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Original Research Article

Bonding performance of a self-adhering flowable composite to indirect restorative materials

Rubens Nazareno Garcia[1, 2]
Caroline Silvestri Silva[1]
Giovana Gochinski Silva[1]
Guilhermo Mocellin[1]
José Ozelame[1]
Laís Fracasso[1]
Matheus Bernhardt Ozelame[1]
Reinaldo Francisco do Nascimento[4]
Ana Cristina Rocha Gomes[3]

Corresponding author:
Rubens Nazareno Garcia
Universidade do Vale do Itajaí – Univali
Curso de Odontologia – Grupo de Pesquisa Biomateriais em Odontologia
Rua Uruguai, 458 – Centro
CEP 88302-202 – Itajaí – SC – Brasil
E-mail: rubensgarcia@univali.br

[1] School of Dentistry, Research Group on Biomaterials in Dentistry, University of Itajai Valley – Itajai – SC – Brazil.
[2] Department of Dentistry, University of Joinville Region – Joinville – SC – Brazil.
[3] DDS and Dental Technician, RPD Dental Laboratory – Joinville – SC – Brazil.
[4] Dental Technician, RPD Dental Laboratory – Joinville – SC – Brazil.

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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 indirect technique. **Material and methods:** Twenty-four samples (5 mm wide, 15 mm length and 2 mm thick / six blocks each substrate) were prepared in the dental prosthetic laboratory. The following materials were used: ceromer (SR Adoro/**AD**, Ivoclar Vivadent), leucite ceramic (IPS Empress Esthetic/EE, Ivoclar Vivadent), zirconia ceramic (ZirCAD/ZI, Ivoclar Vivadent); metal ceramic alloy (Fit Cast SB/ME, Talladium do Brasil). 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. Four Tygon tubings were positioned over each 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 specimens (12 per group) in format of cylinders and samples were stored in distilled water 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): AD + FF = 34.4 ± 4.9; AD + DF = 28.2 ± 4.2; EE + FF = 29.7 ± 5.8; EE + DF = 32.3 ± 6.9; ZI + FF = 23.2 ± 5.4; ZI + DF = 8.5 ± 1.5; ME + FF = 28.9 ± 4.2; ME + DF = 31.7 ± 4.5. **Conclusion:** The efficacy of flowable composites is material-dependent. The self-adhering composite provided lower bond strength only to zirconia ceramic. Comparing with the control group, Dyad Flow showed lower bond strength to the ceromer and zirconia ceramic.

Introduction

The demands from dentists and patients for tooth-colored posterior restorations such as inlays, onlays, and crowns have been increasing in recent years, also the luting techniques for these restorations. For larger restorations, indirect methods are superior alternatives to direct resin composite fillings [18]. The ceromers, leucite-reinforced and zirconia-reinforced ceramics, and the metal-ceramic restorations can represent these materials, which can be luted by either conventional or adhesive technique, or other alternative of luting can be the new self-adhering flowable composite.

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]. A new self-adhering flowable composite, Vertise Flow (Kerr, Orange, CA, USA – named Dyad Flow in Latin America), was recently introduced into 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 [2, 7, 11, 15, 22].

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 indirect 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

Twenty-four samples (5 mm wide, 15 mm length and 2 mm thick / six blocks to each substrate below) were obtained in the dental prosthetic laboratory, according to manufacturer's instructions. The ceromer SR Adoro/**AD** blocks (Batch # R57506 / Exp: 07/2015) were prepared and light-cured in the Lumamat 100 Light Furnace; the leucite-reinforced ceramic IPS Empress Esthetic/**EE** blocks (Batch # KM0305 / Exp: 12/2030) were prepared in the hot pressing technique; the zirconia-reinforced ceramic IPS e.max ZirCAD/**ZI** blocks (Batch # L15418 / Exp: 12/2030) were prepared in the CAD/CAM (Computer-Aided Design / Computer-Aided Manufacturing) technique – all materials from Ivoclar Vivadent, Schaan, Principality of Liechtenstein; and the metal ceramic alloy Fit Cast SB/**ME** blocks (Batch # 121165/Exp: undetermined / Talladium do Brasil, Curitiba, PR, Brazil) were prepared in the lost wax technique. All the samples were sandblasted with aluminum oxide (90 μm / 2.5 Bar / 10 mm distance) and the samples of each substrate were divided into two groups – control and self-adhering flowable composites (n = 3).

For **AD** and **EE** groups, after application of 10% hydrofluoric acid for 1 min (Condac Porcelana, FGM, Joinville, SC, Brazil – Batch # 031211 / Exp.: 12/2013), the samples were rinsed for 1 min, air-dried for 1 min, followed by the application of the silane coupling agent (Monobond-S, monofunctional-3-methacryloxypropyltrimethoxy sylane/3-MPS, Ivoclar Vivadent – Batch # N15219 / Exp.: 02/2013) for 1 min and then air-dried for 30 s. For **ZI** and **ME** groups, it was applied the metal & zirconia primer (Metal/Zirconia Primer, phosphonic acid acrylate in tert-Butyl alcohol, Ivoclar Vivadent – Batch # M68692 / Exp.: 02/2013) for 3 min and then air-dried for 30 s.

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 (table I). According to Shimada *et al.* [18], 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 (12 per group – area: 0.38 mm^2 / by formula πR^2) and samples were stored in distilled water 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
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)	Apply the adhesive, gentle air 5 s, VLC 10 s
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
Dyad Flow or Vertise Flow Self-Adhering Flowable Nanocomposite/A2 pH ≈ 1.9 before VLC pH ≈ 6.5-7 after Kerr, Orange, CA, USA	4398621 Item 34.384 Exp: 06/13	GPDM, prepolymerized filler, 1-μm barium glass filler, nanosized colloidal silica, nanosized Ytterbium fluoride	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. VLC: visible light-curing

Results

ANOVA showed significant differences between flowable composites and among substrates ($p < 0.001$). Tukey test ($p < 0.05$) was applied to investigate the differences. The self-adhering composite provided lower bond strength just on zirconia ceramic. Comparing to the control group, Dyad Flow showed lower bond strength to the ceromer and zirconia ceramic (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)
Ceromer	34.4 ± 4.9 A a	28.2 ± 4.2 B a
Leucite-reinforced ceramic	29.7 ± 5.8 A b	32.3 ± 6.9 A a
Zirconia-reinforced ceramic	23.2 ± 5.4 A c	08.5 ± 1.5 B b
Metal ceramic alloy	28.9 ± 4.2 A b	31.7 ± 4.5 A a

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

Discussion

Based on the findings of the present study, the formulated null hypothesis was accepted for leucite-reinforced ceramic and metal ceramic alloy. However, it was rejected for ceromer and zirconia-reinforced ceramic, because the results differ significantly in shear bond strength to these substrates.

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 against contraction and functional stresses [20]. This study focused on the evaluation of the shear bond strength (SBS) of self-adhering flowable composite Dyad Flow to four substrates used in indirect technique, using microshear methodology proposed by Shimada *et al*. [18]. 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 [17].

Indirect restorations have been used to reduce or minimize polymerization shrinkage of the resin composite direct restorations. One possible reason for this is the small amount of resin cement used in luting procedures. This technique provides better sealing than that of direct composites. Moreover, it is used to facilitate the reproduction of the dental anatomy, in order to improve control of the marginal fit, proximal and occlusal contacts. Regarding to ceromers, when compared to other indirect restorations, as ceramics, they present a more simple fabrication technique, less wear on the antagonist teeth, the possibility of intra-oral repair and lower cost [6]. With regards to ceramics, they are used to achieve esthetic dental restorations because of their superior color, and their clinical success is determined by the bond strength and bonding durability of the resin cement to tooth and ceramic [14].

The application of resin cements for tooth-colored indirect restorations have increased considerably because of their ability to set completely and their greater resistance to occlusal loading when compared to the conventional cementation. This luting usually requires several steps to secure optimal adhesion. However, recently some self-etching primers and self-adhesive resin cements have been introduced, yielding major improvements in bonding to tooth structures. The use of these products is a result of attempts to improve the bonding quality while reducing the number of necessary procedures [18]. To this study, it was speculated that the use of one newly self-adhering composite could result in the similar shear bond strength, although the control group had other flowable composite instead of resin cement.

First of all, the samples of ceromers and leucite-reinforced ceramics were sandblasted with aluminum oxide. Sandblasting of the interior surface of these materials is a common practice in laminates or crown restorations because the roughened surface enables a strong mechanical bond with resin-based dental materials [24]. After that, according to the manufacturer's instructions, the ceromer and leucite-reinforced ceramic was etched with 10% hydrofluoric acid and silanation. The preferred manner of conditioning these surfaces is by etching with hydrofluoric acid, followed by the application of a silane coupling agent to achieve high bond strength. The acid works by creating surface pits via preferential dissolution of the glassy phase from the ceramic matrix and the dissolution of the resin matrix of the ceromer [5]. Treatment of the etched surface with silane increases the wettability and forms a covalent bond [16]. The 3-MPS (silane) is known to promote the adhesion through chemical and physical coupling between metal-composites, ceramic-composites, and composites containing methacrylate groups.

Also, three chemical interaction mechanisms are possible for the bond strength of the flowable composites to other composite, the ceromer, according to the findings of Teixeira *et al.* [19]: 1) the adhesion between the polymer matrices, from both flowable composites and ceromer; 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 regards to acceptable bond strength, as it was possible to observe inside both the control and self-adhering groups. However, by comparing the groups, the DF group showed lower bond strength, with statistical difference between them. It is speculated that the adhesive system Adper Single Bond 2 used prior to flowable composite of the control group helped the wettability of the ceromer substrate, resulting in higher bond strength to ceromer in control group.

As the aforementioned author information about the filler particles, it is interesting to report some information on Dyad Flow filler system. 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. Nanoparticles enhance the polishability of the material and achieve special rheological property; also nanoytterbium fluoride particles give to Dyad Flow a superb radiopacity index for easy detection with X-rays.

The Technical Bulletin Kerr/35104 (2010) also 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.

As can be seen, the resin matrix of the Dyad Flow consists of multifunctional acidic methacrylates. If a high content of acidic functional monomers can react with the substrate like the leucite-reinforced ceramic used in this study, and achieve enough micromechanical and chemical bond strength, it is possible to hypothesize that this self-adhering composite can be used to bond successfully to this type of ceramics surface. Using microshear methodology and Dyad Flow, there is no comparison to the literature until this moment, regarding to bond strength to ceromers and ceramics – just the internal data of the manufacturer Kerr, reported at the Technical Bulletin Kerr/35104 (2010). In this technical report, the porcelain showed 33.9 MPa (without using hydrofluoric acid and silane), while in this study the leucite-reinforced ceramics exhibited 32.3 MPa. Moreover, the composite showed 34.2 MPa in the technical report, while in this study the ceromer presented 28.2 MPa, but they did not inform which was the type of the composite: direct or indirect. The findings of Garcia *et al.* [9] showed similar bond strength to leucite-reinforced ceramics, but the authors used only resin cements – RelyX ARC and RelyX U100, both from 3M ESPE.

Metallic restorations have a long-standing history of clinical use in dentistry. However, increase in patients' aesthetic expectations and demands caused the metallic restorations to be sidelined and led to the development of porcelain-fused-to-metal (PFM) restorations. Despite the brittle nature of porcelain, PFM crowns are widely used because the metallic frameworks afford superior mechanical durability. On the other hand, the use of metals in PFM restorations gives rise to gingival discolorations and metal-related allergies, according to Kuriyama *et al.* [12].

IPS e.maz ZirCAD is an yttrium-stabilized zirconia oxide block. It is suitable for indications that require high strength, including posterior bridges. The composition and physical properties of zirconia oxide-based ceramics differ substantially from silica-based ceramics, such that conventional acid-etching has no positive effects on the resin bond to zirconia oxide ceramics [3]. As resin bond to high-strength ceramics is less predictable, alternative bonding techniques are required to achieve a strong and long-term durable resin bond. Primers containing phosphonic acid compound as the active ingredient, could establish a chemical bond to oxidic surfaces, such as Metal/Zirconia Primer. Hence, they can be an alternative to promote adhesion to oxide ceramics such as zirconia, except for DF. In this study, it was not enough to obtain a reliable bond strength between DF and zirconia. However, the control group showed statistically higher bond strength. Also, it is speculated that the adhesive system Adper Single Bond 2 used prior to flowable composite FF of the control group helped the wettability of the substrate zirconia-reinforced ceramic.

The opposite situation occurred for the metal ceramic alloy Fit Cast SB. In an attempt to improve the bonding of composite to alloys, and according to Di Francescantonio *et al.* [4], there are many methods like electrolytic etching, chemical etchings, adhesive primer application, and silica coating methods for surface treatment of metal alloys. This study also employed Metal/Zirconia Primer to metal ceramic alloy, which showed similar bond strength between the control group FF and the self-adhering composite DF, without statistically significant difference. Sandblasting with aluminum oxide particles removes contaminated layers and creates a roughened surface which provides mechanical interlocking for the composites as well as providing a great surface area for bonding. It has been reported that the sandblasting process could form a passive film made of Ni, Cr and Co oxides, according to the findings of Yoshida *et al.* [23].

Metal/Zirconia Primer, as already described, consists of phosphonic acid acrylate. The manufacturer claimed that it is suitable for oxide ceramic and all type of metal alloy. In this study, the results showed the efficiency of Metal/Zirconia Primer in enhancing shear bond strength between Ni-Cr alloy and the self-adhering flowable composite Dyad Flow. The results were similar to Di Francescantonio *et al.* [4], however they used others primers and others metal ceramic alloys.

Finally, it has been observed that shear bond testing tends to produce cohesive failures of the substrate [8, 10], usually to dental substrates. It is observed that a bigger piece of cohesive fracture in the substrate is pulled out after the transition from adhesive to cohesive fracture [21]. The improvement of the bonding properties of restorative materials have increased the bond strength and changed the failure pattern [13, 17]. This transition is most likely related to the changing stress pattern as the crack progresses across the interface. By using optical microscopy in this study and according to the authors above, it was observed mixed failures within AD and EE groups, followed by cohesive failures (in the ceromer and in the leucite-reinforced ceramic) and adhesive failures. With regards to zirconia-reinforced ceramic (ZI) and metal ceramic alloy (ME), mainly adhesive failures were observed.

Conclusion

Within the limitation of this study, it was observed that the efficacy of flowable composites is material-dependent. The self-adhering composite provided lower bond strength only to zirconia ceramic. Comparing to the control group, Dyad Flow showed lower bond strength to the ceromer and zirconia ceramic. The Dyad Flow can provide acceptable bond strength; however further studies on the properties and action mechanism of this material are necessary.

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