Original article

The effect of thermocycling and acid pre-etch on bond strength to enamel of different self-etch adhesives

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ABSTRACT

Introduction: Self-etch adhesives are becoming more widely used. Their acidity can vary greatly. This determines the degree of infiltration into hard dental tissues and its adhesive ability.

Objective: To determine the effect of thermocycling and pre-etching with orthophosphoric acid on the adhesive resistance of different self-etching adhesives.

Method: The following adhesives were applied to bovine vestibular enamel: 1) Filtek Silorane (FS), 2) Filtek Silorane with acid pre-etch (AFS), 3) Adper Scotchbond 1XT (XT), 4) Adper Scotchbond SE (SE) and 5) Adper Scotchbond SE with acid pre-etch (ASE). All were applied following the manufacturer's instructions. The restored teeth were stored in water (24h, 37 °C) or thermocycled (5000 and 10000 cycles) before being sectioned and subjected to the microtraction test. Two-way ANOVA and Student-Newman-Keuls tests were used for statistical analysis (α=0.05).

Results: XTZ250 achieved the highest values and FS achieved the lowest after all artificial aging. 10000x thermocycling significantly reduced bond strength in all systems. AFS bond strength was 25.7% greater than FS, while ASE was 3.8% greater than SE.

Conclusions: The material and aging influenced bond strength. The ultra-mild self-etch adhesive obtained the lowest values after all aging treatments. Pre-etching was especially beneficial for FS.

KEYWORDS

Self-etch adhesives; Acid etching; Enamel; Thermocycling; Bond strength.

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INTRODUCTION

Self-etch adhesive systems are those that do not require prior application of an etching acid as they contain acidic monomers that are capable of conditioning and infiltrating dental tissue, meaning the risk of discrepancy between both maneuvers is decreased or nonexistent.[1] They are easier to use, and to apply, so their use has increased in the recent years.[2]

Based on the number of steps required for application, self-etch systems can be two or one-step systems. Two-step systems require application of a self-etch primer and then the adhesive resin. One-step systems, also known as "all in one", are products that simultaneously etch, condition and adhere to the tissue.[1]

The principle by which self-etch adhesives bond to the enamel and dentin depend fundamentally on their pH and their ability to chemically interact with them.[2,3] The acidity is used to classify this heterogeneous family of adhesives. This depends on the level of interaction with the hard dental tissues. They are divided into: ultra-mild ($pH>2.5$), mild ($pH\approx 2$), medium strength ($1<pH<2$) and strong ($pH\leq 1$)[3].

The most acidic systems base their function on hybridization of the hard tissues by establishing a micromechanical coupling similar to acid-etching adhesives but much less extensive than that achieved by them.[1,4] Conversely, mild self-etch adhesives base their mechanism on establishing a chemical bond with the hard tissues which, as long as they remain stable over time, increases the quality and longevity of the adhesion.[4,5]

In vitro studies report that strong self-etch adhesives have acceptable performance when they adhere to the enamel.[2] Mild self-etch adhesives have mediocre adhesive ability when bonding to enamel. Occasionally they show good adherence to the dentin due to their mild demineralization and subsequent interaction with the remnant hydroxyapatite.[6,7] This may be contradictory especially after confirming that the maintenance of hydroxyapatite at the interface predisposes to the establishment of a strong bond to the dentin, given that the enamel contains much more hydroxyapatite. This appears to be the exact cause, since it is necessary to obtain a certain degree of micromechanical coupling in the enamel via an etching agent in order to create a resistant interface.[8-11]

All adhesive systems are susceptible to some degree of hydrolytic degradation, but given that degradation of the resin is due to its ability to absorb water, the adhesives' hydrophilic properties modulate their predisposition to suffer this unfavorable phenomenon.[12,13] For this reason, simplified adhesives that combine hydrophilic and hydrophobic monomers can result in interfases that lack an adequate layer of hydrophobic resin that is isolated and free of solvents, which makes them more susceptible to hydrolytic degradation.[14] The best example of hydrolytic degradation is represented by one-step self-etching adhesives that are very rich in highly hydrophilic monomers.[15] They behave as semipermeable membranes even after polymerization.[16]

Etching with orthophosphoric acid prior to application of self-etching adhesives, especially the mild ones, is a technique recommended to improve their bond to the enamel given that it reduces the appearance of marginal defects in both in vitro[8] and in clinical studies.[10,11] In addition, an increase in adhesive ability of several self-etching systems has been observed when this extra step is included[7,17,18], though few studies include acid-etch adhesives as a control material. In addition, the high level of diversity between different self-etch adhesives can cause the previous etching to have a different effect due to the nature and acidity of the adhesive tested.[2]

The objectives of this in vitro study were to determine 1) the bond strength to microtraction to the enamel of different adhesive systems (two self-etch systems with different acidity and one total acid etching adhesive) after three artificial interface aging treatments and 2) the effect of pre-etching with orthophosphoric acid on the bond strength of the systems analyzed.

MATERIALS AND METHODS

Experimental groups

The adhesives used are detailed in Table 1. The experimental groups were the following:

- Group 1: Filtek Silorane Restorative System (FS). Filtek Silorane Adhesive System and a resin composed of low polymerization contraction, Filtek Silorane Low Shrink Posterior Restorative. Due to the new chemical composition of the resin from both materials, they both need to be applied together.
- Group 2: AFS was also reconstructed with the Filtek Silorane system. However, prior to application of the adhesive, 35% orthophosphoric acid Scotchbond Etchant, 3M ESPE) was applied for 15 seconds and rinsed for 10 seconds.
- Group 3: Adper Scotchbond 1 XT (XT) adhesive system, a total acid etch adhesive, and Filtek Z250 microhybrid resin compound.
- Group 4: Adper Scotchbond SE (SE) two-step self-etch adhesive system and Filtek Z250 microhybrid resin compound.
- Group 5: ASE, meaning reconstruction with Adper Scotchbond SE and Filtek Z250, 35% orthophosphoric acid (Scotchbond Etchant, 3M ESPE) was applied for 15 seconds and rinsed for 10 seconds prior to the application of the adhesive.

All materials belong to 3M ESPE company (Minnesota, USA). The color of the resin compounds was A3 VITA in all cases. The technical characteristics and usage instructions for the adhesives evaluated are shown in Table 1.

Sample preparation

Forty-five permanent bovine incisors were used. After being washed and analyzed with a stereoscopic microscope (Olympus SZX7, Hamburg, Germany) to rule out the presence of cavities or cracks, they were refrigerated (4°C) in a distilled water and thymol salt solution for a period of less than six months from the date of extraction. In order to facilitate handling of the teeth, the root was separated from the crown with diamond burrs and the pulp chamber was filled with composite for dual-healing stumps (ParaCore, Coltène-Whaledent) adhered using XP Bond adhesive (Dentsply), with both materials polymerized with the Elipar S10 LED (3M ESPE) unit.

The vestibular surface of the teeth was then polished with 600 grit silicon carbide discs mounted to the polisher (Buehler) under irrigation. This procedure eliminated the original convexity of the vestibular surface and exposed a flat prismatic enamel surface. With a stereoscopic microscope and injecting air, we confirmed that all of the preparations were limited to the thickness of the enamel.

The teeth were then randomly divided into 5 groups (9 teeth per group) according to the 5 experimental groups described above. The adhesives were applied exclusively to the prepared enamel according to the manufacturer's instructions and resin compound blocks were constructed on them. This was always placed using an incremental technique (three increments of composite, 2 mm in height each). The polymerization unit used was LED Demetron I (Kerr), which has a minimum power density of 550 mW/cm2.

The prepared teeth were then subdivided again. In this way, 3 subgroups of 3 teeth each were created from each of the experimental groups based on the following aging treatments:

- Subgroup A: Storage in distilled water at 37°C for 24h.
- Subgroup B: 5000 cycles of thermocycling between 5 and 55°C with a 30-second immersion time.
- Subgroup C: 10000 cycles of thermocycling between 5 and 55°C with a 30-second immersion time.

Once the different aging treatments were concluded, the teeth were sectioned longitudinally with a low-velocity diamond disc using abundant irrigation with water (IsoMet® 5000 Linear Precision Saw, Buehler). The cuts were made along the x- and y-axes in order

Table 1: Adhesive systems evaluated. Information provided by the manufacturer.

Adhesives	Composition	Instructions for use	Type	pH
Filtek Silorane Adhesive System	Self-etch primer: phosphorylated methacrylates, Vitrebond™ copolymer, Bis-GMA, HEMA, water ethanol. Filler: silica treated with silane, initiators, stabilizers.	Self-etch primer: Shake. Apply for 15” in the cavity. Disperse with air injection. Photopolymerize 10”.	Two-step self-etch	2.7 (ultra-mild)
	Adhesive: hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA. Filler (same as the primer).	Adhesive: Shake. Apply in the cavity and distribute uniformly with air. Photopolymerize 10”.		
Adper Scotchbond 1XT	HEMA, Bis-GMA, dimethacrylate, polyacrylic and polythionic acid-based functional metacrylate copolymer, water and ethanol. Nanofiller and photoinitiator.	Acid etch: Apply 35% orthophosphoric acid (Scotchbond Etchant, 3M ESPE) for 15” and rinse for 10”. Remove excess humidity without desiccating.	Two-step total acid-etch	4.7
		Adhesive: Apply two successive layers for 15”. Gently dry with air (2-5”) to evaporate the solvent. Photopolymerize 10”.		
Adper Scotchbond SE	Liquid A (primer): water, HEMA, surfactant, pink coloring.	Liquid A (primer): Apply in the cavity until it is completely stained pink.	Two-step self-etch	1 (strong)
	Liquid B (adhesive): UDMA, TEGDMA, TMPTMA, HEMA, MHP. Nanofiller with zirconium, photoinitiator.	Liquid B (adhesive): Apply actively for 20”. As the pink color applied from Liquid A disappears, indicating activation of the acid part of the adhesive and the start of the self-etch process. Dry with air 10”. Apply a second layer of the adhesive followed by a smooth current of air. Photopolymerize 10”.		

to obtain bar-shaped sections with a quadrangular section and a transverse area, meaning the bonded surface measuring approximately 1 mm^2. An average of 20 specimens were obtained from each tooth that were valid for the subsequent microtraction test.

In order to precisely calculate the bonded surface area of each specimen, their lateral sides were measured using a digital caliper to a 0.001 mm level of precision (Mitutoyo). The samples were then individually submitted to the microtraction test using an Instron 3345 universal trials machine, to which they were glued using cyanoacrylate glue (Loctite Gel) to the machine’s clamps. The microtraction values are expressed in megapascals (MPa). All of the specimens that fractured prior to being submitted to the microtraction test were recorded but excluded from the statistical analysis.

Table 2: Mean values (standard deviation) corresponding to the microtraction strength of the enamel (expressed in MPa), number of samples tested (N), types of failure [adhesive (A), cohesive (C), mixed (M)] and number of pre-test failures (%) for each of the experimental groups based on the type of aging treatment applied.

Aging	24 h					5000x					10000x				
Adhesive system	$\bar{x}$(sd)	n	A/C/M	%	$\bar{x}$(sd)		N	A/C/M	%		$\bar{x}$(sd)	n	A/C/M	%	
FS	23.1 (4.2)	63	58/1/4	2	C 1	22.8 (5.2)	61	54/2/5	3	C 1	18.5 (3.5)	60	57/0/3	2.4	D 2
AFS	29 (4.2)	65	51/6/8	0.3	B 1	27.8 (4)	61	52/5/4	0.5	B 1	23.4 (4)	61	57/1/3	2	B 2
XT	34.1 (4.1)	66	50/4/12	1	A 1	33.4 (4.2)	64	51/2/11	0.8	A 1	31.1 (4)	65	55/2/8	1	A 2
SE	29.4 (3.9)	65	53/3/9	2	B 1	27 (3.8)	61	52/1/8	3	B 2	20.8 (3.7)	60	54/0/6	4.3	C 3
ASE	29.8 (3.7)	62	49/6/7	0.2	B 1	27.1 (4.4)	61	53/2/10	1	B 2	22.9 (4.3)	60	55/0/5	1.2	B 3

Similar letters in the same row mean similar microtraction strength values between the restorative systems after each of the aging treatments.
Similar numbers in the same column mean similar microtraction strength values between the aging treatments for each of the restorative treatments.

The fractured surfaces of all of the specimens were subsequently analyzed with a stereoscopic microscope (Olympus SZX7) in order to determine the type of failure that occurred in each: adhesive (between the adhesive and the enamel-dentin and/or between the adhesive and the composite), cohesive (fracture in the sinus of the enamel-dentin or the composite) or mixed (adhesive and cohesive failure occurring simultaneously). This analysis was performed with a magnification up to 50x and always by a single observer.

Statistical analysis

All of the results obtained were statistically analyzed using IBM SPSS 19 (IBM Corporation, Armonk, New York, USA) for Windows. The accepted level of significance was 0.05 in all cases. First, a descriptive analysis was presented using central tendency measures and the arithmetic mean with the standard deviation used as a measure of dispersion. In order to evaluate how the independent variables (adhesive system used and aging treatment) influenced the quantitative outcome variables (bond strength to the enamel), a two-way ANOVA test was applied. Subsequent comparisons were made using the Student-Newman-Keuls test.

RESULTS

The statistical analysis determined that the bond strength to the enamel was influenced by the adhesive system used and the aging treatment applied. The interaction between both factors was also significant. The means and standard deviations corresponding to the microtraction bond strength test for the systems evaluated are detailed in Table 2.

Influence of the adhesive for each aging treatment

The results obtained are shown in Figure 1. Application of the one-way ANOVA test detected statistically significant differences between the mean microtraction values obtained for the different adhesive systems after each aging treatment, so the next comparison was carried out using the Student-Newman-Keuls test ($p<0.05$).

- 24 h: The highest bond strength values were achieved with XT. The second statistical group included three systems with statistically similar values (in descending order: ASE, SE and AFS). The FS system was in the last place.

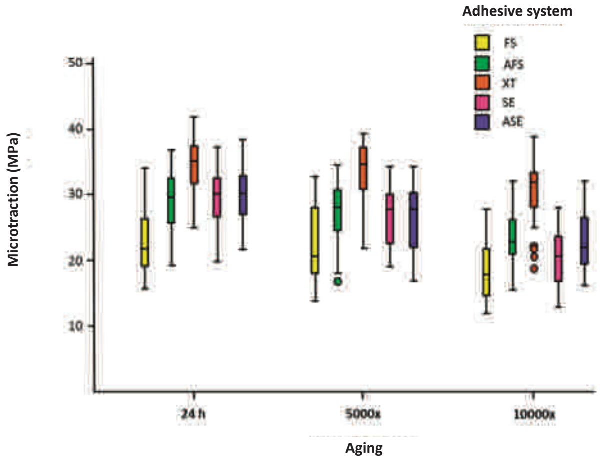

Figure 1. Distribution of the bond strength values after each of the aging treatments obtained for each adhesive system.

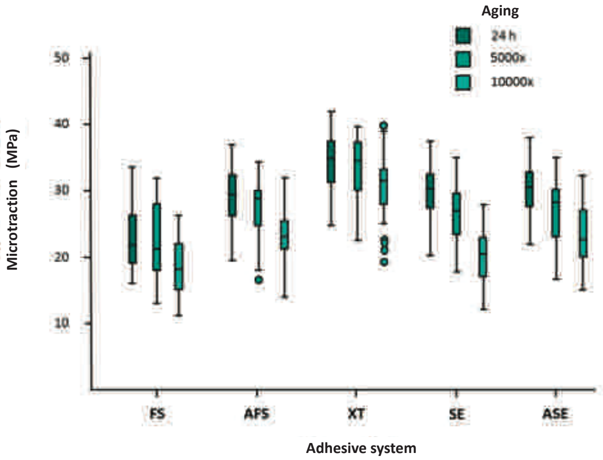

Figure 2. Distribution of the bond strength values obtained for each adhesive system after the different agin treatments.

- 5000 thermocycles: XT was once again in the first place. In the second statistical group, with statistically similar values, were the same three systems as those after 24 hours, though in a different order. In descending order: AFS, ASE and SE. The FS system once again had the lowest strength values.

- 10000 thermocycles: Once again, the bars for the XT system had the highest values. Next, with statistically similar values, were the two groups with self-etch adhesives and reinforcement with acid etching: AFS and ASE. Finally, the values achieved with SE and FS are in third and fourth place, respectively.

Influence of the aging treatment applied for each adhesive

The results obtained are shown in Figure 2 and described below. The one-way ANOVA test detected differences between the microtraction values obtained after the different aging treatments for all of the experimental groups, revealing the following:

- FS, AFS, and XT: the microtraction values obtained were statistically similar after 24h and 5000 thermocycles, being significantly inferior to those registered after 10000x thermocycling.

- SE and ASE: the values decreased statistically with each aging treatment that was applied, meaning the highest values were obtained after 24h of storage, the intermediate values after 5000 thermocycles and the lowest values after 10000 thermocycles.

Observation of the fractured surfaces with a stereoscopic microscope revealed the nature of the failure in each of the samples evaluated. The results are shown in Table 2.

DISCUSSION

The results obtained in this study determined that the bond strength to the enamel is dependent on the restorative system used and the aging treatment applied, as well as the interaction between both variables. Using an intersystem comparison, it is clear that thermal aging reduces the microtraction values achieved by the five experimental groups. However, the intersystem comparison also reveals the important differences in the performance that each of the materials demonstrated.

According to the literature, total acid-etch adhesive systems achieve the best results and are recognized as the gold standard.[1,19] The orthophosphoric acid etching pattern allows the adhesive resin to penetrate between the crystals and into the prisms, as well as allowing for deeper penetration between the interprismatic spaces.

This creates a superficial morphology that is capable of initiating the micromechanical bond that reports the highest levels of adhesion to the enamel[9,19,20], which also makes it easier for the adhesive to achieve greater resistance to the propagation of microfractures.[7,9]

These advantages related to acid etching were also present in our study, given that XT, the only total acid-etch adhesive analyzed, achieved the highest bond strength values after all of the aging treatments. In addition, there was only a significant decrease in its microtraction values after application of more aggressive thermocycling of 10000 cycles (Table 2). The supremacy of the enamel bond of the total acid-etch adhesive over the self-etch systems evaluated is supported by numerous scientific studies.[19-24]

The reduced demineralization capacity of the majority of self-etch adhesives is particularly evident when bonding to the enamel, the dental substrate with the highest inorganic content. Their acidic monomers are limited to acting on the most superficial enamel, which only achieves a mild, flat, uniform etching.[7-25] For this reason and for quite some time, some authors have been promoting the benefits of performing selective etching with orthophosphoric acid, meaning limited to the enamel, prior to applying the self-etch adhesive.[8-11]

In 2009, Erickson et al.[25] analyzed the effect of acid etching once they had the strength values for various self-etch adhesives, including Clearfil SE (Kuraray) and Adper Prompt L-Pop (3M ESPE). As explained below, these two adhesives can be relatively comparable to the two self-etch systems in our study: FS and SE.

On the one hand, Clearfil SE and FS are both mild two-step self-etch systems, given their pH of 2 and 2.7, respectively. Both base their adhesive capacity on the chemical interaction established with the hard dental tissues (via the MDP monomer in the case of Clearfil SE and via the polyalkenoic acid copolymer in FS). To date, their bond strengths to enamel have not been compared directly but their bond to dentin has. Clearfil is considered the gold standard, revealing similar results between them.[26]

On the other hand, SE is a two-step adhesive that nevertheless has very similar performance to a one-step adhesive given that its acidic monomers are not found in its primer, but rather in the adhesive itself, and they are activated only when both liquids are mixed in the oral cavity. This allows it to be assimilated to Adper Prompt L-Pop, a one-step self-etch adhesive that, unlike adhesives from this group, maintains its different components conveniently separated thanks to its characteristic presentation form (mini-lollipop) that are mixed and activated just prior to application. Both adhesive systems base their function on their elevated acidity (they have a pH of 1 and 0.9, respectively), which gives them the ability to etch the enamel in a way that is as similar as possible to orthophosphoric acid.[27]

Erickson et al.[25] found that acid pre-etching improved outcomes for Clearfil SE and Adper Prompt L-Pop by 41 and 27%, respectively. Despite the greater increase in the case of mild self-etch adhesive, the bond values for both were statistically similar to total acid-etch adhesive (control material) after 24h of storage in water (the only aging treatment applied). However, this did not occur in our study given that, despite the fact that the results shown by the acid pre-etch systems were higher than those obtained following their recommended application, they were not statistically comparable to the results obtained with the Adper Scotchbond 1 XT.

The values obtained with AFS were significantly better than those of FS, revealing increases of 28.5, 22.3 and 26.4% (corresponding to the three aging treatments: 24h, 5000 and 10000 thermocycles).

However, the benefit of acid pre-etch was much more discrete in the case of SE, since specimens that had acid applied showed a 10% increase in their microtraction values after 10000 thermocycles and only 1.3 and 0.3% after 24h and 5000 thermocycles, respectively. In fact, this irregular increase in microtraction values for the self-etch systems also had a very variable effect on the distribution of the type of failure that occurred in the specimens. In the case of FS, previous application of acid increased non-adhesive failures by 25%. Conversely, there was an inverse tendency in the case of SE given that there were 6% more failures of this kind when orthophosphoric acid was not applied (Table 2).

This remarkable difference in the influence that acid pre-etching had on the outcome of self-etch adhesives was the result of the actual nature and pH of each of the systems. While FS had a clear inability to demineralize the surface due to its low acidity and benefited from the microporosity created by the acid etching, SE, with its low pH, would be capable of blurring the pattern created by the acid.

Leaving aside the effect of acid pre-etching, in order to analyze the results obtained from the recommended application of the self-etch adhesives, it is necessary to point out that SE achieved statistically higher bond strength values than FS after all of the aging treatments, which is consistent with a previous study.[28] This once again confirms the importance of the acidity of adhesive systems and the micromechanical coupling that results from adequate etching of the enamel surface for adhesive quality.[2,6,27,29]

CONCLUSIONS

Total acid-etch adhesive achieved the highest values and FS, the ultra-mild self-etch system achieved the lowest, after all aging treatments. 10000x thermocycling significantly reduced the values of all adhesive systems. Application of orthophosphoric acid was particularly beneficial for FS since its values were superior to those obtained with the recommended application after all aging treatments.

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