Measurement of Contact Angle and Bond Strength Using 3 Different Self-Etching Primer

Seok-Woo Chang[1], Ho-Beom Kwon[2], Hyun-Mi Yoo[1], Dong-Sung Park[1], Tae-Seok Oh[1], Kwang-Shik Bae[3,4*]

[1]*Department of Conservative Dentistry, The institute of Oral Health Science, Samsung Medical Center, Sungkyunkwan University School of Medicine*
[2]*Department of Prosthodontics, The institute of Oral Health Science, Samsung Medical Center, Sungkyunkwan University School of Medicine*
[3]*Department of Conservative Dentistry, College of Dentistry, Seoul National University*
[4]*Dental Research Institute, Seoul National University*

The purpose of this study was to evaluate the contact angle made by 3 kinds of self etching primers (Clearfil SE Bond, AdheSE, and Tyrian) on dentin and to measure the microtensile bond strength of resin composite to dentin using these self-etching primers.

Contact angle between each of 3 self etching primers and polished dentin surface was measured (n=30) by contact angle analyzer and the result was analyzed by One-way ANOVA.

For the measurement of microtensile bond strength, polished dentin surface was treated with each of 3 self etching primers and dentin adhesives. Z-250 composite resin was built-up with a height of 5 mm on the adhesive-treated surface and light cured for 40s with a halogen light curing unit. Thereafter, each tooth was sectioned into slabs perpendicular to the bonded interface and trimmed (n=45). The microtensile bond strength was measured with universal testing machine and the result was analyzed with Kruskal-Wallis test.

AdheSE group showed the highest contact angle followed by Clearfil SE group and Tyrian group ($p<0.05$). AdheSE group and Clearfil SE group showed significantly higher microtensile bond strength than Tyrian group ($P<0.05$).

(J.K.A.of Stomatognathic Function and Occlusion 2008:24(1):105-112)

Correspondence to:Kwang-Shik Bae, D.D.S., M.S.D. Ph.D.
Department of Conservative Dentistry, College of Dentistry, Seoul National University,
28 Yeongun-dong, Chongro-gu, Seoul, 110-749, Korea
Tel : 82-2-2072-2650, Fax: 82-2-2072-3859
E-mail : baeks@snu.ac.kr
Received: January 23, Last Revision: 2008, March 10, 2008, Accepted: March 25, 2008

INTRODUCTION

For good dentin bonding[1], adhesive must come in close contact with dentin to enhance molecular attraction, chemical adhesion and mechanical attachment. Primer is used to promote dentin wetting by adhesive. In this sense, dentin wetting property of primer is also important in determining the bond strength of resin to dentin.

Toledano et al.[2-3] measured the contact angle between dentin and self-etching primers. They reported that acid etching changed the dentinal surface by removing smear layer and exposing collagen fibers and hydroxyapatite. They also suggested that the surface free energy and wettability of dentinal surface were increased by the changes induced with acid etching[4].

There have been a few studies[5] that evaluated the contact angle and surface energy of dentin. However there have been very few studies[2] on the dentin wetting ability of self-etching primer.

The purpose of this study is to evaluate the contact angle made by 3 kinds of self etching primers on dentin and to measure the microtensile bond strength (MTBS) of resin composite to dentin using these self-etching primers.

MATERIALS AND METHODS

Clearfil SE Bond (Kuraray, Osaka, Japan), AdheSE (Ivoclar/Vivadent, Schaan, Liechtenstein) and Tyrian (Bisco, Schaumburg, U.S.A). (Table 1) were used in this experiment. (Table 1)

1. Specimen preparation

Extracted human molars were used. The roots of these teeth were embedded in self-curing acrylic resin with acrylic resin mold. Occlusal parallel section was cut in order to expose dentin. Cut surfaces were mechanically polished (500 grit) in a circular grinding to obtain a uniform surface roughness.

2. Contact angle measurement

30 specimens processed as explained above were divided into 3 groups of 10 specimens each, according to 3 different self-etching primers used. Phoenix 300 contact angle analyzer (Surface Electro Optics, Suwon, Korea) was used to measure the contact angle in a thermostatic cell at 25o C. One 0.3 μl drop of each self-etching primer was

Table 1. Tested materials

Product	Steps	Composition
Clearfil SE bond primer (Kuraray, Osaka, Japan)	Apply primer for 20 s	HEMA, MDP, hydrophilic dimethacrylate, water,DL-camphoroquinone, N,N-diethanol-p-toluidine
AdheSE primer (Ivoclar/Vivadent, Schaan, Liechtenstein)	Apply primer for 30 s	Phosphoric acid, acrylate, bis-acrylic acid amine, water, initiators, stabilizers
Tyrian primer (Bisco,Schaumburg, U.S.A)	Apply primer for 10 s Air dry for 10 s	Primer A : thymol blue, ethanol and water Primer B : AMPS, Bis MEP, TPO and ethanol

dispensed on polished dentin to make the contact angle measurement. Images were captured at 0, 3 and 6 minutes after dispensing the primers, with a micro-video system in order to evaluate the resin drop spreading. The video signal was transmitted to a computer that provided contact angle values. The significance of the differences of contact angle values between 3 groups was statistically analyzed by one-way ANOVA, Wilcoxon rank sum and two sample T-test with Bonferroni's correction.

3. Microtensile bond strength test

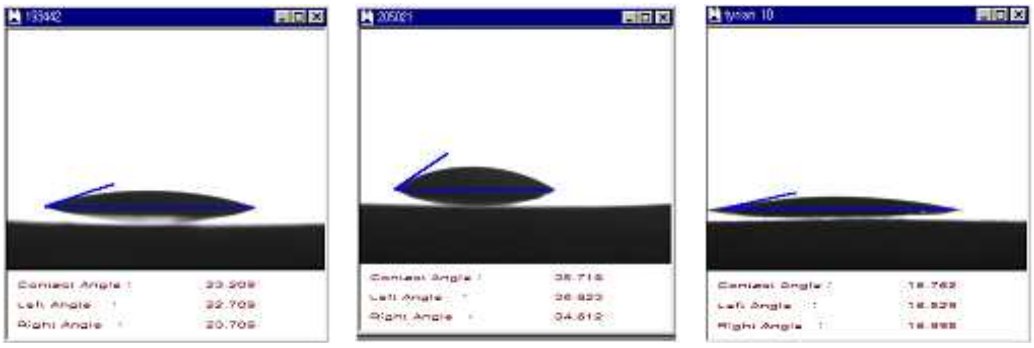
3 kinds of self-etching primers and adhesives were applied following manufacturer's instructions. Table 1 displays mode of application, components and manufacturers of the primer. Composite resin was build up to a height of 5 mm incrementally with Z 250 (3M ESPE, St. Paul, MN, USA). Each layer of composite resin was light cured for 40s with a halogen light curing unit (Elipar TriLight, 3M ESPE, St. Paul, MN, USA). Thereafter, each tooth was sectioned into slabs perpendicular to the bonded interface to produce bonded sections of approximately 0.6 mm thick. In this manner, 45 slabs were obtained, which were divided into 3 groups of 15 slabs each. Slabs were trimmed with a fine diamond bur into hourglass-shaped specimens, with the smallest dimension at the bonded interface (0.6 mm2). The significance of the differences of microtensile bond strength between 3 groups was statistically analyzed by Kruskal-Wallis test with LSD test.

RESULTS

1. Contact angle of self-etching primer is shown in Table 2.

The same letter presents no statistically significant difference. Contact angle of Tyrian could not be measured at 3 minutes after placement because of fast evaporation

Immediately after placement of dentin primer, AdheSE showed the highest contact angle (32.66°) (Fig 1, a). Clearfil SE Bond displayed the intermediary contact angle (22.39°) (Fig 1, b) and Tyrian showed the lowest contact angle (16.91°)



(a) (b) (c)

Fig. 1. contact angle (degrees) measured between polished dentin surface and self-etching primer : (a) AdheSE, (b) Clearfil SE Bond, (c) Tyrian

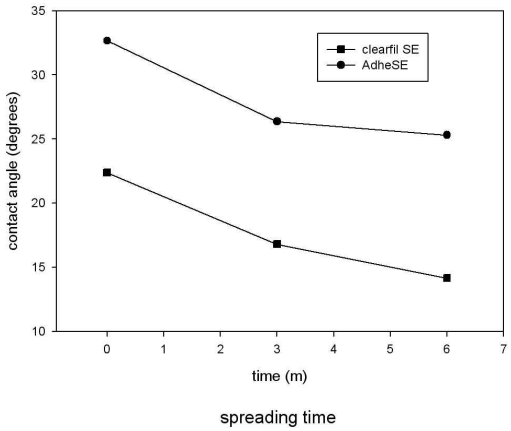


Fig. 2. Spreading times of AdheSE and Clearfil SE Bond measured at 0, 3 and 6 minutes after the placement of each primer. The spreading time of Tyrian could not measured because of fast evaporation.

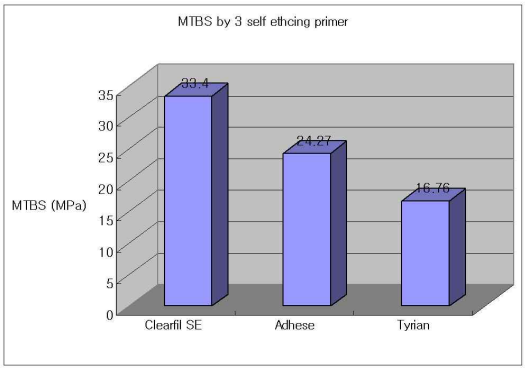


Fig. 3. Microtensile bond strength of Composite bonded to dentin using 3 self-etching primers (Mpa) (MTBS : Microtensile Bond strength)

(Fig 1, c). The significance of differences of these 3 contact angles was statistically analyzed by one-way ANOVA with Bonferroni's correction and showed statistically significant differences among each dentin primers. ($p<0.0004$)

3 minutes after the placement of dentin primer, AdheSE showed higher contact angle (26.37°) than Clearfil SE Bond (16.81°). The significance of difference was analyzed by Wilcoxon rank sum test with Bonferroni's correction. The results showed statistically significant difference ($p = 0.0184$). Tyrian evaporated so fast that the contact angle between Tyrian and polished dentin could not be measured at 3 minutes after placement of Tyrian.

6 minutes after the placement of dentin primer, AdheSE showed higher contact angle (25.31°) than Clearfil SE Bond (14.15°). The significance of difference was analyzed by two sample T-test with Bonferroni's correction. The result showed statistically significant difference ($p<0.0004$).

Clearfil SE Bond and AdheSE showed similar spreading time. The comparison of spreading time of AdheSE and Clearfil SE Bond is shown in Fig. 2.

Table 2. Primer contact angle obtained at 0, 3 and 6 minutes after placement between polished dentin and 3 different dentin primer (mean ± SD) (degrees).

Primer\time (m)	0	3	6
AdheSE (n=10)	32.66 ± 5.56^{b}	26.37 ± 4.49^{a}	25.31 ± 4.31^{a}
Clearfil SE Bond (n=10)	22.39 ± 1.66^{a}	16.81 ± 6.67^{b}	14.15 ± 3.87^{b}
Tyrian (n=10)	16.91 ± 2.19^{c}		

AdheSE and Clearfil SE Bond showed significantly higher microtensile bond strength than Tyrian (p=0.0001). There was no statistically significant difference between the microtensile bond strength of AdheSE and Clearfil SE Bond. The Microtensile bond strength of these 3 self etching primers was showed in Figure 3.

DISCUSSION

Dentin wetting is the first requirement for adhesion to dentin[6]. Miyazaki et al.[7] reported that the wettability of dentin surface is determined by the magnitude of the intermolecular forces between dentin surface and applied liquid. Acid etching was reported to increase roughness[8-9] and wettability of dentin surface[4]. Acid etched surface exposes the hydroxyapatite which presents higher surface energy than those of collagen[4].

There have been some studies that evaluated the contact angle between tooth surface and dental adhesives or resin composites[10]. Rosales-Leal et al. reported that dentin wetting by resins could be correlated to shear bond strength and microleakage[5]. Rosales-Leal et al. studied the dentin wetting ability of four adhesive systems. They evaluated the dentin wetting of scotch bond multipurpose plus (SBMP), syntac single-component, one-step and heliobond by measuring the contact angles. SBMP and syntac showed the lowest contact angle. One-step showed intermediary contact angle and Heliobond showed the highest contact angle. They also evaluated the shear bond strength of these adhesives. The results showed that Heliobond displayed the lowest bond strength, syntac and One-step showed intermediary, and SBMP showed the highest bond strength.

Toledano et al.[2-3] studied the contact angles between dentin surface and dentin primers. They compared the contact angles of Clearfil SE Bond, Etch and Prime, and Scotchbond Multi-purpose plus on polished dentin. However, the relationship between contact angle and shear bond strength was not clearly shown.

In this study, Tyrian demonstrated the lowest contact angle. Clearfil SE Bond and AdheSE showed intermediary and the lowest wetting ability, respectively. However the Microtensile bone strength test showed that Tyrian produced the lowest bond strength and Clearfil SE Bond and AdheSE produced similar bond strength. In this study, the clear relationship between contact angle and microtensile bond strength could not be demonstrated.

Z 250 (3M ESPE, St. Paul, MN, USA) was used in combination with 3 self etching primers to provide the same experimental condition to 3 experimental groups. There was a report that the combination of an adhesive system from one manufacturer with a resin composite from either the same or another manufacturer did not make a difference in bond strength[11].

Aguilar-Mendoza et al.[12] reported that etching of dentin surface with Clearfil SE Bond produced lower contact angle (higher wettability) than AdheSE. Velasques et al. and Atash et al.[13] also reported that Clearfil SE Bond produced significantly higher shear bond strength than AdheSE[14]. On the other hand, Sadek et al.[15] reported that the microtensile bond strength obtained by AdheSE and Clearfil SE Bond showed no statistically significant difference. Sensi et al.[16] also reported that Clearfil SE Bond and AdheSE produced similar shear bond strength and these 2 self etching adhesives produced significantly higher shear bond strength than Tyrian. In this study, Clearfil SE Bond and AdheSE produced similar microtensile bond strength and these 2 self etching systems produced significantly higher microtensile

bond strength than Tyrian. This result is consistent with that of Sensi et al.

Each of the 3 self etching primers used in this study could be classified into strong, mild, and intermediary strong self etching primers by their etching aggresiveness[17]. AdheSE is classified as medium-strong self-etching primer[17]. Clearfil SE Bond is considered to be a mild self-etching primer[13]. Tyrian is classified as a strong self-etching primer[18]. Kenshima et al.[19] investigated the effect of pH of self-etching primers on the bond strength to dentin. In this study, they reported that the microtensile bond strength was not affected by the acidity of self-etching primer. They suggested factors other than pH, such as solvent concentration, the adhesive conversion, cohesive strength of the adhesive system and the hybridization efficiency might have an effect on bond strength[18].

In this experiment, Clearfil SE Bond showed the highest bond strength to dentin, although the difference was not statistically significant. This finding is consistent with many other studies[18-21]. Kenshima et al. reported the high bonding effectiveness of Clearfil SE Bond might be due to 10-methacryloxydecyl dihydrogen phosphate (MDP)[18], which is reported to make very stable bond with hydroxyapatite[22].

Under the experimental condition of this study, it is possible to conclude that the contact angle between dentin and primer alone is not able to explain the differences of microtensile bond strength of composite resin to dentin. Other factors, including the component of bonding system need to be investigated to understand the microtensile bond strength of composite resin to dentin.

CONCLUSION

The result of contact angle measurement showed that Tyrian group showed the lowest contact angle. Clearfil SE Bond group and AdheSE group showed intermediary and the highest contact angle, respectively. The result of microtensile bond strength showed that the Clearfil SE Bond group and AdheSE group showed significantly higher microtensile bond strength than Tyrian group. The relationship between contact angle and microtensile bond strength was not clearly shown.

REFERENCE

1. BEECH DR. Adhesion to teeth: principles and mechanisms in biocompatibility of dental materials FL : CRC Press: D C and Williams; 1982.
2. Toledano M, Osorio R, Moreira MA, Cabrerizo-Vilchez MA, Gea P, Tay FR, et al. Effect of the hydration status of the smear layer on the wettability and bond strength of a self-etching primer to dentin. Am J Dent 2004;17(5):310-4.
3. 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(4):205-10.
4. Toledano M, Osorio R, Perdigao J, Rosales JI, Thompson JY, Cabrerizo-Vilchez MA. Effect of acid etching and collagen removal on dentin wettability and roughness. J Biomed Mater Res 1999; 47(2): 198-203.
5. Rosales-Leal JI, Osorio R, Holgado-Terriza JA, Cabrerizo-Vilchez MA, Toledano M. Dentin wetting by four adhesive systems. Dent Mater 2001;17(6): 526-32.
6. Erickson RL. Surface interactions of dentin adhesive materials. Oper Dent 1992;Suppl 5:81-94.
7. Miyazaki M, Hinoura K, Saito H, Onose H, Moore BK. Influence of light irradiation of dentine primers on dentine-resin bond. J Dent 1995;23(6):371-4.
8. Kinney JH, Balooch M, Haupt DL, Jr., Marshall SJ, Marshall GW, Jr. Mineral distribution and dimensional changes in human dentin during demineralization. J Dent Res 1995;74(5):1179-84.

9. Marshall GW, Jr., Balooch M, Kinney JH, Marshall SJ. Atomic force microscopy of conditioning agents on dentin. J Biomed Mater Res 1995;29(11):1381-7.
10. Paris S, Meyer-Lueckel H, Colfen H, Kielbassa AM. Penetration coefficients of commercially available and experimental composites intended to infiltrate enamel carious lesions. Dent Mater 2007;23(6):742-8.
11. Thomson KB, Peutzfeldt A. Resin composites: strength of the bond to dentin versus mechanical properties. Clin Oral Investig 2007;11(1):45-9
12. Aguilar-Mendoza JA, Rosales-Leal JI, Rodriguez-Valverde MA, Cabrerizo-Vilchez MA. Effect of acid etching on dentin wettability and roughness: Self-etching primers versus phosphoric acid. J Biomed Mater Res B Appl Biomater 2008;84(1): 277-85.
13. Atash R, Van den Abbeele A. Bond strengths of eight contemporary adhesives to enamel and to dentine: an in vitro study on bovine primary teeth. Int J Paediatr Dent 2005;15(4):264-73.
14. Velasquez LM, Sergent RS, Burgess JO, Mercante DE. Effect of placement agitation and placement time on the shear bond strength of 3 self-etching adhesives. Oper Dent 2006;31(4):426-30.
15. 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(4):297-302.
16. Sensi LG, Lopes GC, Monteiro S, Jr., Baratieri LN, Vieira LC. Dentin bond strength of self-etching primers/adhesives. Oper Dent 2005;30(1):63-8.
17. Van Meerbeek B, Vargas M, Inoue S, et al Adhesives and cements to promote preservation dentistry. Oper Dent 2001(supplement, 6):119-44.
18. Kenshima S, Francci C, Reis A, Loguercio AD, Filho LE. Conditioning effect on dentin, resin tags and hybrid layer of different acidity self-etch adhesives applied to thick and thin smear layer. J Dent 2006;34(10):775-83.
19. Kenshima S, Reis A, Uceda-Gomez N, Tancredo Lde L, Filho LE, Nogueira FN, et al. Effect of smear layer thickness and pH of self-etching adhesive systems on the bond strength and gap formation to dentin. J Adhes Dent 2005;7(2):117-26.
20. De Munck J, Vargas M, Iracki J, Van Landuyt K, Poitevin A, Lambrechts P, et al. One-day bonding effectiveness of new self-etch adhesives to bur-cut enamel and dentin. Oper Dent 2005;30(1):39-49.
21. Tay FR, Carvalho R, Sano H, Pashley DH. Effect of smear layers on the bonding of a self-etching primer to dentin. J Adhes Dent 2000;2(2):99-116.
22. 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(6):454-8.

3종의 자가부식 프라이머의 상아질계면 접촉각 및 미세인장결합강도에 관한 연구

[1]성균관대학교 의과대학 삼성서울병원 치과진료부 보존과
[2]성균관대학교 의과대학 삼성서울병원 치과진료부 보철과
[3]서울대학교 치과대학 치과보존학교실
[4]서울대학교 치학연구소

장석우[1] · 권호범[2] · 유현미[1] · 박동성[1] · 오태석[1] · 배광식[3,4*]

본 연구의 목적은 3종의 자가부식 프라이머(Clearfil SE Bond, AdheSE, Tyrian) 사용시 상아질계면의 접촉각 및 미세인장결합강도를 측정하는 것이다. 연마된 상아질 표면에 3종의 자가부식 프라이머를 각각 적용한 후 접촉각을 측정하였으며 (n=30), One-way ANOVA를 사용하여 통계처리하였다. 미세인장결합강도측정을 위해서는 상아질 표면에 3종의 자가부식 프라이머 및 상아질 접착제를 각각 적용한 후 광중합 복합레진 (Z 250)을 5mm 두께로 쌓아 올렸다.

그 후 각 시편을 결합계면에 수직으로 분할한 뒤 (n=45) universal testing machine을 사용하여 미세인장강도를 측정하였고 , Kruskal-Wallis test를 통하여 통계처리하였다.

접촉각 측정결과, AdheSE, Clearfil SE Bond, Tyrian 순으로 높은 접촉각을 보였으며 각 군간 통계적 유의성 있는 차이를 보였다 ($p<0.05$). 미세인장결합강도 측정결과 Clearfil SE Bond군과 AdheSE군은 유의성 있는 차이를 보이지 않았으며, Tyrian군은 Clearfil SE Bond 군이나 AdheSE 군에 비해 유의성 있게 작은 미세인장결합강도를 보였다.

주요어 : 미세인장결합강도, 상아질 접착계면, 자가부식 프라이머, 접촉각

Measurement of contact angle and bond strength using 3 different self-etching primer

교신저자 : **배광식**
110-749 서울시 종로구 연건동 서울대학교 치과대학 보존과
E-mail: baeks@snu.ac.kr
원고접수일: 2008년 1월 23일, 원고수정일: 2008년 3월 10일, 원고채택일: 2008년 3월 25일