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Corresponding Author:
Dr. Boondarick Niyatiwatchanchai,
Department of Operative Dentistry,
Faculty of Dentistry, Chulalongkorn
University, Bankgkok 10330, Thailand.
E-mail: boondarick.n@gmail.com

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Effect of Self-etch Silane Contamination on Dentin Bond Strength to Resin Composite

Chanakan Paaopanchon[1], Boondarick Niyatiwatchanchai[1]

[1]Department of Operative Dentistry, Faculty of Dentistry, Chulalongkorn University, Thailand

Abstract

Objectives: This study aimed to investigate the effect of self-etch silane contamination on dentin bond strength to resin composite when using different adhesive systems.

Methods: 40 flat occlusal dentin surfaces were prepared and randomly divided into 4 groups (n=10): group ER (Optibond FL); group SE (Clearfil SE bond); group SiER and group SiSE (Monobond Etch and Prime (MEP) followed by Optibond FL and Clearfil SE bond, respectively). Microtensile bond strength (μTBS) data was analyzed by two-way and one-way ANOVA followed by the post hoc Tukey honest test. The failure mode data was analyzed using Pearson Chi-square test. After undergoing different contamination procedures (distill water, phosphoric acid, and acidic primer with and without MEP contamination), 6 additional specimens were analyzed by scanning electron microscopy (SEM).

Results: The two-way ANOVA indicated that the adhesive system and silane contamination significantly influenced the μTBS. μTBS (MPa) of all (ER 47.79±3.48; SiER 41.16±11; SE 39.77±3.16; SiSE 35.10±4.12) groups were significantly different from each other, except for the SE versus SiER group. The silane contamination significantly decreased the μTBS for both adhesive systems. Adhesive failure was the most common failure mode for the SiER, SE, and SiSE groups.

Conclusions: Self-etch silane cross-contamination on dentin negatively impacted the μTBS of etch-and-rinse and self-etch adhesive systems. However, the etch-and-rinse adhesive system may be more effective in mitigating the effects of dentin contamination than the self-etch adhesive system.

Keywords: ammonium polyfluoride, bond strength, contamination, dentin, self-etch silane

Introduction

The silane coupling agents are used as adhesion promoters in dentistry between the resin matrix of the resin-based materials and an inorganic substrate. Acid-etching and silane priming of etchable glass ceramic restorations have been mandatory for strong and long-lasting bonding to tooth structures.[1] Despite the variety of etchable ceramics used in dentistry, acid-etching with hydrofluoric acid (HF, 5-10 wt%) and silanization with hydrolyzed 3-methacryloxypropyl trimethoxysilane (MPS) have been instructed for surface pretreatment, before resin luting agents' application.[1-3]

The latest generation of silane primer is known as a self-etch one-bottle system, self-etch silane primer, or single-step pre-hydrolyzed silane solutions. The example of this single bottle ceramic primer is Monobond Etch & Prime (MEP) (Ivoclar Vivadent, Schaan, Liechtenstein). Because it contains a MPS for silanization and a new ammonium polyfluoride, tetrabutyl ammonium dihydrogen trifluoride (TADF), for the etching step. The product also contains a methacrylated phosphoric acid ester.[4]

The fracture of dental ceramics remains a major concern in restorative dentistry. The primary causes of ceramic fracture include microdefects in the material, impact and fatigue loads, improper design, mastication, parafunction, and intraoral occlusal pressures that induce persistent dynamic loading.[5] In addition, cervical recession, microleakage, caries, or discolorations may occur at the margins of restorations.[6] In some situations, ceramic repair may be a more cost-effective and time-saving than re-making the entire ceramic restoration or surgical procedure. In many cases, exposed tooth tissues, such as enamel and dentin, are included in the intraoral repair of ceramics.

It may be difficult to avoid contamination of the dental tissue substrate with self-etch silane during etching, rinsing, or drying procedures. According to ceramic repair protocol, dental application with different adhesive systems may influence the results. Soontornvatin *et al*.,[7] investigated the effect of silane contamination on the μTBS of 3-step etch-and-rinse adhesives on dentin. The study concluded that silane contamination on dentin before the etching step did not affect the dentin bond strengths. However, contamination after etching and priming had a significant negative influence on dentin bond strengths. Lührs *et al*.,[8] investigated the impact of surface contamination on the μTBS of universal adhesives during repair procedures and concluded that contamination with HF acid or an MEP results to a significantly lower bond strength after aging only, but there were no significant differences in the immediate μTBS. In the present, it is unclear whether a MEP would affect dentin bond strength. It is interesting to investigate how MEP contamination affected dentin morphology and dentin bond strength to resin composite when using different adhesive systems.

Therefore, the objective of this study was to investigate the effect of self-etch silane contamination on dentin bond strength to resin composite when using different adhesive systems.

Materials and Methods

Specimen preparation

After approval by the Ethics Committee of the Faculty of Dentistry, Chulalongkorn University No. 053/2022, 46 extracted human third molars that were free of caries, restorations, or cracks were carefully washed under running water and removed blood clots and attached soft tissues by scalpel and ultrasonic scaler (Bransonic, Germany). Then, teeth were immersed in a 0.5% chloramine-T aqueous solution at 4°C and were used within one month after extraction.[9]

All specimens were prepared by sealing root ends with wax and mounting root in a cold-curing acrylic resin base leaving the clinical crown exposed (Figure 1A). The occlusal central groove of the teeth was drilled 1.5 mm in depth using a high-speed cylindrical diamond bur (Jota, Switzerland), ensuring that the dentin exposure was located at bur-end level using a stereomicroscope evaluation (ML9300®, MEIJI, Japan) at 40x magnification. Then, the occlusal enamel was removed perpendicularly to a tooth axis using a low-speed cutting machine (IsoMet 1000, Buehler; Lake Bluff, IL, USA) and stereomicroscope evaluation at 40x magnification to ensure all central area show dentin exposure (Figure 1B). A standardized smear layer on dentin was made using 600–grit silicon carbide paper (TOA, Thailand) with a polishing machine (Nano 2000, Pace technologies, USA) at 100 rounds per minute with 2.27 kg for 30 seconds in one direction under running water, rinsed, and stored in 37°C distill water for 24 hours (Contherm 160M; Contherm Scientific Ltd., Lower Hut, New Zealand) (Figure 1C).[10] Teeth presented with pulp exposure were excluded.

The specimens were randomly divided into 4 groups (n=10 for each group): Group ER and SE: The dentin was subjected to distill water application for 1 minute served as the control followed by water rinsing for 10 seconds and drying with oil-free compressed air for 10 seconds. Then, the ER group was subjected to Optibond FL™ application (Kerr, Orange, CA, USA) as well as the SE group to Clearfil SE bond™ (Kuraray, Kurashiki, Japan). Group SiER and SiSE: 1 coat of MEP (Ivoclar Vivadent, Schaan, Liechtenstein) was applied to dentin according to the manufacturer's instructions followed by Optibond FL™ application for SiER group and Clearfil SE bond™ application for SiSE group. To specify the bonding region of the central dentin, a piece of adhesive tape with a 6x6 mm square shaped hole was located and firmly adhered in the most central area of dentin (Figure 1D). All materials were used strictly according to the manufacturer's instructions as described in Table 1.[7,11-13]

Bonding procedures

Dentin surfaces that were have been etched, primed, and bonded were dried using oil-free compressed air at 0.2 MPa air pressure from 5 cm above the dentin surface using a three-way syringe. An LED light-curing unit (Demi™ LED light-curing system, Kerr, Orange, CA, USA) with an irradiance of 1,000 mW/cm^2 was used to light-polymerize the resin-based materials for 10 seconds at a standardized distance of 2 mm from the bonding surface.

After light-curing of the bonding agents, resin composite buildup with a height of 4.5 mm was created in the central area of each tooth. To form and hold the resin composite onto the dentin surface, a transparent acrylic plate mold 6x6 mm square shaped with 1.5 mm in hight was employed. The resin composite was placed and compacted into the mold (Filtek Z350XT; 3M Oral Care, St. Paul, USA). The excessive material was removed with glass plate. It was made for three increments, each of which was light-cured for 20 seconds with the tip of light curing unit located at a standard distance of 1 mm from the resin composite surface. Before light-curing process of every one specimen, the light energy output was verified at more than 800 mW/cm^2 with a radiometer (Demetron L.E.D Radiometer, Kerr, Orange, CA, USA) throughout the procedure.

After the restorative operation, the specimens were immersed in distilled water and kept in an incubator for 24 hours at a temperature of 37°C.

µTBS testing

After storage, ten specimens were used for each group (n=10). Theses restored teeth were sectioned occluso-gingivally across a bonded interface into stick shaped specimens with 1 mm×1 mm cross-sectional area using a low speed cutting machine (IsoMet® 1000, Buchler, USA).[14]

The four central sticks from each tooth were obtained (Figure 1F, 1G). In contrast, the peripheral area was excluded. Pre-test failures also were excluded from statistical analysis.

According to ISO technical specification 11405:2015, specimens were fixed by their endings to Ciucchi's jig with cyanoacrylate glue (Model Repair II Blue, Dentsply, Ohtawara, Japan) and stressed at a crosshead speed of 1 mm/min until failure in a universal testing machine (EZ-S, Shimadzu, Japan), with a load of 10 kg (Figure 1I). The movement was automatically stopped at the fracture point. The µTBS values were recorded and calculated to the average µTBS (MPa) of each tooth for statistical analysis. Premature failures were excluded from the statistical analysis.

At the ending of the test, the two parts of the sample were stored to investigate the fracture pattern and failure mode.

SEM analysis

SEM was used to examine the specimens' surface morphology of six teeth after undergoing different contamination procedures (distill water or self-etch silane) and after the etching procedure described in Table 1. (Figure 2).

One extra specimen from each group, after contamination procedure and etching procedure, was dehydrated in a series using ethanol solution (Scientific and Technological Research Equipment Centre, Chulalongkorn University, Bangkok, Thailand) (70% for 10 minutes, 95% for 10 minutes and 100% for 20 minutes) then mounted on aluminum stubs, dried in a desiccator, and finally sputter-coated with gold coating. Then, their surfaces were evaluated at magnification of 500X and 3,000X at an acceleration voltage of 15 kV (JSM-6610LV Scanning Electron Microscope JEOL, Japan).

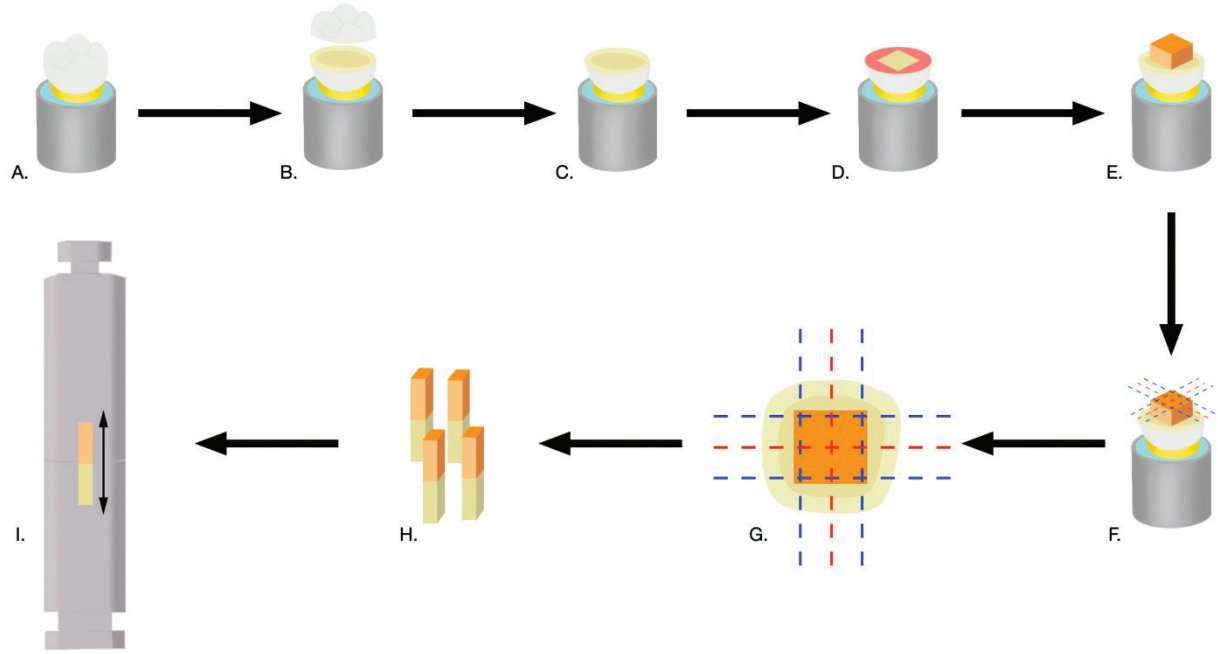

Figure 1: Schematic representation of the procedure for measuring the μTBS of dentin. (A) root mounted in cold-curing acrylic resin, (B) occlusal third removal, (C) standardized smear layer on dentin, (D) 6x6 mm square shaped hole adhesive tape on dentin, (E) composite buildup, (F) serial sectioning, (G) serial sectioning, top view (red dash line: the central reference lines, blue dash line: the adjacent lines), (H) square specimen (1 x 1 x 8-9 mm^3), (I) specimen testing

Table 1: Materials' detail, composition, and manufacturer's instructions

Material and manufacturer	Composition	Manufacturer's instructions
Optibond FL (Kerr, Orange, CA, USA) LOT No. 8308256	Etchant: 37.5% phosphoric acid, silica thickener Primer: HEMA, GPDM, PAMM, ethanol, water, photoinitiator (pH 1.8) Adhesive: TEGDMA, UDMA, GPDM, HEMA, bis-GMA, filler, photoinitiator	**Etch:** Apply Etchant 15 s, rinsed with water 15 s, gently air dry 3 s **Prime:** Apply primer with light scrubbing motion for 15 s, gently air dry 5 s **Bond:** Apply bonding agent with light scrubbing motion for 15 s, remove the excess with a gently air 5 s and light cure for 10 s
Clearfil SE Bond (Kuraray, Kurashiki, Japan) LOT No. 000079	Primer: 10-MDP, HEMA, water, photoinitator (pH1.9) Bond: 10-MDP, bis-GMA, HEMA, hydrophobics dimethacrylates, photoinitator	**Prime:** Apply a layer of primer, wait 20 s, gently air dry 5 s **Bond:** Apply the bonding agent, remove the excess with a gently air 5 s and light cure for 10 s
Monobond Etch&Prime (*MEP*) (Ivoclar Vivadent-Schaan, Lichtenstein) LOT No. Z03024	Butanol, tetrabutyl ammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, bis(triethoxysilyl)ethane, silane methacrylate, colourant, ethanol, water	Actively apply on the ceramic surface for 20 s, let it react for 40 s and wash it with water for 10 s, strong stream of water- and oil-free air for 10 s
Filtek Z350XT (A2 Body) (3M Oral Care, St. Paul, USA) LOT No. NE45910	Bis-GMA, UDMA, TEGDMA, Bis-EMA, non-agglomerated/ non-aggregated 20 nm silica filler, non-agglomerated/ non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/ silica cluster filler	Insert the composite in 2 mm increment and light-cure for 20 s

Abbreviations: HEMA: 2-hydroxyethyl methacrylate; PAMM; Methacroyloxyethyl Phthalate; TEGDMA: triethylene glycoldimethacrylate; Bis-GMA: bisphenol A diglycidyl ether dimethacrylate; 10-MDP: 10-methacryoloyloxydecyl dihydrogen phosphate; GPDM; glycerol phosphate dimethacrylate Bis-EMA; Ethoxylate biphenol A glycol diamethacrylate[7,11-13]

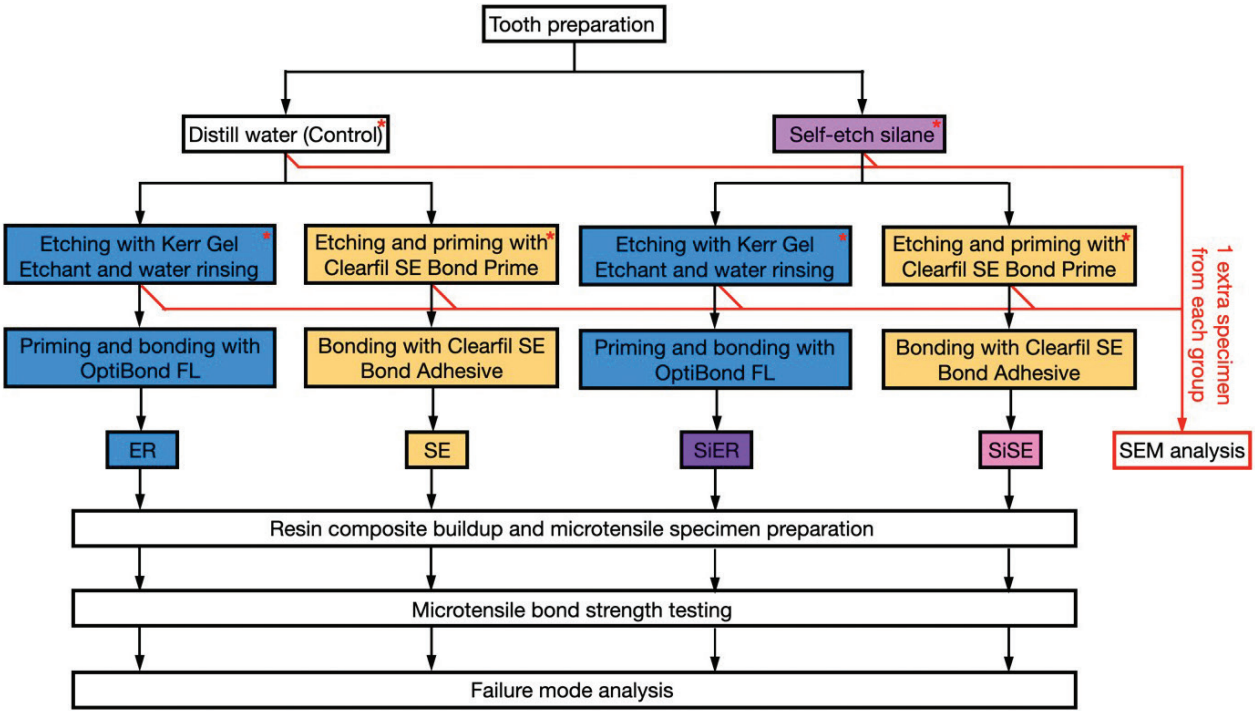

Figure 2: Diagram of study design

Failure mode analysis

After the μTBS testing, the debonded surfaces of all specimens were examined under a stereomicroscope (ML9300®, MEIJI, Japan) at 40x magnification to determine the failure mode. Each specimen was classified into 1 of 4 types as following:

1. Adhesive failure (AF; >80% of the failure area occurred between resin and dentin);
2. Cohesive failure in resin (CFR; >80% of the failure area occurred in dentin/resin, but the majority is in adhesive resin or composite resin);
3. Cohesive failure in dentin (CFD; >80% of the failure area occurred in dentin/resin, but the majority is in dentin);
4. Mixed failure (MF; mixed with adhesive failure between dentin and resin, cohesive failure in resin and/or dentin).[15]

Statistical analysis

Means and standard deviations were calculated and presented in MPa.The Kolmogorov-Smirnov test and Levene's test were used to determine homogeneity. The data was statistically analyzed by 2-way ANOVA and 1-way ANOVA followed by the post hoc Tukey honest significant difference (HSD) test (alpha=0.05), and the failure mode data was analyzed using the nonparametric Pearson Chi-square test with a spreadsheet (Excel Microsoft Office 2010; Microsoft Corp) and a statistical analysis software (SPSS 22.0; SPSS Inc, Chicago, IL, USA.).

Results

μTBS test

The Kolmogorov-Smirnov and Levene's tests indicated that the μTBS data had a normal distribution and homogeneous variances (p=0.54). Mean μTBS data for dentin with and without silane contamination were presented in Table 2. Mean ± standard deviation μTBS of ER was 47.79±3.48 MPa, SiER was 41.16±3.11 MPa, SE was 39.77±3.16 MPa, and SiSE was 35.10±4.12 MPa.The box plots of the μTBS data are shown in Figure 3.

As shown in Table 3, the results of one-way ANOVA indicated that μTBS of all groups were significantly different (p<0.001). Two-way ANOVA revealed that both the adhesive system (p<0.001) and silane contamination (p<0.001) had a significant influence on the μTBS. However, there was no statistically significant interaction between the adhesive system and self-etch silane contamination (p=0.381). Further analysis using the post hoc Tukey HSD test revealed that all groups were signifi-

cantly different from each other, except for the SE versus SiER group (p=0.811) (Table 4). The silane contamination significantly decreased the μTBS for both adhesive systems (p<0.001 for ER versus SiER and p=0.024 for SE versus SiSE). Additionally, the μTBS of the SiER group was significantly higher than the SiSE group (p=0.002), although it was insignificantly higher than the SE group (p=0.811).

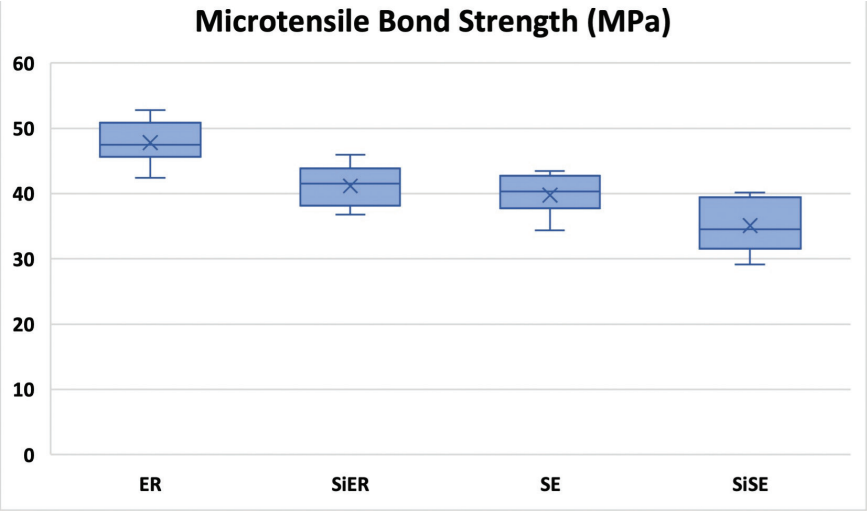

Figure 3: Summary of box plots of μTBS (MPa). Box plot shows the median (+), 25% quartile ([box] bottom line), 75% quartile ([box] top line), maximum (plus error bar), and minimum (minus error bar)

Table 2: Mean μTBS (MPa) on dentin with and without silane contamination

Group	Etch-and-rinse	Self-etch
Control (distill water)	47.79±3.48[a]	39.77±3.16[b]
Silane contamination	41.16±3.11[b]	35.10±4.12[c]

Values are means ±standard deviation (n=10). Means with different superscript letters are statistically different (p<0.05).

Table 3: Summary of 1-way and 2-way ANOVA

Source of Variation	Sum of Squares	df	Mean Square	F	*p*	Partial Eta Squared
1-way ANOVA						
Between groups	824.051	3	274.684	22.556	<0.001	
2-way ANOVA						
Silane contamination	319.300	1	319.300	26.220	<0.001	0.421
Adhesive system	495.180	1	495.180	40.662	<0.001	0.530
Interaction	9.570	1	9.570	.786	0.381	0.021

Table 4: Tukey HSD results

Treatment Pair	Tukey HSD Q statistic	Tukey HSD *p* value	Tukey HSD Inference
ER versus SE	8.015	<0.001	*p<0.05
ER versus SiER	6.629	<0.001	*p<0.05
ER versus SiSE	12.688	<0.001	*p<0.05
SE versus SiER	-1.386	0.811	Insignificant
SE versus SiSE	4.672	0.024	*p<0.05
SiER versus SiSE	6.059	0.002	*p<0.05

ER, etch-and-rinse control; SiER, etch-and-rinse with silane contamination; HSD, honest significant

Failure mode analysis

The analyzed failure mode data of the different groups, as shown in Figure 4, revealed that the most common failure mode for the SiER, SE, and SiSE groups was AF. Particularly, the SiSE group had the highest percentage of adhesive failures at 85%. On the other hand, the ER group predominantly exhibited CFR at a rate of 40%. There were significant differences in failure mode distribution among the groups ($p<0.001$). The failure mode was confirmed by stereomicroscope images, as shown in Figure 5 (original magnification ×40).

SEM analysis

Representative SEM images of dentin surfaces with and without self-etch silane contamination are displayed in Figure 6.

Discussion

According to Van Meerbeek *et al*.,[16] OptiBond FL (Kerr) and Clearfil SE Bond (Kuraray Noritake) were identified as the gold-standard adhesive systems for etch-and-rinse and self-etch techniques, respectively. This designation was based on a comprehensive analysis of laboratory[17] and clinical data[18] through meta-analysis, as well as their exceptional performance in a thirteen-year randomized clinical trial.[19]

This study found that the immediate dentin μTBS of the control ER group was significantly higher than that of the control SE group, which aligns with a previous study.[20] The results indicated that the presence of self-etch silane contamination on dentin leads to a significant decrease in the μTBS for both adhesive systems. However, Liang Chen *et al*.,[15] examined whether the contamination of silane primer would adversely affect tooth adhesion and concluded that silane contamination did not have a negative impact on dentin shear bond strength. According to the use of universal adhesive in self-etch or etch-and-rinse mode, the shear bond strength of the ER and SE groups was insignificantly different. This finding suggests that the choice of adhesive mode does not significantly affect the bond strength in this context.

Furthermore, Soontornvatin *et al*.,[7] investigated the effect of silane primer contamination on the μTBS of two commercial 3-step etch-and-rinse adhesives on dentin. The study indicated that silane contamination on dentin before the etching step did not affect the dentin bond strengths of the 3-step etch-and-rinse adhesives. However, contamination after etching and priming had a significant negative influence on dentin bond strengths.

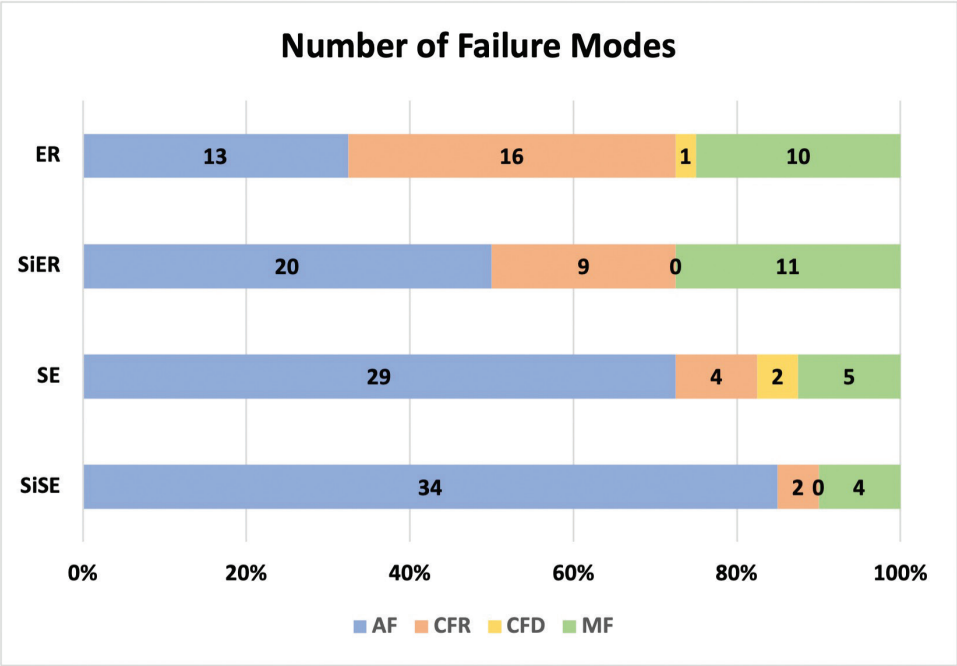

Figure 4: Number of failure modes of resin-dentin bond in each group (n=40). Numbers in each bar represent number of fractional failure modes in each group

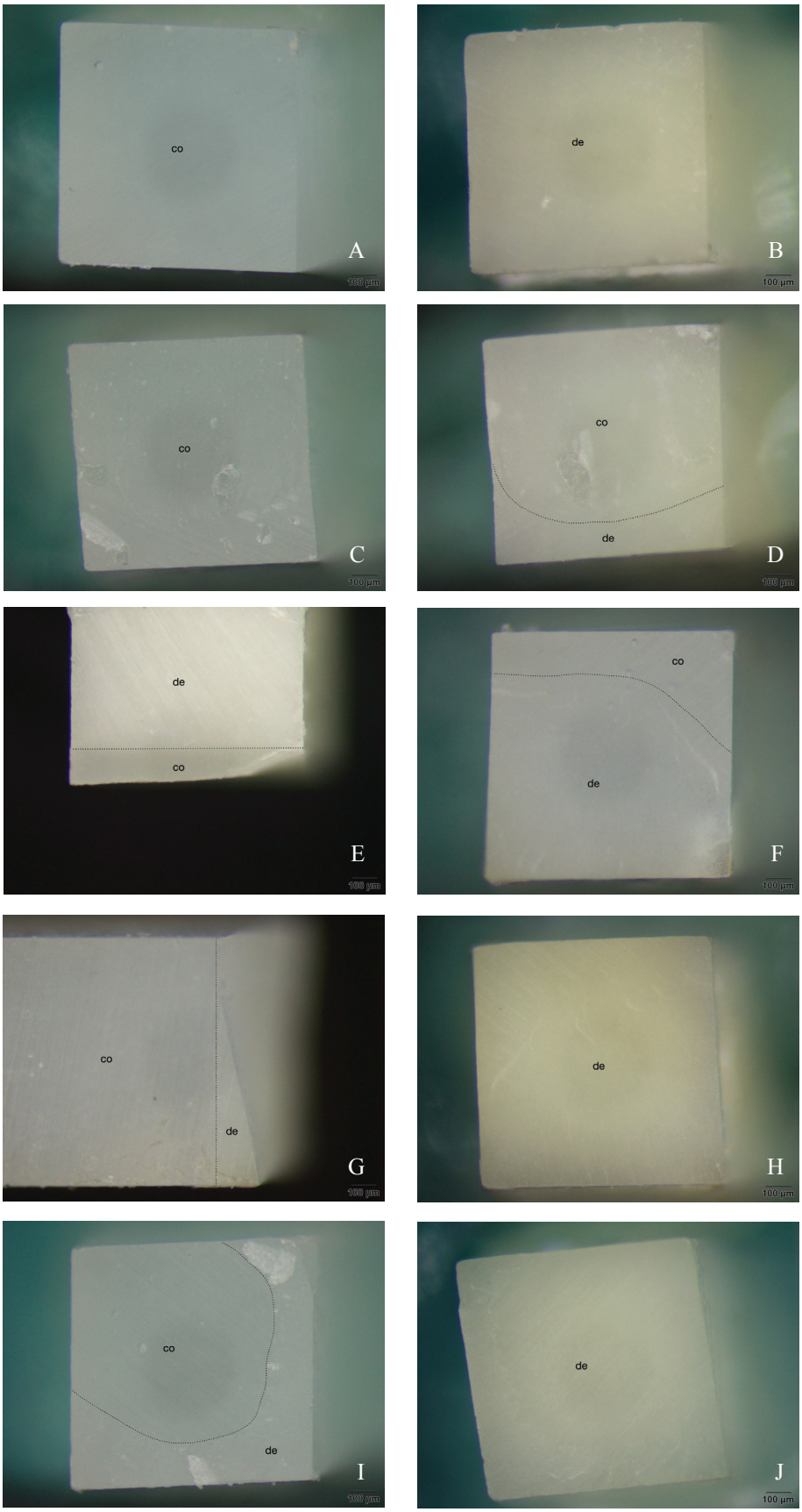

Figure 5: Representative stereomicroscope images of bond failure modes (original magnification ×40). (A) and (B) AF (from group of SiSE's composite and dentin, respectively). (C), (D), (E) CFR (from group of SE's composite, dentin and lateral view of dentin, respectively). (F), (G), (H) CFD (from group of ER's composite, lateral view of composite and dentin, respectively). (I), (J) MF (from group of SiER's composite and dentin, respectively). co: composite; de: dentin; dotted line: resin-dentin interface

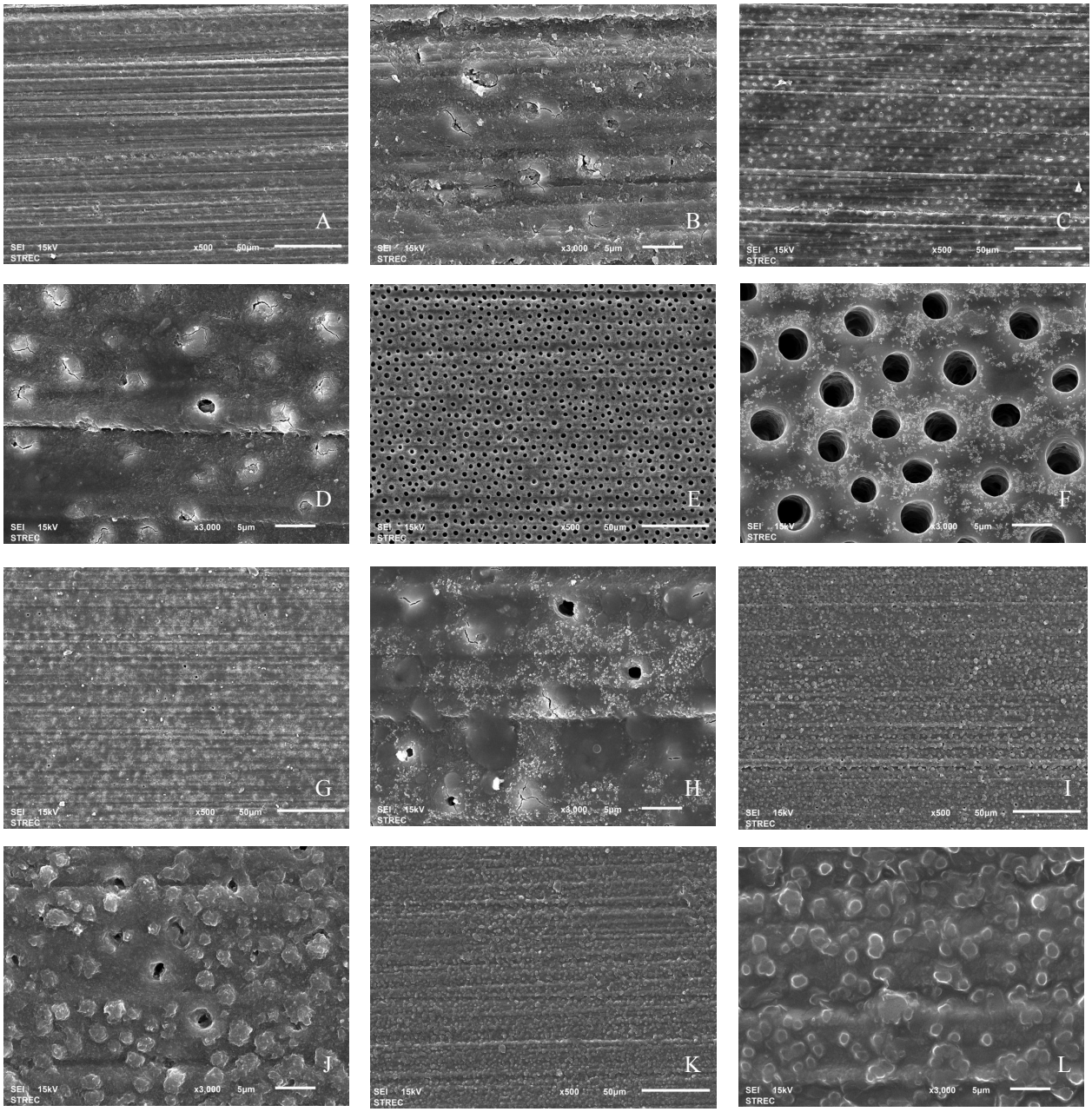

Figure 6: Representative SEM images of dentin surface with and without self-etch silane contamination; (A) and (B) Polished dentin with distill water; A smear layer was visible, and tubules were closed. (C) and (D) Polished dentin with silane contamination; The smear layer was superficially removed, but tubules were sealed. (E) and (F), Phosphoric acid-etched dentin; There was no smear layer visible, and tubules were open. (G) and (H) Silane-contaminated dentin with phosphoric acid etching; A smear layer was mostly visible, and only some tubules were open. (I) and (J) Dentin with self-etched primer; A smear layer was partly visible, and some tubules were partially open. (K) and (L) Silane-contaminated dentin with self-etched primer; occluded tubules and smear layer were clearly visible. (×500 and x3000 indicated the magnification)

Although the present study was designed the silane contamination before etching in SiER group, these two earlier research conclusions differ from ours could be since different silane agents were examined. The silane contained purely MPS was evaluated in two earlier investigations. Conversely, the present study investigated MEP containing a MPS and a new polyfluoride or TADF.[4] These results were consistent with those of Kanzow *et al*.,[21] who found the undefined precipitate formed by the polyfluorides in the MEP led to impaired resin infiltration and reduced bond strength. With MEP, the pretreatment of glass ceramic surfaces for the adhesive luting could be faster due to single application step and less harmful due to the absence of HF acid, and therefore seem to be an interesting option for glass ceramic repair. Regarding the intraoral repair of direct and indirect restorations, consistent protocols are lacking, and the literature presents various approaches.[22] According to Saracoglu *et al*.,[23] it was found that bonding to tooth tissues conditioned with HF acid gel resulted in a significant reduction in enamel and dentin bond strengths to resin composite, regardless of whether it was applied before or after phosphoric acid etching. However, the etch-and-rinse adhesive performed slightly better when HF acid was applied after phosphoric acid etching instead of before. In contrast, a study by Kanzow *et al*.,[21] found a negative significant effect of bovine dentin contamination with HF acid or an MEP before applying universal adhesive in the self-etch mode. In the etch-and-rinse mode of universal adhesive application, the shear bond strength of bovine dentin contamination with HF acid or an MEP showed an insignificant decrease, regardless of whether it was contaminated before or after phosphoric acid etching.

In addition, Lührs *et al*.,[8] investigated the impact of surface contamination on the dentin bond strength of universal adhesives using various etching modes during repair procedures involving HF acid, silane, or MEP. The study also investigated different etching modes before and after thermocycling. The findings indicated that dentin contamination with a silane primer containing 10-MDP before the application of a universal adhesive did not affect bond strength, regardless of the aging process. Compared to the control group, however, contamination with HF acid or an MEP leads to a significantly lower bond strength after aging, but there were no significant differences in the immediate μTBS.

On the other hand, in our study, immediate μTBS of the SiER group was significantly reduced when MEP contamination before phosphoric acid etching. Similarly, in the SiSE group, immediate μTBS was significantly decreased compared to the control SE group. These different results may be influenced by the different methodologies, such as specimen preparation, application sequence, and the type of dental adhesive used.

Additionally, the SiSE group exhibited the lowest performance among all the groups, resulting in an 85% AF. On the other hand, the ER group primarily showed CFR, suggesting that the bond strength between the resin and dentin might be stronger than the strength of the composite resin and/or dentin itself. The immediate μTBS of the SiER group was insignificantly higher than that of the SE group. This finding suggests that the etch-and-rinse adhesive may be more suitable than the self-etch adhesive for self-etch silane contamination dentin.

The results of SEM are shown in Figure 6 with magnifications of ×500 and ×3000. The controlled polished dentin with distill water revealed a distinct smear layer with closed tubules (Figure 6A, 6B). Despite the self-etch silane contamination, the polished dentin appeared to superficially remove the smear layer, allowing for more visible dentinal tubule openings. However, the tubules remained sealed with precipitates (Figure 6C, 6D). These findings align with the previous studies.[8,21] It is possible that an interaction between the fluorides present in the self-etch silane and the dentin surface at a molecular level, similar to the process observed with HF acid, could explain the decrease in μTBS.[8]

It is known that HF acid creates dense amorphous fluoride precipitates on top of the tooth surface, which leads to the sealing of dentin and the closure of dentinal tubule openings. This process inhibits phosphoric acid etching and the infiltration of resin adhesive. However, there is a lack of other studies in the literature that describe the interaction between ammonium polyfluoride primers and dentin. Further research is needed to clarify this phenomenon. Dentin that had been etched with phosphoric acid showed apparent tubules and the complete removal of the smear layer (Figure 6E, 6F). On the other hand, dentin contaminated with self-etch silane, followed by phosphoric acid etching, exhibited a smear layer and only some open tubules (Figure 6G, 6H). This consistent with

the results published by Kanzow *et al*.,[21]. The MEP precipitation partially occluded the dentinal tubules, but it appeared to be less severe compared to HF acid contamination. Furthermore, the sequencing of phosphoric acid etching suggested that performing the etching step prior to self-etch silane contamination rather than after would result in less severe surface precipitates. This outcome is similar to dentin contamination caused by HF acid, which results in thick, amorphous fluoride precipitates. Furthermore, these precipitates cannot be effectively removed by further phosphoric acid etching.[21]

Dentin treated with acidic primer exhibited a smear layer that was partially visible and partially open tubules (Figure 6I, 6J), which was consistent with the previous study.[24] In contrast, self-etch silane contaminated dentin, followed by self-etched primer, revealed occluded tubules and a clearly visible smear layer (Figure 6K, 6L). This result also corresponds to another previous study.[21] Although SE (Clearfil SE Bond) and MEP contain a methacrylated phosphoric acid ester such as 10-MDP.[4] As 10-MDP also bonds to residual calcium ions, the chemical bonding process may be inhibited by the reaction mechanism triggered by the application of MEP. Nevertheless, it was still unclear how the MEP precipitation hampered the MDP-10 interaction and also self-etch adhesive bonding process. It may require further research to identify the molecular interaction.

In this study, only immediate μTBS in dentin was investigated. However, the immediate enamel μTBS and the aged μTBS can be used for further investigation in future studies. Moreover, Fourier transform infrared spectroscopy (FTIR) can be analyzed to examine the alterations to resin monomer, silane, and collagen composition on the dentin surfaces. In the SEM observation, the specimens can be sectioned perpendicular to the resin-dentin interface to determine the thickness of the hybrid layer.

Conclusions

Based on the provided context, the *in vitro* study found that self-etch silane cross-contamination on dentin negatively impacted the μTBS of both etch-and-rinse and self-etch adhesive systems. However, the study suggests that the etch-and-rinse adhesive system may be more effective in mitigating the effects of dentin contamination compared to the self-etch adhesive system.

Conflicts of interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, or company that is presented in this article.

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