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ORIGINAL ARTICLE

Bonding and sealing ability of a new self-adhering flowable composite resin in class I restorations

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Abstract
Objectives The aim of this study is to assess by means of shear bond strength tests (SBS), microleakage analysis (μLKG), and scanning electron microscopy (SEM) the bonding potential and sealing ability of a new self-adhering composite resin.
Materials and methods SBS and μLKG of Vertise Flow (VF, Kerr) were measured and compared to the all-in-one adhesive systems G-Bond (GB, GC), AdheSE One (AO, Ivoclar Vivadent), Adper Easy Bond (EB, 3M ESPE), Xeno V (XV, Dentsply), and iBOND (iB, Heraeus Kulzer). For each system, 20 molars were tested for SBS on dentin (n=10) and enamel (n=10). For μLKG assessment, 12 premolars per group were selected and small, box-shaped cavities were made. After restoration, the teeth were immersed in 50 wt% silver nitrate solution for 24 h. For each group, 10 randomly selected specimens were processed for leakage calculations, while two of the specimens were examined under SEM. Between-group differences in SBS to dentin and μLKG were assessed using Kruskal–Wallis analysis of variance followed by the Dunn's Multiple Range test. Enamel SBS data were analyzed with one-way ANOVA, followed by the Tukey test.
Results On dentin and enamel, VF recorded the lowest SBS values that were statistically comparable to those measured by GB, iB, and AO. μLKG analysis showed the lowest percentage of stained interface for VF. Significantly greater extent of infiltration was seen for iB and EB.
Conclusions Although VF resulted in lower bond strengths values on either dental substrate, better marginal sealing ability was visualized in comparison with all-in-one adhesive systems.
Clinical relevance The results of the present study demonstrated satisfactory in vitro outcome of the self-adhering flowable composite resin VF when used to restore class I cavities.

Keywords Adhesion · Bonding agents · Flowable · All-in-one · Self-adhering

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Introduction

Enhanced adhesion at the interface between dental substrates and restorative materials is crucial for achieving leakage-free and durable restorations. Extensive research efforts have been directed to dentin for the last two decades, focusing on the physical, chemical, and micromechanical aspects of the bonding mechanism [1]. Research advancements have mainly aimed at reducing the sensitivity of the technique. For this purpose, all-in-one adhesive systems have been introduced [1–9]. In these systems, the adhesion is based on the self-etch approach, and the three traditional steps in which the adhesion process is realized, etching, priming, and bonding, are accomplished by a single solution [2, 3, 5, 7–9]. The exclusion of rinsing and drying steps is indeed an attractive clinical advantage of all-in-one systems, since the possibility of cavity contamination is reduced and over-drying and over-wetting issues are limited [10, 11]. In addition, the risk of post-operative sensitivity is reduced with the self-etch approach, which involves simultaneous substrate demineralization and resin infiltration [12].

Flowable composite resins have been proposed as restorative materials. Their low modulus of elasticity favors contraction stress dissipation and marginal integrity preservation [13–17]. Flowable composites have been reported to improve marginal adaptation of restorations in relation to their rheological properties [13, 18, 19]. On the other hand, flowable composites, due to their reduced filler content, show lower mechanical properties when compared with conventional hybrid composites. In particular, the lower filler load of flowables may be responsible for a reduced resistance to deformation during function. Therefore, for the restoration of cavities in high load-bearing areas, the use of flowable composites is recommended only for cavity lining. Conversely, in the restoration of small-sized cavities, as most of the occlusal forces are resisted by the residual tooth structure, the use of flowable composites as stand-alone materials has been proposed [13, 14, 20–23].

A further advancement in adhesive dentistry is represented by the recent introduction of a so-called "self-adhering composite resin", which combines an all-in-one bonding system and a flowable composite, eliminating the need for a separate adhesive application. Incorporation of the bonding agent into a flowable composite holds great potential with respect to saving chair time and minimizing handling errors. No data on the adhesive properties of this simplified restorative material are yet available, and the only retrievable information on the bonding mechanism and performance of this system is that provided by the manufacturer.

Although clinical trials produce the most reliable evidence and translation of in vitro findings to oral-conditions has limitations, laboratory tests are still useful at promptly yielding first-hand information [2, 24]. Specifically, bond strength tests have been considered to provide a quantitative assessment of materials adhesion, based on the concept that the stronger the bond, the better it will resist contraction and functional stresses [2, 3]. Major shortcomings typically related to poor sealing and open margins are marginal staining and bacterial invasion, leading to secondary caries and pulpal damage. Microleakage studies are widely used and referred to as indicators of the materials' sealing ability [1, 24–26].

The aim of the present study was to assess with shear bond strength measurements and microleakage evaluation the adhesive potential and marginal sealing ability of a newly introduced self-adhering flowable composite.

The tested null hypothesis was that statistically similar bond strengths and interfacial sealings are achieved by the new self-adhering flowable composite and by some marketed all-in-one adhesives used in combination with the proprietary flowable composites.

Materials and methods

Shear bond strength tests

A sample of 120 sound extracted molars was collected following informed written consent from the donors. Teeth were stored in 0.5 % Chloramine T solution at 4 °C for preventing bacterial growth for no longer than 3 months until used in the experiment. From the collected sample, two groups of 60 teeth were randomly formed. On teeth meant for the tests on enamel (group E), the roots were cut off 2 mm below the cement–enamel junction with a water-cooled, low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). The crowns were embedded in acrylic resin with their sound buccal or lingual surface displayed. On each tooth, the enamel substrate was wet ground with 320-grit SiC paper (Buehler, Lake Bluff, IL, USA) to create a flat enamel surface, rinsed, and air-dried. Teeth selected for the tests on dentin (group D) were embedded in resin with their long axis perpendicular to the base of the resin block. Then, on each embedded tooth, the occlusal portion was removed, thus exposing a mid-coronal dentin substrate. A standardized smear layer was created by grinding the dentin substrate with 320-grit SiC wet paper (Buehler, Lake Bluff, IL, USA) for 1 min.

Within each group, the following equally sized subgroups (n=10) were randomly formed, based on the adhesive/flowable composite combination to be tested:

- G-Bond/Gradia Direct LoFlo (GC, Tokyo, Japan);
- AdheSE One/Tetric Evo Flow (Ivoclar Vivadent, Schaan, Liechtenstein);
- Adper Easy Bond/Filtek Supreme XT Flow (3M ESPE, St. Paul, MN, USA);
- XenoV/X Flow (Dentslpy Detrey, Kostanz, Germany);
- iBOND/Venus Flow (Heraeus Kulzer, Hanau, Germany);
- Vertise Flow (Kerr, Orange, CA, USA)

The sample size of 10 specimens per group was used in reference to a recent review that surveyed 74 shear bond strength test studies published between 2007 and 2009, and reported 10 or less than 10 specimens per group to be used in the majority (74 %) of the studies [27].

In order to test the materials on a standardized bonding area, an aluminum split mold (Fig. 1a) was used to hold a 3-mm internal diameter silicon mold (Fig. 1b) on the substrate surface. After having prepared the dental substrate according to the manufacturer's instructions (Tables 1 and 2), either the proprietary flowable composite resin or Vertise Flow was applied in a 2-mm-thick layer that was light-cured with a halogen curing device (VIP, Bisco Inc., Schaumburg, IL, USA; 600 mW/cm^2). The specimen preparation procedures were carried out by the same operator (MM). The bonded specimens were left undisturbed for 24 h in 100 %

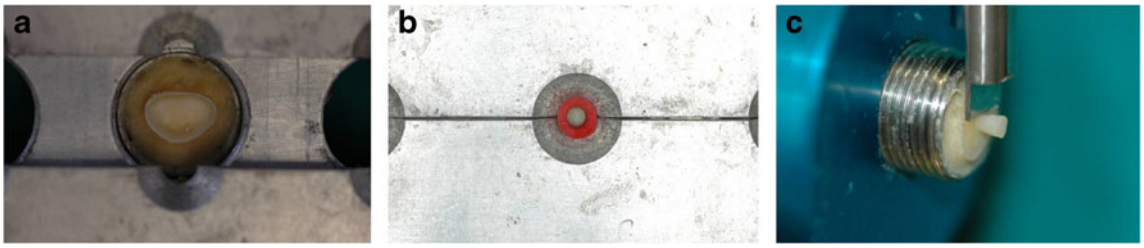

Fig. 1 Preparation of the specimens for shear tests. **a**, **b** Fixing the specimen in the aluminum split mold in order to hold a 3-mm internal diameter silicon mold onto the substrate. **c** Applying a shear load in a direction parallel to the bonded interface

humidity at 37 °C prior to shear bond strength testing. Using a universal testing machine (Triax Digital 50, Controls,132 Milan, Italy), a shear load was applied in a direction parallel to the bonded interface and at a crosshead speed of 0.5 mm/min until failure occurred (Fig. 1c). The load at failure was recorded in newtons. The diameter of the debonded composite cylinder was measured with a digital caliper (Orteam s.r.l, Milan, Italy). Bond strength was then calculated in megapascals by dividing the load at failure by the adhesive surface area (in square millimeter). Failure modes were evaluated by a single operator (CG) under an optical microscope (Nikon type102, Tokyo, Japan) at ×40 magnification, and classified as cohesive within the substrate (enamel/dentin or composite resin), adhesive (between composite resin and enamel/dentin) or mixed (if adhesive and cohesive fractures occurred simultaneously).

Microleakage

Seventy-two extracted human molars were stored in 0.5 % Chloramine T solution at 4 °C to prevent microbial growth. Box-shaped class I cavities were prepared under abundant water cooling on the occlusal surface of each tooth, using a

Table 1 Chemical composition and instructions for use of the tested adhesives

Adhesive	Chemical composition	Application
G-BOND	Acetone, distilled water, 4-methacryloxyethyltrimellitate anhydride, UDMA, triethyleneglycol dimethacrylate (TEGDMA)	Application and leaving undisturbed for 5–10 s;
Batch 0612121	pH=2.0	Air-drying for 5 s;
		Light-curing 10 s;
AdheSE One	Derivatives of bisacrylamide, water, bismethacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxy alkyl methacrylamide, silicon dioxide, catalysts, stabilizers.	Application and agitation for 30 s;
		Air dispersing until there is no water moving;
Batch K10655	pH=1.5	Light-curing for 10 s
Adper Easy Bond	HEMA, bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylate functionalized polyalkenoic acid, Finely dispersed bonded silica filler with 7-nm primary particle size, ethanol, water, initiators based on camphorquinone, stabilizers.	Application and agitation for 20 s;
Batch 346502	pH=2.4	Air-drying for 5 s;
		Light-curing for 10 s;
Xeno V	Bifunctional acrylates, acrylic acid,acid phosphoric functionized ester,acid acrylicate water, tertiary butanol, phosphine oxide initiator, stabilisator.	Application twice;
Batch 0803001397	pH<2	Agitation for 20 s;
		Air-drying for 5 s;
		Light-curing for 20 s;
i BOND	UDMA, 4META, glutaraldehyde, acetone, water, photo initiators, stabilizers	Application and agitation for 20 s;
Batch 010062	pH=2	Air-drying for 5–10 s;
		Light-curing for 20 s;
Vertise Flow	GPDM;	Air-drying of the cavity;
Batch 3172311	Prepolymerized filler, 1-μm barium glass filler, nano-sized colloidal silica, nano-sized Ytterbium fluoride;	Application of <0.5-mm layer;
	pH=1.9	Brushing for 15–20 s;
		Light-curing for 20 s;

Table 2 Manufacturer and content of the flowable composites

Flowable	Composition	Manufacturer
Gradia Direct LoFLO Batch 0804071 Shade A3.5	HDR prepolymerized filler 20 μm; Nano-silica filler 7 nm; Fluoro-alumino-silicate glass 1.7 μm; UDMA and multifunctional methacrylate resin;	GC, Tokyo, Japan
Tetric Evo Flow Batch L08447 A2	Bisphenol A glycidyl dimethacrylate (bis-GMA), UDMA, decandiol dimethacrylate (37.6 %)[a]; Barium glass Filler, ytterbium trifluoride, mixed oxide, highly dispersed silicon dioxide (41.1 %); Prepolymer (20.4 %); Additives, catalysts, stabilizers (0.9 %); Pigments (<0.1 %);	Ivoclar Vivadent, Schaan, Liechtenstein
Filtek Supreme XT Flowable Batch 70U 3913A3 Shade A3	Bis-GMA, TEGDMA, ethoxylated bisphenol A glycol dimethacrylate (bis-EMA); Dimethacrylate polymer; photoinitiatior; Silica nanofiller 75 nm, zirconia nanofiller 5–10 nm, bound zirconia/silica nanocluster 0.6–1.4 μm,	3M ESPE, St. Paul, MN, USA
X Flow Batch 0806002781 Shade B1	Strontium alumino sodium fluoro phosphate Silicate glass, di- and multifunctional acrylate and methacrylate resins, -decamethylene glycol (DGDMA), highlydispersed silicon dioxide, UV stabilizer, Ethyl-4-dimethylaminobenzoate, camphorquinone, Butylated hydroxytoluene (BHT), iron pigments, titanium dioxide, filler 61%[a]	Dentslpy Detrey Kostanz, Germany
Venus Flow Batch 010120 Shade A3	Bis-GMA, TEGDMA, low filler with the particle size of 0.7 μm Ba glass	Heraeus Kulzer, Hanau, Germany
Vertise Flow Batch 3172311 Shade A2	GPDM; Prepolymerized filler, 1-μm barium glass filler, nano-sized colloidal silica, nano-sized Ytterbium fluoride.	Kerr, Orange, CA, USA

[a] Percentage by weight

cylindrical diamond bur (FG315, Intensiv, Grancia, Switzerland) mounted on a high-speed handpiece. Cavities were approximately 2 mm in depth, 2 mm in the mesio-distal, and 2 mm in the bucco-lingual direction. A new bur was used after five preparations. Teeth were then randomly divided into six groups ($n=12$), and cavities were restored using the same materials (Tables 1 and 2) and the same curing device as in shear bond strength testing. The specimen preparation procedures were carried out by the same operator (MM). After 24-h storage in 100 % humidity and 37 °C temperature, restored teeth were covered with two layers of fast-setting nail varnish applied up to within 1 mm of the bonded interface. Before their dehydration, teeth were immersed into 50 wt% silver nitrate solution ($AgNO_3$), and left undisturbed for 24 h, in the dark. The silver-impregnated teeth were thoroughly washed with distilled water and placed into a photo-developing solution for 8 h (Dental X-Ray Developer, Kodak Co, Rochester, NY, USA). The teeth were again abundantly rinsed with water. Within each group, two specimens were kept for scanning electron microscopy (SEM), while the remaining 10 were each one cut into two halves with the low-speed diamond saw under water cooling (Isomet, Buehler Ltd, Lake Bluff, IL, USA). The obtained sections were kept moist until the microscopic observations took place. A digital image of each section was acquired and recorded by a different operator (AV) using a photo-camera (D80, Nikon Co, Tokyo, Japan) equipped with a Medical-Nikkor lens (Nikon Co, Tokyo, Japan) at ×2 magnification [28–30]. In order to quantify the microleakage on the digital image of each tooth half, blinded measurements of the length of the stained tooth-composite interface were carried out, by the same operator who prepared the specimens, in pixels and related to the total interfacial length, also measured in pixels, using the image analysis software Digimizer V.3.0.0 (MedCalc Software, Mariakerke, Belgium). The percentage of microleakage was thus calculated:

$$(Length\ of\ stained\ interface / Total\ interfacial\ length) \times 100.$$

The calculations were performed on both sides of each slice, but only the side exhibiting the higher leakage was considered in the statistical analysis.

Scanning electron microscopy

Two randomly selected specimens per group were processed for SEM observations of interfacial leakage. A 2-mm-thick slab containing the interface of interest was sectioned from each restored tooth and polished with wet SiC papers of increasingly finer grit (600, 1,000, 1,200, Buehler, Lake Bluff, IL, USA). The interface was brought into relief by etching with 32 % silica-free phosphoric acid gel (Uni-Etch, Bisco, Schaumburg, IL), followed by brief deproteinization with a 2 % sodium hypochlorite solution for 60 s. After rinsing with de-ionized water, specimens were dehydrated in an ascending series of aqueous ethanol solutions to absolute ethanol, and dried using hexamethyldisilazine (HMDS, Carlo Erba, Rodano, Italy). Specimens were then mounted on aluminum stubs, coated with a 15–20-nm-thick layer of gold by means of the SC7620 Sputter Coater device (Polaron Range, Quorum Technologies, England), and inspected by a scanning electron microscope (JSM-6060LV, JEOL, Tokyo, Japan) in low-vacuum mode at ×500 and ×1,000 magnifications at an average voltage of 19 kV. The observations were carried out by a single operator (FP).

Statistical analysis

Shear bond strength data

As the distribution of the pooled data from dentin and enamel was not normal, it precluded the use of two-way analysis of variance, assessing the influence on bond strength of adhesive, substrate, and between-factor interaction. Therefore, separate analyses were applied to dentin and enamel data. The Levene's test revealed that homogeneity of group variances was violated for dentin data. Therefore, the Kruskal–Wallis analysis of variance was applied, followed by the Dunn's multiple range test for multiple comparisons. Having checked that enamel bond strength data had normal distribution and homogeneous group variances, the one-way analysis of variance (ANOVA) was applied, followed by the Tukey test for post hoc comparisons. A series of independent samples *t* test was performed in order to assess whether for each adhesive significantly different levels of bond strength were achieved on the two tested substrates.

Microleakage

The microleakage data distribution was not normal according to the Kolmogorov–Smirnov test. Therefore, the Kruskal–Wallis ANOVA was applied, followed by the Dunn's multiple range test for post hoc comparisons. In all the analyses, the level of significance was preset at $\alpha=0.05$.

Results

Descriptive statistics of dentin and enamel shear bond strength and microleakage are reported in Tables 3, 4, and 5, respectively, that also indicate the statistical significance of between-group differences. For dentin bond strength and microleakage data (Tables 3 and 5, respectively) median values and interquartile ranges, were reported, as the use of these non-parametric statistics is indicated with data sets that do not have normal distribution or homogeneous group variances [31]. The mean shear bond strength on dentin and enamel is graphically shown in Fig. 2.

Shear bond strength

The adhesive system was found to be a significant factor for shear bond strength to dentin and enamel ($p<0.001$). On dentin, Vertise Flow recorded the lowest bond strength values, although they were still statistically comparable to those measured by G-Bond and iBond (Table 3). Also on the enamel substrate, Vertise Flow produced the lowest shear bond strengths, although these values were statistically comparable to those of AdheSE One and iBond (Table 4). All the tested materials achieved statistically similar adhesion levels in enamel and dentin except for AdheSE One, which gave significantly higher bond strength on dentin.

Microleakage

Adhesive system turned out to be a significant factor also for sealing ($p<0.001$). Vertise Flow exhibited the lowest percentage of infiltrated interface. iBond and EasyBond showed a significantly greater extent of stained interface than Vertise Flow (Table 5).

SEM evaluation

All the adhesive materials appeared well adapted onto the substrate. Nevertheless, no distinct hybrid layer could be seen in any of the observed specimens (Fig. 3). Silver nitrate deposits were detected in all specimens (Fig. 3a, b, d, e, f) with the exception of the AdheSE One (Fig. 3c). The tracer infiltration into dentin was documented in adhesive layers and dentinal tubules of specimens bonded with Vertise Flow, Xeno V, and EasyBond (Fig. 3a, d, f). In iBOND specimens, the dye mostly collected within the adhesive layer, while few dentinal tubules appeared to be penetrated (Fig. 3e). In G-Bond specimens, dentinal tubules were free of dye infiltration and silver nitrate was only detected within the adhesive layer (Fig. 3b).

Table 3 Descriptive statistics of shear bond strengths on dentin

Adhesive	*N*	Mean (MPa)	Standard deviation	Median (MPa)	25–75 %	Significance $p<0.05$
EasyBond	10	12.2	3.6	12.2	9–14.2	A
AdheSE One	10	11.3	5.7	10.9	6.1–17.1	A
Xeno V	10	10.7	4.7	11.1	7.2–14.6	A
G-Bond	10	6.9	3.2	7	4.6–9.1	AB
iBond	10	5.8	1.2	5.7	4.7–6.7	AB
Vertise Flow	10	3.4	1.6	3.6	2.8–4.1	B

Different letters label statistically significant differences according to the post hoc tests ($p<0.05$)

Discussion

Based on the findings of the present study, the formulated null hypothesis has to be rejected, as the results obtained with the self-adhering composite differ significantly from the other adhesive materials in shear bond strength to dentin and enamel, as well as in interfacial microleakage.

Vertise Flow is a new self-adhering, flowable composite resin, whose bonding mechanism relies on the adhesive monomer glycerol phosphate dimethacrylate (GPDM). Specifically, the phosphate group of GPDM is responsible for acid etching. The dimethacrylate functional groups are involved in cross-linking reactions with other methacrylate monomers, thus providing mechanical strength to the adhesive material [Kerr Technical Bulletin]. Based on the pH declared form the manufacturer (1.9), Vertise Flow can be expected to interact with dental substrate similarly to a mild self-etch adhesive.

In the present study, Vertise Flow recorded the lowest values of bond strength to dentin and enamel. Among the possible reasons for such a result, the wettability of the material should be considered. Proper wettability of an adhesive material onto a substrate enables a close adhesive–substrate interaction [32, 33]. In comparison with the other tested adhesive systems, the self-adhering flowable composite Vertise Flow is more viscous, does not contain solvent, and has lower wettability. These properties could represent a drawback for the material's ability to wet self-etched collagen fibrils. As theoretically the interaction of the acidic composite matrix could be enhanced by active application, the manufacturer recommends brushing the first layer of material onto the entire cavity surface for 20 s. Based on this study's findings, active application did not enhance the bond strength of Vertise Flow to levels comparable to those of the other adhesives tested.

It is worth mentioning that although the authors are aware that manufacturer's reports may possibly be biased, they could not avoid referring to such sources as the only currently available information on these newly launched system.

Although the shear bond strength of Vertise Flow was the lowest measured in this study, the microleakage evaluation of the self-adhering flowable composite was the lowest of the tested materials. Hygroscopic expansion and relatively low polymerization shrinkage might be advocated as possible reasons for such satisfactory performance. Concerning the hygroscopic expansion, it is known that acid resins absorb more water than neutral resins [34, 35]. Among the several factors that have been reported to affect the amount of water sorption, the chemical nature of matrix monomers and matrix/filler content have to be considered [36]. It has been observed that in adhesive monomers with polymerizable and functional groups linked by spacer groups, the molecular design influences the hygroscopic expansion of the resulting polymer [4]. A hygroscopic expansion higher than that of urethane dimethacrylate (UDMA)-based polymers has recently been reported for Vertise Flow and related to the hydrophilic acid phosphate group and the spacer group in the adhesive monomer GPDM [37]. It can be speculated that, by compensating for polymerization shrinkage [38], hygroscopic expansion of Vertise Flow might have contributed to the better sealing performances showed by the material in the present study. The satisfactory sealing performance of Vertise Flow can also be accounted for the uniqueness of the dynamics in its adhesion/polymerization process. During "traditional" resin-based restorative procedure, an adhesive solution and a restorative composite are used in sequence, thus the curing of the restorative material occurs after bonding is accomplished. Polymerization stress

Table 4 Descriptive statistics of shear bond strengths on enamel

Adhesive	*N*	Mean (MPa)	Standard deviation	Significance $p<0.05$
EasyBond	10	12.1	5	A
Xeno V	10	10.4	4	AB
G-Bond	10	7.7	1.9	ABC
AdheSE One	10	6	4	BCD
iBond	10	5	1.8	CD
Vertise Flow	10	2.6	2.6	D

Different letters label statistically significant differences according to the post hoc tests ($p<0.05$)

Table 5 Descriptive statistics of marginal sealing

Adhesive	*N*	Mean (%)	Standard deviation	Median (%)	25–75 %	Significance $p<0.05$
Vertise Flow	10	18.4	9.4	15.5	13.1–22.9	A
G-Bond	10	23.6	21.6	19.1	0–47.1	AB
AdheSE One	10	29.4	8.5	29.9	24–35.9	ABC
Xeno V	10	32.6	4	32.3	31.3–35	ABC
iBond	10	59.7	35.3	76.7	46–82.2	BC
EasyBond	10	73.4	26	68.1	58.7–100	C

Different letters label statistically significant differences according to the post hoc tests ($p<0.05$)

of the restorative composite may act as a competitor of the bond just established by the adhesive with the dental substrate [39]. In the case of the adhesive-free composite Vertise Flow, bonding and polymerization process of the resin occur simultaneously. As the viscous-elastic flow can occur simultaneously with the bonding process, it can be speculated that the competition between bonding and curing stress is reduced, thus favoring marginal adaptation of the material.

In the present study, the bond strength data did not correspond with microleakage observations. This difference was distinctive for Vertise Flow and EasyBond that expressed opposite results in these experimental parameters. The discrepancies found in the present study are in line with previous investigations, where no association was observed between bond strength and interfacial microleakage [40–44]. Furthermore, it has been suggested that comparing the results of these two in vitro methodologies can lead to misleading interpretations [40–42].

It might be argued that only simplified materials were compared with the new system in the present study. Recent research advancements have mainly aimed at reducing technique sensitivity and chair time. From this perspective, the elimination of a bonding step can be considered as a breakthrough. Therefore, it seemed reasonable to consider the all-in-one systems followed by the application of a flowable resin as the comparative product group for this newly formulated material.

Single-step adhesives tested in the present study differed significantly in their bonding potential (Table 3, Fig. 2).

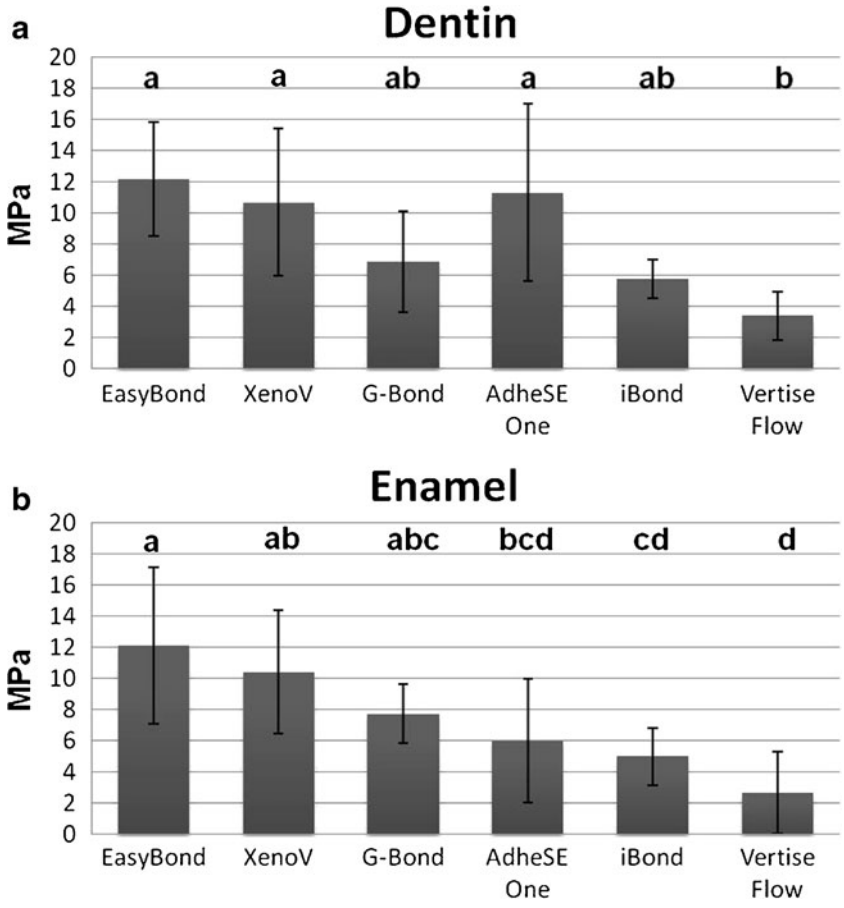

Fig. 2 Bar charts of mean shear bond strength to dentin (**a**) and enamel (**b**). *Error bars* represent standard deviations. Different *capital letters* label statistically significant differences according to the post hoc tests ($p<0.05$)

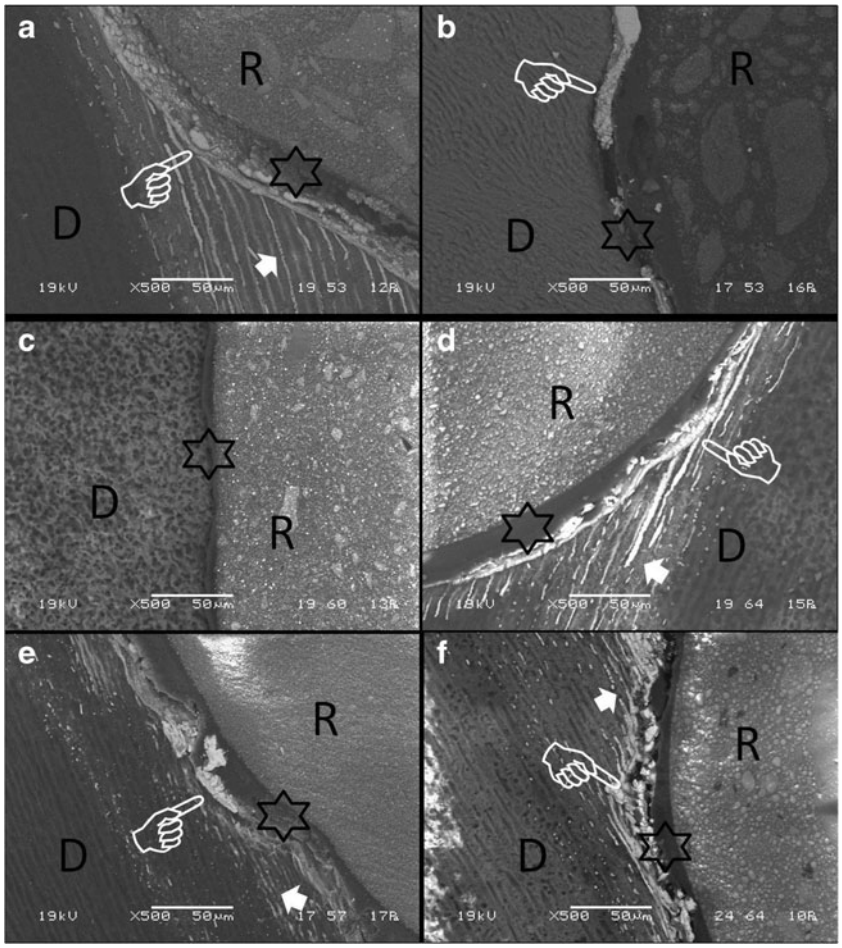

Fig. 3 Scanning electron microscopic images of silver nitrate penetration (*pointer*) at the dentin adhesive material interface (*star*) in class I cavities (magnification ×500, bar 10 μm). The *arrowheads* point out silver nitrate-impregnated dentinal tubules. **a** Vertise Flow, **b** G-Bond, **c** AdheSE One, **d** Xeno V, **e** iBOND, and **f** Adper EasyBond; *D* dentin, *R* composite resin

Such finding is in agreement with previous studies and could be related to the heterogeneity in chemical composition of the adhesive solutions [45–49]. In particular, solvent is an important component of all-in-one adhesives. Besides, keeping adhesive in a homogenous solution, solvent enhances the penetration of hydrophilic functional monomers into the dental substrates [3]. Indeed the tested all-in-one adhesives differed for the type of solvent (Table 1**)**. EasyBond and Xeno V, the two adhesives that in the present study achieved the highest bond strength to dentin and enamel, contain alcohols as a solvent. This finding is in line with the results of a previous investigation that reported a higher bonding potential for all-in-one adhesives containing alcohol [50]. Conversely, a distinctive feature of EasyBond is the presence of 2-hydroxyethyl methacrylate (HEMA). HEMA is a small-dimension, water-soluble methacrylate monomer that enhances the wetting properties of the adhesive solution. Additionally, this monomer prevents phase-separation reactions, by promoting the miscibility of hydrophilic and hydrophobic components of the adhesive [6, 51]. Hence, reduced amount of water droplets within hybrid layers were documented for HEMA-rich adhesives [8]. Furthermore, it was pointed out that inclusion of HEMA in the composition of all-in-one adhesives contributed to enhanced 24-h bond strength. This advantage is offset by the relative higher permeability of HEMA to water [52]. This may explain the greater extent of silver nitrate penetration that was observed in EasyBond specimens (Fig. 3f), despite the high bond strength developed on dentin and enamel. The hydrophilicity of HEMA has also been reported to expose the bond of HEMA-rich adhesives to water degradation with time [53]. However, no inference on bond stability can be made in the present study that only assessed the 24-h bond strength.

The all-in-one adhesives that had the lowest bond strength to dentin, iBond, and G-Bond contain acetone and water as solvents and do not include HEMA. In previous research, the lack of HEMA was suggested as a possible explanation for the relatively weak adhesion [54].

Shear bond strength test method was selected because the microtensile is a very sensitive technique and, when materials or substrates with relatively low bond strength values are tested, specimens tend to fail prematurely during preparation [55]. It was specifically pointed out that a high frequency of premature failures was recorded when testing microtensile bond strength of all-in-one adhesives to enamel [5]. As the present study was aimed at assessing bonding potential of simplified systems to dentin as well as to

enamel, authors considered it reasonable to minimize the stress applied to specimens during their preparation and to use the shear method, where the only pre-stress prior to testing is the removal of the mold. Besides technique sensitivity, shear was preferred over microtensile testing as it has been demonstrated that microtensile bond strength measurements are relevantly affected by the mechanical properties of the overlaid restorative composite that in our protocol had to differ among experimental groups [56, 57].

Regarding the clinical relevance of bond strength tests, this was shown to be related to the time frame of the experiment, rather than to the test method itself [58]. Moreover, according to a recent review, owing to the simplicity of the method, shear bond strength test remains popular for evaluating the adhesion of dental materials to tooth substrates [27].

Based on SEM examinations all the tested systems exhibited a rather superficial interaction with the dental substrates and no distinct hybrid layer was evident at the used magnifications. This microscopic picture is typical of all-in-one adhesives [50]. Silver nitrate depositions in the adhesive layers of Vertise Flow, G-Bond, Xeno V, iBond, and Easy Bond identified areas of residual water or other solvent that created a defective seal (Fig. 3a, b, d, e, f).

The low-vacuum mode was used for SEM evaluation in this study. This SEM modality, in contrast to traditional SEM observation in high-vacuum mode, permits the detection of interfacial staining such as silver nitrate and may prevent sample artifacts such as failure at the tooth-restoration interface.

To conclude, discordant results emerged for the new material in the present study. When compared with five combinations of self-adhesive systems and the proprietary flowable composites, the bond strength values recorded by Vertise Flow to dentin and enamel were the lowest, while the sealing ability was superior to the other materials. Ongoing in vivo studies are expected to clarify whether the sealing ability and bond strength of Vertise Flow self-adhering flowable composite resin will yield clinical success.

Conclusion

Vertise Flow, when used to restore class I, provided satisfactory sealing ability despite the relatively low bond strength recorded on enamel and dentin. The outcome of microleakage suggests that adequate marginal seal can expectedly be achieved in the clinical setting, while the clinical acceptability of Vertise Flow retention should be verified with in vivo trials.

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Conflict of interest The authors declare that they have no conflict of interest.

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