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ORIGINAL ARTICLE

Evaluation of a self-adhering flowable composite in terms of micro-shear bond strength and microleakage

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Abstract

Purpose. The objective of this study was to evaluate dentin bond strength and microleakage of a self-adhering flowable resin with or without adhesive resin. ***Materials and methods.*** Dentin pieces were randomly assigned to three groups ($n = 10$): Group OR (Optibond+Revolution Formula 2 Flow); Group VF (Vertise Flow); and Group OVF (Optibond+Vertise Flow). Then specimens were stressed in shear at a cross-head speed of 1 mm/min until the bond failed. Moreover, microleakage was determined by restoring Class V cavities. A statistical analyses was carried out using Mann-Whitney U and Kruskal-Wallis tests ($p < 0.05$). ***Results.*** Group OVF specimens produced the highest mean shear bond strength. In addition, the results showed that the marginal seal in group VF was similar to that in group OR. ***Conclusion.*** Self-adhering flowable composite resin combined with adhesive resin provided stronger dentin bond strength and a better marginal seal than when it was used individually. VF is a useful material with an acceptable bond strength and marginal seal.

Key Words: *self-adhering, flowable composite, micro-shear, microleakage*

Introduction

The first generation of flowable composites was introduced in 1996 to restore Class V lesions [1]. The flowable composites can be easily inserted into small cavities and are expected to exhibit better adaptation to the internal cavity wall compared to the conventional restorative composites, which are more viscous [2–5]. The conventional wisdom is that the use of flowable composites would result in less microleakage, internal voids and post-operative sensitivity [6]. Today, there are wide-ranging applications for them, such as small Class III and Class V cavities, pits and fissures, a base for composite restorations, amalgam margin repairs, enamel defects and incisal edge repairs in anterior sites. In addition, recently they are being used as filled adhesives [1,7].

The flowable composite materials are a modification of restorative resin composites, so they tend to contain a lower filler content (weight: 60–70%; volume: 46–65%) than their hybrid analogs (weight: 70–80%; volume: 60–75%) [5,8]. Reduced filler loading of flowable composites leads to enhanced flow and reduced elastic modulus [4,9]. Thus, this material may act as a stress-breaker [10,11]. Nevertheless, they demonstrate higher polymerization shrinkage and have inferior mechanical properties because of the lower filler content. Stress from shrinkage results in cracked enamel rods, marginal gaps and open margins. Microleakage around resin composite restorations occurs because of these gaps [3].

The latest news in adhesive dentistry is the development of a self-adhering flowable composite resin. It is a direct composite resin restorative material that has an adhesive resin incorporated into the flowable composite resin. Self-adhering flowable composite resin is based on bonding technology that utilizes glycerophosphate dimethacrylate (GPDM) [12], a functional monomer, to etch enamel and dentin. It also contains hydroxy-ethyl methacrylate (HEMA), another functional monomer, most commonly used in dental adhesives to enhance wetting and resin penetration in dentin. This resin bonds in two ways: primarily through the chemical bond between the phosphate functional groups of a GPDM monomer and calcium ions of the tooth and, secondarily, through a micromechanical

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bond as a result of an inter-penetrating network formed between the polymerized monomers of self-adhering flowable composite resin and collagen fibers (as well as the smear layer) of dentin [13]. However, very few studies have been conducted to test self-adhering flowable composite resin. Two studies showed that self-adhering flowable composite resin has the highest hygroscopic dimensional expansion [14] and water sorption [15] after 150 days of water immersion when compared to conventional flowable and composite resins.

Self-adhering flowable composite resin not only has low microleakage but also makes a strong bond to dentin. It is also advocated that it creates a tenacious bond to tooth structure and protects against microleakage. Moreover, the bond strength is comparable to that of other self-etch adhesives for both dentin and enamel [13]. Therefore, further work is required to evaluate the performance of this resin. The purpose of this study was to evaluate the micro-shear bond strength and amount of microleakage between dentin and a self-adhering flowable resin with or without adhesive resin. The null hypothesis was that self-adhering flowable composite resin has improved bond strength to dentin and a reduced degree of microleakage.

Materials and methods

Micro-shear test evaluation

Thirty caries-free, freshly extracted human third molars were selected and stored in physiological saline solution at 4°C for less than 1 month. Teeth were then sectioned ~ 1.0 mm thick and parallel to the occlusal surface from the upper middle coronal region using a water-cooled diamond saw (Isomet, Buehler, Lake Bluff, IL). The smear layer on the occlusal dentin was standardized using 600 grit silicon carbide (SiC) paper. The materials used for each group are presented in Table I. Slices were randomly assigned to the following three groups ($n = 10$), according to the treatments applied and performed on each:

- Group OR (Optibond All-In-One/Revolution Formula 2): An all-in-one, self-etch adhesive resin (Optibond All-In-One) was applied to the tooth slice according to the manufacturer's instructions. Prior to irradiation of adhesive resin, a cylinder of tygon microbore tube (Tygon, Norton Performance Plastic Co, Cleveland, OH) with an internal diameter of 0.7 mm and a height of 1 mm was cut and mounted on the tooth to restrict the bonding area. After the photo-curing of the bonding resin with a halogen light curing unit (Hilux, Benlioglu Dental, Ankara, Turkey), the cylinder was filled with flowable composite (Revolution Formula 2) and irradiated according to the manufacturer's instructions. To ensure that a minimal output intensity of 600 mW/cm^2 was employed for the experiments, the output of the unit was periodically checked.
- Group VF (Vertise Flow): A cylindrical tygon microbore tube was placed on the dentin slice without applying adhesive resin and immobilized using cotton pliers. Then, self-adhering flowable composite resin (Vertise Flow) was put into the cylinder with the dispensing tip and rubbed with a blunt probe for 15 s to obtain a thin layer. Excess material was removed with a brush and light cured for 20 s. The cylinder was filled with VF again and then irradiated according to the manufacturer's instructions.
- Group OVF (Optibond All-In-One/VF): Adhesive resin was applied and a cylindrical tygon microbore tube was bonded to the dentin slices as described in Group OR. Then, VF was put into a cylinder with the dispensing tip and rubbed with a blunt probe for 15 s to obtain a thin layer. Excess material was removed with a brush and light cured for 20 s. The cylinder was then filled with VF again and irradiated according to the manufacturer's instructions.

In this way, very small cylinders of resin, ~ 0.7 mm in diameter and 1 mm in height, were bonded to the surface. The specimens were left in water at room temperature for 1 h prior to the removal of the tygon tube and then they were stored in water at 37°C for 24 h. After the storage period, the tygon tube was removed to reveal a composite cylinder with a cross-sectional area of 0.5 ± 0.02 mm^2. The specimens were checked under a stereomicroscope (SMZ 800, Nikon, Tokyo, Japan) at a magnification of 20 × prior to micro-shear testing. Specimens which showed a detectable interfacial defect were excluded.

The specimens were attached to the testing device (Bencor-Multi-T, Danville Engineering Co., San Ramon, CA) with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA) and placed under micro-shear stress until failure in a universal testing machine (LF Plus, LLOYD Instrument, Ametek Inc., Leicester, UK) at a crosshead speed of 1.0 mm/min. A thin wire (diameter 0.2 mm) was looped around the resin cylinder, making contact with half of the cylinder base and held flush against the resin/tooth interface. Ten specimens in each group were tested. Maximum loads at bond failure were recorded in Newtons (N) and bond strengths were then calculated in mega pascals (MPa). Mean bond strengths were statistically evaluated using Kruskall-Wallis and Mann-Whitney U-tests ($\alpha = 0.05$).

Microleakage test

Fifteen human mandibular premolars extracted for routine orthodontic treatment. Teeth had no caries, crack, fracture or restoration. After extraction they

Table I. Materials used in microshear bond strength and microleakage tests.

Materials/ Manufacturers	Description	General composition	Manufacturers' instruction for use
Optibond All-in-one (Kerr Corporation, Orange, CA)	All-in-one self-etch adhesive	GPDM, Co-monomers including mono- and di-functional methacrylate monomers, water, acetone and ethanol, CQ, nano-sized fillers, including discrete nano-silica sodium hexafluorosilicate	Apply 20 s with scrubbing motion, apply second application for 20 s with scrubbing motion, gently air dry and light cure for 10 s
Revolution Formula2 (Kerr Corporation, Orange, CA)	Flowable light-cure, hybrid composite resin	Uncured methacrylate ester monomers (38–53%), inert mineral fillers, activators and stabilizers	Dispense a layer of Revolution Formula 2 directly onto the preparation along the axial and gingival margins. Use a brushing action to allow the material to flow into the marginal interfaces and recesses of the preparation. Incremental layers of Revolution Formula 2 should be no more than 2 mm. Light cure each increment for 20 s.
Vertise Flow (VF) (Kerr Corporation, Orange, CA)	Self-adhering flowable composite	GPDM and methacrylateco-monomers Pre-polymerized filler, barium glass, nano-sized colloidal silica, nano-sized ytterbium fluoride	Dispense Vertise™ Flow onto preparation with provided dispensing tip. Use provided brush to apply Vertise Flow to the entire cavity wall with moderate pressure for 15–20 s to obtain a thin layer (<0.5 mm). Light cure for 20 s. After lining the cavity wall build the restoration with more Vertise™ Flow in increments of 2 mm or less. Light cure each increment for 20 s.

were stored in a physiological saline solution at 4°C for less than 1 month. A cervical cavity of 4.0 mm mesiodistal length, 2.0 mm occlusogingival height and 1.5 mm depth was prepared at the cemento–enamel junction on both buccal and lingual surfaces of each tooth with a diamond bur in a water-cooled high-speed hand piece. Burs were replaced after all three preparations. Tooth structure was cleaned with distilled water and dried before the following procedure. The samples were randomly and equally divided into three groups of five samples each. All samples were restored according to the grouping: OR, VF and OVF. Following bonding and filling procedures recommended by the manufacturers (Table I). Finishing and polishing procedures were performed immediately after filling.

Teeth were stored in water at 37°C for 24 h. After this storage period, all *in vitro* specimens were thermocycled between 5 ± 2°C and 55 ± 2°C for 30 s at each temperature level for a total of 1000 cycles (Nova, Nova Ticaret, Konya, Turkey). For evaluation of leakage by dye penetration, the apices were sealed with autopolymerized acrylic resin and the teeth were painted with two coats of nail varnish so that only the restorations and a 1 mm band of the surrounding surface were exposed. They were then immersed in 0.5% basic fuchsine dye solution at 37°C for 24 h. After removal from dye, the teeth were then rinsed thoroughly and cleaned with a bristle brush to remove surface dye. Then, each tooth was sectioned longitudinally across the center of the restoration, with a low-speed diamond saw under water cooling. The cavity walls (including both occlusal and gingival margins) of each prepared segment were observed under the stereomicroscope (SMZ 800, Nikon, Tokyo, Japan) at 40 × magnification. Dye penetration was evaluated along the occlusal and gingival walls. The extent of leakage was assessed, using the following scores [16].

- Score 0: No dye penetration;
- Score 1: Dye penetration within $^1/_2$ of occlusal or gingival wall;
- Score 2: Dye penetration extending to the end of occlusal or gingival walls;
- Score 3: Dye penetration through the gingival or occlusal wall to 1/3 of axial wall;
- Score 4: Dye penetration through the gingival or occlusal wall to 2/3 of axial wall; and
- Score 5: Dye penetration throughout the axial wall.

The sections were observed by two different operators under a stereomicroscope. If the two investigators disagreed on the score assigned to a certain specimen, they re-evaluated the sections and reached a consensual score. Figure 1 demonstrates individual examples of scoring. Statistical analyses were carried out by using Kruskal-Wallis and Mann-Whitney U-tests ($p < 0.05$).

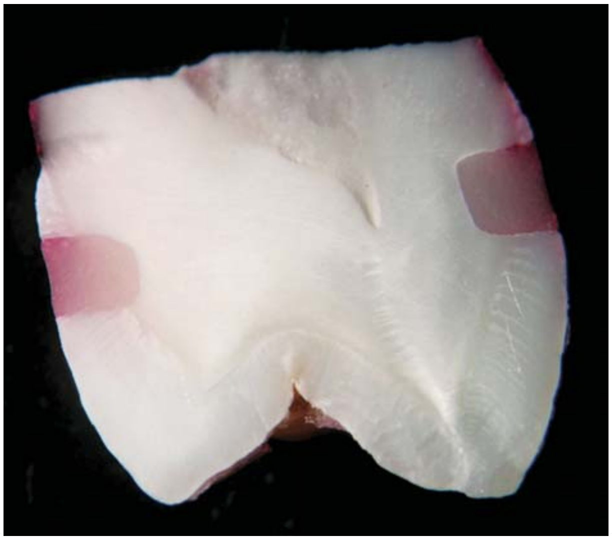

Figure 1. Microleakage evaluation of occlusal and gingival margin. Scores are 1 for the gingival margins and 0 for the occlusal margins.

Results

The mean micro-shear bond strengths for each group of specimens are presented in Table II. A statistically significant difference in micro-shear bond strength was found among groups ($p < 0.05$). In addition, the highest mean force value was observed in group OVF specimens and this was followed by group OR specimens. Furthermore, there was a statistically significant difference in bond strength between group OR and group VF ($p = 0.019$), group OR and group OVF ($p = 0.023$) and group VF and group OVF ($p = 0.003$).

Statistical results of the Kruskal-Wallis test for microleakage scores of the groups are shown in Table III (KW = 6.43 for gingival margin and KW = 1.66 for occlusal margin). According to the Mann-Whitney U-test, there was a significant difference in gingival margin microleakage between groups OVF and VF ($p < 0.05$). However, the microleakage analysis revealed no significant differences between other groups in the occlusal and gingival margins ($p > 0.05$). The lowest mean microleakage score was observed in group OVF specimens in the gingival margin.

Discussion

The most frequently used tests are bond strength tests to screen adhesives. The micro-shear bond strength was chosen because it has many advantages including small bonding surface area, easier sample preparation, use of lesser amount of material and fewer teeth and suitable for testing of enamel bonding. Furthermore, when evaluating this test method for bonding substrate, similar regions on the axial surfaces of enamel and dentin slices were bonded for all specimens to eliminate interference from substrate variations [17].

Table II. Mean shear bond strength (MPa) and SD of each group.

Groups	*n*	*M*	SD
OR	10	29.33^{a}	5.19
VF	10	23.70^{b}	5.28
OVF	10	35.08^{c}	7.00

KW = 11.93 and groups with different superscripted letters significantly different ($p < 0.05$).

Table III. Dye penetration ratios of three groups along the cavity wall.

		Gingival margin		Occlusal margin	
Groups	*n*	*M*	SD	*M*	SD
OR	20	1.65^{ab}	2.08	1.35^{ab}	1.72
VF	20	1.90^{a}	1.33	1.30^{ab}	0.92
OVF	20	0.85^{b}	0.67	0.95^{b}	0.6

Groups with different superscripted letters significantly different ($p < 0.05$).

This study found that a self-adhering flowable composite resin with adhesive resin, OVF, significantly increased dentin bond strength and reduced microleakage scores. Therefore, the null hypothesis was accepted. *In vitro* self-adhering resin bonding and microleakage performance are being described for the first time in this study. In the existing literature, there are only two studies of self-adhering flowable composite resin and they evaluated hygroscopic dimensional changes and diffusion and concurrent solubility of self-adhering and new resin–matrix composites during water sorption/desorption cycles [14,15]. Therefore, the results of the present study were compared with the results reported by the manufacturer. Contrary to the manufacturer's report, VF demonstrated the lowest bond strength to dentin. Diametric and volumetric changes of flowable composite resin may significantly affect its bonding performance due to the hydrophilic acidic phosphate group and the short spacer group in the adhesive monomer GPDM. Bui et al. [18] examined the shear bond strength of VF to dentin along with other self-etch adhesive systems. They reported that the shear bond strength of VF (25.1 MPa) was lower than that of Optibond All-In-One (27 MPa); however, this difference was not statistically significant. In this study, VF demonstrated a lower bond strength than OR. This result agreed with that of Bui et al.

Although VF incorporates adhesive technology found in Optibond products to create bonds to tooth structure, VF and OR groups exhibited different bond strengths. Adding other fillers may reduce the bond strength of VF. Miyazaki et al. [19] suggested that filler in the adhesive resin might decrease the wetting of the dentin surface because of the higher viscosity of filled resins. This would decrease the penetration of monomers, thus reducing the shear bond strength.

A clinically important limit for dentin bonding is not yet known. The bond strength of ~ 20 MPa appears to be acceptable clinically for enamel bonding. The theoretical limit for dentin bonding strength may actually be higher than that of enamel because dentin is more resistant to shear fracture [20]. However, a systematic review [21] revealed that there were significant differences in the pooled mean bond strength, as the weighted bond strength means per adhesive class have the following ranges: 31 MPa for 3-step etch&rinse adhesives, 29 MPa for 2-step self-etch adhesives, 26 MPa for 2-step etch&rinse adhesives and 20 MPa for 1-step self-etch adhesives. In the present study, VF had a 23.7 MPa bond strength. This result shows that the micro-shear bond strength of VF can be compared favorably to other 1-step self-etch adhesive systems.

Ferrari [13] reported that VF exhibited less microleakage than commercially available self-etch adhesive/flowable composite systems for Class I restorations. In the present study, the microleakage analysis revealed that there was no statistically significant difference between groups OR and VF. This result was not in accordance with what Ferrari reported.

Compared to the conventional shear bond strength, the specimen size in micro-shear bond strength test can be greatly reduced in this way; the micro-shear test is a good method to examine adhesion of small areas [22–24]. However, it has also been demonstrated that microtensile and micro-shear bond strength test methods present similar results on bond strengths of self-etch adhesives to dentin [24]. Nevertheless, the micro-shear bond strength test is less demanding in terms of specimen production and bonding areas can be much better controlled with the use of a microbore tube [25]. In this study, it was important to control the test area used. Therefore, the micro-shear bond strength test was performed to measure the bond strength of groups tested.

Self-adhering flowable composite resin was selected for testing because it is a new material which eliminates the need for a separate bonding application. It not only reduces clinical application time but also lessens the complexity of the technique, the risk of making errors during application and helps to reduce post-operative sensitivity for the clinician. Therefore, this will greatly simplify the direct restorative process [12]. Furthermore, it was advocated that using a bonding agent with Vertise Flow will enhance the bond strengths to both dentin and enamel [13]. Thus, Vertise Flow was also tested with adhesive resin. On the other hand, a limitation of this study is that only one adhesive resin was evaluated and scanning electron microscopy (SEM) was not conducted to evaluate the bonding mechanism and characterize the failure patterns of the bonding surface.

Conclusions

Within the limitation of this study, self-adhering flowable composite resin used with an adhesive system provided stronger bond strength and a better marginal seal than individual usage of self-adhering flowable composite resin. The clinicians can reliably use self-adhering flowable composite resin with an adhesive resin in Class V cavities.

***Declaration of Interest*:** The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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