

Research Article

Comparative Analysis of the Shear Bond Strength of Flowable Self-Adhering Resin-Composites Adhesive to Dentine with a Conventional Adhesive

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Introduction

The development of the restorative materials that show adhesion with tooth structure is an interesting area of research. The lack of adherence of restorative material to the tooth structure may lead to several problems such as secondary caries, marginal leakage (infiltration of fluids, bacteria and ions into the gaps between restoration and cavity walls) [1] and post-operative sensitivity. Adhesion to dentin is more difficult as compared with enamel. This lack of adhesion to dentin is attributed to its organic composition and fluid that is present in the dentinal tubules. Currently, flowable restorative composites (nano-fillers added to low viscosity monomers) are ideal for many reasons such as pit-and-fissure sealant, small Class III and V restorations, enamel flaws and incisal edge repairs [2]. Flowable composites generally result in higher shrinkage and lower elastic modulus (due to lesser filler content) than conventional non-flowable composites [3]. The interfacial shrinkage stresses between dental tissue and composites indicate the potential to increase the possibility of marginal leakage. Three-step conventional adhesive systems (etch + primer + bond) may result in high resin to dentin adhesion, however, factors such as excessive etching may influence the performance due to the possibility that the resin monomers may not be able to penetrate into the open dentinal tubules. In addition, there is a possibility of collagen collapse on air drying after etching [4]. A huge number of new adhesive systems has emerged (universal

Abstract

This study aimed to measure and compare the Shear Bond Strength (SBS) of a self-adhering flowable resin composite to dentin with a conventional flowable composite, and evaluate the various modes of failure. Thirty (n=30) human premolar teeth were randomly allocated to two groups (Group I: Vertise Flow; Group II: Filtek Ultimate) of 15 specimens each, and SBS was measured for specimens from each group by applying shear load using a Universal Testing Machine (UTM). Modes of failure were observed with an optical microscope. An independent samples T test was performed to test the Levene's assumption of homogeneity of variance across both groups, with the critical value set at 0.05. The results revealed, that the self-adhering flowable composite (Group I: Vertise™ Flow) resulted in lower shear bond strength as compared with the conventional flowable resin composite (Group II: Filtek Ultimate). Moreover, group I predominantly exhibited non-cohesive failure which reflects a poor bonding to dentin. On the other hand, Group II showed mixed failure for most of the samples, which demonstrate strong adhesive bonding. Therefore, it could be concluded that the self-adhering flowable resin composite (Group I) results in lower SBS to dentin as compare with conventional flowable composite.

Keywords: Shear bond strength; Dental adhesives; Flowable resin composites; Self-adhering composites

or multi-mode) and different operative techniques for each group of materials have been introduced [5-7]. As a result, it becomes a challenge for the dentists to choose an adhesive system for routine use at the clinic. Therefore, it is essential for the practitioners to be aware of the physical and chemical characteristics of these materials and its association with structures to select and use them correctly.

The bonding between the restorative material and tooth substrate greatly influences the mechanical, biological, and aesthetic properties. It has been always a great challenge to bond resin-based composites to dentin in comparison to enamel bonding [8,9]. The bond strength of resin composites to dentin could be assessed *via* numerous techniques. However, SBS is considered as a facile, and commonly adopted method for measuring the bond strength of restorative materials [9,10]. So far, very little literature is available on the SBS of self-adhering flowable composites. Therefore, in the current study we have adopted the SBS method to compare the adhesion properties of conventional flowable resin composite with that of self-adhering resin composite.

Generally, the self-etch adhesive bonding methods are being used as an alternative to conventional flowable resin composites, mainly due to the effortful handling of the latter ones [9,11-13]. However, besides the clinical advantages associated with these systems, their etching potential and bonding performance still need to be tested

Table 1: Materials used in the current study.

Group Name	Product Name	Manufacturer	Composition	Instructions for use
Group I	Vertise Flow	Kerr, Orange, CA, USA	GPDM adhesive and methacrylate co-monomers, monomer, Prepolymerized filler containing barium glass filler, nano-sized colloidal silica, nano-sized Ybf3 (70 wt. %).	Brush a thin layer (<0.5mm) of Vertise Flow for 15-20 s, Light cure for 20s. Build additional layers (2mm or less) then light cure for 20s.
Group II	Filtek Ultimate Flow	3M ESPE, St Paul, MN, USA	BisGMA, TEGDMA and Procrylat resins, Ybf3 filler, zirconia/silica cluster filler, silica filler.	Place the composite into the cavity (2mm) Light cure for 20s.
	Single Bond Universal	3M ESPE, St. Paul MN, USA	MDP Phosphate Monomer, Dimethacrylate resins Vitrebond Copolymer, HEMA, Filler, Ethanol, Water, Initiators, and Silane.	Apply a single coat of the adhesive to the required area on tooth by rubbing for 20 seconds; gentle stream of air was directed over the material for 5 seconds then LED light cure.

Bis-GMA: Bisphenol-Aglycidylmethacrylate; GPDM: Glycero-Phosphate Dimethacrylate; TEGDMA: Triethylene Glycol Dimethacrylate; Ybf3: Ytterbium Fluoride; MDP: Methacryloxydecyl Dihydrogen Phosphate; HEMA: 2 Hydroxyethyl Methacrylate.

under various clinical circumstances [10,14,15]. Therefore, there has been increased interest in self-adhering flowable composite technology. Recently, Rengo et al. [16], showed that self-adhering flowable resin composite resulted in higher micro-leakage in dentin interfaces as compared with enamel interfaces. Moreover, they observed that etching of dentin using phosphoric acid reduced the sealing performance when a self-adhering flowable resin composite was applied in Class V cavities. Etching is carried out to open dentinal tubules so that the adhesive could penetrate to the dentinal tubules. So by etching the dentinal tubules are opened to enhance penetration of the adhesive. This will not only block the micro-leakage channels but also increase the interlocking and subsequently improve the shear bond strength.

In order to simplify the procedure, the manufacturers have launched adhesive systems, which combines the primer and the adhesive in a one-step. This new class of flowable composites are called "Self-Adhering Resin Composites" (SRCs). They do not require a separate acid conditioning step and moist post-rinse control. They are considered simplified adhesive materials and offer some advantages over conventional etch-and-rinse systems such as reduction of postoperative sensitivity and prevention of procedural errors related to clinical application of conventional bonding agents (such as over-drying and over-wetting) and reduction of chair time [14,17,18]. Another advantage is that infiltration of adhesive resin tends to occur simultaneously with the self-etch process [19]. As the rinsing and drying steps are excluded by the use of SRCs, therefore, the chances of cavity contamination, over-drying and overwetting issues are reduced [14].

These new SRCs have been proposed as an adhesive-free restorative material indicated for the restoration of small class I cavities, class V cavities, and noncarious cervical lesions as well as for lining in class I and II restorations [20]. Despite the ease of clinical use and other advantages of these SRCs, the durability and clinical service are still the matter of great concern for many dental practitioners [21]. Limited studies are available about their physical and mechanical properties [17,22,23].

The aim of this study was to evaluate the SBS of a self-adhering flowable composite and a conventional flowable composite to biodentine and mode of failure of these two components in order to evaluate their adhesion properties and their clinical performance.

Materials and Methods

Materials

The materials used in this study are summarized here along with

the supplier's details. The information regarding flowable composites used in this study is given in Table 1.

Methods

30 human premolar teeth were used in the study. Following extraction, teeth were thoroughly cleaned with pumice to eliminate any surface debris and were kept in 0.1% thymol solution at room temperature before the experiment. The teeth were then inspected under magnification to ensure that they do not have cracks, fractures or flaws (step 1). The teeth were then embedded in self-cure acrylic resin (which cures itself) in a plastic mold (2x2 inch).

In order to eliminate the coronal enamel, a low-speed cutting machine (Isomet, Buehler, Ltd, Evanston, Ill) was used for sectioning all the teeth (step 2). Subsequently, the specimens were embedded in self-cure acrylic resin in a plastic mold. To obtain uniformity, the embedded teeth occlusal surface and acrylic resin were kept at the same level in the mold. The specimen's surface was then wet ground with various silicon carbide papers (320, 400, 600) to further smooth the dentinal surface. All the specimens' surfaces were inspected with a dissecting microscope to confirm the complete removal of enamel fragments (step 3). The specimens were then stored in the water container in an incubator at 37°C for 24h before bonding. The specimens were randomly segregated into two groups of 15 specimens (step 4) each and named as Group I: Vertise Flow and Group II: Single Bond Universal + Filtek Ultimate. The single bond universal liner was applied (step 5), rubbed for 20 seconds, streamed gently with air to ensure uniform distribution and light cured (step 6).

A cylindrical polyvinyl siloxane putty mold with 3.0x3.0 mm (H x D) dimensions was positioned on the surface of dentin and the resin composite was filled in the mold (step 7 and 8) and irradiated for 20 seconds using a LED curing unit (Elipar Free Light II; 3M/ ESPE, USA 1,200mW/cm²). The polyvinyl siloxane putty were removed with a sharp blade (step 9 and 10), and the specimens were examined for defects under a light stereomicroscope at ×10 magnification. The samples were then kept in 100% relative humidity for 24h at 37°C. Figure 1 shows a stepwise scheme for the preparation of the sample.

Shear bond testing

Specimens were mounted on the universal testing machine (Instron 5965) and the shear force was applied at a crosshead speed of 1.0mm/min through a knife-edge blade using 5kN load cell. SBS was measured in MPa by dividing the peak load at failure with the specimen surface area.

In shear bond tests, actually it is a tensile stress that causes

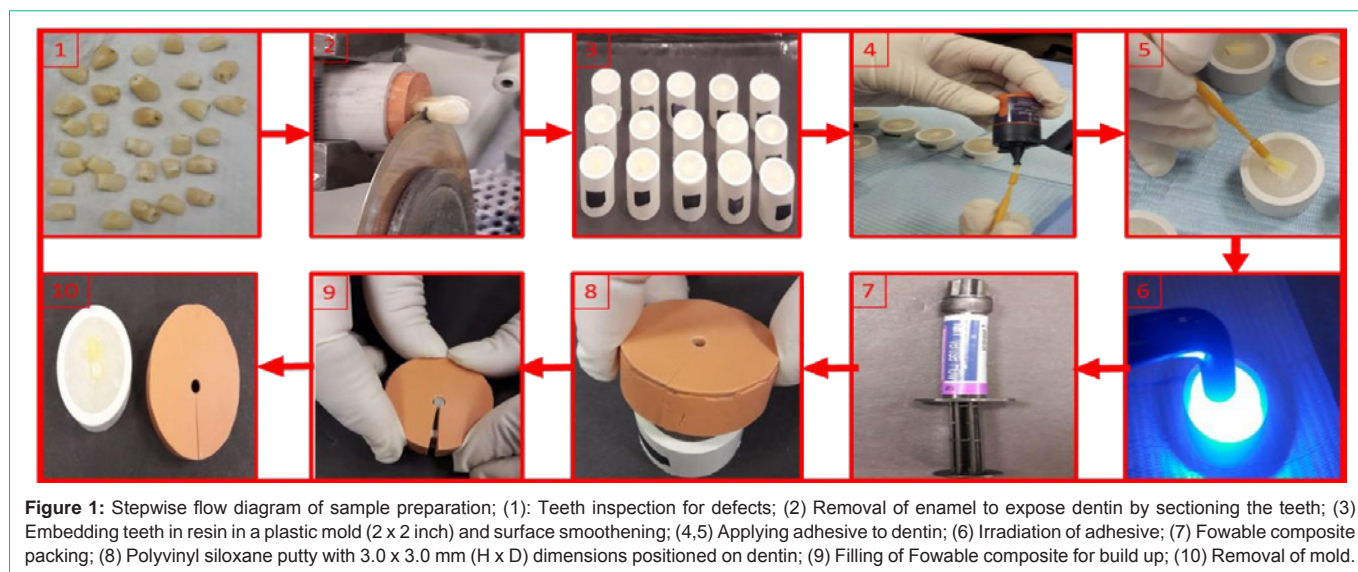


Figure 1: Stepwise flow diagram of sample preparation; (1): Teeth inspection for defects; (2) Removal of enamel to expose dentin by sectioning the teeth; (3) Embedding teeth in resin in a plastic mold (2 x 2 inch) and surface smoothing; (4,5) Applying adhesive to dentin; (6) Irradiation of adhesive; (7) Fowable composite packing; (8) Polyvinyl siloxane putty with 3.0 x 3.0 mm (H x D) dimensions positioned on dentin; (9) Filling of Fowable composite for build up; (10) Removal of mold.

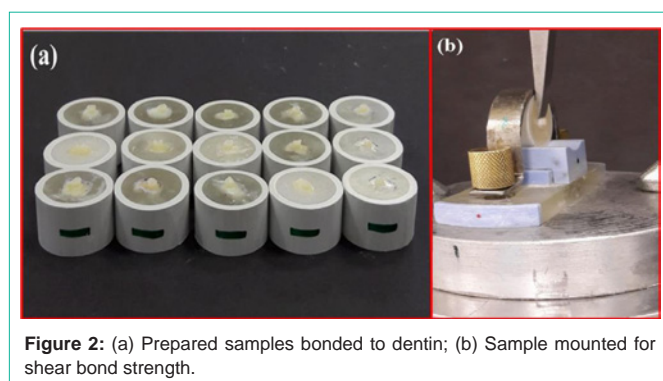


Figure 2: (a) Prepared samples bonded to dentin; (b) Sample mounted for shear bond strength.

Table 2: Shear bond strength for Group I specimen (n=15).

Sample No.	Maximum Load [N]	Shear Bond Stress at Maximum Load [MPa]
1	139.9	19.8
2	123.9	17.5
3	137.2	19.4
4	117.1	16.6
5	136.9	19.4
6	120.5	17
7	114.1	16.1
8	125.3	17.7
9	133.9	18.9
10	120.8	17.1
11	111.9	15.8
12	112.6	15.9
13	111.5	15.8
14	105.4	14.9
15	119.1	16.8
Mean	122	17.3
Standard Deviation	10.7	1.5

debonding. The results may vary from different studies because, the stress distribution at the interface is very heterogeneous. In addition, it is noteworthy to mention that, the position and configuration of the loading device greatly affect the stress distribution at the bonded interface and as a result may cause variation in the results (Figure 2a). The force that causes debonding of the specimen increases by increasing distance of loading device from the interface due to bending moment of the cylindrical specimen. Therefore to avoid variation in the results and keep consistency, we tried our best to keep the position of the loading device accurate during the course of testing. However, SBS results obtained for a specific material from different studies may not be associated due to variation in the bonding substrate, specimen preparation, storage conditions, and loading method [24].

For statistical analysis, independent samples T test was performed to test the Levene’s assumption of homogeneity of variance across both groups, with critical value set at 0.05, using SPSS. The samples mounted on the SBS testing machine are shown in Figure 2b.

Results

Shear bond strength group I and group II

The obtained results for each individual specimen of group I are depicted in Table 2. The mean SBS demonstrated by group I was 17.3 MPa at a maximum mean load of 122N.

Similarly, the results obtained for each individual specimen of group II are depicted in Table 3. The mean SBS demonstrated by group II was 25.2MPa at a mean maximum load of 178.2N.

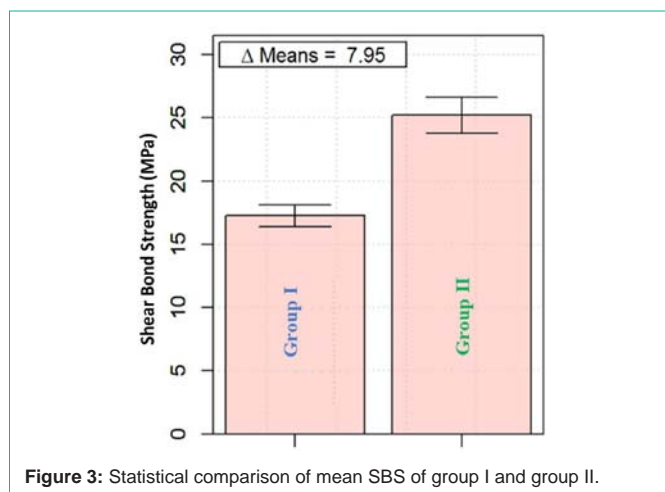
The results of the Levene’s test for equality of variances in the SBS of group I and group II are illustrated in Figure 3. The obtained results revealed that the mean SBS of group II is statistically greater than that of group I (critical-value <0.05). Therefore, the null hypothesis is rejected.

Modes of failure

Figure 4 and 5 presents the fracture patterns for each sample (n=15) for both group I and group II, respectively. Non-cohesive failure was the most dominant mode of failure in Group I. All

Table 3: Shear bond strength for Group II specimen (n=15).

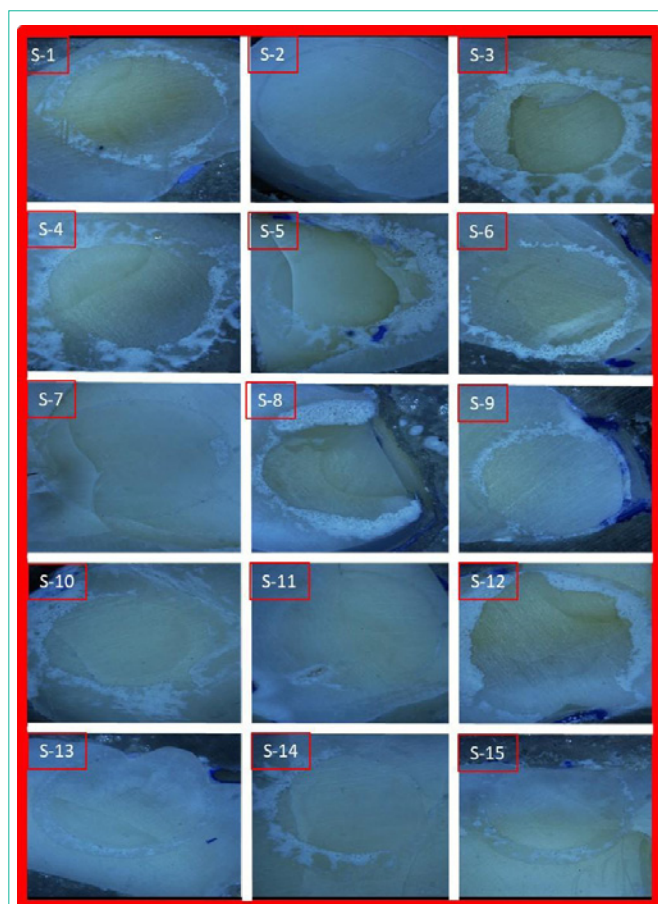
Sample No.	Maximum Load [N]	Shear Bond Stress at Maximum Load [MPa]
1	168.7	23.9
2	167.1	23.6
3	191.5	27.1
4	194.6	27.5
5	149.5	21.2
6	197.2	27.9
7	205.1	29
8	143.5	20.3
9	191.1	27
10	177.1	25
11	177.8	25.1
12	170.2	24.1
13	192.7	27.3
14	188.4	26.7
15	158.9	22.5
Mean	178.2	25.2
Standard Deviation	18.3	2.6

**Figure 3:** Statistical comparison of mean SBS of group I and group II.

the samples exhibited non-cohesive failure in group I, except S-3 (Figure 4) which resulted in cohesive failure. On the other hand, mostly cohesive failure was observed for Group II. Adhesive failure was observed for four samples S-2, S-7, S-9 and S-10 (Figure 5) in Group II only, the rest of 12 (S-1 to 6, S-8, and S-11 to S-15) samples exhibited cohesive failure.

Discussion

The present study revealed that the mean SBS of conventional flowable resin composite (Filtek Ultimate) is considerably greater than that of SRC (Vertise Flow). Therefore, the first null hypothesis was rejected. The bonding of resin composites to enamel and dentin is carried out by removing minerals from dental hard tissue and replacing it with resin monomers which upon polymerization create micromechanical interlocking in the porosities. The bonding process of Vertise™ Flow depends on the monomer adhesive (GPDM),

**Figure 4:** Microscopic analysis of the modes of failure n=15 (Group I).

because due to the presence of the phosphate group, it promotes etching and develops chemical interaction with dental specimens [25]. In current study, the lower bond strength of Vertise Flow may be associated with its limited ability to demineralize and infiltrate dentin substrate, which may result in poor micromechanical interlocking, ineffective sealing of dentinal tubules and poor wetting of dentin collagen fibrils. The self-adhering resin composite should be able to quickly interact with dentin and acquire optimal wetting characteristics in short period of time [26-28]. The bond strength of SRC can be partially related to their chemical interaction with dentin hydroxyapatite [29].

As per manufacturer, there are two ways by which Vertise Flow interact with tooth surfaces. The first mechanism involves chemical interaction of calcium ions in tooth with the functional phosphate groups in the GPDM monomers (inside resin), whereas the second bonding mechanism occurs as a result of micromechanical etching of the tooth caused by the low pH (pH 1.9) of the resin material, which is analogous to that of numerous self-etching materials [30]. The SBS of Vertise™ Flow has been studied by other researchers with and without using an adhesive. Their results revealed that, with an adhesive system, Vertise™ Flow had greater bond strength values compared to its individual use. They also found that the SBS of a conventional flowable composite: Filtek Ultimate Flow is higher than that of self-adhering resin composite (Vertise™ Flow), which is in line with our current study [30]. The high SBS of Filtek Ultimate could

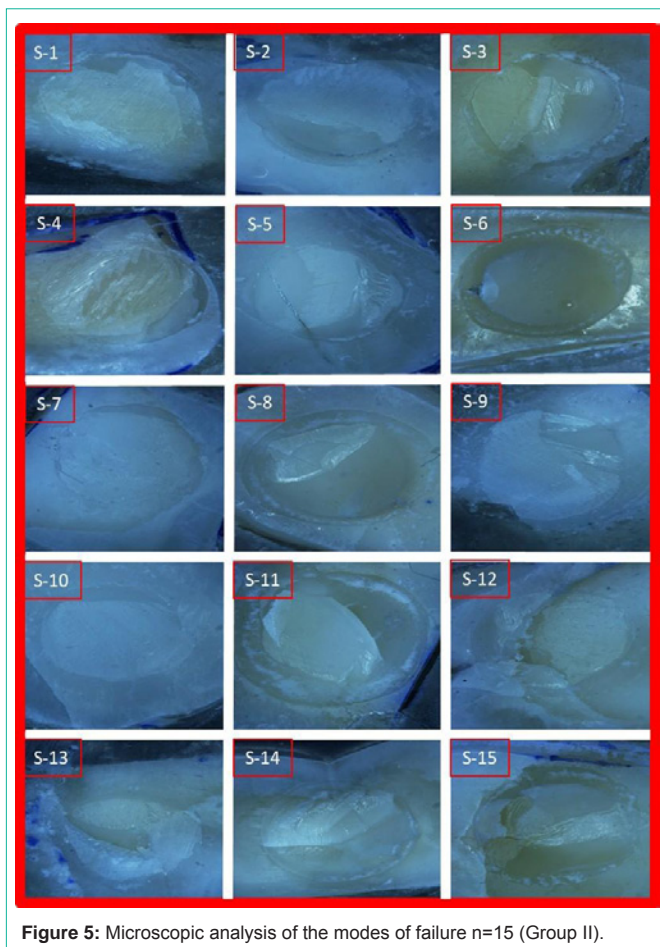


Figure 5: Microscopic analysis of the modes of failure n=15 (Group II).

be attributed to the wetting properties of the adhesives to dentin. Previous studies reported that optimum bonding could be achieved by having specific wetting properties of the adhesive resin similar to the conditioned dentin [31,32]. Similar to our study, different *in vitro* studies revealed that Vertise™ Flow has displayed lower SBS values than all-in-one adhesive, [33] self-etch (two-step) and etch & rinse adhesive systems [34].

It has been observed from microscopic analysis (Figure 4 and 5) that the non-cohesive failure mode was predominantly observed in Group I. However, on the other hand, the most cohesive failures occurred in Group II, which may be attributed to the higher bond strength, forecasting an efficient bonding [35]. In addition, due to the high number of cohesive failure, it could be inferred that the stress distribution is balanced during the mechanical testing of bond strength [36]. Sometimes, the stresses are not homogeneously distributed either due to the uneven surfaces or due to large filler size in the adhesive. It may lead to underestimated values, mixed or adhesive failure.

Conclusion

Within the limitations of this study, the self-adhering flowable composite: Vertise™ Flow had resulted in the lowest SBS as compared with the conventional flowable resin composite (Filtek Ultimate) showing the greater SBS. It could be concluded from the results that reducing the bonding procedures (avoiding etch and

rinsing steps), reduces the bond strength. In addition Vertise™ Flow predominantly exhibited non-cohesive failure, which reflects a poor bonding to dentin. Moreover, the low SBS of Vertise™ shows its limited ability to interact with dentin, demineralize it and infiltrate into dentin substrate, which may result in poor micromechanical interlocking ineffective sealing of dentin tubules and poor wetting of dentin collagen fibrils. On the other hand, the conventional flowable composite (Filtek Ultimate) showed mix failure for most of the samples, which demonstrates its favorable wetting properties (due to single bond universal), good chemical interaction with dentin and subsequently resulted in strong adhesive bonding. Further clinical trials are required to evaluate the data acquired, in laboratory on the new self-adhering flowable composites, under clinical situations in dentistry. The *in vitro* and clinical evaluation will help to improve the performance of Vertise™ Flow for the long-term application of the restorations.

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