Bond performance and mechanical properties of self-adhesive flowable resin composites

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This thesis is based on the published article listed below with additional data.

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Summary

Since 2009, self-adhesive flowable composites, which can be used for direct restorations without any adhesive application, have been available. While this approach is simpler to use, clinicians are still reluctant to remove the adhesive step entirely from the restoration procedure. Early studies of the first generation of materials reported poor bonding and mechanical properties, which may be the reason many clinicians avoid using them. Recently, several products in this category have been developed and the manufacturers claim that their bonding performance has been improved. However, there is currently minimal research reporting the bonding and mechanical properties of newly developed self-adhesive flowable composites. The purpose of this study was to investigate the bonding and mechanical properties of self-adhesive flowable composites.

The three self-adhesive flowable composites used to date were: Constic (CO), Fusio (FU), and Vertise Flow (VF). The two newer version of self-adhesive flowable composites were: Fit SA F03 (FS3) and Fit SA F10 (FS10). Two hundred and twenty-five bovine mandibular incisors were used for the shear bond strength (SBS) tests. Prepared enamel specimens were divided into groups of n = 75 for testing with and without etching. For the etching group, the enamel surfaces were etched with 35% phosphoric acid for 15 s, water rinsed, and air-dried. The mold insert was clamped against the enamel or dentin surface and filled with self-adhesive flowable composite. The mold insert was removed, and the finished specimens were stored in water at 37°C for 24 h. After the storage period, SBS testing was carried out using a universal testing machine at a crosshead speed of 1.0 mm/min.

One hundred bovine mandibular incisors were used for the microleakage evaluation. Hemispherical cavities were prepared and the cavities were divided into groups of n = 50 for testing with and without enamel etching. The cavities were restored with the self-adhesive flowable composites according to each manufacturer's instructions. The restored teeth were then stored in water at 37°C for 24 h. The microleakage specimens were subjected to a thermal shock tester for 2,000 cycles. Specimens were then stored in 0.1% basic fuchsin solution at room temperature for 24 h. Specimens were sectioned with a diamond-impregnated disk near the center of the specimen longitudinally. The microleakage scores of sectioned specimens were evaluated using a digital microscope. The dye penetration levels of the samples were scored as follows: 0, No dye penetration; 1, Dye penetration up to half of enamel thickness; 2, Dye penetration of more than half of enamel thickness but short of enamel-dentin junction; 3, Dye penetration up to 1/3 of the dentin; 4, Dye penetration of more than 1/3 of dentin and less than 2/3; 5, Dye penetration of more than 2/3 of dentin.

Twelve specimens of each material were prepared for simulated occlusal (localized) wear testing. The materials were cured with the LED light curing unit. After storage in water at 37° C for 24 h, the material surfaces were polished flat up to a 4,000-grit SiC papers. This study used an Alabama wear testing machine. Stainless steel ball bearings served as the wear antagonists and load was applied in cycles at 2 Hz frequency. Each specimen was profiled using a noncontact optical profilometer. After the completion of 400,000 wear cycles, the specimens were profiled again using the profilometer. Simulated occlusal wear was determined by comparing the before and after data sets in a software. The volume loss (mm³) and maximum depth (µm) of the wear facets were determined.

The surface microhardness of the cured materials was measured. The material was placed into a polytetrafluoroethylene cylindrical mold and the specimens were light irradiated for 20 s. Twelve flat specimens for each group were prepared, and then they were stored under dark conditions for 24 h in a 100% humid environment at 37°C. KHN was measured from the indentation after application of a 1.961 N load for 15 sec using a microhardness tester. Three measurements per specimen were conducted in different locations, and then the mean values

were calculated. SEM observations were performed the facets of the specimens after wear testing and the bonding interfaces between tooth substrates and restorations.

The SBSs of the tested materials were 6.5–12.2 MPa to ground enamel, 22.5–32.5 MPa to etched enamel, and 1.3–4.2 MPa to dentin. The SBSs were different depending on the material and substrates. The microleakage scores of tested materials were 1.08–2.22 in the specimens without enamel etching and 1.22–2.35 in the specimens with enamel etching, showing significance difference depending on the material. The facets on tested materials after wear testing showed 0.099–0.447 mm³ of volume loss and 148.6–365.3 μ m maximum facet depth. The size of the facet in SEM observations showed the same rank order as the volume loss and maximum depth of facet. KHN of the tested materials ranged from 36.6 to 53.3. SEM observations of material-tooth interfaces showed good adaptation regardless of substrate, but the types of fillers in the materials were different depending on the material.

The results of this study suggested that the clinical use of restorations using selfadhesive flowable composites with phosphoric acid etching to enamel may be acceptable in small and shallow cavities that are mainly composed of enamel and are not in a stress bearing area. Further research studies measuring the clinical wear rate of self-adhesive flowable composites using an intraoral situation might offer more relevant information.

Introduction

The clinicians on the American Dental Association Clinical Evaluators (ACE) Panel member dentists had different preferences for bonding techniques, with 57% preferring 2-step etch-&rinse (E&R), 15% each favoring 2-step self-etch and 1-step self-etch, and 13% supporting 3step E&R. On the other hand, self-etch or universal adhesives are among the top five bestselling adhesive systems (1). Thus, it appears that, although most clinicians still believe that the E&R technique is superior, they tend to use simplified adhesives in the clinic (2).

Regarding direct composite materials, the first bulk-fill flowable composite was SureFil SDR (Dentsply), launched in 2009. The popularity of bulk-fill flowable composite has been steadily increasing in recent years (3). The ACE Panel Report showed that 70% of clinicians claimed that they favored the incremental filling technique for posterior composites, with only 26% favoring bulk-fill, and 3% preferring other methods (4). On the other hand, several bulk-fill composites are found among the top five best-selling composites. Again, this indicates that clinicians seem to favor simpler bonding and filling techniques for direct restorations.

Since 2009, self-adhesive flowable composites, which can be used for direct restorations without any adhesive application, have been available (5). Vertise Flow (Kerr) was the first such product, and other similar products have been developed and available for more than 10 years. While this approach is simpler to use, clinicians are still reluctant to remove the adhesive step entirely from the restoration procedure. Early studies of the first generation of materials reported poorer bonding and mechanical properties, which may be the reason many clinicians avoid using them (6,7). Recently, several products in this category have been developed and the manufacturers claim that their bonding performance has been improved (8). However, there is currently minimal research reporting the bonding and mechanical properties of newly developed self-adhesive flowable composites.

The purpose of this study was to investigate the bonding and mechanical properties of self-adhesive flowable composites. The null hypothesis tested was that there would be no differences in bonding or mechanical properties among the tested materials.

Materials and Methods

Study materials

The three self-adhesive flowable composites used to date were: Constic (CO, DMG), Fusio (FU, Pentron Clinical), and Vertise Flow (VF, Kerr). The two newer version of self-adhesive flowable composites were: Fit SA F03 (FS3, Shofu) and Fit SA F10 (FS10, Shofu). All study materials are listed in Table 1.

Shear bond strength (SBS)

Two hundred and twenty-five bovine mandibular incisors were used for the SBS tests. The prepared teeth were mounted in self-cured acrylic resin (Tray Resin II, Shofu) and placed under tap water to reduce temperature rise of the resin during the polymerization reaction. The enamel and dentin surfaces were then ground flat using a model trimmer. The labial surfaces of the coronal central portion were ground under running tap water using P120-and P400-grit silicon carbide (SiC) papers (Sankyo Fuji Star), in an automatic grinding machine (Minitech, Presi). This method was used to prepare enamel (n = 150) and dentin (n = 75) surfaces with a standardized surface texture and smear layer, as specified by ISO 29022 (9). Prepared enamel specimens were divided into groups of n = 75 for testing with and without etching. For the etching group, the enamel surfaces were etched with 35% phosphoric acid (Ultra Etch, Ultradent Products) for 15 s, water rinsed, and air-dried. The mold insert (2.38 mm diameter and 2.67 mm height for the restoration) was clamped against the enamel or dentin surface and filled in one increment up to 2.0 mm height with self-adhesive flowable composite. The exit of the light-emitting diode light curing unit (Elipar DeepCure-S LED Curing Light, 3M Oral Care)

was fixed on the top surface of the mold insert and light irradiated. The mold insert was removed, and the finished specimens were stored in water at 37°C for 24 h. After the storage period, SBS testing was carried out using a universal testing machine (Type 5500R, Instron) at a crosshead speed of 1.0 mm/min. The SBSs (MPa) were calculated by dividing the peak load at failure by the bonding area.

Microleakage evaluation

One hundred bovine mandibular incisors were used for the microleakage evaluation. Hemispherical cavities (4.0 mm in diameter, 2.0 mm in depth) were prepared (centered towards the coronal end of the labial surfaces) using a diamond point (Diamond Point B HP-42, Shofu) in a water-cooled high-speed handpiece. After finishing the cavity preparations, a digimatic micrometer and periodontal probe were used to check the cavity size. Prepared cavities were divided into groups of n = 50 for testing with and without enamel etching. The ISO technical specification indicated that at least 10 specimens per group should be prepared (10). The cavities were restored with the self-adhesive flowable composites according to each manufacturer's instructions. The restored teeth were then stored in water at 37°C for 24 h. The top surface of the restorations was polished using a polisher (Super-Snap Rainbow Technique Kit, Shofu). The microleakage specimens were subjected to a thermal shock tester (TTS-1, Thomas Kagaku) between 5°C and 55°C in water baths for 2,000 cycles, in which each cycle comprised 30 s of immersion and a transfer time of 5 s.

The apex of the root of the tooth was covered with self-cured acrylic resin and the entire surface was coated with nail varnish, except for the restoration and 1 mm of tooth surface adjacent to the restoration. Specimens were then stored in 0.1% basic fuchsin solution (Fujifilm Wako Chemical) at room temperature for 24 h. After being removed from the solution, the specimens were washed with water, and sectioned with a diamond-impregnated disk near the center of the specimen longitudinally from the incisal edge to the apex. The microleakage

scores of sectioned specimens were evaluated using a digital microscope (VHX-6000, Keyence). The dye penetration levels of the samples were scored as follows: 0, No dye penetration; 1, Dye penetration up to half of enamel thickness; 2, Dye penetration of more than half of enamel thickness but short of enamel-dentin junction; 3, Dye penetration up to 1/3 of the dentin; 4, Dye penetration of more than 1/3 of dentin and less than 2/3; 5, Dye penetration of more than 2/3 of dentin.

Simulated occlusal wear test

Twelve specimens of each material were prepared for simulated occlusal (localized) wear testing (11). Custom fixtures were machined from stainless steel to have a cylindrical cavity 6.5 mm in diameter and 4.0 mm in depth. Two increments of the materials (approximately 2.0 mm in thickness) were cured with the LED light curing unit. After storage in water at 37°C for 24 h, the material surfaces were polished flat to a P4,000-grit surface using a sequence of SiC papers.

This study used an Alabama wear testing machine with a four-station plastic water bath (12,13). The custom wear fixtures were mounted in the bath, and a brass cylinder was then placed around each of them. The water slurry of PMMA was added until it had a depth of approximately 6 mm over the surface of the resin. Stainless steel ball bearings (r = 2.387 mm) served as the wear antagonists and were mounted inside a collet assembly. The wear challenges were delivered by mounting these assemblies on spring-loaded pistons. Load was applied in cycles at 2 Hz frequency, reaching a maximum of 78.5 N. The antagonists rotate by approximately 30° during the application of the load, and then counter-rotate to return to the starting position as the load is relaxed. One test consisted of 400,000 cycles over a period of about 55 h.

Each specimen was profiled using a noncontact (Proscan 2100, Scantron) with built-in software before wear challenging, to provide pretest digitized contours. The specimens were

cleaned with water in an ultrasonic bath and then profiled again using the profilometer. Simulated occlusal wear was determined by comparing the before and after data sets in a software. The volume loss (mm³) and maximum depth (μ m) of the wear facets were determined for each of the five self-adhesive flowable composites.

Knoop hardness number (KHN) measurement

The surface microhardness of the cured self-adhesive flowable composites was measured. The material was placed into a polytetrafluoroethylene cylindrical mold (6.0 mm in diameter, 2.0 mm in height) and covered with a transparent matrix tape. The specimens were light irradiated for 20 s. One flat surface for each specimen was polished using a sequence of SiC papers up to P2,000-grit (Sankyo Fuji Star). Twelve flat specimens for each group were prepared, and then they were stored under dark conditions for 24 h in a 100% humid environment at 37°C. KHN was measured from the indentation after application of a 1.96 N load for 15 s using a microhardness tester (Via-S, Matsuzawa). Three measurements per specimen were conducted in different locations, and then the mean values were calculated.

Statistical analysis

Statistical analysis was conducted with a commercial statistical software package (SPSS Statistics Base, IBM). Because the Kolmogorov-Smirnov test confirmed the normal distribution of the SBSs and facet depths (volume loss and maximum depth), a two-way analysis of variance (ANOVA) for SBSs and one-way ANOVA for volume loss and maximum depth of wear facets with Tukey's post-hoc honestly significant difference test (significance level of 0.05) were used for data analysis. In addition, the microleakage score was assessed using the Kruskal-Wallis test at significance level of 0.05. One-way ANOVA followed by Tukey's HSD test (significance level of 0.05) was used for comparisons within subsets of the KHN data.

Scanning electron microscopy (SEM) observations of wear facets

A tabletop SEM (TM4000II, Hitachi) was used to observe the facets of two selected specimens that showed values close to the mean volume loss and maximum depth in each group after wear testing. A thin coating of gold-palladium alloy was applied in a sputter coater (Emitech SC7620 Mini Sputter Coater, Quorum Technologies) and observation was conducted at an operating voltage of 15 kV.

SEM observation of bonding interfaces

Field-emission SEM (ERA 8800FE, Elionix) was used to observe the bonding interfaces between tooth substrates and restorations. Two specimens for each group were prepared for three different substrates and five different materials. Bonded specimens were sectioned near the center of the specimen and the bonding interface was polished up to P4,000-grit SiC paper. Then, the surface was polished up to 1.0 µm-grit diamond paste (DP Paste, Struers) and ultrasonically cleaned for 30 s. The polished specimens were dehydrated using a sequence of different concentrations of aqueous solutions of tert-butanol, up to 100%, and a freeze dryer (Model ID3, Elionix). The bonding interfaces of the dehydrated specimens were argon ion etched using a compact ion shower system (EIS-200ER, Elionix) for 40 s. A thin gold-alloy coating was applied in a sputter coater (Quick Coater SC-701, Sanyu Electron) and observation was conducted at an operating voltage of 15 kV.

Results

SBS

The results for the SBS of self-adhesive flowable composites are shown in Table 2. The SBSs of the tested materials were 6.5–12.2 MPa to ground enamel, 22.5–32.5 MPa to etched enamel, and 1.3–4.2 MPa to dentin. The SBSs were different depending on the material and substrates.

The rank order of the SBS to ground enamel and dentin was FS3 = FS10 = CO > FU = VF, and that to etched enamel was $FU = CO \ge VF > FS3 = FS10$ (Table 2).

Microleakage scores

The results for the microleakage score of self-adhesive flowable composites are shown in Table 3. The microleakage scores of tested materials were 1.08-2.22 in the specimens without enamel etching and 1.22-2.35 in the specimens with enamel etching, showing significance difference depending on the material. The rank order of the microleakage score was VF > FU = CO > FS3 = FS10, regardless of the presence or absence of enamel etching (Table 3).

Occlusal wear and SEM observations of wear facets

The results of simulated occlusal wear in volume loss and maximum facet depth of selfadhesive flowable composites are shown in Table 4. The facets on tested materials after wear testing showed 0.099–0.447 mm³ of volume loss and 148.6–365.3 μ m maximum facet depth. The rank order of the volume loss and maximum depth of facet on materials was FS3 = FS10 > CO > FU = VF, and there was excellent correlation (r = 0.987) between volume loss and maximum depth values.

Representative SEM observations of facets on tested materials (after the wear tests) are shown in Figs. 1 and 2. The size of the facet in SEM observations showed the same rank order as the volume loss and maximum depth of facet.

KHN

The values of KHN of self-adhesive flowable composites are shown in Table 5. KHN of the tested materials ranged from 36.6 to 53.3 in the following order: FU > CO > FS3 > FS10 > VF. FU showed a significantly higher and VF showed a significantly lower KHN than the other materials. No significant difference was observed between FS3 and FS10.

SEM observation of bonding interfaces

Representative SEM observations of the bonding interfaces of self-adhesive flowable composites are shown in Figs. 3–7. SEM observations of material-tooth interfaces showed good adaptation regardless of substrate, but the types of fillers in the materials were different depending on the material. The observed fillers in CO, FU, and VF were irregular, while FS3 and FS10 used mainly spherical fillers.

Discussion

Self-adhesive flowable composites contain functional monomers, which can be defined as polymerizable monomers bearing both hydrophilic and hydrophobic moieties in their structure. Phosphoric acid and carboxylic acid groups are widely used as the hydrophilic acid moieties in functional monomers (14). Monomers with phosphoric acid groups, such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) in CO, phosphoric acid monomer in FS3 and FS10, glycerol phosphate dimethacrylate (GPDM) in VF, and a monomer with carboxyl groups, 4-methacryloxyethyl trimellitate acid (4-MET) in FU, were used in these materials (Table 1), and could, in theory, facilitate chemical adhesion to the tooth substrate.

However, the observed enamel and dentin bond strengths of the materials after 24 h in self-etch mode were less than 10 MPa, which is significantly lower than those previously reported for composites with universal adhesives in self-etch mode under the same bonding test conditions (15). It is possible that the self-adhesive composites have limited bonding properties to tooth substrates due to their weak demineralization and chemical bonding abilities as compared to composites used with adhesive systems. In this study, the bond strengths of the materials to etched enamel were significantly higher than those in self-etch mode to enamel and dentin. This was a result of the effects of phosphoric acid etching on enamel, which include increases in wettability, surface free energy, and surface roughness and area (16). Nevertheless,

the observed bond strengths of the materials to etched enamel were lower than those previously reported for composites with a separate adhesive (15,17). Therefore, the chemical bonding ability of self-adhesive flowable composites is still limited when compared to that of conventional one-step, multiple-step, or universal adhesive systems as shown in the literature (15,17).

When the tested materials were compared, the rank order of bond strengths was FS3 = FS10 > CO > FU = VF to enamel and dentin in self-etch mode and FU = CO > VE > FS3 = FS10 to etched enamel. Interestingly, the rank order of microleakage scores (FS3 = FS10 > CO = FU > VF) for enamel cavities with or without etching was similar to that of bond strength to enamel and dentin in self-etch mode, and strong correlations were found between these variables (r = 0.820–0.950). In addition, the order of wear properties (FS3 = FS10 > CO > FU = VF) was like that of bond strengths to etched enamel, and again, strong correlations were found (r = 0.814–0.851).

The materials FS3 and FS10 include a surface pre-reacted glass-ionomer (S-PRG) filler based on giomer technology (18). This means that the material can form chemical bonds due to an acid-base reaction and, as a result, undergo higher swelling in water than composites (19). On the other hand, higher wear resistance was observed in CO, FU, and VF than in the FS3 and FS10, meaning that the physical properties of the former were stronger than those of the latter. Thus, in bonding to etched enamel, the stronger physical properties of the CO, FU, and VF may provide better mechanical interlocking to the etched enamel surface.

When the bond strength of self-adhesive flowable composites between enamel and dentin was measured in self-etch mode, the bond strength to dentin (1.3–4.2 MPa) was significantly lower than that to enamel (6.5–12.2 MPa). Of course, the enamel bond strength was also lower than the previously reported bond strength with composites using adhesive systems. However, based on these results, this problem can be overcome using enamel etching

(enamel with etching: 22.5–32.5 MPa), which can give bond strengths comparable or superior to those of composites using adhesive systems. Therefore, self-adhesive flowable composites are recommended for small and shallow cavities that are mainly composed of enamel in pits and fissures, and in the cervical area. In addition, phosphoric acid etching of enamel is essential for the usage of these materials.

No difference in microleakage was observed between surfaces with and without phosphoric acid etching. This is somewhat interesting because acid etching of enamel greatly changes the substrate surface. The SEM observations of the bonding interfaces showed good adaptation in all conditions; thus, these materials may be able to adhere closely to both etched and un-etched enamel. In the results of the microleakage test, the scores of self-adhesive flowable composites (1.08–2.35) were less than 3, indicating that dye penetrated less than 1/3 of the dentin, and that the penetration was mostly within the enamel. A previous study reported that microleakage tests with dye penetration do not correlate with any clinical parameters, such as post-operative hypersensitivity, retention, or marginal staining (20). Therefore, there is no use in asking whether those scores are clinically acceptable.

Ujiie et al. (11) recently reported that the simulated occlusal wear (measured with the same method used in this study) of popular flowable composites on the market was $0.025-0.148 \text{ mm}^3$ in volume loss and $98.1-210.6 \mu \text{m}$ maximum facet depth. From the results of this study, FU and VF seem to have occlusal wear properties within the range of previously tested flowable composites, but FS3, FS10, and CO showed weaker occlusal wear resistance (12). A previous study reported on flowable composites [G-ænial Universal Flow, GC: volume loss of 0.025 mm³, maximum depth of 98.1 µm; G-ænial Bulk Injectable, GC: volume loss of 0.026 mm³, maximum depth of 103.8 µm (13)] that can be used in occlusal contact and have similar occlusal wear resistance to a representative nanofilled composite [Filtek Supreme Ultra, 3 M Oral Care: volume loss, 0.026 mm³, maximum depth, 102.8 µm (21); and volume loss, 0.034

mm³, maximum depth, 110.6 μ m (13)]; however, care should be taken in the selection of materials, as not all flowable composites have adequate wear resistance.

Much higher levels of wear than 8 μ m/year for the materials tested could be expected in this study. The results suggest that even the self-adhesive composites, which show similar occlusal wear to conventional flowable composites, might not be indicated for restorations in occlusal contact areas. However, the data of Barkmeier et al. (22) were reported on an early generation paste-type composite developed for use in posterior dentition. Therefore, further research studies measuring the clinical wear rate of flowable composites using an intraoral scanner might offer more relevant information.

The null hypothesis, that there would be no differences in bonding and mechanical properties among the self-adhesive composites, was rejected. The results of this study suggested that the clinical use of restorations using self-adhesive composites with phosphoric acid etching to enamel may be acceptable in small and shallow cavities that are mainly composed of enamel and are not in a stress bearing area. In addition, S-PRG fillers in self-adhesive composites have been shown to release ions responsible for remineralizing tooth structure (23), which might be a good option for high caries-risk patients, or for the restoration of small cavities in pit and fissure areas.

Conclusions

- 1. The SBSs of the tested materials were 6.5–12.2 MPa to ground enamel, 22.5–32.5 MPa to etched enamel, and 1.3–4.2 MPa to dentin.
- The microleakage scores of tested materials were 1.08–2.22 in the specimens without enamel etching and 1.22–2.35 in the specimens with enamel etching, showing significance difference depending on the material.

- 3. The facets on tested materials after wear testing showed 0.099–0.447 mm³ of volume loss and 148.6–365.3 μ m maximum facet depth.
- 4. KHN of the tested materials ranged from 36.6 to 53.3.
- 5. SEM observations of material-tooth interfaces showed good adaptation regardless of substrate, but the types of fillers in the materials were different depending on the material.

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Tables and Figures

Material (code)	Manufacturer	Resin matrix	Filler type	Filler load
Constic (CO)	DMG	Bis-GMA, EBADMA, HDMA, HEMA TEGDMA, UDMA, 10-MDP	Prepolymerized particles, Ba-glass, SiO ₂ , YbF ₃ , ZnO	65.0 wt%
Fusio (FU)	Pentron	HEMA, TEGDMA, UDMA, 4-MET,	Ba-glass	65.0 wt%
Vertise Flow (VF)	Kerr	GPDM, HEMA, MEHQ	Amorphous silicon nanosized, silanized Ba-glass	, 70.0 wt%
Fit SA F03 (FS3)	Shofu	UDMA, HEMA, phosphoric acid monomer	S-PRG filler, glass powder YbF ₃	68.2 wt%
Fit SA F10 (FS10)	Shofu	UDMA, HEMA, phosphoric acid monomer	S-PRG filler, glass powder YbF_3	65.7 wt%

Table 1 Materials used in the study

Bis-GMA, bisphenol A glycidyl methacrylate; EBADMA, ethoxylated bisphenol A dimethacrylate; HDMA, hexanediol dimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 4MET, 4-methacryloyloxyethyl trimellitate; GPDM, glycerol phosphate dimethacrylate; MEHQ, hydroquinone monomethyl ether; SPRG, surface pre-reacted glass-ionomer

Table 2 Shear bond strength (MPa)

Code	Enamel without etching	Enamel with etching	Dentin
СО	9.1 (2.1) ^{bB}	28.2 (4.6) ^{aA}	2.1 (1.7) ^{abC}
FU	7.4 (2.1) ^{bcB}	32.5 (5.1) ^{aA}	1.8 (1.2) ^{bC}
VF	6.5 (1.6) ^{cB}	25.6 (3.3) ^{abA}	1.3 (1.1) ^{bC}
FS3	12.2 (2.2) ^{aB}	23.7 (4.3) ^{bA}	4.2 (2.0) ^{aC}
FS10	$10.2 (3.2)^{abB}$	22.5 (3.9) ^{bA}	3.5 (1.5) ^{aC}

n = 15. Values are given as mean shear bond strength (SD).

The same lower-case letter in a column indicates no significant difference (p > 0.05) between estimates in different rows.

The same capital letter within individual rows indicates no significant difference (p > 0.05) between estimates in different columns.

Table 3	Microl	eakage	score
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Code	Enamel without etching	Enamel with etching
СО	1.81 (0.19) ^{bB}	2.21 (0.25) ^{aA}
FU	1.67 (0.28) ^{bB}	2.23 (0.21) ^{aA}
VF	2.22 (0.32) ^{aA}	2.35 (0.33) ^{aA}
FS3	1.26 (0.28) ^{cA}	1.34 (0.32) ^{bA}
FS10	1.08 (0.22) ^{cA}	1.22 (0.11 ^{)bA}

n = 10. Values are given as mean microleakage score (SD).

The same lower-case letter in a column indicates no statistically significant difference (p > 0.05) between estimates in different rows.

The same capital letter within individual rows indicates no statistically significant difference (p > 0.05) between estimates in different columns.

Code	Volume loss	Maximum depth
CO	0 228 (0 02 4)b	261 2 (20 p)b
0	$0.228 (0.034)^2$	201.5 (39.8)
FU	$0.099 (0.018)^{c}$	148.6 (32.9) ^c
VF	0.129 (0.024) ^c	195.1 (40.1) ^c
FS3	0.367 (0.125) ^a	327.4 (34.0) ^a
FS10	0.447 (0.128) ^a	365.3 (41.4) ^a

Table 4 Simulated wear in volume loss (mm³) and maximum depth (μ m)

n = 12. Values are given as mean volume loss and maximum depth (SD).

The same lower-case letter in a column indicates no statistically significant difference (p > 0.05) across rows.

Table 5 Knoop hardness number

Code	KHN	
СО	47.5 (1.0) ^b	
FU	53.3 (1.0) ^a	
VF	36.6 (3.0) ^d	
FS3	45.6 (1.6) ^{bc}	
FS10	44.4 (1.0) ^c	

n = 12. Values are given as mean knoop hardness number (SD). The same lower case letter indicates no statistically significant difference (p > 0.05).



Fig. 1 Representative SEM images of facets for self-adhesive flowable resin composites after Creighton University occlusal wear test at magnification of x60.



Fig. 2 Representative SEM images of facets for self-adhesive flowable resin composites after Creighton University occlusal wear test at magnification of x1,000.



Fig. 3 Representative SEM images of bonding interfaces for Constic. E, enamel; EE, etched enamel; and D, dentin.



Fig. 4 Representative SEM images of bonding interfaces for Fusio. E, enamel; EE, etched enamel; and D, dentin.



Fig. 5 Representative SEM images of bonding interfaces for Vertise Flow. E, enamel; EE, etched enamel; and D, dentin.



Fig. 6 Representative SEM images of bonding interfaces for Fit SA F03. E, enamel; EE, etched enamel; and D, dentin.



Fig. 7 Representative SEM images of bonding interfaces for Fit SA F10. E, enamel; EE, etched enamel; and D, dentin.