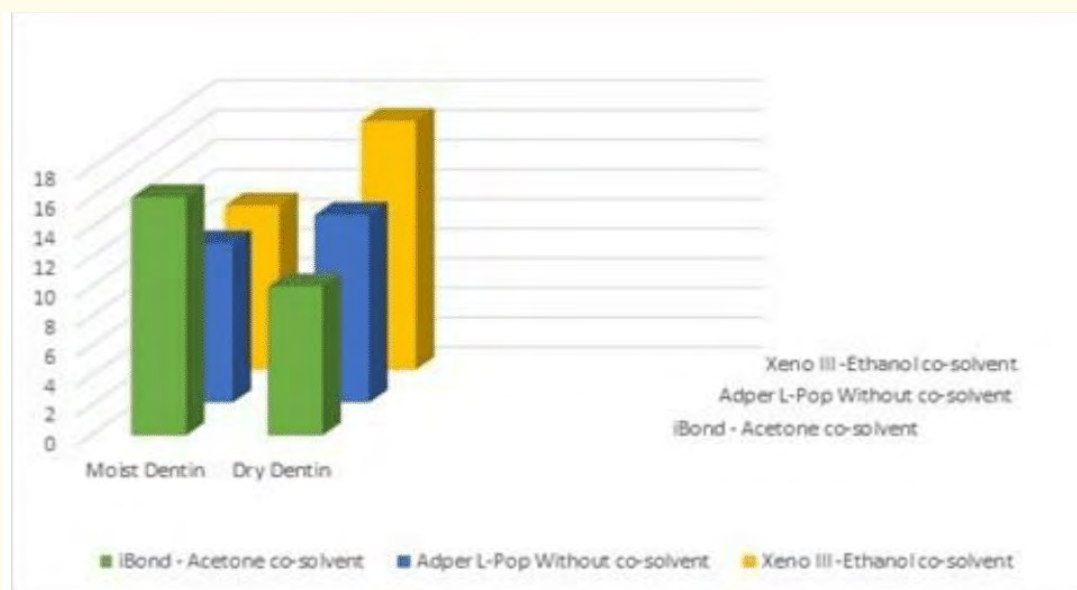


Figure 4: Shear bond strengths of three different self-etch adhesives on different dentin conditions.

The results of one-way ANOVA test (Table 3) showed that the mean shear bond strength of Xeno III to dry dentin (16.87 ± 4.14 MPa) was significantly higher than that of moist dentin (11.07 ± 2.44 MPa) at (p -value = 0.001). The mean shear bond strength of Adper Prompt L-Pop to dry dentin (12.65 ± 2.19 MPa) was higher than that of moist dentin (10.73 ± 2.31 MPa) but without significant difference at (p -value = 0.073). The mean shear bond strength of iBond to dry dentin (10.92 ± 2.01 MPa) was significantly lower than that of moist dentin (16.05 ± 3.4 MPa) at (p -value = 0.001).

The result of the Bonferroni multiple comparison test at 95% confidence level (Table 3) showed that the mean shear bond strength of Xeno III to dry dentin was significantly higher than that of Adeper Prompt L-Pop and iBond to dry dentin at p -values equal to 0.01 and 0.0001, respectively. The mean shear bond strength of iBond to moist dentin was significantly higher than that of Adeper Prompt L-Pop

and Xeno III to moist dentin at (p -value = 0.001). There were no significant differences between iBond and Adper Prompt L-Pop to dry dentin (p -value = 0.599), and between Xeno III and Adper Prompt L-Pop to moist dentin (p -value = 1.000).

Most manufacturers suggest that the bonding site prior to the application of self-etch adhesive systems should be dry, gently-dry, moist, with slightly shiny appearance, left slightly but visibly moist with a shiny surface, or not desiccated. The instructions of the manufacturers of the bonding systems used in this study were the same for all 3 systems, which were “lightly dry to moist surface”. These instructions are not quantitative but are open to individual interpretation of the appearance or the status of the surface.

For Xeno III self-etch adhesive there was a significant difference between the mean shear bond strength to dry dentin (16.87 ± 4.14 MPa) and that of moist dentin (11.07 ± 2.44 MPa). This reduction in bond strength to moist dentin compared to that of the dry one may be due to the presence of excessive water in bonding site, which came from water in moist dentin and water component in the adhesive itself. This may assist in the reduction of the effectiveness of ethanol cosolvent in accelerating water elimination and also may compromise the diffusion of adhesive into the dentin substrate [10,11]. The high bond strength value obtained to dry dentin contradicts with the manufacturer’s instructions, which recommend the use of slightly moist dentin.

For Adper Prompt L-Pop self-etch adhesive there was no significant difference between the mean shear bond strength to dry dentin (12.65 ± 2.19 MPa) and that of moist dentin (10.73 ± 2.31 MPa). These low values of shear bond strengths may be due to the absence of co-solvents (ethanol or acetone) in this brand of adhesive, which adversely affects the adhesive performance and makes it difficult to eliminate excess water in moist dentin and water ingredient in adhesive itself or water component of adhesive when applied on dry dentin [8,12]. This is shown by having a higher shear bond strength with dry dentin compared to moist dentin, despite that it was not statistically significant.

For iBond self-etch adhesive there was a significant difference between the mean shear bond strength to dry dentin (10.92 ± 2.01 MPa) and that of moist dentin (16.05 ± 3.4 MPa). The high bond strength to moist dentin may be due to the presence of acetone co-solvent which aids in the elimination of excessive water in moist dentin, without compromising the polymerization [13]. On the other hand, excessive air-drying of dentin may evaporate the water from the dentin substrate leading to reduced diffusion of adhesive into dentin and minimizing the dissociation of acidic monomers. This results in decreased bond strength to dry dentin [8].

The difficulty in achieving the balance between the moist and the dry dentin makes the dentin bonding technique extremely sensitive. The clinician should have a clear and thorough understanding of the chemical composition and adhesive mechanism of various self-etch adhesive systems.

Xeno III with ethanol co-solvent ingredient showed the most effective bonding when applied on dry dentin; while, iBond with acetone co-solvent ingredient presented the most effective bonding when applied on moist dentin. In the absence of co-solvent ingredients in Adper Prompt L-Pop self-etch adhesive, the bond effectiveness was low on both types of dentin with no significant difference between them.

Conclusion

Based on the findings of this study, it can be concluded that:

- The highest mean shear bond strength to dry dentin was seen when the Xeno III containing ethanol co-solvent ingredient was used.
- The highest mean shear bond strength to moist dentin was seen when the iBond containing acetone co-solvent ingredient was used.
- In the absence of a co-solvent ingredient in self-etch adhesive (Adper Prompt L-Pop), the mean shear bond strengths to dry and moist dentin were low with no significant difference between them.

Conflict of Interest

Authors declare that there is no conflict of interest.

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