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Original Research Article

Bonding performance of a self-adhering flowable composite to substrates used in direct technique

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Abstract

Introduction: Simplified restorative materials may be a logical next step for dental manufacturers. **Objective:** The aim of this study was to evaluate the shear bond strength of a self-adhering flowable composite to four substrates used in direct technique. **Material and methods:** Eighteen samples (5 mm wide, 15 mm length and 2 mm thick) of bovine teeth – uncut enamel/**UE**, cut enamel/**CE**, median dentin/**MD** (6 samples each) – and blocks (also 6 samples) of the nanocomposite/**NC** Filtek Z350 XT (3M ESPE) were used, and samples of each substrate were divided into two groups (n = 3). Two flowable composites (Control/**FF** – Filtek Z350 XT Flow/3M ESPE, and the self-adhering/**DF** – Dyad Flow/Kerr) were bonded to the four substrates. For all groups and in each sample, four Tygon tubings were positioned over the sample, which were filled in with the composites FF and DF, and visible light-cured for 20 s. The tubings were removed to expose the cylinder-shape specimens (12 per group) and samples were stored in relative humidity at 37±2°C for one week. After this period, each sample was attached to testing machine and the specimens were submitted to the shear bond strength test at speed of 1.0 mm/min, until failure. The results were analyzed by two-way ANOVA and Tukey test ($p < 0.05$). **Results:** The means (SD) were (in MPa): UE + FF = 20.8 ± 1.0; UE + DF = 23.9 ± 3.1; CE + FF = 22.7 ± 1.8; CE + DF = 29.6 ± 5.4; MD + FF = 24.8 ± 4.5; MD + DF = 20.8 ± 3.2; NC + FF = 25.9 ± 6.2; NC + DF = 28.4 ± 5.1. **Conclusion:** The efficacy of flowable composites is material-dependent. The self-adhering composite provided higher bond strength on both cut enamel and nanocomposite. Comparing to the control group, Dyad Flow showed lower bond strength to median dentin, however higher bond strength to cut enamel.

Introduction

The adhesive dentistry is in a constant state of evolution. The etch-and-rinse adhesive approach pioneered by Buonocore in 1955 [4] is still used by the dentists [14]. However, the use of self-etch adhesives allows a simpler, less time-consuming and less technique-sensitive clinical procedure [26]. Self-etch adhesive approach to the dental substrate involves simultaneous demineralization and infiltration of the tooth surface to the same depth, ensuring complete penetration of the adhesive [5], and chemical interaction between functional monomers and residual hydroxyapatite [30].

Flowable composites were first introduced in 1995 to restore Class V lesions. They have excellent handling properties, low viscosity, and superior injectability. Easy handling is a highly desired characteristic because it reduces the working time of clinicians and chairside time of patients, according to Bayne *et al.* [1]. Following the same characteristics, a new self-adhering flowable composite, Vertise Flow (Kerr, Orange, CA, USA – named Dyad Flow in Latin America), was recently introduced in the market, as well as the Fusio Dentin Liquid (Pentron Clinical, Orange, CA, USA). These adhesive-free composites are claimed to rely on chemical and micromechanical interaction between material and tooth structures or other substrates, achieved with incorporation of an acidic adhesive monomer into the flowable composites [8, 14].

Owing to the novelty of this material, it seemed interesting to investigate further on the bonding performance of this new self-adhering flowable composite Dyad Flow. The aim of this study was to evaluate the shear bond strength of a self-adhering flowable composite to four substrates used in direct technique. The tested null hypothesis was that statistically similar bond strengths are achieved by the self-adhering flowable composite and the flowable composite of the control group.

Material and methods

Eighteen samples (5 mm wide, 15 mm length and 2 mm thick) of bovine teeth and six samples (blocks) of one nanocomposite were used in this study. The roots of the bovine teeth were removed with aid of a flexible diamond disc (KG Sorensen, Cotia, SP, and Brazil) under refrigeration. Their crowns were sectioned using the same diamond disc, also under refrigeration, to obtain six samples of uncut enamel/UE; six samples of cut enamel/CE (underwent abrasion with 200- and 600-grit silicon-carbide sandpaper); and six samples of median dentin flat surfaces (1 mm below enamel-dentin junction). Immediately prior to adhesive procedures, dentin samples underwent abrasion with 600-grit silicon-carbide sandpaper to create fresh smear layer. Also, six samples (blocks) of the nanocomposite/**NC** Filtek Z350 XT (3M ESPE) were used.

Samples of each substrate were divided into two groups (n = 3) and the products were used according to manufacturer's instructions. Two flowable composites (Control/**FF** – Filtek Z350 XT Flow/3M ESPE, and the step-less self-adhering/**DF** – Dyad Flow/Kerr) were bonded to the four substrates (table I). According to Shimada *et al.* [23], for all groups and in each sample, four Tygon tubings (TYG-030, Saint-Gobain Performance Plastic, Maime Lakes, FL, USA) were positioned over the sample, which were filled in with the composites FF and DF, and visible light-cured (VLC) for 20 s (LED Bluephase – 1.200 mW/cm^2 – Ivoclar Vivadent, Schaan, Principality of Liechtenstein). The tubings were removed to expose the specimens in format of cylinders (area: 0.38 mm^2/ by formula πR^2) and samples were stored in distilled water in relative humidity at 37±2°C for one week. After this period, each sample was attached to the universal testing machine (Emic DL 1000, São José dos Pinhais, Pr, Brazil) and the specimens were submitted to shear bond strength test (SBS), applied at the base of the specimen/substrate cylinder with a thin wire/0.25 mm, at speed of 1.0 mm/min – until failure. The results were analyzed by two-way ANOVA (two flowable composites and four substrates) and Tukey test ($p < 0.05$).

Table I – Materials used

Material	Batch #	Composition	Protocol
Acid Gel Dentalville do Brasil Joinville, SC, Brazil	351 Exp: 07/13	Phosphoric acid 37% and clorhexidine 2%	Phosphoric acid 30 s to enamel and 15 s to dentin), wash, dry 5 s, apply the adhesive, gentle air 5 s, VLC 10 s
Adper Single Bond 2 Dental Adhesive pH ≈ 4.7 3M ESPE St. Paul, MN, USA	N2I1104BR Exp: 11/13	Bis-GMA, HEMA, UDMA, dimethacrylates, ethanol, water, camphorquinone, photoinitiators, copolymer of polialcenoic acid, silica (5 nm)	
Filtek Z350 XT Flow VLC Flowable Nanocomposite/A2 3M ESPE St. Paul, MN, USA	1211700713 Exp: 12/13	Bis-GMA, TEGDMA, Bis-EMA, silane-treated ceramic, silica, zirconium oxide – 55 vol% / 65 wt%	Apply and VLC 20 s
Filtek Z350 XT VLC Nanocomposite /A2B 3M ESPE St. Paul, MN, USA	N312081BR Exp: 06/14	Bis-GMA, UDMA, TEGDMA, and Bis-EMA. Non-agglomerated/non-aggregated 20 nm silica filler, non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles) – 63.3% vol% / 78.5% wt	Apply and VLC 20 s
Dyad Flow or Vertise Flow Self-Adhering Flowable Nanocomposite/A2 pH ≈ 1.9 before VLC pH ≈ 6.5-7 after VLC Kerr, Orange, CA, USA	4398621 n. 34384 Exp: 06/13	GPDM, prepolymerized filler, 1-μm barium glass filler, nanosized colloidal silica, nanosized Ytterbium fluoride	Phosphoric acid 30 s just to enamel, wash, 5 s maximum air dry, apply, brush a thin layer (<0.5 mm) with pressure for 15-20 s, VLC 20 s

Composition as provided by respective manufacturer: Bis-GMA, bisphenol glycidyl dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-EMA, bisphenol A polyethylene glycol dimethacrylate; GPDM, glycerol phosphate dimethacrylate

Results

The ANOVA showed significant differences between flowable composites and among substrates ($p < 0.001$). To investigate the differences, Tukey test was applied the ($p < 0.05$). The self-adhering composite provided higher bond strength on cut enamel and nanocomposite. Comparing to the control group, Dyad Flow showed lower bond strength to median dentin, however higher bond strength to cut enamel (table II).

Table II – SBS means (±SD) in MPa and Tukey test ($p < 0.05$)

	Flowable composite	
	Filtek Flow (FF/control)	**Dyad Flow (DF/self-adhering)**
Uncut enamel	20.8 ± 1.0 A b	23.9 ± 3.1 A b
Cut enamel	22.7 ± 1.8 B a	29.6 ± 5.4 A a
Median dentin	24.8 ± 4.5 A a	20.8 ± 3.2 B b
Nanocomposite	25.9 ± 6.2 A a	28.4 ± 5.1 A a

Means followed by the same lower case within columns and capital letters within rows did not significantly differ by Tukey test ($p < 0.05$)

Discussion

Based on the findings of the present study, the formulated null hypothesis has not to be rejected to uncut enamel and nanocomposite. However, it has to be rejected to cut enamel and median dentin, because the results differ significantly in shear bond strength on these substrates.

Although clinical trials produce the most reliable evidences, and translation of in vitro findings to oral conditions has limitations, laboratory tests are still useful at promptly yielding first-hand information. 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 [26, 28]. This study focused on the evaluation of the shear bond strength (SBS) of the self-adhering flowable composite Dyad Flow to four substrates used in direct technique, using microshear methodology proposed by Shimada *et al*. [23]. This type of mechanical test solves problems related to tension propagations at the bonded interface in larger areas. It presents the advantage that several specimens can be obtained from one sample without cutting it, being easier and cheaper than the microtensile test, when the samples need to be cut to obtain the specimens [22].

Adhesion to the enamel surface is based on the infiltration of resin monomers into etched enamel. An optimum etching pattern is observed when using an etch-and-rinse technique/ER [13]. Self-etching adhesive systems/SE also has been advocated for this type of procedures as a suitable replacement for etch-and-rinse systems [18]. However, there is some concern about the ability of these SE systems to etch enamel, as many studies find that bond strengths of composite to enamel provided with ultra-mild, mild or intermediary-strong SE systems are lower when compared to ER systems [7, 12, 16, 19]. Notwithstanding, SE systems seem to perform well in clinical studies [20], and the selective etching of enamel with phosphoric acid has been demonstrated as a potential technique to use with SE systems to improve their bonding performance on enamel [3, 15, 29].

For this study, according to the manufacturer's instructions, the uncut and cut enamel was etched with phosphoric acid in the control groups, and additionally etched with phosphoric acid in the DF groups. There was statistical difference to uncut and cut enamel, inside each group, to both flowable composites (FF and DF), with higher SBS to cut enamel. By comparing the groups (control and self-adhering), there was statistical difference to cut enamel, and DF showed stronger SBS than control group. According to Van Meerbeek *et al*. [26], pre-etching enamel significantly improves the bonding effectiveness, since phosphoric acid significantly enhances the surface energy of enamel and thus provides significantly more micro-retention [21].

Di Hipólito [6] describes the action mechanism of resin monomers with substrate enamel, that is, due to a sequence of interdependent phenomena. Possibly, factors such as increased inter and intra enamel spaces to infiltration of resin monomers, breaking the surface tension of the liquid monomer, and the formation of a smaller contact angle with the surface of the enamel, are responsible for the higher bond strength for the cut enamel, within both groups (FF and DF). Also the exposition of the prismatic layer may have favored the quality of the demineralization process and infiltration of the flowable composite DF, which showed higher bond strength compared to the control group.

Based on the pH 1.9 declared from the manufacturer (before VLC), it is possible to speculate that Dyad Flow can interact similarly to a mild self-etch adhesive, that was enough to demineralize the uncut and cut enamel, but specially the cut enamel, and also interacting with the nanocomposite Filtek Z350 XT (both cut enamel and this nanocomposite showed higher SBS to DF group). To the manufacturer's instructions, its acidic phosphate group etches the tooth structure and creates chemical bonds with the calcium. Specifically regarding to microshear bond strength test and enamel substrate, there is no comparison in the literature until this moment, just to dentin substrate.

The Technical Bulletin Kerr/35104 (2010) shows that the Dyad Flow has one common element in all Kerr bonding agents, that is, the GPDM adhesive monomer, a phosphate functional group that creates a chemical bond with the calcium ions of the tooth. GPDM monomers ensure a tenacious bond to both enamel and dentin, evidenced by the strength known to all generations of the OptiBond adhesive family. A GPDM adhesive monomer acts like a coupling agent. On one hand, it has an acidic phosphate group for etching the tooth structure and also for chemically bonding to the calcium ions within the tooth structure. On the other hand, it has two methacrylate functional groups for copolymerization with other methacrylate monomers to provide increased crosslinking density and enhanced mechanical strength for the polymerized adhesive.

The dentin is a more heterogeneous and physiologically dynamic substrate than enamel. Inside the control group, there was no statistical difference between median dentin and other substrates, except to uncut enamel, that showed lower SBS. But, to the DF group, median dentin and uncut enamel showed the lowest SBS. Also there was difference comparing the substrate dentin between control and DF groups, with higher SBS to the control group. Garberoglio and Brännström [9] showed that the number and diameter of dentinal tubules increase with deepness. At superficial dentin, 96% of the area is occupied by intertubular dentin, 3% by peritubular dentin and only 1% by dentinal fluid. There is an inverse relationship, however, for the area closer to pulp, when 66% of the area is occupied by peritubular dentin, 12% by intertubular dentin and 22% by dentinal fluid. According to Swift Junior *et al.* [24], differences in composition and morphology in relation to deepness may directly influence the behavior and mechanical properties of dentin against chemical and physical agents to which dentin is submitted during the operative procedures, such as the application of the self-adhering flowable composite used in this study.

Dyad Flow recorded lower bond strength to median dentin, also without statistical difference to uncut 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, and this property could represent a drawback for the material's ability to wet self-etched collagen fibrils [8].

Despite of the description of this manufacturer (Kerr), no chemical analytic data on the bonding potential of GPDM are available. It indicates that GPDM "etches" rather than "bonds" to hydroxyapatite [30]. To achieve self-adhesiveness, it is speculated that a relatively viscous (flowable) composite should contain a functional monomer that rather possesses an effective chemical bonding potential, as it cannot penetrate deeply [21]. Anyway, the findings of this study agree with the findings of Bektas *et al.* [2], regarding the dentin substrate and microshear bond strength methodology. For that study, the Vertise Flow also showed lower SBS, comparing to the control group.

Concerning to the Dyad Flow filler system, again according to the Technical Bulletin Kerr/35104 (2010), the type, proportion, and size of each filler particles were carefully chosen for optimized wetting, mechanical strength, and polishability. Dyad Flow consists of 4 filler types: 1) a prepolymerized filler, 2) a 1-micron barium glass filler, 3) a nanosized colloidal silica, and 4) a nanosized Ytterbium fluoride. The average particle size of Dyad Flow is 1 micron. The pre-polymerized filler (PPF) enhances the handling characteristics of the material, making it smooth and easy to manipulate. Furthermore, PPFs help minimize shrinkage due to a "pre-shrunk," or prepolymerized, nature. Nanoparticles enhance the polishability of the material and achieve special rheological property; also, the nanoytterbium fluoride particles give to Dyad Flow a superb radiopacity index for easy detection with X-rays.

Not all composites are equal in their ability to bond to other materials. Highly hydrophobic matrices, advanced monomers to polymers conversion, and formulations which a large proportion of filler particles could discourage the interaction of materials. Three chemical interaction mechanisms are possible for the bond strength of the flowable composites to the nanocomposite, according to the findings of Teixeira *et al.* [25]: 1) the adhesion between the polymer matrices, from both flowable composites and the nanocomposite; 2) the adhesion between the fillers particles exposed of both composites; and 3) the formation of a micro-network of the polymer chains and the fillers particles of both composites. This latter mechanism would likely dominate and produce the greatest contribution with regard to acceptable bond strength, as it was possible to observe inside the control group and inside the self-adhering group, and also among the groups, with no statistical difference among them.

It has been observed that shear bond testing tends to produce cohesive failures of the substrate [10, 11]. The improvement of the bonding properties of restorative materials have increased the bond strength and changed the failure pattern [17]. This transition is most likely related to the changing stress pattern as the crack progresses across the interface. It is usually observed that a bigger piece of cohesive fracture in the substrate is pulled out after the transition from adhesive to cohesive fracture occurs [27]. Using optical microscopy and according to the authors above, it was observed mixed failures to all groups, including mainly cohesive failures in the substrates.

Conclusion

Within the limitation of this study, it was observed that the efficacy of flowable composites is material-dependent. The self-adhering composite provided higher bond strength on cut enamel and nanocomposite. Comparing to the control group, Dyad Flow showed lower bond strength to median dentin, however higher bond strength to cut enamel. The Dyad Flow can provide acceptable bond strength, however more studies about the properties and action mechanism of this material are necessary in the future.

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