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ORIGINAL ARTICLE

Microshear bond strength to dentin of self-adhesive flowable composite compared with total-etch and all-in-one adhesives

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KEYWORDS
microshear bond strength;
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thermocycling

Abstract *Background/purpose:* Because of the lack of data on long-term survival of a flowable self-adhesive composite (SAC) restoration, the purpose of this study was to compare the microshear bond strengths (μSBSs) of flowable resin composites to dentin, either with self-adhesive ability or with the combined use of a total-etch adhesive and all-in-one adhesive, before and after thermocycling.
Materials and methods: Coronal dentin specimens of 60 extracted sound third human molars were divided into three groups ($n = 20$) as follows: Group 1, flowable SAC (VF); Group 2, total-etch adhesive + flowable composite (FL); Group 3, all-in-one adhesive + flowable composite (AL). For each adhesive, half of the specimens were subjected to μSBS testing after 24-hour water storage, and the other half of the specimens were subjected to 5000 thermocycles followed by μSBS testing. The morphologies of the adhesive interfaces were evaluated under a scanning electron microscope. Data were analyzed using one-way analysis of variance (ANOVA) and independent *t* test.
Results: One-way ANOVA showed similar results for both 24-hour water storage and thermocycled groups. The FL group showed the highest μSBS values ($P < 0.001$). The VF and AL groups were not statistically significantly different. Thermocycling had no effect on μSBS values ($P = 0.578$). The interfacial observation revealed that VF had a gap at the resin–dentin interface. By contrast, both FL and AL specimens had distinct adhesive layers without any gap formation.

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Conclusion: The results from this study indicated that laboratory bonding effectiveness of flowable SAC was approximately that of all-in-one adhesive.

Introduction

To meet the increasing esthetic demands of patients, a tooth-colored material with simplified clinical steps is highly desirable, not only for clinical efficiency but also for less technique sensitivity. Therefore, a flowable self-adhesive composite (SAC) has been developed as it does not require any pretreatment of the tooth.[1] The SAC can bond directly to the tooth using self-etching adhesive technology combined with a conventional flowable resin composite. The composition of SAC is similar to other flowable resin composites but includes acidic (functional) monomers such as glycerol phosphate dimethacrylate (GPDM) and 4-methacryloxyethyl trimellitic acid (4-MET) used in current dental adhesives.[2] Thus, the SAC can simultaneously demineralize and infiltrate the tooth structure, resulting in micromechanical retention.[1,3,4] In addition, the bonding performance may be enhanced by the additional chemical treatments, depending on the acidic monomer used.[5,6]

In this study, the bonding performance of an SAC was investigated using the microshear bond strength (μSBS) test. Recent studies have reported the bond-strength values of SAC when tested immediately after application.[3,4,7] However, long-term survival of the tooth—restoration interface in the oral cavity with temperature change, chewing loads, and chemical attack is a challenge.[8] Thus, an artificial aging process is more likely to represent long-term clinical bonding performance. In this study, aging by thermocycling was selected, as recommended by the International Organization for Standardization (ISO).[9]

The aim of this study was to compare μSBS values of SAC with flowable resin composite combined with a total-etch or an all-in-one adhesive to dentin before and after thermocycling. The null hypotheses were as follows: (1) there was no difference in μSBS of SAC, total-etch adhesive, and all-in-one adhesive to dentin after both 24-hour water storage and 5000-cycle thermocycles; (2) there was no difference in μSBS of each material tested before and after thermocycling.

Materials and methods

Sixty sound third human molars were used following approval of the protocol by the Human Research Ethics Committee, Faculty of Dentistry, Chulalongkorn University. The teeth were extracted, cleaned, and immersed in 0.1% thymol solution at room temperature for 7 days, stored in distilled water at 4°C, and used within 6 months. The teeth were sectioned at the occlusal third of the crown and approximately 2 mm below the cementoenamel junction (Figure 1A) with a slow-speed diamond saw (Isomet; Buehler, Lake Bluff, IL, USA) under water cooling. The resulting dentin specimens (Figure 1B) were embedded with the coronal surface exposed in polyvinyl chloride tubes using epoxy resin (Figure 1C), polished using wet 600-grit silicon carbide paper for 60 seconds to create standardized smear layer, randomly divided into three groups according to the adhesive systems, and restored according to the respective manufacturers' instructions (Table 1).

After the adhesive application, three clear cylindrical plastic tubes, 0.8 mm internal diameter × 1.0 mm height (Tygon tubing; Norton Performance Plastic Co, Cleveland, OH, USA), were placed on the flat dentin surface (Figure 1D) and subjected to adhesive light curing for 20 seconds. After curing, each tube was filled with flowable resin composite: Group 1 (VF), shade A3.5 Vertise Flow (Kerr Corporation, Orange, CA, USA); Group 2 (FL), OptiBond FL (Kerr Corporation) + shade A3.5 Premise Flowable (Kerr Corporation); Group 3 (AL), OptiBond all-in-one (Kerr Corporation) + shade A3.5 Premise Flowable (Kerr Corporation) and light cured for 40 seconds. The light output intensity was not less than 800 mW/cm^2, checked using a radiometer (Kerr Corporation).

All specimens were stored in water at 37°C for 24 hours. The plastic tubes were removed and specimens were examined under a 10× magnification stereomicroscope (ML 9300; MEIJI TECHNO, Saitama, Japan) to evaluate the integrity of the resin—dentin interface. Any specimens with interfacial gaps, bubbles, or any defects were excluded from the study. For each adhesive, half of the specimens were subjected to μSBS test immediately after the tube removal and the other half of the specimens were subjected to thermocycling between 5°C and 55°C for 5000 cycles. The dwell time and transfer time were 30 seconds and 10 seconds, respectively.

Specimens were mounted in a universal testing machine (EZ-S; Shimadzu, Tokyo, Japan), a 0.4-mm thick blade placed parallel and adjacent to the resin—dentin interface and specimens were tested to failure at a crosshead speed of 1.0 mm/min. All specimens were analyzed for mode of failure using a stereomicroscope at 45× magnification and categorized as adhesive, cohesive in dentin, cohesive in resin composite, or mixed. Representative specimens were randomly selected for fractographic examination by scanning electron microscopy (SEM) at 2000× magnification (JSM-5410LV; JEOL, Tokyo, Japan)

For dentin—resin interface observations, the resin-bonded specimens were prepared as for μSBS test specimens and sectioned perpendicular to the adhesive interface using a slow-speed saw under running water to obtain 2-mm thick dentin slices. The sectioned surfaces were sequentially polished with 600-, 800-, and 1,000-grit silicon carbide papers under running water, followed by treatment with 1.0- and 0.4-μm aluminum oxide polishing pastes and cleaning with an ultrasonic device. The specimens were immersed in 1M hydrochloric acid for 30 seconds followed by treatment with 5% sodium hypochlorite for 5

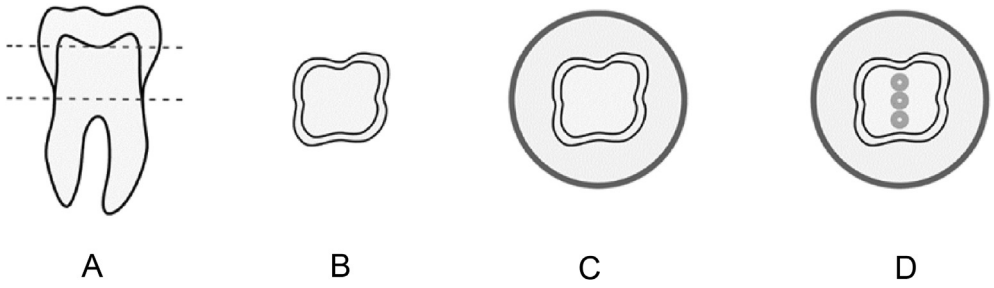

Figure 1 Specimen preparation. (A) A tooth was sectioned to obtain a flat dentin surface; (B) a dentin specimen; (C) a dentin specimen embedded in polyvinyl chloride tube; (D) the plastic tubes placed on the dentin specimen.

Table 1 Materials (modification from manufacturers' Material Safety Data Sheet and instructions for use).

Materials	Composition	pH	Filler (%wt)	Elastic modulus (GPa)	Application
Vertise Flow (Kerr Corporation, Orange, CA, USA) (VF)	GPDM, HEMA, PPF, nano-sized ytterbium fluoride, 1-μm barium glass filler, nano-sized colloidal silica	1.9	70	Approximately 9	• Wash the dentin surface thoroughly with water spray and air dry with maximum air pressure for 5 s • Dispense Vertise Flow onto the dentin surface with a dispensing tip. • Use brush provided to apply Vertise Flow with moderate pressure for 15—20 s to obtain a thin layer (<0.5 mm).
OptiBond FL (Kerr Corporation) (FL)	Etching: 37.5% phosphoric acid and silica thickener				• Place 37.5% phosphoric acid on dentin for 15 s • Rinse with water for about 15 s • Gently air dry for a few seconds (do not desiccate).
	Primer: HEMA, GPDM, PAMM, ethanol, water, and photo-initiator	1.9	0		• Apply primer with a light scrubbing motion for 15 s • Gently air dry for about 5 s until the dentin surface is slightly shiny.
	Adhesive: TEGDMA, UDMA, GPDM, HEMA, bis-GMA, filler, and photo initiator	2.6	48		• Apply adhesive with a light scrubbing motion for 15 s • Blow thin with a light application of air.
OptiBond all-in-one (Kerr Corporation) (AL)	Bis-GMA, GPDM, HEMA, ethanol, acetone, water, filler, ytterbium fluoride, photoinitiator, accelerator, stabilizer, and water	2.5	7		• Wash the dentin surfaces thoroughly with water spray and air dry (do not desiccate). • Apply adhesive to the dentin surface using the disposable applicator brush. • Scrub with a brushing motion for 20 s • Apply a second application with a brushing motion for 20 s • Dry with gentle air first and then medium air for at least 5 s
Premise flowable (Kerr Corporation)	Bis-EMA, TEGDMA, and silica nanofiller		72.5	Approximately 9	• Apply 2-mm thick layer • Light cure for 40 s

bis-EMA = ethoxylated bisphenol A glycol dimethacrylate; bis-GMA = ethoxylated bisphenol A glycol dimethacrylate; GPDM = glycerol phosphate dimethacrylate; HEMA = hydroxyethylmethacrylate; PAMM = phthalic acid monoethyl methacrylate; PPF = prepolymerized filler; TEGDMA = triethylene glycol dimethacrylate; UDMA = urethane dimethacrylate.

minutes to achieve decalcification and deproteinization. The specimens were dehydrated with a graded series of ethanol followed by treatment with hexamethyldisilazane for 10 minutes, and then gold sputter-coated and examined using an SEM at 500× and 1500× magnifications.

Statistical analysis

Data were analyzed using SPSS Statistics for Windows, version 22.0 (SPSS Inc., Chicago, IL, USA). The μSBS data were analyzed with one-way analysis of variance (ANOVA).

The effect of thermocycling was analyzed using an independent *t* test. The distribution of the failure modes was analyzed with a Chi-square test. Statistical significance was set as $P < 0.05$.

Results

Bond strength data and failure mode are presented in Table 2. The FL group specimens had significantly higher μSBS values than the other groups ($P < 0.001$) in both nonthermocycled and thermocycled groups. The μSBS values of the VF and AL group specimens were not statistically significantly different. One-way ANOVA and independent *t* test showed that the adhesive system significantly affected μSBS values ($P < 0.001$), whereas thermocycling had no effect on μSBS values ($P = 0.578$). SEM images of all groups also showed no difference between nonthermocycled and thermocycled groups of each material tested.

There was no significant difference in the failure mode distribution between the groups ($P > 0.99$). Adhesive failure predominated, with a few mixed failures but no cohesive failures. The VF group showed the highest number of adhesive failures (93.3%) for both nonthermocycled and thermocycled groups. The other groups showed a similar result (ranging from 70% to 83%).

Representative fractographic images are shown in Figure 2. VF specimens (Figures 2A and 2B) show the dentinal tubules; some were empty and the others were occluded. The intertubular dentin was partially covered with SAC. FL specimens (Figures 2C and 2D) showed an adhesive layer completely covering the dentin surface. Dentinal tubules were hardly visible. AL specimens (Figures 2E and 2F) show mostly empty dentinal tubules.

The dentin–resin interfacial observations are shown in Figures 3–5. VF specimens (Figures 3A–3D) showed gaps at the interface with a small number of resin tags. FL specimens (Figures 4A–4D) demonstrated cone-shaped resin tags continuously along the interfaces with a distinct hybrid layer. The adhesive layer thickness is about 25–30 μm. AL specimens (Figures 5A–5D) had 10- to 15-μm thick adhesive layers. Some cylindrical resin tags could be observed.

Discussion

In this study, the μSBS values of SAC were not significantly different from all-in-one adhesives, but significantly lower than the total-etch adhesive in both nonthermocycled and thermocycled groups. The μSBS values of all adhesives were not statistically different between nonthermocycled and thermocycled groups. Thus, the first hypothesis, that is, no difference in μSBS of each material bonded to dentin after both 24-hour water storage and 5000-cycle thermocycles, was rejected. By contrast, the second hypothesis, that is, no difference in μSBS of each material tested before and after thermocycling, was accepted.

The total-etch or three-step etch-and-rinse adhesive, which is a gold standard, was selected as a control group due to favorable long-term laboratory and clinical performance.[4,10,11] Moreover, the all-in-one or one-step self-etching adhesive was used in this study as it is recognized as the simplest to use clinically. Thus, the bonding effectiveness of the simplified SAC should be at least comparable with the all-in-one adhesive.[4]

SAC is not recommended by the manufacturer to be used in combination with an adhesive. Thus, a different resin composite was selected in this study. It is well-known that type and composition of a resin composite significantly influence the bond strength value.[12,13] In addition, when using resin composites with different elastic moduli, different bond strength values may result.[14] Hence, a conventional flowable resin composite was selected in combination with a total-etch adhesive and an all-in-one adhesive for comparison with the SAC, instead of using a conventional resin composite.

Previous studies showed low microtensile bond strength (μTBS) values of SAC (3.4–17.7 MPa) and a high amount of pretest failures (10%–66.7%).[3,4,7] Thus, the μSBS test was selected to overcome the sensitivity of specimens' preparation and the limitations of μTBS testing, which are the skill required for specimen preparation, the difficulty of measuring very low bond strengths, and the high number of pretest failures.[15] The μSBS test is easy to execute and it is easy to control the bond area using plastic tubes, allowing regional mapping or depth profiling and decreasing the number of pretest failures.[15–18] In addition, there is no

Table 2 μSBS values and failure modes.

Adhesive system	μSBS ± SD (MPa)	Mode of failure (*n*/%)			
		Adhesive	Mixed	Cohesive in dentin	Cohesive in resin composite
24-h water storage					
VF	22.1 ± 6.13^{aA}	28/93.3	2/6.7	—	—
FL	32.2 ± 8.94^{bB}	25/83.3	5/1.7	—	—
AL	24.4 ± 6.21^{aC}	21/70	9/30	—	—
5000-cycle thermocycling					
VF	21.1 ± 5.39^{cA}	28/93.3	2/6.7	—	—
FL	31.8 ± 6.80^{dB}	21/70	9/30	—	—
AL	23.9 ± 7.14^{cC}	22/73.3	8/26.7	—	—

Small letters indicate significant differences in μSBS values among the adhesive systems after 24-hour water storage or 5000-cycle thermocycling ($P > 0.05$). Capital letters indicate significant differences between μSBS values after 24-hour water storage or 5000-cycle thermocycling for the same adhesive system ($P > 0.05$).

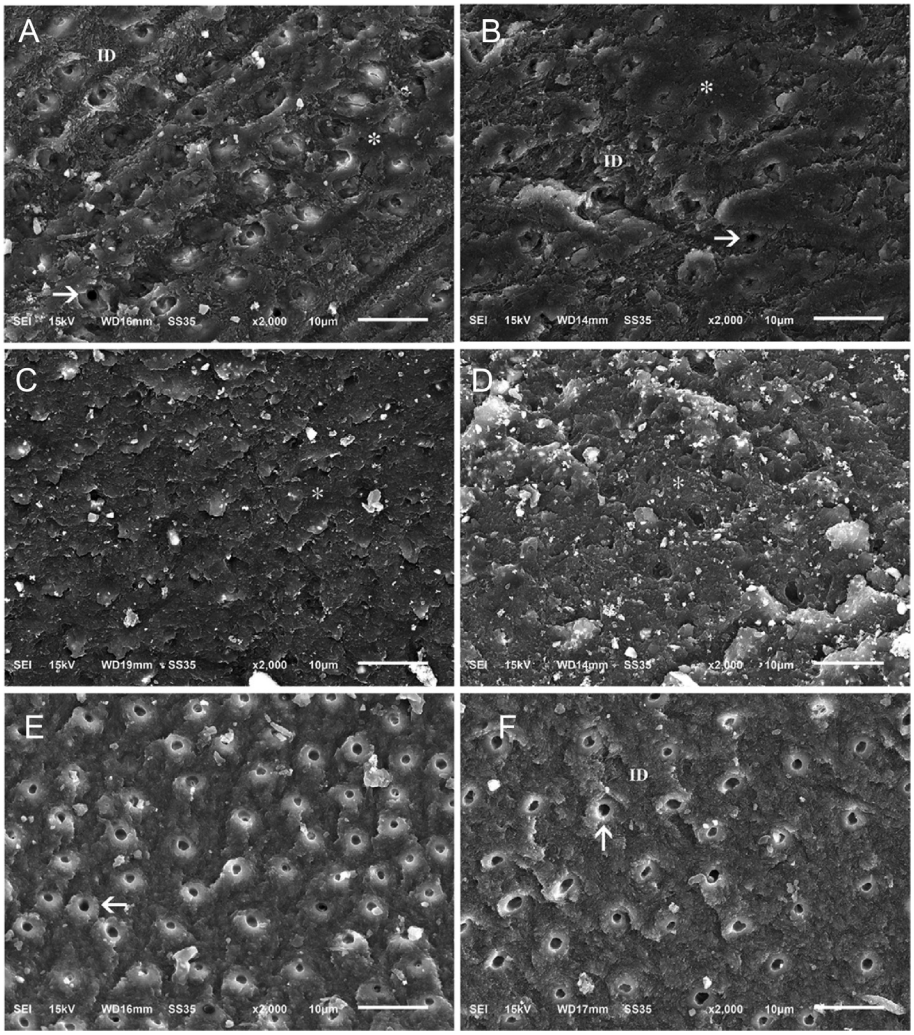

Figure 2 Scanning electron microscopic images of the dentin surface after microshear bond strength testing. (A and B) VF groups; (C and D) FL groups; (E and F) AL groups; (A, C, and E) nonthermocycled groups; (B, D, and F) thermocycled groups. Arrow = empty dentinal tubule; asterisk = self-adhesive composite or adhesive; ID = intertubular dentin.

stress from specimen preparation at the interface prior to testing, except for mold removal. Thus, the μSBS test is suitable for testing the bond strength of SAC, which some authors have reported to be relatively low with specimens tending to fail during preparation.[3,4,7] The results from this study revealed no pretest failures and higher bond strength values than in previous studies.[3,4,7]

The ISO Technical Report 11405 considered that 500-cycle thermocycling in water between 5°C and 55°C is an appropriate artificial aging process.[9] Some studies indicated that 500 cycles were not sufficient to mimic long-term bonding effectiveness and might not have any effect on bond strength values.[19–21] Thus, 5000-cycle thermocycling, which represents a 6-month clinical service,[19] was used in this study.

The result revealed that for all groups 5000 thermocycles did not affect μSBS. This result was consistent with that of the previous studies, which reported that thermocycling did not affect the bond strength.[10,13] This may be a result of surrounding tooth structure and resin composite thermally protecting the interfacial components from hydrolytic degradation, which can be accelerated by heat.[10,20,21] Furthermore, the low C factor (about 1:5) of specimens generated inadequate repetitive expansion/contraction stress at the interface to affect the μSBS value.[10,20,21] Similar to μSBS, SEM interfacial morphology of all groups showed the same characteristic for both nonthermocycled and thermocycled groups.

The total-etch adhesive showed the highest μSBS for both nonthermocycled and thermocycled groups. This finding is consistent with previous studies, which have indicated that the total-etch adhesive represented high bonding performance in both laboratory and clinical studies.[4,10,11] In addition, it has been suggested that the high filler load and the thick layer of this adhesive could absorb stress at the tooth—restoration interface.[22]

SAC has the highest amount of filler load among tested adhesives and does not contain any solvent, thus it showed the most viscous consistency with low wettability. As a result, SAC could not penetrate deeply into demineralized tooth structure, hence sufficient micromechanical interlocking was unlikely to happen. Consequently, the bonding effectiveness was reduced. In this respect, the SAC showed the lowest μSBS in this study. The SAC should contain a

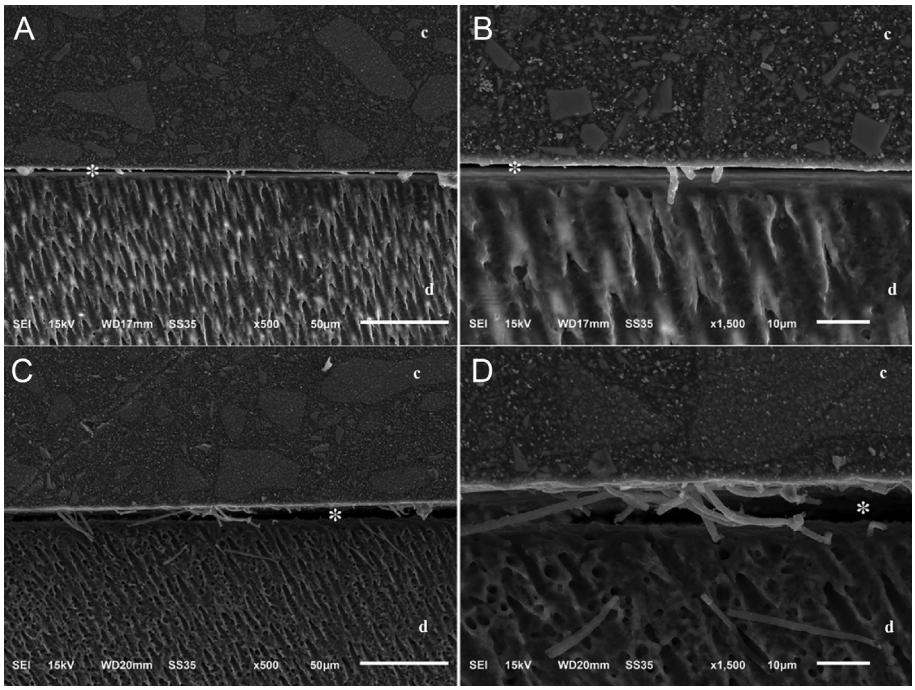

Figure 3 Scanning electron microscopic observation of the dentin—resin interface from the VF groups showed the interfacial gaps (asterisk) with a small number of cylindrical-shaped resin tags. The hybrid layer was not clearly observed. (A) Non-thermocycled specimen; (B) higher magnification; (C) thermocycled specimen; (D) higher magnification. c = resin composite; d = dentin.

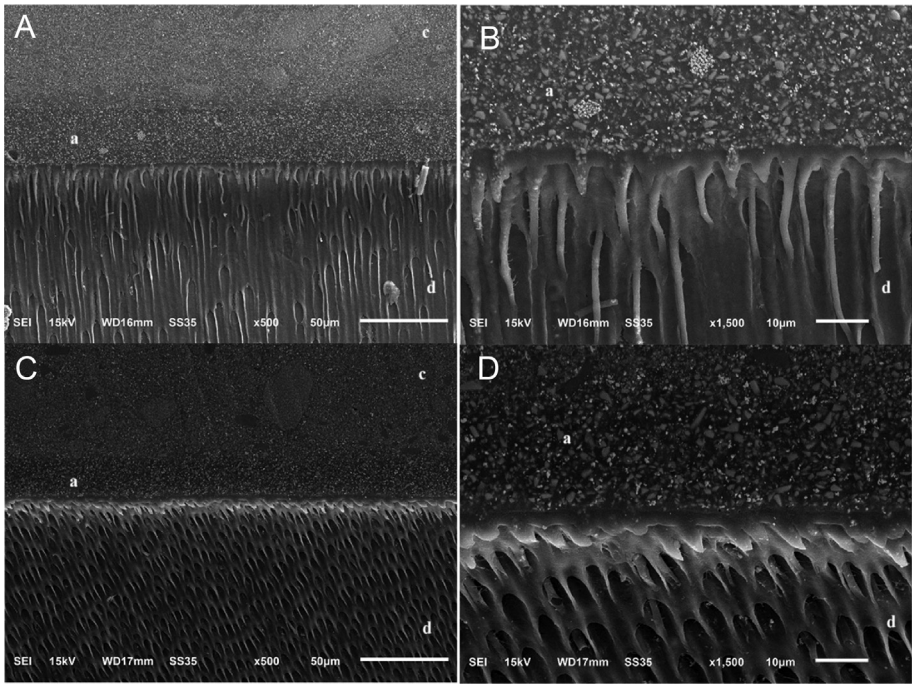

Figure 4 Scanning electron microscopic observation of the dentin—resin interface from the FL groups showed the approximately 25—30-µm thick adhesive layer (a) with the distinct hybrid layers and cone-shaped resin tags continuously along the interfaces. (A) nonthermocycled specimen; (B) higher magnification; (C) thermocycled specimen; (D) higher magnification. c = resin composite; d = dentin.

functional monomer with effective chemical bonding potential to accomplish self-adhesiveness, as it cannot penetrate deeply to perform precise micromechanical interlocking.[3,4] The SAC used in this study contains GPDM as a functional monomer. From the fractography evaluation, empty dentin tubules and exposed intertubular dentin were observed. Moreover, SEM interfacial observations showed a gap at the dentin—SAC interface without a distinct hybrid

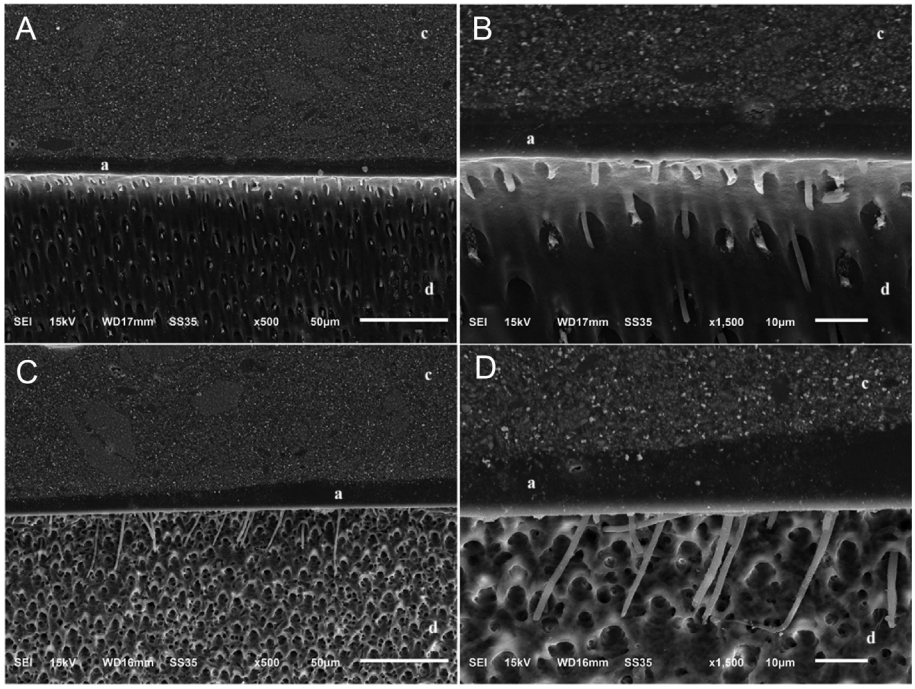

Figure 5 Scanning electron microscopic observation of the dentin—resin interface from the AL groups showed the approximately 10—15-μm adhesive layers (a) with cylindrical-shaped resin tags. The hybrid layer was not clearly observed. (A) Nonthermocycled specimen; (B) higher magnification; (C) thermocycled specimen; (D) higher magnification. c = resin composite; d = dentin.

layer. These findings may suggest that GPDM may not perform as effectively as expected.

SEM interfacial observations demonstrated gaps between SAC and dentin in both nonthermocycled and thermocycled groups. The gap could be derived from the poor permeability and wettability of the first layer of SAC applied to dentin. Besides, the water was not able to diffuse through the first layer of the highly viscous SAC, which functioned as a semipermeable membrane. Thus, it manifested as a gap.[3,4,23] The water may have originated from residual water from specimen preparation or intrinsic water from dentin.[23,24] This finding was consistent with previous studies that showed gaps and bubbles at the dentin—SAC interface.[3,4,7]

Corresponding to previous studies, the bond strength of the all-in-one adhesive was lower than that of the total-etch adhesive.[25—27] The all-in-one adhesive comprises highly hydrophilic functional monomer, which leads to high amount of water uptake and acts as a semipermeable membrane.[11,28] Moreover, the high concentration of solvent may cause incomplete evaporation, leading to an inappropriate thickness of adhesive layer and the formation of bubbles within the adhesive layer.[10,11,29] Because of its relatively high pH (about 2.5), the all-in-one adhesive superficially demineralizes dentin and forms a superficial interaction between adhesive and demineralized dentin,[11,28] thus forming a thin hybrid layer and few resin tags. Together with the finding that the GPDM may not chemically bond to the tooth, this may be the cause of the inferior bonding performance.

As a result of our study, the chemical bonding potential of GPDM could play an important role in the bonding performance of SAC and the all-in-one adhesive. Unfortunately, the chemical bonding of GPDM to hydroxyapatite may not occur and has yet to be proved.[5,6,30] Therefore, μSBS values of the SAC and the all-in-one adhesive used in this study were significantly lower than those of the total-etching adhesive. This may suggest the use of other monomers with a proven chemical bonding to hydroxyapatite, for example, 4-MET[5,6] for self-adhesive potential rather than GPDM. In agreement with other studies, SAC using 4-MET had higher bond strength values than SAC using GPDM.[3,4]

Further investigation is needed to confirm the clinical effectiveness of SAC. In addition, not only bonding effectiveness affects stability and success of a restoration, but also other properties such as wear rate, water sorption, and solubility need to be considered.

This laboratory study demonstrated that the μSBS value of the SAC was comparable with the all-in-one adhesive, but lower than the total-etch adhesive, in both nonthermocycled and thermocycled groups. Aging by 5000 thermocycles cycling did not affect the μSBS values of any of the adhesives tested.

Conflicts of interest

The authors have no conflicts of interest relevant to this article.

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References

1. Hanabusa M, Mine A, Kuboki T, et al. TEM interfacial characterization of an experimental self-adhesive filling material bonded to enamel/dentin. *Dent Mater* 2011;27:818—24.
2. Ferracane JL. Resin composite—state of the art. *Dent Mater* 2011;27:29—38.
3. Fu J, Kakuda S, Pan F, et al. Bonding performance of a newly developed step-less all-in-one system on dentin. *Dent Mater J* 2013;32:203—11.
4. Poitevin A, De Munck J, Van Ende A, et al. Bonding effectiveness of self-adhesive composites to dentin and enamel. *Dent Mater* 2013;29:221—30.
5. Moszner N, Salz U, Zimmermann J. Chemical aspects of self-etching enamel—dentin adhesives: a systematic review. *Dent Mater* 2005;21:895—910.
6. Van Landuyt KL, Snauwaert J, De Munck J, et al. Systematic review of the chemical composition of contemporary dental adhesives. *Biomaterials* 2007;28:3757—85.
7. De Munck J, Luehrs AK, Poitevin A, Van Ende A, Van Meerbeek B. Fracture toughness versus micro-tensile bond strength testing of adhesive-dentin interfaces. *Dent Mater* 2013;29:635—44.
8. Van Meerbeek B, De Munck J, Yoshida Y, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent* 2003;28:215—35.
9. International Organization for Standardization. *Technical Report ISO/TS 11405: Dental Materials: Testing of Adhesion to Tooth Structure*. Geneva, Switzerland: International Organization for Standardization, 2003.
10. De Munck J, Van Landuyt K, Peumans M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res* 2005;84:118—32.
11. De Munck J, Van Meerbeek B. The current status of bonding to dentin anno 2007. *Int J Oral Sci* 2007;6:45—60.
12. Van Noort R, Noroozi S, Howard IC, Cardew G. A critique of bond strength measurements. *J Dent* 1989;17:61—7.
13. De Munck J, Mine A, Poitevin A, et al. Meta-analytical review of parameters involved in dentin bonding. *J Dent Res* 2012;91:351—7.
14. Leloup G, D'Hoore W, Bouter D, Degrange M, Vreven J. Meta-analytical review of factors involved in dentin adherence. *J Dent Res* 2001;80:1605—14.
15. Roeder L, Pereira PNR, Yamamoto T, Ilie N, Armstrong S, Ferracane J. Spotlight on bond strength testing—unraveling the complexities. *Dent Mater* 2011;27:1197—203.
16. McDonough WG, Antonucci JM, He J, et al. A microshear test to measure bond strengths of dentin-polymer interfaces. *Biomaterials* 2002;23:3603—8.
17. Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: a critical literature review. *Dent Mater* 2010;26:e78—93.
18. Münchow EA, Bossardi M, Priebe TC, et al. Microtensile versus microshear bond strength between dental adhesives and the dentin substrate. *Int J Adhes Adhes* 2013;46:95—9.
19. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent* 1999;27:89—99.
20. Amaral FLB, Colucci V, Palma-Dibb RG, Corona SAM. Assessment of *in vitro* methods used to promote adhesive interface degradation: a critical review. *J Esthet Restor Dent* 2007;19:340—53.
21. Morresi AL, D'Amario M, Capogreco M, et al. Thermal cycling for restorative materials: does a standardized protocol exist in laboratory testing? A literature review. *J Mech Behav Biomed Mater* 2014;29:295—308.
22. Can Say E, Nakajima M, Senawongse P, et al. Microtensile bond strength of a filled vs unfilled adhesive to dentin using self-etch and total-etch technique. *J Dent* 2006;34:283—91.
23. Tay FR, Pashley DH, Suh BI, Carvalho RM, Itthagarun A. Single-step adhesives are permeable membranes. *J Dent* 2002;30:371—82.
24. Van Landuyt KL, Snauwaert J, De Munck J, et al. Origin of interfacial droplets with one-step adhesives. *J Dent Res* 2007;86:739—44.
25. Sidhu SK, Omata Y, Tanaka T, et al. Bonding characteristics of newly developed all-in-one adhesives. *J Biomed Mater Res* 2007;80:297—303.
26. Perdigao J, Gomes G, Gondo R, Fundingsland JW. *In vitro* bonding performance of all-in-one adhesives. Part I—microtensile bond strengths. *J Adhes Dent* 2006;8:367—73.
27. Hanabusa M, Mine A, Kuboki T, et al. Bonding effectiveness of a new 'multi-mode' adhesive to enamel and dentine. *J Dent* 2012;40:475—84.
28. 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.
29. Cardoso MV, de Almeida Neves A, Mine A, et al. Current aspects on bonding effectiveness and stability in adhesive dentistry. *Aust Dent J* 2011;56(Suppl 1):31—44.
30. Sezinando A, Perdigao J, Regalheiro R. Dentin bond strengths of four adhesion strategies after thermal fatigue and 6-month water storage. *J Esthet Restor Dent* 2012;24:345—55.