



Original Article

บทวิทษภกร

Microtensile bond strength of four contemporary dental adhesives

Vichuda Sereepanpanich, D.D.S., Grad. Dip. in Clin. Sc. (Operative Dentistry)¹

Sirivimol Srisawasdi, D.D.S., M.S., Ph.D.²

¹Graduate Student, Department of Operative Dentistry, Faculty of Dentistry, Chulalongkorn University

²Department of Operative Dentistry, Faculty of Dentistry, Chulalongkorn University

Abstract

Objective This study evaluated microtensile bond strength (MTBS) of two newly developed self-etch adhesives compared to a two-step self-etch adhesive and a three-step total-etch adhesive, and determined the effect of surface moisture on MTBS of the new single-step self-etch adhesive.

Materials and methods Twenty-five extracted human third molars were ground flat to expose mid-coronal dentin, and teeth were randomly divided into five experimental groups, which were Adper Scotchbond Multipurpose, Clearfil SE Bond, Adper SE Plus Self-Etch Adhesive, Adper Easy Bond Self-Etch Adhesive, and Adper Easy Bond Self-Etch Adhesive in moist condition groups. Each of the adhesives was used to bond resin-based composite to the dentin surface. After 24 hours of water storage, each specimen was sectioned into 8 beams. All beams were tested for MTBS at a cross-head speed of 1 mm/min. Data were statistically analyzed using one-way ANOVA and Tamhane's multiple comparison tests ($\alpha = 0.05$).

Results One-way ANOVA revealed significant difference in mean MTBS values among the groups ($p < 0.05$). Tamhane's multiple comparison tests showed that Adper Scotchbond Multipurpose exhibited the highest mean MTBS value, however, it was not significantly different from the mean MTBS value obtained from Clearfil SE Bond and Adper Easy Bond Self-Etch Adhesive in moist condition. Adper Easy Bond Self-Etch Adhesive showed significant differences in mean MTBS between moist and dry condition, in which moist condition improved the mean MTBS. Adper SE Plus Self-Etch Adhesive exhibited the lowest MTBS, which was significantly different from MTBS obtained from all groups except that from Adper Easy Bond Self-Etch Adhesive.

Conclusion The dentin bond strengths produced by Adper Scotchbond Multipurpose three-step total-etch adhesives and Clearfil SE Bond two-step self-etch adhesive were higher than that of the newly developed simplified adhesives (Adper SE Plus and Adper Easy Bond). MTBS of Adper Easy Bond single-step self-etch adhesive significantly increased when bonded to moist dentin.

(CU Dent J. 2009;32:191-202)

Key words: microtensile bond strength; self-etch adhesive

Introduction

Patient's demand for esthetic restorations has generated an interest in the advancement of adhesive dentistry. Buonocore, in 1955, established that treatment of enamel surface with an aqueous acid solution resulted in the formation of resin tags by penetration of the resin monomer into the resulting microporosities, and a micromechanical bond was formed after polymerization.¹ Enamel bonding technique has become standard and is well accepted, however, bonding to dentin has evolved significantly with the development of various bonding systems.² Several classifications of the dental adhesives have been suggested in scientific literatures. A functional-based classification has recently been suggested, consisting of three main groups of adhesives³: total-etch or etch and rinse adhesives, self-etch adhesives and glass-ionomer based adhesives. This classification is simple and has proven to be more practical. As it is based on the applied adhesion strategy, this classification provides a dentist or a researcher with background information on the adhesion mechanism, and on the characteristics of an adhesive system.

Enamel and dentin bonding has progressed from multi-step systems to simplification of the application procedure in order to abate technique-sensitivity and reduce working time.⁴ Originally, three-step total-etch systems were found to be complicated, consisting of multi-bottle system that required separate application of etchant, primer, and bonding resin.³ However, with long term clinical track records, three-step total-etch adhesives have been considered as the gold standard, and in spite of the rather elaborate and lengthy working procedure, they can achieve very satisfactory results.⁵ *Self-etch adhesives do not require a separate etch and rinse step, as they contain acidic monomers that simultaneously etch and prime enamel and dentin.³ As a result, the dissolved smear layer and demineralization products are not rinsed away, but incorporated in the adhesive resin to form hybrid layer.⁶ Initial self-etch

systems consisted of an acidic primer, followed by a hydrophobic bonding resin. Recently, all-in-one adhesive or one-step self-etch adhesives have been brought onto the market, which combine etchant, primer and bonding resin into one solution.^{7,8} Morphological features of the hybrid layer produced by self-etch adhesives depend a great deal on the aggressiveness of the functional monomers. Consequently, three categories of self-etch adhesives can be made according their acidity: mild ($\text{pH} \geq 2$), intermediate ($\text{pH} \sim 1.5$) and strong self-etch adhesives ($\text{pH} \leq 1$).³ While bonding to enamel using self-etch systems remains a concern, especially for mild self-etch adhesives, bonding to dentin has reached reasonable results.^{9,10} Some two-step self-etch adhesives have been documented with adequate *in vitro* bond strengths that came in the vicinity of that of total-etch adhesives.^{11,12} Recent studies have reported good clinical results for a self-etch adhesive, Clearfil SE Bond (Kuraray, Tokyo, Japan), which is a mild two-step self-etch adhesive with a pH of around 2. Studies regarding Clearfil SE Bond reported high retention rates in non-carious class V cavities after 2 years¹³ and 3 years.¹⁴ Mild self-etch adhesives demineralize dentin only very shallowly, leaving hydroxyapatite crystals around the collagen fibrils available for possible additional chemical interaction, which may be a plausible explanation for good performance on dentin of this class of self-etch adhesives.¹⁵

Compared to total-etch adhesive, many advantages have been pointed out for self-etch adhesives. It has been suggested that they improved the efficiency in clinical procedures by omitting the obligatory rinsing step in total-etch adhesive, thus reducing both the technique sensitivity and the chair time.¹⁶ As the smear layer and smear plugs were not removed prior to actual bonding procedure, wetting of dentin by dentinal fluid from the dentin tubules was prevented,¹⁷ and potential postoperative sensitivity was reported to be reduced.¹⁸ Perdigão et al., however, could not observe any difference in postoperative sensitivity between a

three-step total-etch adhesive and a self-etch system.¹⁹ Concerns surrounding the self-etch adhesives included the ability of the self-etching primer to penetrate a thick smear layer and the reduced potential for demineralization in the subsurface dentin due to neutralization of the primer by the mineral components of the smear layer.⁶

Most newly developed adhesives are currently released onto the market with little supportive clinical data. A clinical trial is the most valid way to evaluate the quality and efficacy of adhesive systems. However, clinical trials usually are expensive and require a long observation period, therefore, *in vitro* screening of newly developed adhesives remains essential to quickly evaluate their bonding effectiveness to dentin. Adhesion analyses of dental adhesives have been performed using various testing methods, including bond strength, microleakage and contraction gap size measurements.²⁰ Bond strength of dentin adhesives has frequently been evaluated using shear and tensile bond strength tests. These simple tests used large surface areas (7–12 mm²), and served well when resin-dentin bond strengths were relatively low (10–15 MPa). However, with the improvements in dental adhesive systems, the bond strengths became high enough to cause cohesive failures in dentin. Frequency of cohesive failures of dentin was reported to be as high as 80% when bond strengths reached 25 MPa.²¹ A microtensile method of bond strength testing was developed by Sano et al.²² One advantage of this method was that the bonded interface of small (1 mm²) specimen had a better stress distribution during loading, therefore, there were fewer cohesive failures in dentin found compared to conventional testing.²¹ This was thought to be due to a reduction in flaw density. Using this method often resulted in higher apparent bond strengths at failure than found when using large specimens.²³ Owing to the bonded interface of small specimens, it permitted multiple specimens to be prepared from each tooth. Thus, there was a trade-off between the extra labor involved in using this method,

and the extra data that could be obtained per tooth.²⁴ Since then, the microtensile bond strength (MTBS) testing has become commonly used to evaluate *in vitro* performance of dental adhesive systems.

The application methods for newly developed adhesive systems have been simplified, and the manufacturers' instructions have become clearer for achieving optimum clinical performance. Although adhesive systems have become simpler, careful management is still required, especially regarding the matter of influence of surface moisture on bonding performance.^{25–28} Researches investigating the effect of surface moisture on the MTBS of the single-step self-etch adhesive have been scarce.

This study investigated the MTBS of two newly developed self-etch adhesives compared to a conventional two-step self-etch and a three-step total-etch adhesive, and determined the effect of dentin surface moisture on the MTBS of newly developed single-step self-etch adhesive. The null hypotheses were there was no difference between the MTBS of four adhesives to dentin, and dentin surface moisture did not affect the MTBS of the newly developed single-step self-etch adhesive.

Materials and methods

The specimen preparation of the study is schematically presented in Fig. 1. Twenty-five intact, non-carious, and non restored extracted human third molars were obtained for the study with consent. All the selected teeth were debrided and stored in a 0.1% thymol solution at 4°C for up to 1 month following extraction. The root of tooth was embedded into an autopolymerizing resin, leaving the clinical crown exposed. The occlusal third of the embedded tooth was removed to expose mid-coronal dentin using a slow-speed saw with a diamond-impregnated disc (Buehler, Lake Bluff, IL, USA) under water cooling.

Dentin surfaces were evaluated for absence of enamel and/or pulp exposure using a stereomicroscope (ML 9300; Meiji Techno Co. Ltd., Tokyo, Japan) at 40X. Smear layer on dentin was created by grinding the surface with a 180-grit silicon carbide paper under running water.^{29,30} Teeth were randomly divided into five experimental groups containing five teeth each as followed:

Group 1: Adper Scotchbond Multipurpose (3M ESPE, St. Paul, MN, USA)

Group 2: Clearfil SE Bond (Kuraray, Osaka, Japan)

Group 3: Adper SE Plus Self-Etch Adhesive (3M ESPE, St. Paul, MN, USA)

Group 4: Adper Easy Bond Self-Etch Adhesive (3M ESPE, St. Paul, MN, USA)

Group 5: Adper Easy Bond Self-Etch Adhesive in moist condition (3M ESPE, St. Paul, MN, USA)

Dentin surface was rinsed with running water for 10 seconds and dried with a gentle air stream for 10 seconds, with the exception of group 5 that the effect of surface moisture on the performance of Adper Easy Bond Self-Etch Adhesive was determined. In group 5, dentin surface was prepared by depositing 30 μ l of distilled water on the surface with a micropipette

(Acura manual 825 Volume 10–100 μ l, Socorex, Switzerland) and blotted to remove excess water using a filter paper (#1 Whatman, Maidstone, UK) for 30 seconds. The tooth was bonded according to the manufacturers' instructions (Table 1).

After curing of the adhesive, a light-cured resin composite, Filtek Z350 Shade A1 (3M ESPE, St. Paul, MN, USA,) was built up to approximately 4 mm in height by incremental placement onto the treated dentin surface. Each 2 mm increment was polymerized for 40 seconds using visible light-polymerization unit (Elipar TriLight Curing Light; 3M ESPE, St. Paul, MN, USA) with 500 mW/cm² intensity. The light guide was held perpendicularly and within 1 mm of the surface. The light output from the light-polymerizing unit was checked using a radiometer (SHOFU Lite-Checker; Shofu Inc., Kyoto, Japan) throughout the experiment.

After storage in distilled water at 37°C for 24 hours, teeth were sectioned perpendicular to the adhesive-tooth interface using low speed cutting machine (Isomet 1000; Buehler Ltd., Lake Bluff, IL, USA) to obtain rectangular sample of about 1 x 1 mm² and 8 mm long. Eight beams were retrieved from the 2 widest slabs of each tooth. Five teeth from each group yielded 40 beams for bond strength evaluation.

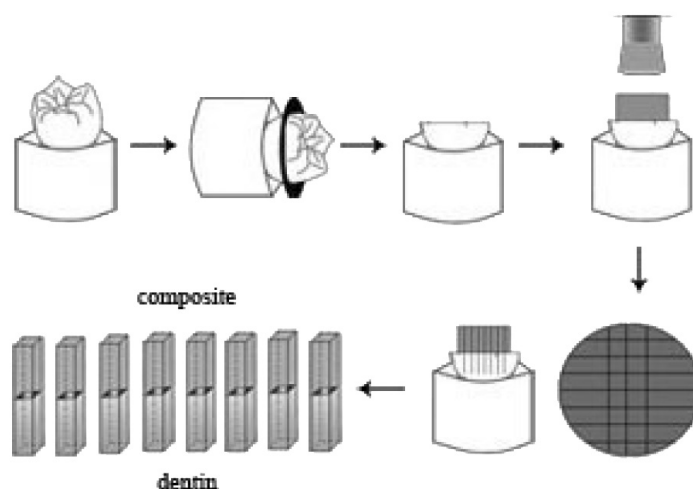


Fig. 1 Schematic diagram of the specimen preparation for the microtensile bond test.

Table 1 Adhesive systems evaluated.

Adhesive (Manufacturer)	Type	Composition	General procedures
1. Adper Scotchbond Multi-purpose (3M ESPE, St. Paul, MN, USA) Lot No. 20060921	Three-step total-etch adhesive	Etchant: 35% phosphoric acid Primer: HEMA, polyalkenoic acid copolymer, water Bond: HEMA, Bis-GMA, tertiary amines, photoinitiator	1. Etchant 15 s 2. Rinse 10 s and blot dry 3. Primer application 10 s 4. Air stream 5. Bond application 6. Gentle air stream 7. Light polymerize 10 s
2. Clearfil SE Bond (Kuraray, Osaka, Japan) Lot No. 81144	Two-step self-etch adhesive	Primer: 10-Methacryloyloxydecyl Dihydrogen phosphate (MDP), HEMA, hydrophilic dimethacrylates, photoinitiators, amine, water. Bond: MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, colloidal silica, photoinitiator	1. Primer application 20 s 2. Gentle air stream 3. Bond application 4. Gentle air stream 5. Light polymerize 10 s
3. Adper SE Plus Self-Etch Adhesive (3M ESPE, St. Paul, MN, USA) Lot No. MFG135L002-AB (primer) MFG 135L001-AC2 (bond)	Two-step self-etch adhesive	Primer: water, HEMA, surfactant, pink colorant Bond: UDMA, TEGDMA, hydrophobic trimethacrylate (TMPTMA), HEMA phosphates, methacrylated phosphates (MHP), bonded zirconia nanofiller, photoinitiator	1. Primer application to obtain continuous red-colored layer 2. Moderate bond scrub 20 s to red color disappear 3. Air stream 10 s 4. Second coat bond application 5. Gentle air stream 6. Light polymerize 10 s
4. Adper Easy Bond Self-Etch Adhesive (3M ESPE, St. Paul, MN, USA) Lot No. 301596	Single-step self-etch adhesive	HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylate functionalized polyalkenoic acid, bonded silica nanofiller, ethanol, water, photoinitiator	1. Gentle bond rub 20 s 2. Air stream 5 s 3. Light polymerize 10 s

HEMA, 2-Hydroxyethyl methacrylate; Bis-GMA, adduct of bisphenol A and glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

The dimension of each beam was measured using a digital caliper (Mitutoyo Co., Japan). All beams were then attached to the apparatus with a cyanoacrylate adhesive (Model Repair II Blue; Dentsply–Sankin, Ohtawara, Japan) and stressed to failure in tension using a universal testing machine (LR 10K; Lloyd Instruments Ltd, Fareham, Hants, UK) at a cross-head speed of 1 mm/min. The MTBS (MPa) at breaking point was recorded automatically by the testing machine.

All data were analyzed statistically using SPSS 13.0 for Windows (Chicago, IL, USA). Since the MTBS data were normally distributed, the data were analyzed using one-way ANOVA and Tamhane's multiple comparison tests, with statistical significance set at $\alpha = 0.05$.

Results

Table 2 showed means and standard deviations for MTBS values obtained with the five experimental groups. One-way ANOVA revealed significant difference in mean MTBS values among the groups ($p < 0.05$). Further statistical analysis using Tamhane's multiple comparison tests revealed that Adper Scotchbond Multipurpose exhibited the highest mean MTBS value, which, however, was not significantly different from

the mean MTBS value obtained from Clearfil SE Bond and Adper Easy Bond Self-Etch Adhesive used in moist condition. Adper Easy Bond Self-Etch Adhesive showed significant differences in mean MTBS between moist and dry condition, in which moist condition improved the mean MTBS. The lowest MTBS was obtained from the Adper SE Plus Self-Etch Adhesive group, which was statistically different from all groups except Adper Easy Bond Self-Etch Adhesive.

Discussion

In the present study, bond strengths of four contemporary dental adhesive systems were assessed utilizing the non-trimming method of microtensile bond testing, which was created by a natural progression of slab reduction into small beams (Fig. 1). This technique is currently considered to be a reliable adhesion test, for it allows the loading stress to be more uniformly distributed by testing of small-sized specimens.^{3,24} With this method, multiple specimens can be obtained from single tooth, and the coefficients of variation associated with testing are usually between 10% and 25%²⁴, providing more accurate results. Indeed, the coefficients of variation in the present study

Table 2 Microtensile bond strength of adhesives tested.

Adhesive	No. of Samples	Mean (SD) Bond Strength, Megapascals
Adper Scotchbond Multipurpose	40	68.5 (14.8) a
Clearfil SE Bond	40	67.1 (11.7) a
Adper SE Plus Self-Etch Adhesive	40	40.3 (20.3) b
Adper Easy Bond Self-Etch Adhesive	40	50.2 (15.7) b
Adper Easy Bond Self-Etch Adhesive (Moist condition)	40	69.9 (17.1) a

SD: Standard deviation.

Same symbols indicate mean values that were not significantly different ($p > 0.05$).

were within the acceptable limits of frequency for Adper Scotchbond Multipurpose, Clearfil SE Bond and Adper Easy Bond Self-Etch Adhesive in moist condition. Conversely, Adper SE Plus Self-Etch Adhesive and Adper Easy Bond Self-Etch Adhesive exhibited a wide spread of the bond strength values, which was probably due to their poor adhesion, resulting in the interface more susceptible to the cutting procedures.

Within the limits of this *in vitro* study, Clearfil SE Bond two-step self-etch adhesive revealed bond strength close to that obtained with the conventional adhesive Adper Scotchbond Multipurpose, which was in agreement with previous studies.^{11,12} Adper Scotchbond Multipurpose is a three-step total-etch adhesive, with the first step involving etching and rinsing to remove smear layer exposing a microporous network of collagen that is nearly deprived of hydroxyapatite.³¹ Priming follows, which enables the applied resin layer to wet the etched surface. The bonding resin, which is a Bis-GMA and HEMA resins combined with an initiation system, seals the dentinal surface and provides an interface for bonding to composites. Clearfil SE Bond is a two-step self-etch adhesive characterized by a relatively mild pH (pH = 2). When the primer is applied, it partially demineralizes dentin to a depth of 1 μm , which is sufficient to obtain mechanical interlocking through hybridization.³ As no rinsing of the primer is needed, it remains diffused throughout the dentin tissues, preventing collapse of the collagen network. For both Adper Scotchbond Multipurpose and Clearfil SE Bond, complete absence of solvents in the bonding resin allows formation of a highly cross-linked polymer with high degree of conversion. Moreover, the hydrophobic bonding component creates a coat, which prevents the adhesive layer from behaving as a permeable membrane after polymerization that expedites water sorption within the adhesive layer.³²

Adper SE Plus two-step self-etch adhesive has been developed by separating the aqueous and acidic

components to minimize hydrolysis of the acidic phosphates for improved shelf life. According to the documentations included in the materials package (Table 1), the primer agent of the Adper SE Plus Self-Etch Adhesive system (aqueous primer) includes water and HEMA, while the bonding resin (acidic adhesive) contains a hydrophilic acidic monomer, methacrylated phosphates (MHP), as the functional monomer, along with UDMA and TEGDMA. The bonding technique claims to provide a solvent-free, hydrophobic overcoat similar to the bonding philosophy of Adper Scotchbond Multipurpose adhesive. In the present study, Adper SE Plus Self-Etch Adhesive exhibited the worst bond strength mean and the highest variance. The poor performance of Adper SE Plus Self-Etch Adhesive may be due to the water/acidic monomer ratio in an aqueous mixture, which might not be stable for each application. Therefore, the increase or decrease in water concentration may affect the degree of ionization of the acidic monomer. Moreover, the acidic adhesive of Adper SE Plus Self-Etch Adhesive contained TEGDMA, which absorbed more amount of water after polymerization than Bis-GMA.³³

Investigation of dentin surface moisture during the bonding procedure is of clinical relevance. The results of this study suggested that extrinsic dentin wetness had an effect on the MTBS of the single-step self-etch adhesive Adper Easy Bond. An explanation for the increase in bond strength might be related to the chemical nature of this adhesive system. Calcium salt created by the phosphate monomer was highly insoluble. According to the adhesion-decalcification concept, the less soluble the calcium salt of an acidic molecule, the more intense and stable the molecular adhesion to a hydroxyapatite-based substrate.^{15,31} Furthermore, in the presence of water, acidic functional group diffused into the softened tooth surface and theoretically formed ionic bonds with calcium of hydroxyapatite.³⁴ Our finding is not in agreement with the study of Chiba et al.²⁸ They demonstrated that Adper

Prompt L-Pop (3M ESPE, St. Paul, MN, USA), which was a strong single-step self-etch adhesive (pH = 0.8-1), was more sensitive to moisture at the dentin surface, and showed the reduction in bond strengths in wet condition. Similar to Adper Easy Bond, Adper Prompt L-Pop contains the phosphoric acid ester as a functional monomer. An explanation was that moisture on dentin surface might dilute the adhesives applied to the dentin, thus negating the etching effect of the adhesive. Another explanation might be imperfect polymerization of adhesives due to excessive water present on the dentin surface. Thus, evaporation of water present on the dentin surface is important for achieving optimum bond strength. A prescribed amount of water is an essential component to provide the medium for ionization of acidic monomer.³⁵ From this study, it should also be noted that a moist dentin surface is essential for optimal bond strength of Adper Easy Bond, even though the manufacturer recommends that it works on both wet and dry tooth surfaces. The manufacturer's instruction might need to emphasize on using this material on moist dentin for a better performance. However, the present finding should be interpreted with caution, as the results were obtained under laboratory conditions and it remains to be determined whether a similar trend occurs *in vivo*.

Conclusions

Under the condition of this study, the results led to a rejection of the first null hypothesis: the dentin bond strengths produced by Adper Scotchbond Multipurpose three-step total-etch adhesives and Clearfil SE Bond two-step self-etch adhesive were higher than that of the newly developed simplified adhesives, Adper SE Plus and Adper Easy Bond. In addition, when Adper Easy Bond single-step self-etch adhesive was bonded to moist dentin surface, the resultant mean MTBS value was significantly higher than when bonded to dry surface. Therefore, the

second hypothesis tested in this study was also rejected.

Acknowledgement

The authors would like to thank Assistant Professor Dr. Suchit Poolthong for all his invaluable advices throughout this investigation.

The authors also thank the staffs of the Dental Material Science Research Center, the Faculty of Dentistry, Chulalongkorn University for their assistance in laboratory experiments.

This investigation was supported in part by 3M ESPE (Thailand).

References

1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res.* 1955;34:849-53.
2. Kugel G, Ferrari M. The science of bonding: from first to sixth generation. *J Am Dent Assoc.* 2000;131 Suppl:20S-25S.
3. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent.* 2003;28: 215-35.
4. Van Meerbeek B, Perdigao J, Lambrechts P, Vanherle G. The clinical performance of adhesives. *J Dent.* 1998;26:1-20.
5. Van Meerbeek B, Van Landuyt K, De Munck J, Hashimoto M, Peumans M, Lambrechts P, et al. Technique-sensitivity of contemporary adhesives. *Dent Mater J.* 2005;24:1-13.
6. Tay FR, Pashley DH. Aggressiveness of contemporary self-etching systems. I: Depth of penetration beyond dentin smear layers. *Dent Mater.* 2001;17: 296-308.

7. Frankenberger R, Perdigao J, Rosa BT, Lopes M. "No-bottle" vs "multi-bottle" dentin adhesives--a microtensile bond strength and morphological study. *Dent Mater.* 2001;17:373-80.
8. Chan KM, Tay FR, King NM, Imazato S, Pashley DH. Bonding of mild self-etching primers/adhesives to dentin with thick smear layers. *Am J Dent.* 2003;16:340-6.
9. Inoue S, Vargas MA, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, et al. Microtensile bond strength of eleven contemporary adhesives to enamel. *Am J Dent.* 2003;16:329-34.
10. Toledano M, Osorio R, de Leonardi G, Rosales-Leal JI, Ceballos L, Cabrerizo-Vilchez MA. Influence of self-etching primer on the resin adhesion to enamel and dentin. *Am J Dent.* 2001;14:205-10.
11. Sadek FT, Goracci C, Cardoso PE, Tay FR, Ferrari M. Microtensile bond strength of current dentin adhesives measured immediately and 24 hours after application. *J Adhes Dent.* 2005;7:297-302.
12. Omar H, El-Badrawy W, El-Mowafy O, Atta O, Saleem B. Microtensile bond strength of resin composite bonded to caries-affected dentin with three adhesives. *Oper Dent.* 2007;32:24-30.
13. Turkun SL. Clinical evaluation of a self-etching and a one-bottle adhesive system at two years. *J Dent.* 2003;31:527-34.
14. Peumans M, Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Three-year clinical effectiveness of a two-step self-etch adhesive in cervical lesions. *Eur J Oral Sci.* 2005;113:512-8.
15. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, et al. Comparative study on adhesive performance of functional monomers. *J Dent Res.* 2004;83:454-8.
16. Cho BH, Dickens SH. Effects of the acetone content of single solution dentin bonding agents on the adhesive layer thickness and the microtensile bond strength. *Dent Mater.* 2004;20:107-15.
17. Itthagarun A, Tay FR. Self-contamination of deep dentin by dentin fluid. *Am J Dent.* 2000;13:195-200.
18. Tay FR, King NM, Chan KM, Pashley DH. How can nanoleakage occur in self-etching adhesive systems that demineralize and infiltrate simultaneously? *J Adhes Dent.* 2002;4:255-69.
19. Perdigao J, Geraldini S, Hodges JS. Total-etch versus self-etch adhesive: effect on postoperative sensitivity. *J Am Dent Assoc.* 2003;134:1621-9.
20. International Organization for Standardization/ Technical Specification (ISO/TS)11405:2003, Dental materials--testing of adhesion to tooth structure.
21. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. *Dent Mater.* 1995;11:117-25.
22. Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, et al. Relationship between surface area for adhesion and tensile bond strength--evaluation of a micro-tensile bond test. *Dent Mater.* 1994;10:236-40.
23. Cardoso PE, Braga RR, Carrilho MR. Evaluation of micro-tensile, shear and tensile tests determining the bond strength of three adhesive systems. *Dent Mater.* 1998;14:394-8.
24. Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, et al. The microtensile bond test: a review. *J Adhes Dent.* 1999;1:299-309.
25. Asaka Y, Miyazaki M, Takamizawa T, Tsubota K, Moore BK. Influence of delayed placement of composites over cured adhesives on dentin bond strength of single-application self-etch systems. *Oper Dent.* 2006;31:18-24.
26. Chiba Y, Yamaguchi K, Miyazaki M, Tsubota K, Takamizawa T, Moore BK. Effect of air-drying time of single-application self-etch adhesives on dentin bond strength. *Oper Dent.* 2006;31:233-9.
27. Yamamoto A, Tsubota K, Takamizawa T, Kurokawa H, Rikuta A, Ando S, et al. Influence of light intensity on dentin bond strength of self-etch systems. *J Oral Sci.* 2006;48:21-6.

28. Chiba Y, Rikuta A, Yasuda G, Yamamoto A, Takamizawa T, Kurokawa H, et al. Influence of moisture conditions on dentin bond strength of single-step self-etch adhesive systems. *J Oral Sci.* 2006;48:131-7.
29. Koibuchi H, Yasuda N, Nakabayashi N. Bonding to dentin with a self-etching primer: the effect of smear layers. *Dent Mater.* 2001;17:122-6.
30. Tani C, Finger WJ. Effect of smear layer thickness on bond strength mediated by three all-in-one self-etching priming adhesives. *J Adhes Dent.* 2002;4:283-9.
31. Yoshioka M, Yoshida Y, Inoue S, Lambrechts P, Vanherle G, Nomura Y, et al. Adhesion/decalcification mechanisms of acid interactions with human hard tissues. *J Biomed Mater Res.* 2002;59:56-62.
32. Carvalho RM, Tay FR, Giannini M, Pashley DH. Effects of pre- and post-bonding hydration on bond strength to dentin. *J Adhes Dent.* 2004;6:13-7.
33. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, et al. Systematic review of the chemical composition of contemporary dental adhesives. *Biomaterials.* 2007;28:3757-85.
34. Yoshida Y, Van Meerbeek B, Nakayama Y, Snauwaert J, Hellems L, Lambrechts P, et al. Evidence of chemical bonding at biomaterial-hard tissue interfaces. *J Dent Res.* 2000;79:709-14.
35. Wang Y, Spencer P. Continuing etching of an all-in-one adhesive in wet dentin tubules. *J Dent Res.* 2005;84:350-4.

กำลังแรงยึดแบบดึงระดับจุลภาคของสาร ยึดติดร่วมสมัยสี่ชนิด

วิชาชุด เสรีพันธุ์พานิช ท.บ., ป.บัณฑิต (ทันตกรรมหัตถการ)¹

ศิริวิมล ศรีสวัสดิ์ ท.บ., M.S., วท.ด.²

¹นิสิตบัณฑิตศึกษา สาขาทันตกรรมหัตถการ ภาควิชาทันตกรรมหัตถการ คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

²ภาควิชาทันตกรรมหัตถการ คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

บทคัดย่อ

วัตถุประสงค์ เพื่อศึกษากำลังแรงยึดแบบดึงระดับจุลภาคของเซลล์เอทซ์อีซีพีที่ถูกพัฒนาขึ้นใหม่ 2 ชนิด เปรียบเทียบกับเซลล์เอทซ์อีซีพีชนิด 2 ชั้นตอน และโททอลเอทซ์อีซีพีชนิด 3 ชั้นตอน และเพื่อหาผลของความขึ้นบนผิวฟันที่มีต่อค่ากำลังแรงยึดแบบดึงระดับจุลภาคของเซลล์เอทซ์อีซีพีชนิดชั้นตอนเดียวชนิดใหม่

วัสดุและวิธีการ นำฟันกรามมนุษย์ซี่ที่สามจำนวน 25 ซี่มาตัดให้ได้ผิวเนื้อฟันบริเวณกึ่งกลางตัวฟันที่เรียบ และแบ่งฟันออกเป็น 5 กลุ่มการทดลองโดยการสุ่ม ได้แก่ กลุ่มแอตเปอร์สก็อตซ์บอนด์มัลติเพอร์โพส กลุ่มเคลียร์ฟิล เอสอีบอนด์ กลุ่มแอตเปอร์เอสอีพลัสเซลล์เอทซ์อีซีพี กลุ่มแอตเปอร์อีซีบอนด์เซลล์เอทซ์อีซีพี และกลุ่มแอตเปอร์อีซีบอนด์เซลล์เอทซ์อีซีพีในสภาพผิวฟันที่มีความขึ้นนำแอตเปอร์แต่ละกลุ่มมาใช้ยึดติดเรซินคอมโพสิตกับผิวเนื้อฟันที่เตรียมไว้ ภายหลังจากนำฟันตัวอย่างไปแช่ในน้ำกลั่นเป็นเวลา 24 ชั่วโมง แ่งขึ้นทดสอบเรซิน-เนื้อฟัน (1 ตารางมิลลิเมตร) ถูกนำไปทดสอบกำลังแรงยึดแบบดึงระดับจุลภาคที่ความเร็วหัวกด 1 มิลลิเมตรต่อวินาที ผลการทดลองถูกวิเคราะห์โดยใช้การวิเคราะห์ความแปรปรวนทางเดียว และใช้การทดสอบแทมเฮนส์ทดสอบความแตกต่างค่าเฉลี่ยแบบพหุคูณ ที่ระดับนัยสำคัญ 0.05

ผลการศึกษา จากการวิเคราะห์ความแปรปรวนทางเดียวแสดงให้เห็นว่ามีความแตกต่างอย่างมีนัยสำคัญของค่าเฉลี่ยกำลังแรงยึดแบบดึงระดับจุลภาคในกลุ่มการทดลอง โดยการทดสอบความแตกต่างค่าเฉลี่ยแบบพหุคูณเผยให้เห็นว่าแอตเปอร์สก็อตซ์บอนด์มัลติเพอร์โพสมีค่ากำลังแรงยึดเฉลี่ยสูงสุด แต่ไม่แตกต่างจากค่ากำลังแรงยึดเฉลี่ยที่ได้จากเคลียร์ฟิลเอสอีบอนด์ และแอตเปอร์อีซีบอนด์เซลล์เอทซ์อีซีพีในสภาพผิวฟันที่มีความขึ้นอย่างมีนัยสำคัญ ค่ากำลังแรงยึดเฉลี่ยของแอตเปอร์อีซีบอนด์เซลล์เอทซ์อีซีพีมีความแตกต่างอย่างมีนัยสำคัญระหว่างกลุ่มที่ใช้ยึดติดกับสภาพผิวฟันที่มีความขึ้น และกลุ่มที่ใช้ยึดติดกับสภาพผิวฟันที่แห้ง โดยสภาพผิวฟันที่มีความขึ้นให้ค่าแรงยึดเฉลี่ยที่สูงขึ้น ส่วนแอตเปอร์เอสอีพลัสเซลล์เอทซ์อีซีพีแสดงค่ากำลังแรงยึดเฉลี่ยต่ำที่สุด โดยแตกต่างจากค่ากำลังแรงยึดเฉลี่ยที่ได้จากทุกกลุ่มการทดลองอย่างมีนัยสำคัญยกเว้นกลุ่มแอตเปอร์อีซีบอนด์เซลล์เอทซ์อีซีพี

สรุป ค่ากำลังแรงยึดกับเนื้อฟันของแอดเปอร์สก็อตซ์บอนด์มัลติเพอร์โพส และเคลียร์ฟิลเอสอีบอนด์มีค่าสูงกว่ากำลังแรงยึดของเซลฟ์เอทซ์แอดฮีซีฟที่ถูกพัฒนาขึ้นใหม่ (แอดเปอร์เอสอีพลัสเซลฟ์เอทซ์แอดฮีซีฟ และแอดเปอร์อีซีบอนด์เซลฟ์เอทซ์แอดฮีซีฟ) อย่างไรก็ตามเมื่อนำแอดเปอร์อีซีบอนด์เซลฟ์เอทซ์แอดฮีซีฟมาใช้ยึดติดกับผิวเนื้อฟันที่มีความชื้น ค่าเฉลี่ยกำลังแรงยึดแบบดึงระดับจุลภาคมีค่าสูงขึ้นอย่างมีนัยสำคัญ

(ว ทันต จุฬาฯ 2552;32:191-202)

คำสำคัญ: กำลังแรงยึดแบบดึงระดับจุลภาค; เซลฟ์เอทซ์แอดฮีซีฟ
