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Shear Bond Strength and Tooth-Composite Interaction With Self-Adhering Flowable Composites

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Clinical Relevance

The investigation of bonding performance and the assessment of tooth-composite interaction are helpful in the evaluation of restoration systems. Distinct differences between self-adhering flowables regarding the adhesive performance were observed, so that the clinical use must be pursued cautiously.

SUMMARY

Purpose: To evaluate the tooth-composite interaction (A) and shear bond strength (SBS; B) of self-adhering flowables.

Methods and Materials: (A) Thirty-two human molars with one Class V cavity were restored with Vertise Flow (VF), Fusio Liquid Dentin (FLD), an experimental self-adhering flowable (EF), or Adper Prompt-L-Pop/Filtek Supreme XT Flowable (PLP). Teeth were prepared according to laboratory standard and stored in water (24 hours, 37°C). Microleakage (ML; percentage interface length at enamel [E]/dentin [D]) and tooth-composite interaction were investigated. (B) The buccal surface of 160 embedded human molars was abraded to expose an enamel/dentin area of diameter ≥3 mm. Composite specimens were produced on enamel/dentin with VF, FLD, EF, or PLP. Prior to loading, 80 samples were water stored (24 hours, 37°C) and 80 thermocycled (5°C-55°C, 1500 cycles). The SBS was measured, and failure modes were classified by scanning electron microscopy.

Statistics: Kruskal-Wallis, Mann-Whitney U, and Fisher exact tests were performed (α=0.05).

Results: (A) At enamel margins, EF and VF showed significantly lower ML than did FLD and PLP ($p_i \leq 0.009$; 81%-89%); in dentin, lower values resulted with FLD and VF compared with PLP and EF ($p_i \leq 0.01$; 77%-94%). Adhesive tags at E were consistently verifiable with EF and VF but irregularly with FLD and PLP. At D, tags were detectable with all systems. (B) In all groups, SBS decreased by up to 97% after thermocycling. It was generally diminished with self-adhering flowables (E: 50%-98%, D: 59%-98%; $p_i < 0.02$). More cohesive defects were observed with PLP ($p_i < 0.009$).

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Conclusion: Tooth-composite morphology and bond strength indicate that the clinical use of self-adhering flowables must be pursued cautiously.

INTRODUCTION

In the mid-1990s, flowable composites were developed wherein the low viscosity permits the composite to shape itself to fit cavity areas that are difficult to access.[1,2] These conventional flowables require a separate adhesive to bond to hard dental tissues. However, the interface between tooth and restoration remains the weak point of an adhesive filling.[3,4] One reason frequently discussed in this context is the technique sensitivity of current adhesive restoration approaches.[5] Simplification of clinical procedures is therefore one driving force in current material development and research.[6,7] While in the past, most attempts at optimization have been directed at reducing the time needed and the number of steps required for application, dentists are now increasingly calling for more user-friendly and less error-prone systems.

A particularly innovative route was to develop a product that combines the restorative material with the benefits of a bonding system.[6,7] Recently, flowable self-adhering composites have been introduced that appear to promise a combination of easy handling and simplified, time-saving procedures thanks to the absence of additional etching and bonding steps. The manufacturers claimed these composites were suitable for use as a filling material in small restorations and as a lining material with a bonding quality comparable to self-etching bonding systems.[8,9]

Since the commercial launch of self-adhering flowables, only a few studies investigating bonding performance have been published. None of these studies offer conclusive arguments about the clinical potential of this material group.[10-12] Whereas Poitevin and others[11] warned against routine clinical use, Bektas and others[13] judged Vertise Flow to be a useful material with acceptable bond strength and marginal seal. Stiffness was described as similar to packable composites,[14] whereas water sorption, hygroscopic expansion, and mass reduction were regarded as critical.[15,16] Furthermore, it remains unclear whether preliminary etching of the tooth substrate is beneficial.[11,12,17,18] Initiating bond strength evaluations found divergent values for self-adhering flowables compared with conventional ones.[19-21] Also, very few studies regarding the aging behavior of this material have been published.[22,23] Longevity in particular is a severe challenge for the tooth-composite interaction.[24] Against this background, we decided to examine this new material class in order to provide further information to help assess its adhesive potential.

This study aimed to assess the composite-tooth bond of an experimental self-adhering flowable composite in comparison to two established systems in terms of microleakage, tooth-composite interaction, and shear bond strength (SBS) to enamel and dentin both before and after thermocycling. As a control, a flowable composite in combination with a 1-step self-etch adhesive was used.

METHODS AND MATERIALS

Noncarious human molars without cracks, stored in chloramine-t-trihydrate (0.5%, 4°C), were used in this study. Collection of the tooth specimens took place with informed consent and was approved by the ethics committee. The teeth were prepared according to ISO/TS 11405 within three months after extraction.[25] Three self-adhesive flowables (experimental flowable [EF], Vertise Flow [VF], Fusio Liquid Dentin [FLD]) and a flowable composite in combination with a self-etch adhesive (Filtek Supreme XT Flowable/Prompt-L-Pop [PLP]) were tested (Table 1).

The study was divided into evaluation of tooth-composite interaction (part A) and SBS to enamel and dentin (part B).

Part A: Tooth-Composite Interaction

Cavity Preparation and Restoration—Thirty-two mixed, oval Class V cavities were prepared with a rounded cylindrical diamond bur (107 μm, 836KR.314.014, Komet/Gebr. Brasseler GmbH & Co. KG, Lemgo, Germany) at high speed with water cooling. Preparations had a standard size (approximately 1.5 mm deep, 3 mm wide in incisal-apical orientation, 4 mm wide in mesiodistal orientation) and were placed facially at the cemento-enamel junction with half of the cavity margin above (enamel) and half below this line (dentin). The enamel cavity margins were given a 0.5 mm bevel with a finishing diamond bur (46 μm, 8836KR.314.014, Komet/Gebr. Brasseler GmbH & Co. KG).

Immediately after preparation of each cavity, eight teeth were randomly assigned to one of four groups EF, VF, FLD, or PLP, and restorations were placed following the manufacturer's instructions (Table 1). Light curing of the composite

Table 1: *Used Materials*.

Material (Group)	Type	Manufacturer	Composition[a] [Lot Number]	Bonding Procedure/Application
Experimental Flowable (EF)	Self-adhering flowable, experimental	DMG mbH, Hamburg, Germany	Unknown [F-142890]	Clean the tooth, remove all residuals with water spray, dry the tooth, apply product onto the cavity surface, massage a thin layer (~0.5 mm) into the entire surface for 20 s using the brush, light cure for 20 s, build up layers with a maximum of 2-mm thickness and light cure each for 20 s.
Vertise Flow (VF)	Self-adhering flowable	Kerr GmbH, Rastatt, Germany	GPDM, HEMA, prepolymerized filler, 1-μm barium glass filler, nano-sized colloidal silica, nano-sized ytterbium fluoride [3461596]	Wash the tooth thoroughly with water spray and air dry at maximum air pressure for 5 s, dispense product onto preparation with a dispensing tip, brush a thin layer (<0.5 mm) onto entire cavity surface with moderate pressure for 15-20 s, light cure for 20 s, build additional layers in increments of 2 mm or less, light cure each increment for 20 s.
Fusio Liquid Dentin (FLD)	Self-adhering flowable	Pentron Clinical Technologies LLC, Wallingford, CT, USA	UDMA, TEGDMA, HEMA, 4-MET, silane treated barium glass, amorphous silica, minor additives, photo curing system [201091]	Remove any potential debris with water and thoroughly clean the cavity, air dry briefly (2-3 s) to remove excess water and form a moist surface with no water pooling, dispense a 1-mm increment directly onto the tooth surface, gently rub the material into the preparation using the needle tip for 20 s, light cure initial layer for 10 s, add subsequent layers with a maximum thickness of 2 mm, and light cure each for 10 s; the final layer should be cured for additional 10 s.
Adper Prompt-L-Pop™/Filtek Supreme XT Flowable (PLP)	self-etch adhesive	3M Espe AG, Seefeld, Germany	*first blister:* methacrylate phosphates, bis-GMA, phosphoric acid, photo initiators; *second blister:* water, HEMA, polyalkenoic acid polymer [405560]	Remove loose debris by spraying with water, use two to three brief blows of air to dry the cavity, mix PLP as stated in the manual, brush the adhesive onto the entire cavity surface, massage it in for 15 s applying pressure, use a gentle stream of air to thoroughly dry the adhesive to a thin film, rewet the brush tip with adhesive and apply a second coat, use a gentle stream of air again, light cure the adhesive for 10 s.
	flowable composite	3M Espe AG, Seefeld, Germany	Bis-GMA, TEGDMA, functionalized dimethacrylate polymer, substituted dimethacrylate, silane treated ceramic, silane treated silica [N166300]	Dispense flowable, place maximum 2-mm-thick increments directly from the dispensing tip and light cure each for 20 s.

Abbreviations: 4-MET, 4-methacryloxyethyltrimetellitic acid; bis-GMA, bisphenol-glycidyl methacrylate; GPDM, glycerol phosphate dimethacrylate; HEMA, hydroxyethylmethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *As described by the corresponding manufacturer.*

layers was performed with a minimum intensity of 1000 mW/cm^2 at the minimum distance from the light source (HS LED 1200, Henry Schein Dental Deutschland GmbH, Langen, Germany). Restorations were finalized with a finishing diamond bur and silicone polishers (Politip F, 533602; Politip P, 533584; Ivoclar Vivadent AG, Schaan, Liechtenstein).

Specimen Preparation: Evaluation of Microleakage—The restored teeth were stored in double-distilled water (24 hours, 37°C) and immersion fixed in buffered glutaraldehyde (5%, 0.1 M sodium phosphate buffer, pH 7.2, 24 hours, 4°C). For microleakage assessment, the teeth were soaked three times in fresh buffer (0.1 M sodium phosphate buffer, pH 7.2, one hour each, 20°C). The apices of

the teeth were sealed with composite resin and the whole tooth surface was covered with two coats of nail polish, with the exception of a 1 mm window around the restoration. Microleakage was tested using a standardized tracer penetration method. The samples were immersed in basic ammoniacal silver nitrate ($AgNO_3/NH_4OH$, 3 M, pH 9.5, 24 hours, 37°C) in darkness and rinsed in distilled water (60 seconds, 20°C) before they were incubated in photo-developing solution (TETENAL Europe GmbH, Norderstedt, Germany) under fluorescent light (Konrad Benda Laborgeräte, Wiesloch, Germany, eight hours, 20°C). After rinsing in distilled water again (60 seconds, 20°C), the roots were cut 2 mm under the restoration margin and the crown was embedded in Stycast 1266 (Emerson & Cuming ICI Belgium N.V., Westerlo, Belgium). Restorations were then sectioned longitudinally under water cooling (n=3 each, 200 μm thickness, Leitz 1600 Sägemikrotom, Ernst Leitz Wetzlar GmbH, Wetzlar, Germany). Each section was examined stereomicroscopically with a digital microscope camera (20×, ProgRes CT3, JENOPTIK Laser, Optik, Systeme GmbH, Jena, Germany) with accessory operation and control software. Microleakage was measured separately for enamel and dentin in terms of length of $AgNO_3$ penetration and stated as the percentage of the interface length.

Specimen Preparation: Tooth-Composite Interaction Features—The sections were decalcified (2% HCl, 10 seconds), deproteinized (10% NaOCl, 30 seconds), rinsed with distilled water, dehydrated in ascending ethanol baths (30% to 100%), immersed in hexamethyldisilazane (Carl Roth GmbH & Co. KG, Karlsruhe, Germany), and air dried. All samples were mounted on specimen holders, gold sputtered (20 nm; Edwards Sputter Coater S150B, BOC Edwards Ltd., Crawley, UK) and examined by scanning electron microscopy (SEM; 30× to 2000×, 25 kV accelerating voltage, LEO 1430 vp, Carl Zeiss Microscopy GmbH, Oberkochen, Germany) in the backscattered and secondary electron mode.

The tooth-composite interaction was characterized by tag formation at enamel (yes/no) and dentin (intra, peritubular, and lateral resin penetration). For each of the three sections per tooth, the interfacial gap formation (adhesive failure) was scored for the interface between restoration and enamel as well as dentin (1 to 5):

- 1: no adhesive failure at enamel or dentin interface
- 2: <33% adhesive failures of the interface length
- 3: 33% to 50% adhesive failures of the interface length
- 4: >50% adhesive failures of the interface length
- 5: total adhesive failure (100%)

The mean score for each tooth was determined separately for enamel and dentin.

Part B: SBS Measurements

Specimen Preparation: Evaluation of SBS—One hundred sixty human molars were embedded in Stycast 1266 (Emerson & Cuming ICI Belgium N.V.) and randomly assigned to the groups EF, VF, FLD, or PLP (n=40). In each group, the buccal surfaces of the teeth were wet abraded with a 120-grit silicon-carbide paper until enamel or dentin (n=20) surfaces with a diameter greater than 3 mm were exposed (Struers DAV-P, Struers GmbH, Willich, Germany). The surfaces were polished for 60 seconds under water cooling with a 600-grit silicon carbide paper to standardize the smear layer. It had already been ascertained that the specimens had the right diameter of flat surface, and the dentin specimens were additionally assessed for the absence of enamel (25×, stereomicroscope SM 20, Carl Zeiss, Jena, Germany). In accordance with ISO, a split mold (polytetrafluoroethylene, 3 mm diameter, 3 mm high) was positioned on the exposed tooth surfaces with a strong clamp to define and limit the bonding area and to produce a standardized composite test specimen. Composite cylinders were bonded to enamel and dentin according the manufacturer's instructions (Table 1) and light cured as already mentioned. Specimens that failed immediately during the bonding procedure were documented as "pretesting failure" (PTF) and were considered statistically.

Test specimens were stored in distilled water (24 hours, 37°C). From each group, 20 samples ($n_{enamel}=10$, $n_{dentin}=10$) were thermocycled (TC, 5°C-55°C, 1500 cycles; Willytec Thermalcycler V 2.8, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany). Specimens that debonded during this procedure were recorded as "testing failure" (TF) and were considered statistically.

Immediately after removal from water, the SBS was measured according to ISO. Each specimen was positioned in a Universal Testing Machine (Z 010, software testXpertII, version 2.2, Zwick GmbH & Co. KG, Ulm, Germany) and loaded at a cross-head speed of 0.75 ± 0.25 mm/min.

Specimen Preparation: Evaluation of Failure Modes—Debonded composite cylinders and enamel/

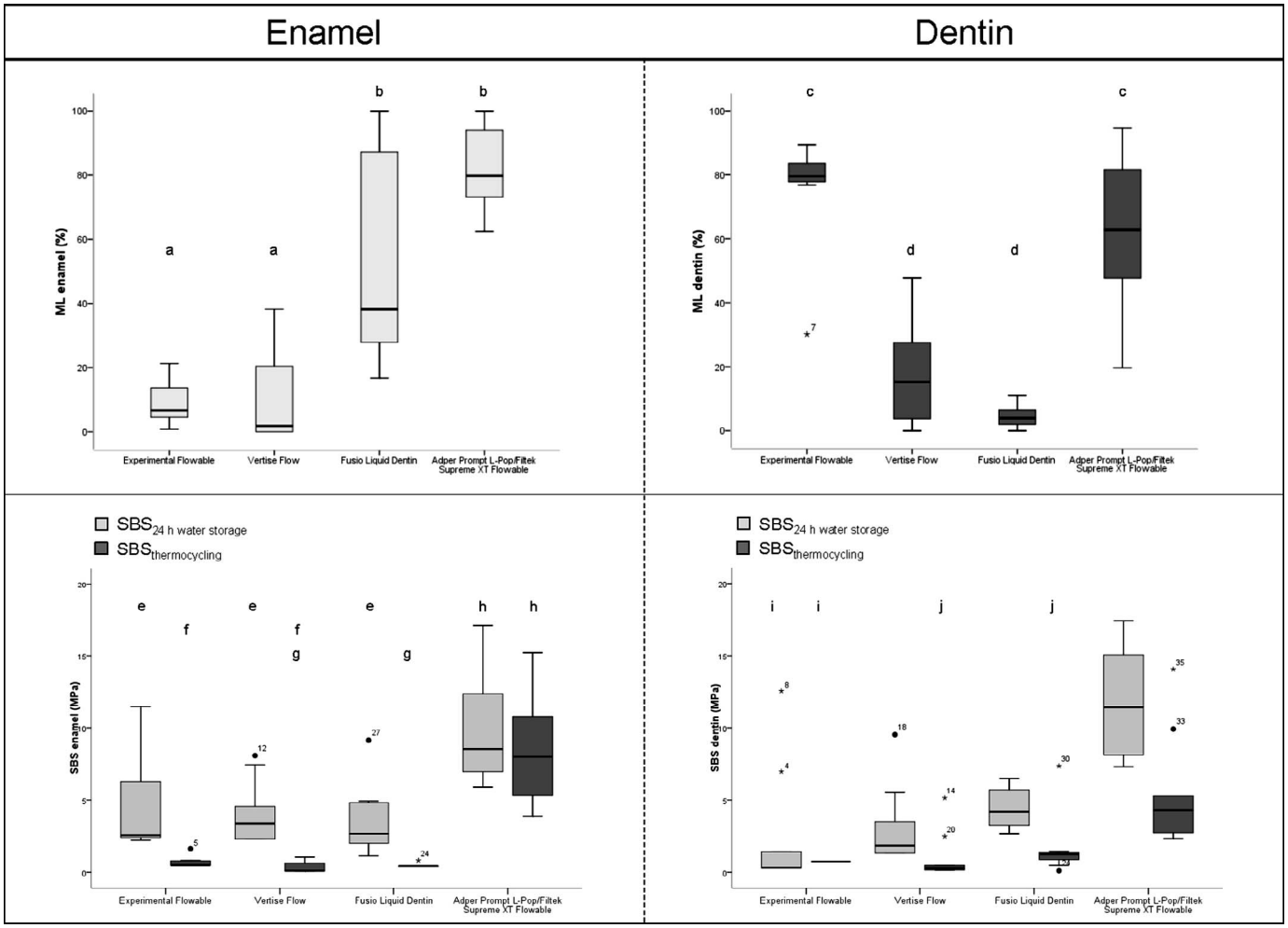

Figure 1. *Microleakage and shear bond strength on enamel and dentin. Groups with the same superscript letters are not significantly different ($p_i > 0.05$).*

dentin samples were mounted on specimen holders before being gold sputtered (20 nm, Edwards Sputter Coater S150B) and examined by SEM (30×, 20 kV, CamScan CS 24, Cambridge Scanning Corp Ltd, Cambridge, UK) in the backscattered and secondary electron mode. SEM images were digitized (EPSON Expression 1680 Pro, Seiko Epson Corp, Nagano, Japan) and stitched (Adobe Photoshop CS 4 Extended, Adobe Systems Inc, San José, CA, USA). Using the stitched SEM images, failure modes were scored by cohesive/adhesive failure percentages (1 to 6):

- 1: 100% cohesive failure at composite, adhesive, or enamel/dentin
- 2: 75% to <100% cohesive/1% to <25% adhesive
- 3: 50% to <75% cohesive/25% to <50% adhesive
- 4: 25% to <50% cohesive/50% to <75% adhesive
- 5: 1% to <25% cohesive/75% to <100% adhesive
- 6: 100% adhesive failure between composite and enamel/dentin

Statistical Analysis

The data were subjected to statistical analysis using SPSS (PASW Statistics 18, SPSS Inc, Chicago, IL, USA). The significance level was set at $\alpha=0.05$.

Part A—Microleakage mean values ± standard deviations were determined; medians and percentiles were illustrated with boxplots (Figure 1). Differences between groups regarding microleakage formation and adhesive failures were analyzed using Kruskal-Wallis and 2-tailed Mann-Whitney U-test. Materials were ranked based on statistically significant differences with grades 1 to 4. Grade 1 corresponded to the most favorable of the four values (lowest mean value of microleakage) and grade 4 to the most unfavorable.

Table 2: *Microleakage/Adhesive Defects (Score) at Enamel and Dentin Interface*[a]

	Enamel				Dentin			
	EF	**VF**	**FLD**	**PLP**	**EF**	**VF**	**FLD**	**PLP**
Microleakage, %	9.0 (6.7)$_a$	10.3 (16.1)$_a$	52.9 (33.8)$_b$	82.1 (13.4)$_b$	75.2 (18.6)$_c$	17.6 (16.3)$_d$	4.4 (3.6)$_d$	62.3 (24.5)$_c$
Score	2.0$_e$	2.2$_e$	4.5$_f$	3.9$_f$	4.5$_{g,h}$	3.6$_{g,i,j}$	3.3$_i$	4.3$_{h,j}$

[a] *Mean (standard deviation). Means with same subscript letters are not significantly different ($p>0.05$).*

Part B—SBS mean values ± standard deviations were determined; medians and percentiles were illustrated with boxplots (Figure 1).

PTFs and TFs were replaced by a singular imputation with the lowest measured SBS value in each group, differences between groups, and differences within a group (without vs with thermocycling), and differences regarding fracture mode were analyzed using Kruskal-Wallis and 2-tailed Mann-Whitney U-test. Differences in the appearance of PTFs and TFs were analyzed using a 2-tailed Fisher exact test. Materials were ranked based on statistically significant differences with grades 1 to 4. Grade 1 corresponded to the most favorable of the four values (highest mean value of SBS) and grade 4 to the most unfavorable.

RESULTS

Part A

Microleakage was always observed in different occurrences (Table 2; Figure 1). At enamel interfaces, EF and VF showed significantly lower microleakage than did FLD and PLP (81%-89%) and were both ranked 1.5 compared with 3.5 for FLD and PLP (Table 3). Within dentin cavity segments, FLD and VF showed significantly lower microleakage than PLP and EF (77%-94%), which is consistent with the material ranking of 1.5 compared with PLP and EF, with rank 3.5.

The tooth-composite interaction was generally based on adhesive tag formation at enamel. The dentin tooth-composite interaction showed product-specific characteristics. Typical SEM images are shown in Figures 2 and 3. At enamel, the experimental flowable always showed distinct tags, whereas these arose frequently and lightly with VF and sparsely in the case of FLD and PLP. At dentin, in the PLP group, tags appeared as slightly peritubular anchored and regularly lateral branched. With EF, tags were thinner and partially branched; with FLD and VF, they were thin, sparse, and without branching.

Interfacial adhesive failures occurred with all systems at enamel and dentin (Table 2). At enamel, in the self-adhering flowables group, EF and VF showed significantly lower score values than FLD. At dentin, FLD exhibited the lowest score in the group, with a significant difference from EF.

Part B

The SBSs are illustrated in boxplots in Figure 1. Tables 4 and 5 summarize the mean values and standard deviations, number of PTFs and TFs, and scores of failure modes after water storage and after thermocycling.

After 24 hours of water storage, higher values of SBS were found in the control group ($p_i<0.004$) irrespective of the tooth substrate. The experimental flowable did not have a significantly higher SBS to enamel ($p_i>0.237$) than the other two self-adhering flowables. For the self-adhering flowables, the highest bond strength to dentin was seen with FLD ($p_i<0.0029$) and the lowest value with EF ($p_i<0.043$). In the materials ranking, first rank was given to the control, followed in descending order by FLD, VF, and EF (Table 3).

Table 3: *Ranking of Materials by Specific Parameters*

	Enamel				Dentin			
	EF	**VF**	**FLD**	**PLP**	**EF**	**VF**	**FLD**	**PLP**
Microleakage	1.5	1.5	3.5	3.5	3.5	1.5	1.5	3.5
Adhesive defects	1.5	1.5	3.5	3.5	3.5	1.5	1.5	3.5
$SBS_{24h\ water}$	3	3	3	1	4	3	2	1
$SBS_{thermocycling}$	2	3.5	3.5	1	4	2.5	2.5	1
Tooth-composite interaction	Distinct tags	Frequent smooth tags	Sparse tags	Sparse tags	Thin, partly branched tags	Sparse tags	Sparse tags	Peritubular anchored, branched tags

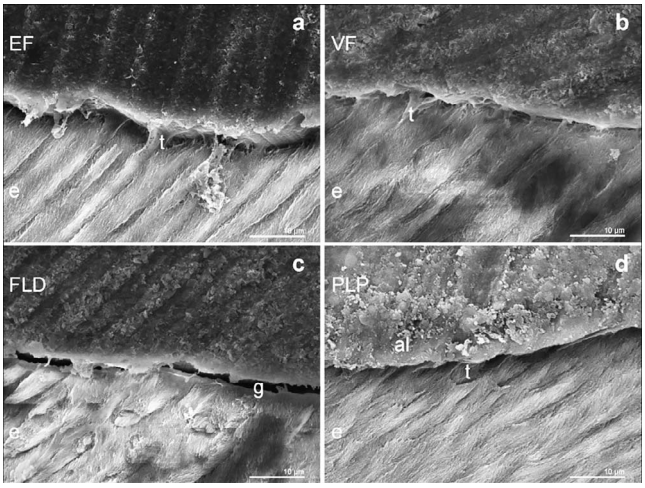

Figure 2. *Typical scanning electron microscope images of enamel-resin interface. (a): Distinct enamel tags with EF. (b): Light tags with VF. (c): A large gap can be observed between FLD and enamel. Tags were not verifiable in this image. (d): A thin adhesive layer between resin and enamel; sparse enamel tags were observed with PLP. e, enamel, t, tag; g, gap; al, adhesive layer.*

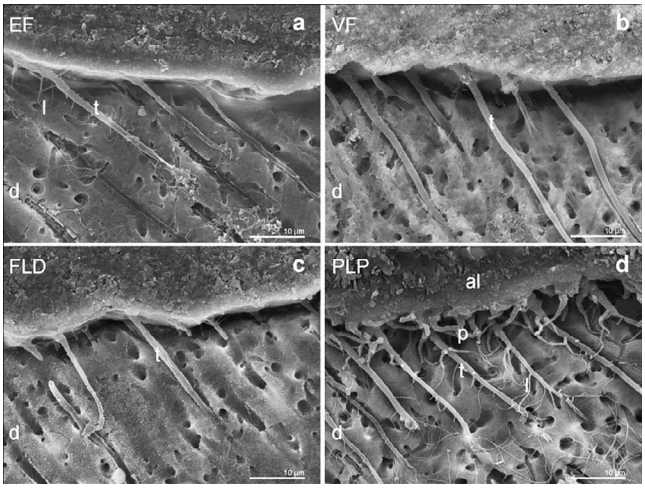

Figure 3. *Typical scanning electron microscopy images of dentin-resin interface. (a): Thin and partial branched tags produced by EP. (b, c): Tags are thin, sparse, and not laterally branched with VF/FLD. (d): Peritubular anchored and regularly lateral branched dentin tags produced by PLP are shown. d, dentin; t, tag; l, lateral branches; p, peritubular resin penetration; al, adhesive layer.*

After thermocycling, generally higher values of SBS were found in the control group ($p_i<0.001$). On enamel within the group of self-adhering flowables, EF showed higher SBS than FLD ($p_i<0.005$). At the dentin surfaces, only one specimen survived aging with EF, which is why statistical analysis was not performed for EF. FLD showed significantly higher SBS than VF. Irrespective of the tooth substrate, SBS values decreased after TC in all groups. Within the self-adhering flowables group, the reduction of SBS was up to 90%; with PLP, it was up to 53% after thermomechanical loading. After TC, the ranking of the systems was changed only marginally (Table 3).

The quantity of PTFs differed between the materials. While FLD showed no pretesting failure at all, this phenomenon occurred in all other groups. The flowables EF and VF presented more failed specimens in comparison with FLD and PLP ($p_i<0.02$).

No TF occurred within the control group, while failures were observed in the groups of flowables with varying frequencies of 3/10 to 8/10 (E) and 0/10 to 8/10 (D). The experimental flowable showed the highest rates of total failures (15 of 20) on enamel and dentin in comparison with all others ($p_i<0.033$).

Failure mode analysis revealed significantly more cohesive defects in the control group compared with the self-adhering flowables ($p_i<0.009$). Among the self-adhering flowables with VF on enamel, significantly more cohesive defects were observed compared with FLD ($p=0.033$). No other significant differences were found between the self-adhering flowables ($p_i>0.057$); for FLD without TC, the modes were all adhesive.

DISCUSSION

Currently, only a few self-adhering flowable materials are commercially available, and in consequence, only a very few investigations regarding the bonding performance of the materials have been carried out. In this study, we tested the leading products in the market and one experimental material of the same class.

The literature might lead to the expectation that these simplified materials with no additional adhe-

Table 4: *Shear Bond Strength to Enamel and Dentin, After 24 Hours of Water Storage*[a]

	Enamel				Dentin			
	EF	VF	FLD	PLP	EF	VF	FLD	PLP
PTF, n	2	2	0	0	5	3	0	1
SBS, MPa	4.4 (3.0)$_a$	4.0 (2.1)$_a$	3.5 (2.3)$_a$	9.8 (3.6)	2.4 (4.1)	3.0 (2.6)	4.4 (1.3)	11.6 (3.5)
Score	5.8$_{b,c}$	5.5$_b$	6.0$_c$	3.8	6.0$_d$	5.6$_d$	6.0$_d$	3.4

[a] *Mean (standard deviation). Means with same subscript letters are not significantly different ($p>0.05$).*

Table 5: *Shear Bond Strength to Enamel and Dentin, After Thermocycling*[a]

	Enamel				Dentin			
	EF	VF	FLD	PLP	EF	VF	FLD	PLP
PTF/TF, n	2/4	1/3	0/8	0/0	5/4	3/0	0/0	0/0
SBS, MPa	0.7 $(0.4)_a$	0.4 $(0.4)_{a,b}$	0.5 $(0.1)_b$	8.3 (3.7)	0.7 (0.0)	1.0 $(1.6)_c$	1.6 $(2.1)_c$	5.4 (3.7)
Score	5.9_d	5.4_d	5.6_d	3.7	5.9_e	5.7_e	5.8_e	2.9

[a] *Mean (standard deviation). Means with same subscript letters are not significantly different ($p>0.05$).*

sive system would bond less efficiently with tooth substrate when compared with multistep etch-and-rinse or self-etch reference materials.[11,24] Nonetheless, hopes were pinned on the new self-adhering composites, with the expectation that their bonding effectiveness might be similar to the already simplified one-step self-etch adhesives combined with flowable composites. For this reason, we chose a restoration system consisting of a one-step self-etch adhesive with a flowable composite as control (Adper Prompt L-Pop, Filtek Supreme XT Flowable). Materials from the same manufacturer were combined to reduce the risk of side effects caused by unexamined interaction between substances.[5] An important element in assessing a restoration system's performance is evaluating the testing system itself by comparing it with a long-standing adhesive system as a control under the same laboratory conditions.[6,26,27]

The investigation of the tooth-composite bond and bond failures allows a complex assessment of restoration systems. Therefore, the evaluation of microleakage and interfacial bond failures in three-dimensional cavities was combined with the two-dimensional evaluation of SBS. With regard to silver nitrate penetration, the comparison between groups revealed significant differences. Regarding the ranking of the systems, VF achieved top positions (rank 1.5) for both tooth structures. The other tested self-adhering flowables achieved either a high rank in enamel and a low one in dentin (EF 1.5/3.5) or vice versa (FLD 3.5/1.5). These results are in contrast with others,[18] who found no significant differences between the microleakage of VF and FLD in enamel and a lower microleakage in dentin with VF compared with FLD. A possible reason for the deviation of the present results from previous ones is the study design; Celik and others[18] used thermocycling for artificial aging before microleakage measurement, which may enhance the effect of this parameter and influence the materials' ranking.

Regarding the relationship between microleakage and tooth-composite interaction at enamel, it is notable that the lowest amounts for microleakage and adhesive failure (EF and VF) corresponded with the most intensive interlocking of resin with enamel by tag formation and vice versa; high values of microleakage (FLD and PLP) were associated with poor micromechanical interlocking. For dentin, this correlation was not seen, as EF and PLP with an acceptable adhesive interlocking (tag formation, peritubular anchoring, lateral branches) showed the highest rates of microleakage. In contrast, low levels of microleakage generally corresponded with fewer adhesive failures both at enamel and dentin.

Thermocycling as a standard artificial aging method was used prior to the SBS measurements.[28] The thermocycling regimen of ISO TS 11405[25] was modified because there is a reference that the recommended number of 500 cycles is too low to achieve a realistic aging effect. Gale and Darvell stated that 10,000 cycles are equivalent to clinical use of one year.[29] Thus, the 1500 cycles applied in the present study may not simulate long-term clinical use, but they are sufficient to discriminate between materials that cannot withstand a wet environment on one hand and those that can on the other. In the current study, significant differences in SBS between the materials were found before and after artificial aging. For both tooth substrates, the self-adhering flowables showed significantly lower bond strength before and after thermal loading than the control. Furthermore, on dentin, FLD was more effective in bonding than the other two self-adhering materials. This was confirmed in a similar microtensile bond strength evaluation.[11] TC decreased SBS significantly in all cases, which is also in line with the findings of other working groups.[22,23]

Differences between the groups may be the result of a number of factors, but the largest influence is probably associated with the composition (Table 1), the rheological potential, and the types of the functional monomer.[30,31] FLD contains 4-methacryloxyethyltrimetellitic acid (4-MET) as a functional monomer, whereas VF contains glycerol phosphate dimethacrylate (GPDM). No information about the

chemical composition of the EF is given by the manufacturers.

4-MET is a well-known and frequently used acidic functional monomer with good adhesive durability on dentin.[32] It partially demineralizes dentin, leaving hydroxyapatite partially attached to collagen within a submicron hybrid layer. The residual hydroxyapatite interacts chemically with carboxyl groups of 4-MET.[31] At enamel, FLD showed higher microleakage, inferior micromechanical interlocking, but equivalent SBS compared with the other self-adhering materials. At the dentin surfaces, regarding microleakage and SBS, FLD performed better than the other flowables, although the micromechanical interlocking was poor. The fracture analysis also showed high rates of adhesive failures, which also confirms that FLD has weak micromechanical anchoring. On the basis of these observations, it can be speculated that FLD might be less acidic, resulting in a weaker enamel bonding and that the chemical bond between 4-MET and dentin is stronger compared with the other functional monomers.

Data regarding the bonding effectiveness and chemical analyses of the interfacial hybrid zone of the functional monomer in VF, GPDM, have been rare until now. The manufacturer points out that, on one hand, the phosphate functional group of the GPDM monomer will create a chemical bond with the calcium ions of the tooth. On the other hand, GPDM holds two methacrylate functional groups for copolymerization with other methacrylate monomers.[33] GPDM is also used by the manufacturer in the well-known etch-and-rinse adhesive OptiBond FL, which has performed excellently in numerous clinical and laboratory studies.[34,35] However, in a two-dimensional geometry (SBS measurements), VF did not perform as well as FLD. Looking at the microleakage values and adhesive defect formation in SEM images, the material achieved best sealing rates, independent of tooth structures. This could be the result of a chemical bond caused by the acidic phosphate group in the GPDM monomer.[11] Otherwise, looking at failure mode characterization, VF showed more cohesive defects than the other self-adhering composites regardless of the tooth substrate, which is also an indicator of a stronger micromechanical or chemical anchoring. The experimental system was able to seal the enamel sufficiently, supported both by microleakage values and tooth-composite interaction. Compared with the other self-adhering materials, EF showed a weaker sealing and bond at dentin. This may be an indication of a more acidic composition—with good bonding on enamel and less efficient performance on dentin. Whether the differences in efficiency are related to a potentially acidic mode of function remains speculation, because no information about the chemical composition is given by the manufacturers.

As a result, self-adhering composites should provide a strong chemical bond to the tooth on account of filler content and viscosity. One potential explanation of the lower SBS values with the self-adhering materials could be that the higher viscosity of the flowable resin composites compared with a separate adhesive system hinders deep penetration into the dentin tubules and between collagen fibers, which would improve sealing of the tooth structures and SBS values.

In accordance with several working groups, distinct differences in the bonding effectiveness of the materials were also found with the occurrence of PTFs.[11,36] The correct handling of this phenomenon is often discussed.[6,37,38] Excluding all PTFs from statistical analysis overestimates the mean bond strength and should be avoided because a high proportion of PTFs is typically associated with low SBS, measured for those specimens that resist debonding prior to testing.[39] Alternatively, researchers may assign an SBS value of 0 MPa or a value equivalent to the lowest measured for each PTF. Using 0 MPa for calculation in the event of PTF occurrence penalizes the tested materials severely, as there was low bond strength above 0 MPa. In case of a high incidence of PTFs, methods with data imputation will inevitably result in data distribution that is skewed. Because of this debate, we decided to assign the lowest measured SBS value within the respective group to a PTF.

In part, group differences regarding the evaluation of microleakage, adhesive defects, and degree of tooth-composite interaction and corresponding material ranking are in conflict with the ranking of SBS measurements. In contrast to the evaluation of tooth-composite interaction features in three-dimensional Class V restorations with flat specimens, a pure material science parameter is determined. C-factor, other physical outcomes resulting from three-dimensional geometry, and chemical effects remain unconsidered.

In general, the question can be raised as to whether short-term experimental studies can forecast clinical outcomes. Whereas some authors have questioned the value of experimental studies predicting clinical reliability in general,[40] the outcomes of prior laboratory investigations are often highlighted as indispensable and beneficial for characterizing adhesive

systems prior to product launch.[27,41-43] In addition, De Munck and others[4] and Peumans and others[35] have retrospectively concluded that the bonding effectiveness found *in vitro* correlates to a certain extent with their *in vivo* findings. In our opinion, clinical studies with self-adhering composites as the definitive filling material should be initiated only when more promising laboratory results are available and when these results are comparable with established reference systems.

CONCLUSION

The study indicates that the self-adhering flowables exhibit significantly lower SBS to dental tissue than the self-etching control PLP. An individual evaluation of every newly launched self-adhering restorative appears necessary as these materials differ considerably in bonding failure mode and tooth-composite interaction. If microleakage and SBS values of clinically successful restoration materials are used as a benchmark, the self-adhering flowables tested in this study are currently not suitable as a permanent filling material *in vivo*.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee of the University of Leipzig, Germany. The approval code for this study is 299-10-04102010.

Conflict 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, and/or company that is presented in this article.

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