Micro-Shear Bond Strength of Different Adhesives to Human Dental Enamel

Nagayassu MP * / Shintome LK ** / Arana-Chavez VE *** / Fava M ****

*The **aim** of this study was to evaluate the micro-shear bond strength of 5 adhesive systems to enamel, one single-bottle acid-etch adhesive (O), two self-etching primers (P) and two all-in-one self-etching adhesives (S). **Method**: Sixty premolar enamel surfaces (buccal or lingual) were ground flat with 400- and 600-grit SiC papers and randomly divided into 5 groups (n=12), according to the adhesive system: SB2 - Single Bond 2 (O); CSE - Clearfil SE Bond (P); ADS - AdheSE (P); PLP - Adper Prompt L-Pop (S); XE3 - Xeno III (S). Tygon tubing (inner diameter of 0.8mm) restricted the bonding area to obtain the resin composite (Z250) cylinders. After storage in distilled water at 37º C for 24h and thermocycling, micro-shear testing was performed (crosshead speed of 0.5mm/min). Data were submitted to one-way ANOVA and Tukey test (a=5%). Samples were also subjected to stereomicroscopic and SEM evaluations after micro-shear testing. Mean bond strength values (MPa±SD) and the **results** of Tukey test were: SB2: 36.36(±3.34)a; ADS: 33.03(±7.83)a; XE3: 32.76(±5.61)a; CSE: 30.61(±6.68)a; PLP: 22.17(±6.05)b. Groups with the same letter were not statistically different. It can be **concluded** that no significant difference was there between SB2, ADS, XE3 and CSE, in spite of different etching patterns of these adhesives. Only PLP presented statistically lower bond strengths compared with others.*

J Clin Pediatr Dent 35(3): 301–304, 2011

INTRODUCTION

Acid etching was proposed for enamel[1] with the aim to dissolve the hydroxyapatite crystals either at the rod cores or at the rod peripheries for creating a superficial microporosity[2] Placed onto etched enamel, the monomers of adhesive systems penetrate into microporosities thus forming resin tags that yield good micromechanical retention.[3] Nowadays, a number of adhesive systems are used on dentin, some of them needing previous acid etching, while others already contain weak acids among their components. The adhesive systems are usually applied onto both enamel and dentin with the same time of application.[4] However, acid etching promotes the complete removal of some micrometers of the outer enamel because of its high mineral and small amounts of organic content,[5] whereas in dentin the acids remove hydroxyapatite crystals while exposing the organic components, in special collagen fibrils.[6]

Among the current adhesive systems there are two main groups, the first that includes weak acids, and the second that contains acidic monomers.[4] Even though many authors have stated that these two conditioning agents promote a slight superficial etching of enamel that can be a disadvantage for the success of adhesive procedures,[7] they are routinely used when both enamel and dentine are simultaneously conditioned.[8] Moreover, some conditioning agents do not require rinsing whereas others are already included in the adhesive system.

Few studies have devoted analysis to adhesive characteristics of the main types of adhesive systems to enamel. The present investigation has been addressed with the purpose to evaluate the micro-shear bond strength of different adhesives to the outer enamel surface.

MATERIALS AND METHOD

This study was approved by the Research Ethics Committee of the Sã o Jose dos Campos School of Dentistry - UNESP (Protocol No. 024/2006-PH/CEP).

Forty healthy premolars, extracted for orthodontic reasons, of patients of both sexes ranging from 14-16 years of age were used, after obtaining free and informed consent from their parents. In order to perform the microshear tests, thirty teeth were used, the remaining 10 teeth were used for

* Marcos Paulo Nagayassu, Post-graduated (PhD) in Restorative Dentistry, School of Dentistry of Sã o José dos Campos, UNESP. Sã o José dos Campos, SP. Brazil.

** Luciana Keiko Shintome, Post-graduated (PhD) in Restorative Dentistry, School of Dentistry of Sã o José dos Campos, UNESP. Sã o José dos Campos, SP. Brazil.

*** Victor E Arana-Chavez, Full Professor, Division of Oral Biology, School of Dentistry, University of Sã o Paulo. Sã o Paulo, SP. Brazil.

**** Marcelo Fava, Associate Professor, Department of Children Clinical Dentistry, School of Dentistry of Sã o José dos Campos, UNESP. Sã o José dos Campos, SP. Brazil.

Send all correspondence to Dr. Victor E. Arana-Chavez, Division of Oral Biology, School of Dentistry, University of Sã o Paulo.

05508-900 Sã o Paulo, SP, Brazil.

E-mail: vearana@usp.br

micromorphological analysis by SEM.

The teeth were stored in a 0.5% chloramine solution for no longer than one week. After this, the roots were sectioned and the crowns cleaned with rubber cups and pumice stone paste (S.S.White Brasil Ltda, Brasil.) and water for 10s. Then they were cut in the mesio-distal direction using double-faced diamond disks (KG Sorensen Ind. e Com. Ltda, Brasil.).

For the mechanical test, each face of the sectioned crown was embedded in fast polymerizing colorless acrylic resin (Jet - Clássico Artigos Odontoló gicos Ind. Bras.), by using silicone molds in order to obtain blocks with 3 cm length, 2 cm width, and 1 cm depth, with the enamel surface parallel to the horizontal plane. The enamel surface of the specimens was flattened by using 400 and 600 grain water abrasive papers (3M do Brasil), mounted in a polishing machine (Struers DP10 - Panambra), with copious water to obtain a smooth, flat enamel surface approximately 3 mm in diameter. The specimens were randomly divided into 5 groups (n=12) according to the adhesive system used (Table 1).

In Group 1, the specimens were etched with a 37% phosphoric acid (Dentsply) for 15 s, followed by air/water spray washing for 20 s and drying with an air jet for 3 s. Then, the Single Bond 2 adhesive system was applied in accordance with the manufacturer's recommendations. In groups 2 to 5, the specimens did not receive acid etching before application of the correspondent adhesive, which were placed in accordance with the manufacturers' recommendations.

After light curing the adhesives for 10 s, samples were placed in a metal device that has a two-piece Teflon matrix with an orifice inside it, measuring 2 mm in diameter and 2 mm high. To make the samples for the microshear bond strength test Tygon R-3603 tubes (Norton Performance Plastic Co.), with an outer diameter of 2mm by 1 mm high, and 0.8 mm inner diameter were used, for the purpose of delimiting the bonding area. By means of the Teflon matrix, the Tygon tube was immobilized over the tooth enamel, and the resin composite Filtek Z250 (3M ESPE) was applied with a calcium hydroxide applicator (SS White Duflex) in a single increment, and light activated for 40 s, in order to obtain small cylinders for the mechanical test. After this, the test specimen was removed from the matrix and complementary light activation was performed for 40 s. After 1h at room temperature, the Tygon tube was gently removed with a #15 scalpel blade (Med Blade). All the test specimens were stored in distilled water at 37º C in a oven for 24 hours. After this, they were thermocycled (Ética Equipamentos Cientí ficos S.A.) with baths for 30 s between 5(±2) and 55(±2)º C, totaling 500 cycles.

Table 1. Division of the experimental groups

Group	Adhesive System	Composition
1	*Single Bond 2* (*3M ESPE*)	Etching: 37% Phosphoric acid Adhesive: 2-hydroxyethyl methacrylate (HEMA), Bisphenol A diglycidyl ether dimethacrylate (Bis-GMA), ethanol, water
2	*Clearfil SE Bond* (*Kuraray*)	Primer A: 10-methacryloyloxy decyl diydrogenphosphate (MDP) Primer B: HEMA, water Adhesive: MDP, Bis-GMA, HEMA
3	*AdheSE* (*Ivoclar Vivadent*)	Primer: dimethacrylates, phosphonic acid acrylate, water Adhesive: dimethacrylates, HEMA, SiO2
4	*Adper Prompt L-Pop* (*3M ESPE*)	Methacrylated phosphoric acid esters, water, Bis-GMA, HEMA
5	*Xeno III* (*Dentsply*)	HEMA, water, ethanol, 2,6-Di-tert-butyl-p hydroxyl toluene, Pyro-EMA-SK, PEM-F, Urethane dimethacrylate, EPD, p-diethyl amine ethyl benzoate

After thermocycling, the test specimens were submitted to microshear bond strength evaluation performed in a universal test machine EMIC DL2000 with a 10kg load cell. To place the test specimens correctly, a metal base was used and 0.2 mm diameter orthodontic wire was fixed to the top mobile extremity of the machine. When the test was performed, this wire was placed at the adhesive interface, forming a loop around the resin composite cylinder. The speed used was 0.5mm/min and the microshear bond strength values were expressed in MPa.

The data were submitted to the single factor analysis of variance (ANOVA) with a level of significance of 5%. After this, the Tukey multiple comparison test was carried out to verify at which levels the difference between the groups occurs, and which of these produced the highest mean bond strength values.

After the bond strength tests, the surfaces of the samples were examined under a stereoscopic microscope Stemi 2000C (Carl Zeiss), at 16x magnification, to determine the type of fracture that occurred in the region of rupture between the enamel and composite resin in each specimen. The fractures were classified into: type a (adhesive fracture at the resin/adhesive interface); type b (cohesive fracture in enamel: fracture in the enamel dental structure); type c (cohesive in resin composite: fracture in the resin composite cylinder body), and type d (mixed fracture, with the fracture involving the resin, adhesive and dental structure).

For the micromorphological analysis of the etched enamel surface, ten teeth were used with their crowns previously prepared as previously described in order to obtain 20 enamel surfaces (buccal or lingual), and randomly subdivided into the five experimental groups (n=4).

Using the central region of the vestibular or lingual face, for Group 1, the specimens were etched with a 37% phosphoric acid for 15 s, followed by air/water spray washing for 20 s and drying with an air jet. For groups 2 and 3, only the self-etching primer of the adhesive systems Clearfil SE Bond and AdheSE were applied, respectively. For Groups 4 and 5, the acid solution resulting from the mixture of the components of the self-etching adhesive systems Adper Prompt L-Pop and Xeno III were applied, respectively. The etched enamel surfaces of the specimens in Groups 2 to 5 were completely washed with alternate baths of acetone for 20 s and ethanol for 20 s, with the purpose of removing the self-etching primers and resinous components of the adhesive systems used.

After etching the enamel surface, all the test specimens were dehydrated in ascending grades of ethanol (70%, 80%, 90% and 100%), for 15 min each. After this, they were air-dried on a filter paper and mounted on aluminum stubs, and sputter coated with gold in a Sputter ion appliance (SCD/050 - Bal-Tec). Specimens were evaluated in a scanning electron microscopy (JSM-6100 - Jeol), operating at 10kV at an original magnification of 750X.

RESULTS

The statistical description is presented in Table 2, and the graphic representation of the points around the mean (Dot Plot) and corresponding columns, means and standard deviation, are shown in Figure 1

The result of the *ANOVA* (single factor) test was p=0.0001. By means of the Tukey multiple comparison test (5%) two homogeneous groups were established.

After analysis under stereomicroscope, the test specimens were classified according to the types of fracture (Table 3).

Figure 2 shows the representative appearances of dental enamel from specimens belonging to the five different adhesives used. Phosphoric acid produced a potentially retentive and porous structure with dissolution of enamel prisms cores across entire surface. Self-etching primers revealed a very slight pattern of demineralization as well as unetched sites. Self-etching adhesives produced a surface with slightly greater dissolution than self-etching primers, with shallow depressions.

Table 2. Statistical description of the bond strength data (MPa) obtained in the microshear bond strength test (n=12), and results of the Tukey Multiple Comparison Test (5%), according to five different adhesive systems

Adhesive Systems	Mean ± Standard Deviation	Homogeneous Groups*	
Single Bond 2	36.36 ± 3,34	A	
AdheSE	33.03 ± 7,83	A	
Xeno III	32.76 ± 5,61	A	
Clearfil SE Bond	30.61 ± 6,68	A	
Adper Prompt L-Pop	22.17 ± 6,05		B

*sets that present equal letters indicate no statistically significant difference.

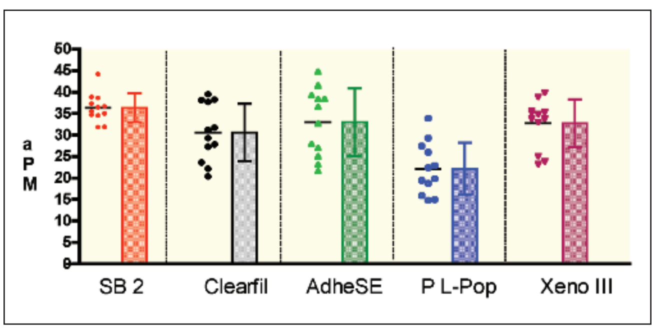

Figure 1. Dot Plot Graph and corresponding mean and standard deviation of the microshear data (in MPa), according to the groups.

Table 3. Types of fracture according to the experimental condition

	Experimental Conditions	Fracture Adhesive	Cohesive enamel	Cohesive Resin	Mixed
1	*Single Bond 2*	10			2
2	*Clearfil SE Bond*	11			1
3	*AdheSE*	10			2
4	*Adper Prompt L-Pop*	12			
5	*Xeno III*	11			1

DISCUSSION

The present study showed that the two self-etching primers (Clearfil SE Bond and AdheseSE) and only one self-etching adhesive (Xeno III) exhibited a similar microshear bond strength on human dental enamel than that of the single component adhesive (Single Bond 2), while the other self-etching adhesive (Adper Promp-L Pop) presented less bond strength.

A microshear bond strength test was used because of the higher friability of enamel when compared to dentin. In fact, studies focusing dentin usually applied microtensile tests, but they need a previous preparation of the adhesive surface. On the other hand, microshear bond tests use a very small bonding area, which avoids the problem of non-uniformity of the stress distribution along the adhesive interface.[9] A scanning electron microscopy analysis was also done in order to determine how the microporosities created by the adhesive systems could influence on microshear bond strength. The self-etching primer/adhesives groups were rinsed with acetone and alcohol in order to remove resin components and evaluate only the etching effect on the enamel surface.[10]

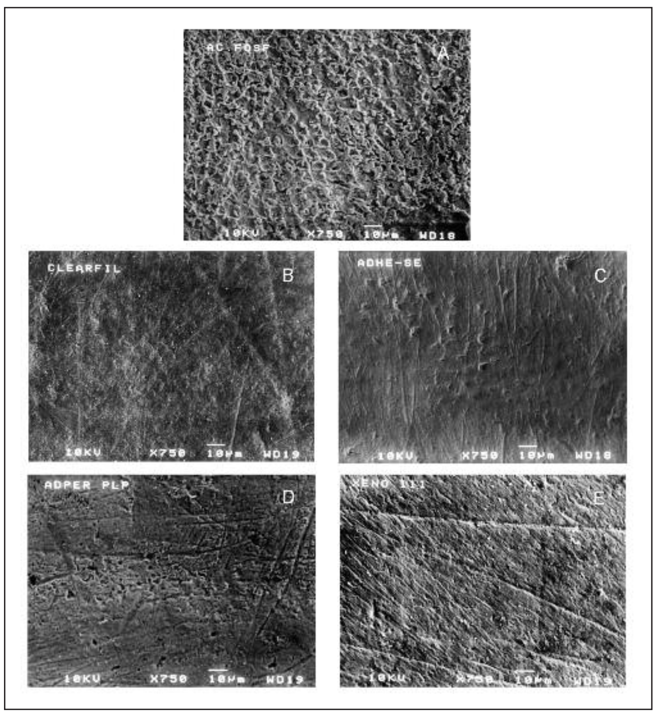

Figure 2. Scanning electron micrographs obtained after the application of: a) 37% phosphoric acid for 15 s; b) self-etching primer of the adhesive system Clearfil SE Bond; c) self-etching primer of the adhesive system AdheSE; d) acid solution resulting from the mixture of the components of the adhesive system Adper Prompt L-Pop; e) acid solution resulting from the mixture of the components of the adhesive system Xeno III. Original magnifications: 750X.

Although the micromorphological analysis revealed that AdheSE, Xeno III and Clearfil SE Bond yielded discreet enamel etching patterns, they obtained statistically similar performance to that of Single Bond 2, which requires previous etching with phosphoric acid. Many factors could tinfluence on the degree of demineralization of self-etching systems, such as the type and concentration of the acid solution, time of etching, formulation of the acidic monomers and buffering capacity of hydroxyapatite.[11]

Bond strength to enamel would therefore be the product of the slight increase in micromechanical retention added to the chemical interaction,[1] since the functional monomers of the self-etching systems chemically interact with the enamel hydroxyapatite in spite of their low etching capacity.[12] The absence of relationship between the depth of demineralization and the strength of bonds produced by the more aggressive self-etching adhesive systems on enamel shown in the present investigation is consistent with previous works.[13,14,15,16,17] This means that other factors, apart from the etching pattern, may have a more important role on the bond strength values.[18] Variation in adhesive viscosity, surface tension, acidity of the self-etch system, chemical interaction of acidic monomers with enamel, water concentration, cohesive strength of the adhesives are important features to be considered.[19,20]

The acidity of the etching agents contained in the different adhesive systems used in the present study varied from 0.8 (Adper Promp-L Pop) up to 2.0 (Clearfil SE Bond). This is a key factor that could have influenced on the microshear bond strength findings. Indeed, in spite of their retentive etching pattern, the self-etching systems with more acid pH obtained the lowest bond strength values. It means that other factors, like the presence of solvents within the polymer, may render a thin the adhesive layer, possibly weakening the polymer formed.[13,20]

Nevertheless, in spite of the difference between the microshear bond strength values, all the adhesive systems evaluated obtained values of bond strength to enamel ranged from 22.17 to 36.36MPa, irrespective of the etching method used on each material. This bond strength mean (20 MPa) is considered necessary for the adequate maintenance of the adhesive on enamel.[3,15] On the other hand, the obtained values are below the tensile strength of enamel,[2] which could be responsible for the high predominance (90%) of adhesive failures, results similar to those obtained in previous investigations.[4,15,16]

CONCLUSION

Based on the experimental conditions under which the present study was conducted, it could be concluded that:

The highest bond strength values obtained corresponded to the single component adhesive Single Bond 2, however, with no statistically significant difference for the self-etching primers Clearfil SE Bond and AdheSE as well as for the self-etching adhesive Xeno III

The performance of the self-etching adhesive Adper Prompt L-Pop was significantly low to that of the others.

REFERENCES

1. Brackett WW, Ito S, Nishitani Y, Haisch LD, Pashley DH. The microtensile bond strength of self-etching adhesives to ground enamel. Oper Dent, 31: 332–7, 2006.
2. Giannini M, Soares CJ, Carvalho RM. Ultimate tensile strength of tooth structures. Dent Mater, 20: 322–9, 2004.
3. Hara AT, Amaral CM, Pimenta LAF, Sinhoreti MAC. Shear bond strength of hydrophilic adhesive systems to enamel. Am J Dent, 12: 181–4, 1999.
4. El-Araby AM, Talic YF. The effect of thermocycling on the adhesion of self-etching adhesives on dental enamel and dentin. J Contemp Dent Pract, 8: 17–24, 2007.
5. Erickson RL, De Gee AJ, Feilzer AJ. Fatigue testing of enamel bonds with self-etch and total-etch adhesive systems. Dent Mater, 22: 981–7, 2006.
6. Arana-Chavez VE, Massa LF. Odontoblasts: the cells forming and maintaining dentine. Int J Biochem Cell Biol, 36: 1367–73, 2004.
7. Hayakawa T, Kikutake K, Nemoto K. Influence of self-etching primer treatment on the adhesion of resin composite to polished dentin and enamel. Dent Mater, 14: 99–105, 1998.
8. Kaaden C, Powers JM, Friedl KH, Schmalz G. Bond strength of self-etching adhesives to dental hard tissues. Clin Oral Investig, 6: 155–60, 2002.
9. Braga RR, Meira JB, Boaro LC, Xavier TA. Adhesion to tooth structure: a critical review of “macro” test methods. Dent Mater, 26: 38–49, 2010.
10. Di Hipó lito V, de Goes MF, Carrilho MR, Chan DC, Daronch M, Sinhoreti MA. SEM evaluation of contemporary self-etching primers applied to ground and unground enamel. J Adhes Dent, 7: 203–11, 2005.
11. Perdigã o J, Gomes G, Duarte S Junior, Lopes MM. Enamel bond strengths of pairs of adhesives from the same manufacturer. Oper Dent, 30: 492–9, 2005.
12. Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P, Van Meerbeek B. Bond strength of a mild self-etch adhesive with and without prior acid-etching. J Dent, 34: 77–85, 2006.
13. Moura SK, Pelizzaro A, Dal Bianco K, de Goes MF, Loguercio AD, Reis A et al. Does the acidity of self-etching primers affect bond strength and surface morphology of enamel? J Adhes Dent, 8: 75–83, 2006.
14. Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives. Part II: etching effects on unground enamel. Dent Mater, 17: 430–44,. 2001.
15. Perdigã o J, Lopes L, Lambrechts P, Leitã o J, Van Meerbeek B, Vanherle G. Effects of a self-etching primer on enamel shear bond strengths and SEM morphology. Am J Dent, 10: 141–6, 1997.
16. Shimada Y, Senawongse P, Harnirattisai C, Burrow MF, Nakaoki Y, Tagami J. Bond strength of two adhesive systems to primary and permanent enamel. Oper Dent, 27: 403–9, 2002.
17. Weerasinghe DS, Nikaido T, Wettasinghe KA, Abayakoon JB, Tagami J. Micro-shear bond strength and morphological analysis of a self-etching primer adhesive system to fluorosed enamel. J Dent, 33: 419–26, 2005.
18. Abdalla AI, El Zohairy AA, Abdel Mohsen MM, Feilzer AJ. Bond efficacy and interface morphology of self-etching adhesives to ground enamel. J Adhes Dent, 12: 19–25, 2010.
19. Burrow MF, Kitasako Y, Thomas CD, Tagami J. Comparison of enamel and dentin microshear bond strengths of a two-step self-etching priming system with five all-in-one systems. Oper Dent, 33: 456–60, 2008.
20. Moura SK, Reis A, Pelizzaro A, Dal-Bianco K, Loguercio AD, Arana-Chavez VE, Grande RH. Bond strength and morphology of enamel using self-etching adhesive systems with different acidities. J Appl Oral Sci, 17: 315–25, 2009.