Effect of smear layer and surface roughness on resin-dentin bond strength of self-etching adhesives

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The purpose of this study was to evaluate the effect of surface roughness and smear layer on the micro-shear bond strength (µSBS) of two self-etching adhesives, Clearfil SE Bond (SE) and Clearfil Tri-S Bond (S3). Flat dentin surfaces were prepared with SiC papers (600-, 180- and 120-grit) and diamond burs (extra fine, medium and coarse). They were further divided into smear-covered and smear-free surfaces and bonded with respective adhesives. µSBS test was performed after water storage at 37°C for 24 h. Smear layer thickness measurement was evaluated by scanning electron microscope (SEM). Surface roughness was examined by contact stylus profilometer. Smear layer thickness and surface roughness were significantly different among all groups (p<0.05). No difference in µSBS was observed among surface prepared by SiC whereas bur-cut smear layer had negative effect on µSBS, especially for S3. Surface roughness from different surface preparations had no influence on µSBS (p<0.05).

Keywords: Self-etching adhesives, Smear layer, Surface preparation, Surface roughness, Micro-shear bond strength

INTRODUCTION

Self-etching adhesives have been claimed to be user-friendly due to their simpler application and less technique sensitivity compared to etch-and-rinse adhesives⁴. Without phosphoric acid etching, smear layer is not completely removed and the remaining portion is then incorporated into hybrid layer. There are many studies reported the adverse effect of smear layer when bonding with self-etching systems, especially mild-self etching adhesives⁴⁻⁶.

Surface preparation methods affect the performance of self-etching adhesives evaluated by the bond strength test⁵⁻⁹ and create distinct characteristics of smear layer⁴⁻⁶. Compared between two commonly used surface preparation methods, SiC papers produce more irregular surfaces and thicker smear layer while dental burs, which is applied in the clinical setting, produce a thinner but denser smear layer⁴⁻⁶. However, both smear layer characteristics and surface roughness demonstrate paradoxical effects on bond performance. For instance, if the smear layer is mechanically eliminated, the remaining intact underlying dentin surface topography roughened from abrasive papers or cutting burs may enhance the bonding ability of mild self-etching adhesive. The presence of rough surface increases the wetting ability of bonding between the adhesives and the surface¹¹. Even though recent studies have reported that the dentin surface preparation had an influence on the characteristic of smear layer⁴ and also affected the bonding performance of self-etching adhesives⁹, there is limited data regarding the independent effect of smear layer and underlying surface roughness on the bond strength of self-etching adhesives.

Therefore, the aim of this study was to evaluate the effects of smear layer and surface roughness on resin-dentin bond strength of self-etching adhesives. The null hypotheses tested were that: 1) there is no effect of different smear layers and 2) surface roughness on the bond strength of self-etching adhesives.

MATERIALS AND METHODS

Tooth selection

Four hundred and six extracted human upper premolars from orthodontic patients aged from 18 to 25 years were used in this study. The inclusion criteria of the selected teeth were free from decay, cracks, restoration and pathologic enamel and dentin. The teeth were stored as anonymous specimens in 0.1% thymol solution and used within 3 months of storage. The teeth were collected under the ethical approval of the Faculty of Dentistry/Faculty of Pharmacy, Mahidol University Institutional Review Board (MU-DT/PY-IRB 2013/006.2501).

Smear layer-covered dentin preparation

The mid-coronal dentin discs, approximately 2 mm thick, were prepared perpendicular to the long axis of teeth using a low speed diamond saw (Isomet™, Buehler, Lake Bluff, IL, USA). Smear layer-covered dentin discs were prepared with 6 different surface preparations: 600-, 180- and 120-grit SiC paper (Buehler) and extra fine (15 µm), medium (80 µm) and coarse (125 µm) tapered diamond burs (856, Intensiv, grancia, Switzerland). For SiC-prepared surface, dentin discs were manually polished with grit-specific SiC paper at a constant weight under running water for 60 s. For diamond bur-prepared surfaces, specific burs were...
utilized in a high-speed handpiece with copious water spray for a 5 light pressure strokes in order to make a uniform surface as described by Saikaew et al.9.

**Smear layer-free dentin preparation**

To obtain smear layer-free dentin, specimens were prepared in the same manner as described above, after which the smear layer was removed by gently brushing with an ultra-soft toothbrush for 60 s under running water and subjected to ultrasonication (CREST 257D, CREST-Ultrasonics, Trenton, NJ, USA) in distilled water for 45 min at 25±2°C. A pilot study (data not shown) to confirm the smear layer removal was performed using a scanning electron microscope (SEM, JSM 6610LV, JEOL, Peabody, MA, USA).

**Smear layer thickness measurement**

The specimens in smear layer-covered dentin group were used to observe the thickness of the smear layer (n=3 per group). A carborundum disk was used to pre-cut 3–4 mm transversal grooves on the pulpal side of the dentin disc. Subsequently, the teeth were fixed in 2.5% glutaraldehyde in 0.1 M Sorensen Phosphate Buffer (pH 7) at 4°C for 12 h, rinsed with 0.2 M Sorensen Phosphate Buffer for 1 h in three different changes, and rinsed for 1 min with deionized water. The discs were then dehydrated in increasing concentrations of ethanol: immersing for 20 min in 25, 50 and 75% ethanol, 10 min in 95% ethanol and 50 min in 100% ethanol. The discs were then placed in hexamethyldisilazane (HMDS) for 10 min and allowed to air-dry for 10 min13. Finally, the discs were carefully divided along the groove by a scalpel blade and hammer. The dentin surfaces were then coated with gold and observed by SEM at magnification of ×1,000 and ×3,000. The thickness of the smear layer was randomly determined at three sites in each specimen, and the mean thickness of the smear layer was calculated. The data were analyzed using one-way analysis of variance (ANOVA) and Duncan test (α=0.05).

**Surface roughness measurement**

Three specimens for each smear-free group were used. For each specimen, investigations were done randomly at 4 different positions. The surface roughness parameters (the surface area roughness, Sa, and the developed surface area ratio, Sdr14) were collected with a stylus profilometer (Talyscan 150®, Taylor Hobson, Leicester, England). With the 2 µm-radius stylus tip, the surface was measured every 5 µm in 201 profiles in order to cover 1 mm² of the surface area. A cut-off value of 0.08 mm was applied to the profile in order to transform the real profile into long wave components which were responsible for waviness profile and short wave components which were used to form roughness profile. The mean values and standard deviations of Sa and Sdr were calculated and analyzed using one-way ANOVA and Duncan test (α=0.05).

**Micro-shear bond strength (µSBS) test**

The specimens in the smear layer-covered dentin and smear layer-free dentin groups with 6 different surface roughness were randomly divided into 2 subgroups, depending on the adhesive used: Clearfil SE Bond (SE, Kuraray Medical, Osaka, Japan) and Clearfil S3 bond (S3, Kuraray Noritake Dental, Tokyo, Japan). Each adhesive was applied following the manufacturer’s instructions (Table 1). A plastic tube, 0.8 mm in diameter and 1.0 mm in height, was placed on the uncured adhesive surface. The adhesives were polymerized to stabilize the plastic tube with a light curing unit (Bluephase G2, Ivoclar Vivadent, Schann, Liechtenstein). After curing, a resin composite (Filtek™ Z250, 3M ESPE, St. Paul, MN, USA) was placed into the plastic tube and cured for 20 s. Fifteen specimens per group were prepared. All specimens were stored in distilled water at 37°C for 24 h. The plastic tubes were carefully removed and the specimens were checked under a light microscope before testing. Specimens with any defect at the bonding interfaces were excluded from the study. The restored specimens were placed on a universal testing machine (Instron 5566H, Instron, Buckinghamshire, UK) with a

| Table 1 | Compositions of adhesives used in the study |
|---------|------------------------------------------|
| **Adhesive** | **Composition** | **pH** | **General application** |
| Clearfil SE Bond (01173A) | Primer: 10-MDP, HEMA, Hydrophilic dimethacrylate, Di-camphorquinone, N,N-diethanol-p-toluine, water Bond: 10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, di-camphorquinone, N,N-diethanol-p-toluine, silanated colloidal silica | 2.0 | 1. apply primer 20 s 2. dry with mild air flow 3. apply bonding 4. air flow gently 5. light cure for 10 s | |
| Clearfil S3 Bond (00040C) | 10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, di-camphorquinone, ethanol, water, silanated colloidal silica | 2.7 | 1. apply adhesive 20 s 2. high pressure air flow 5 s 3. light cure for 10 s | |

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, Bis-GMA: bisphenol-A-diglycidyl methacrylate

*The pH of adhesives was obtained from references20,22. 
cyanoacrylate adhesive (Model Repair II Blue, Dentsply, SANKIN, Tokyo, Japan). A thin wire was looped around the resin cylinder and gently held flush against the resin-dentin interface. A shear force was applied at a crosshead speed of 1.0 mm/min until failure occurred. μSBS was expressed in MPa, and data were analyzed by three-way ANOVA and Duncan tests (α=0.05).

SEM observation of fractured dentin
The fractured dentin surfaces were examined under SEM to determine the mode of failure at magnifications of ×100 and ×500. Fractured modes were classified into 4 groups: A, Adhesive failure; CC, Cohesive failure within composite resin; CD, cohesive failure within dentin; or M, mixed failure. Statistical evaluation was performed via Kruskal-Wallis tests for non-parametrical analysis (α=0.05).

SEM observation of resin-dentin interface
Three teeth were examined for each group. The teeth were prepared in the same manner as the specimens for the bond strength measurements. The specimens were vertically sectioned through the bonded interface with the low-speed diamond saw. All cut specimens were stored in buffered formalin for 24 h and rinsed for 1 h. Each specimen was embedded in an epoxy resin (AKASEL, Vanlose Sand 2, Melrose, Denmark). The sectioned sides of the specimens faced the outside of the epoxy resin. The sectioned surfaces were ground with 600-, 800-, 1000- and 1200- grit SiC papers. Subsequently, the specimens were further polished with diamond paste at particle sizes of 6, 3, 1 and 0.25 µm. The specimens were then subjected to 10% phosphoric acid treatment for 3 s followed by 3 min of 5.25% sodium hypochlorite immersion. Finally, all specimens were coated with gold, and observed under SEM.

RESULTS

Smear layer thickness
Table 2 shows the means and standard deviations of the smear layer thickness. The thickness of the smear layer was significantly different among all groups (p<0.05) and increased with increased abrasive size for each instrument. The thickest smear layer was observed on the surface abraded by 120-grit SiC paper. The burs-prepared smear layers were thinner than those prepared by the corresponding grit SiC papers.

| Surface preparation (abrasive size in µm) | Mean thickness (SD, µm) |
|------------------------------------------|------------------------|
| 600-grit SiC paper (14.5)                | 1.35 (0.19)            |
| 180-grit SiC paper (78)                 | 2.10 (0.17)            |
| 120-grit SiC paper (116)                | 2.40 (0.25)            |
| extra fine diamond bur (15)             | 0.95 (0.14)            |
| medium diamond bur (80)                 | 1.47 (0.16)            |
| coarse diamond bur (125)                | 2.24 (0.19)            |

SD=standard deviation
Smear layer thickness of all groups were statistically significant difference (p<0.05).

Dentin surface roughness measurement of smear-free surfaces
The smoothest surfaces were found in the 600-grit SiC-prepared group. Surface roughness gradually increased with 180- and 120-grit SiC, respectively. The roughness within the diamond bur group gradually increased with increasing grit sizes from extra fine to medium to coarse diamond bur. Both Sa and Sdr values of all groups are shown in Table 3. The data indicated statistically significant difference among all groups (p<0.05). Representative SEM images of smear-free surface are shown in Fig. 1. SiC-prepared surfaces were more homogenized whereas bur-prepared surfaces were wavy.

µSBS test
The data comparing the μSBS on SiC-prepared dentin and bur-cut dentin are shown in Figs. 2 and 3, respectively. For SiC-prepared surfaces, the results indicated a significant effect of adhesive (F=156.734, p<0.001) on μSBS whereas effect of surface roughness (F=0.320, p=0.727) and smear layer (F=1.720, p=0.191) were not significant. There was an interaction between adhesive and smear layer (F=5.345, p=0.022). For bur-cut dentin, there were significant effects of adhesive (F=162.335, p<0.001) and smear layer (F=106.906, p<0.001) but no significant effect of surface roughness was found (F=0.901, p=0.408). There was an interaction between adhesive and surface created by diamond bur (F=3.144, p=0.046).

SE performed higher μSBS compared to S3 regardless of surface conditions (Figs. 2 and 3). For both adhesives, there was no difference in μSBS between
Table 3  Dentin surface roughness. Comparison of dentin surface roughness with different coarseness and without smear layer (n=12/group)

| Surface preparation         | Mean of surface roughness parameters (SD) |
|-----------------------------|-------------------------------------------|
|                             | Sa* (µm)  | Sdr* (%) |
| 600-grit SiC paper          | 0.14 (0.03) | 0.10 (0.03) |
| 180-grit SiC paper          | 0.50 (0.02) | 0.64 (0.05) |
| 120-grit SiC paper          | 0.84 (0.07) | 1.47 (0.21) |
| Extra fine diamond bur      | 0.24 (0.02) | 0.19 (0.03) |
| Medium diamond bur          | 1.08 (0.07) | 1.73 (0.27) |
| Coarse diamond bur          | 1.45 (0.11) | 2.95 (0.32) |

SD: standard deviation, Sa: surface area roughness, Sdr: developed surface area ratio
Both Sa and Sdr of all groups were statistically significant difference (p<0.05).

*Sa is measured as the arithmetic mean of the absolute values of the surface departure from the mean plane of the sampling area
*Sdr is known as the developed interfacial area ratio, which is the percentage of additional surface area contributed by the texture as compared to an ideal plane the size of the measurement region.

![Fig. 1](image)

Fig. 1  Representative SEM images of smear-free surfaces (×3,000).
(A) Dentin surface prepared by SiC 180-grit (top view). (B) Dentin surface prepared by coarse diamond bur (top view). (C) Dentin surface prepared by SiC 180-grit (vertical view). (D) Dentin surface prepared by coarse diamond bur (vertical view).

smear-free and smear-covered dentin when bonded to SiC prepared dentin (Fig. 2). On the other hand, the adverse effect of smear layer on µSBS was detected when bonded to bur-prepared dentin. The bond between adhesives to smear-covered dentin demonstrated lower µSBS compared to those bonded to smear-free dentin.

More profound effect was observed in S3 with coarse diamond bur-prepared surface.

Fracture mode analysis
In general, adhesive failure was predominantly observed, followed by mixed failure. Figures 2 and 3 show the
Observation of the prepared surface and the resin-dentin interface
Representative resin-dentin interfaces imaged by SEM are shown in Fig 4. The hybrid layer thicknesses of both adhesives were approximately 1–2 µm. The resin tags of SE were in conical shapes while the S3 resin tags were cylindrical. When comparing the resin-dentin interface of smear-free and smear-covered groups, no difference was detected between the groups using SE. In contrast, major differences were found in groups using S3. The fewer and thinner resin tags were observed in smear-covered dentin compared to smear-free dentin with the same roughness. It was also noted that the resin tags of S3 were poorly formed especially in the coarse diamond
DISCUSSION

Smear layer thickness increased with the increased grit size of the abrasives (Table 2), consistent with previous studies\(^2,15,16\). However, with the different abrasive methods, the smear layers created by SiC papers were thicker than those of diamond burs. Additionally, smear layers created by different grit-size SiC papers had no effect on \(\mu\)SBS of both adhesives tested (Fig. 2). This observation is in agreement with previous studies which performed bond strength test with SiC prepared dentin. They reported that smear layer thickness has no influence on bond strength of self-etching adhesives\(^7,8,10,17\). On the contrary, adverse effect of smear layer was reported by studies performed with bur-prepared dentin. They reported that smear layer thickness has no influence on bond strength of self-etching adhesives\(^7,8,10,17\). On the contrary, adverse effect of smear layer was reported by studies performed with bur-prepared dentin\(^3,5,18\). This might be explained by the different characteristics of smear layer caused by the different cutting speed. The higher cutting speed of diamond burs could force the smear layer into dense and thin layer whereas the smear layers created by SiC paper were thicker but loosely organized\(^2,10\). The loosely organized smear layer created by SiC papers allows both SE and S3 to readily penetrate and form a strong bond to underlying dentin. On the contrary, adverse effect of bur-prepared smear layer on \(\mu\)SBS between these two adhesives was observed (Fig. 3). Likewise, smear-free dentin demonstrated significantly higher bond strength than those of smear layer-covered dentin. Therefore, the first null hypothesis was rejected \((p<0.001)\) since the smear layer characteristics had a significant effect on bond strength of self-etching adhesives.

For SE, there was no statistically significant difference in mean \(\mu\)SBS when applied with different abrasive sizes (Figs. 2 and 3), however, the mean \(\mu\)SBS (slightly) reduced in the bur-cut smear layer group. More profound effect was observed in S3 group where the decreased bond strengths associated with greater abrasive size of diamond bur. As some authors suggested the active application of adhesives to improve the bonding performance of mild self-etching adhesives\(^16,19\), this might indicate the buffering effect of smear layer, especially the bur-cut smear layer. In addition, pH of S3 is 2.7\(^20\) which is considered as ultra-mild self-etching adhesive\(^1\), hence, it might be difficult for S3 to infiltrate through dense smear layer of bur-cut dentin. Our SEM images also supported this hypothesis since resin tags of S3 with bur-cut dentin were short and poorly formed compared to those with smear-free dentin (Fig. 4). The evidence of insufficient resin infiltration through bur-cut dentin was also demonstrated in previous studies by \(\text{SEM}\)\(^5\) and \(\text{TEM}\)\(^6\). In addition, S3 is one-step self-etching adhesive which was reported as compromise materials due to some shortcomings\(^1\). Therefore, it might be concluded that the quality of hybrid layer, especially with the one-step, ultra-mild self-etching adhesive, was affected by surface preparation.

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Fig. 4  Representative SEM images of resin-dentin interface prepared by coarse diamond bur.

(A) SE bonded to smear-free surface. (B) S3 bonded to smear-free surface. (C) SE bonded to smear covered surface. (D) S3 bonded to smear covered surface.

C=resin composite, A=adhesive layer, H=hybrid layer, D=dentin
A number of studies have reported the effects of smear layer and surface roughness on adhesive performance. However, most of these studies performed the experiment only on smear layer-covered dentin. Fractured specimens have been used to prepare smear layer-free dentin, although uncontrollable and varying degrees of surface roughness may be found on the fractured surfaces. Furthermore, chemical removal of smear layer using Ethylenediaminetetraacetic acid (EDTA) was also reported but, due to an etching effect, the altered mechanical properties of the treated dentin can be expected. Therefore, in this study, we attempted to mechanically remove the smear layer using a modified method from Nakabayashi and Saimi to maintain dentin surface topography. From our pilot experiment, only ultrasonication was unable to completely remove smear layer from dentin surface. Thus, the additional mechanical removal with ultra-soft toothbrush was applied prior to ultrasonic step. Our preliminary study also confirmed that there was no difference in roughness profile between the dentin surface before and after smear layer removal. This modified technique may be the most realistic method to remove the smear layer without affecting the underlying dentin surface topography. Therefore, with smear-free dentin, the real effect of surface roughness on resin-dentin bond strength can be evaluated.

As the resin-dentin bond strength of adhesive depends mainly on micro-mechanical interlocking through hybrid layer, it has been suggested that the increase in surface area of substrate by increasing roughness can improve the bond strength of adhesive. There is also a suggestion that a balance between roughness and smear layer thickness should be obtained in order to produce higher bond strengths for the self-etching adhesive like SE. However, in our study, although the surface roughness parameters (Sa and Sdr) were significantly higher when we used rougher abrasives (Table 3), but with larger surface area, the bond strengths were not significantly different (Figs. 2 and 3). It is also worth noting that despite using a coarse diamond bur which created the most irregular surface (Fig. 1D), the surface area was increased only by 2.95% (Table 3). Therefore, the second null hypothesis that there is no effect of surface roughness on bond strength of self-etching adhesives was not rejected.

In clinical practice, there are some situations that cavity preparation with diamond bur is necessary preparation for indirect restorations. According to our results, the increased surface roughness created by diamond burs had no benefit on bond strength of self-etching adhesives. Contrarily, bur-cut smear layer caused negative effect on bond strength of self-etching adhesives especially with one-step, ultra-mild self-etching adhesive. Therefore, it might be suggested that fine-grit diamond burs are recommended for cavity preparation in case of using self-etching adhesives. Future study should be performed on the bonding modification technique to improve the bond strength with regular or coarse diamond burs.

### CONCLUSION

Using the surface preparation methods demonstrated in this study, surface roughness had no effect on the µSBS of self-etching adhesives. SiC-prepared smear layer had no influence on bond strength of both adhesives tested. On the contrary, the efficacy of self-etching adhesives was reduced when applied on bur-cut smear layer, especially for S3.

### REFERENCES

1. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. Dent Mater 2011; 27: 17-28.
2. Sattabansuk V, Vachiramon V, Qian F, Armstrong SR. Resin-dentin bond strength as related to different surface preparation methods. J Dent 2007; 35: 467-475.
3. Semeraro S, Mezzanzanica D, Spreatcio D, Gagliani M, Re D, Tanaka T, Sidhu SK, Sano H. Effect of different bur grinding on the bond strength of self-etching adhesives. Oper Dent 2006; 31: 317-323.
4. Koibuchi H, Yasuda N, Nakabayashi N. Bonding to dentin with a self-etching primer: the effect of smear layers. Dent Mater 2001; 17: 122-126.
5. Ermis RB, De Munck J, Cardoso MV, Coutinho E, Van Landuyt KL, Poitevin A, Lambrechts P, Van Meerbeek B. Bond strength of self-etch adhesives to dentin prepared with three different diamond burs. Dent Mater 2008; 24: 978-985.
6. Mine A, De Munck J, Cardoso MV, Van Landuyt KL, Poitevin A, Van Ende A, Matsumoto M, Yoshida Y, Kuboki T, Yatani H, Van Meerbeek B. Dentin-smear remains at self-etch adhesive interface. Dent Mater 2014; 30: 1147-1153.
7. 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: 283-289.
8. Keshishma S, Reis A, Uceda-Gomez N, Tancredo Lde L, Filho LE, Nogueira FN, Loquercio AD. 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: 117-126.
9. Saakp W, Chowdhury AFMA, Fukuyama M, Kakuda S, Carvalho RM, Sano H. The effect of dentine surface preparation and reduced application time of adhesive on bond strength. J Dent 2018; 47: 63-70.
10. Reis A, Grandi V, Carlotto L, Bortoli G, Patzlaff R, Rodrigues Accorinte MdL, Dourado Loguercio A. The effect of different bur grinding preparation methods. J Dent 2007; 35: 467-475.
11. Ayad MF, Rosenstiel SF, Hassan MM. Surface roughness of dentin after tooth preparation with different rotary instrumentation. J Prosthet Dent 1996; 75: 122-128.
12. Ayad MF, Rosenstiel SF, Salama M. Influence of tooth surface roughness and type of cement on retention of complete cast crowns. J Prosthet Dent 1997; 77: 116-121.
13. Perdigao J, Lambrechts P, Van Meerbeek B, Vanherle G, De Baere E. Field emission SEM comparison of four postfixation drying techniques for human dentin. J Biomed Mater Res 1995; 29: 1111-1120.
14. Culi P, Alaeddin S, Wenerberg A, Karlsson S. In vitro dentin pretreatment: surface roughness and adhesive shear bond strength. Eur J Oral Sci 1999; 107: 400-413.
15. Oliveira SSA, Pugach MK, Hilton JP, Waterabe LG, Marshall SJ, Marshall Jr GW. The influence of the dentin smear layer on adhesion: a self-etching primer vs. a total-etch system. Dent Mater 2003; 19: 758-767.
16. Thanatvarakorn O, Prasansuttiporn T, Takahashi M, Thittaweerat S, Foxton RM, Ichinose S, Tagami J, Nakajima
M. Effect of scrubbing technique with mild self-etching adhesives on dentin bond strengths and nanoleakage expression. J Adhes Dent 2016; 18: 197-204.

17) 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: 99-116.

18) Koase K, Inoue S, Noda M, Tanaka T, Kawamoto C, Takahashi A, Nakaoki Y, Sano H. Effect of bur-cut dentin on bond strength using two all-in-one and one two-step adhesive systems. J Adhes Dent 2004; 6: 97-104.

19) 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: 340-346.

20) Iwasa M, Tsugita K, Shimamura Y, Ando S, Miyazaki M, Platt JA. pH changes upon mixing of single-step self-etching adhesives with powdered dentin. J Adhes Dent 2011; 13: 207-212.

21) Ogata M, Harada N, Yamaguchi S, Nakajima M, Tagami J. Effect of self-etching primer vs phosphoric acid etchant on bonding to bur-prepared dentin. Oper Dent 2002; 27: 447-454.

22) Vanlanduyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, Van Meerbeek B. Systematic review of the chemical composition of contemporary dental adhesives. Biomaterials 2007; 28: 3757-3785.

23) Suyama Y, Luhrs AK, De Munck J, Mine A, Poitevin A, Yamada T, Van Meerbeek B, Cardoso MV. Potential smear layer interference with bonding of self-etching adhesives to dentin. J Adhes Dent 2013; 15: 317-324.

24) Kusunoki M, Itoh K, Oikawa M, Hisamitsu H. Measurement of shear bond strength to intact dentin. Dent Mater J 2010; 29: 199-205.

25) Nakabayashi N, Saimi Y. Bonding to intact dentin. J Dent Res 1996; 75: 1706-1715.

26) Nakabayashi N, Ashizawa M, Nakamura M. Identification of a resin-dentin hybrid layer in vital human dentin created in vivo: durable bonding to vital dentin. Quint Int 1992; 23: 135-141.