



Micro-shear Bond Strength of Self-Adhesive Versus Conventional Low-viscosity Composite Resins: *In vitro* Study

**Cassia Thaís Iurkiv Zanatta ^{a*},
Poliana Maria de Faveri Cardoso ^a, Veridiana Camilotti ^a,
Márcio José Mendonça ^a and Julio Katuhide Ueda ^a**

^a *Department of Restorative Dentistry, School of Dentistry, UNIOESTE – Western State University of Paraná, Cascavel, PR, Brazil.*

Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Aims: This study aimed to evaluate the micro-shear bond strength of a self-adhesive versus conventional low-viscosity composite resins adhered to enamel.

Study Design: *In vitro* study.

Place and Duration of Study: Dental Clinic of the Western State University of Paraná, between October 2022 and September 2023.

Methodology: In this *in vitro* study, the crowns of ten bovine incisor teeth were separated from the roots and embedded in polyvinyl chloride cylinders with acrylic resin. The teeth were divided into

*Corresponding author: E-mail: cassaiurkiv@gmail.com;

two groups (n=5) according to the type of low-viscosity composite resin used on the enamel, resulting in a total of fifteen specimens: one group with self-adhesive composite resin and another with conventional composite resin. Specimens measuring 3 × 2 mm were prepared with low-viscosity composite resin and applied to the adhesive areas according to their respective groups. The micro-shear test was carried out after 7 days of immersion in distilled water in a universal mechanical testing machine with a 50 kg load cell. The statistical analysis in this study employed the Shapiro-Wilk test to assess data normality. Due to non-normal distribution, the non-parametric Mann-Whitney U test was utilized for group comparisons ($p < 0.001$). Supplementary analyses were performed using the Dwass-Steel-Critchlow-Fligner (DSCF) test for multiple comparisons ($p < 0.001$) to detect intergroup differences. Fractures were analyzed using a stereoscopic magnifying glass at 40x magnification.

Results: The lowest micro-shear bond strength was observed in the self-adhesive composite resin group, while the highest was observed in the conventional composite resin group. On average, the micro-shear bond strength was higher for the conventional composite resin group compared to the self-adhesive composite resin group ($p < 0.001$). Most fractures in the conventional composite resin group were mixed, whereas in the Self-Adhesive composite resin group they were predominantly adhesive.

Conclusion: Based on this study, it can be concluded that self-adhesive flowable composite resin exhibited lower enamel bond strength values compared to conventional flowable composite resin.

Keywords: Composite resins; flowable composite; shear strength; in vitro; dental clinics; clinical procedures; filler content.

1. INTRODUCTION

Using low-viscosity composite resins, also known as flow or fluid resins, has become a common practice in dental clinics [1,2]. These resins were originally designed for Class V cavities [3]. However, they are now used in a variety of clinical procedures. This includes the restoration of small caries lesions, the sealing of pit and fissures, and even as a lining for composite resin restorations [4]. In smaller cavities, these resins can be easily inserted. Additionally, they are expected to provide better adaptation to the cavity walls when compared to higher-viscosity composite resins [5]. Because of their lower filler content, these composite resins show a lower modulus of elasticity and greater polymerization contraction when compared to conventional composite resin [6]. A noteworthy advancement in dentistry is the introduction of low-viscosity self-adhesive composite resins. These represent the combination of two functional groups: the all-in-one adhesive system and the low-viscosity composite resin [7, 8]. Incorporating the functional monomer glycerophosphate methacrylate (GPDM) into the chemical composition of composite resins has simplified the stages of direct restorative procedures [3,7,9]. According to the manufacturer's claims, this monomer exhibits acidic properties, conditions the tooth structure, binds to the calcium in the tooth structure, and has two methacrylate functional groups, which can copolymerize with other methacrylate monomers

[9,10]. Therefore, using these composite resins decreases the time needed for application and minimizes clinical errors and technique sensitivity. However, there have been reports indicating that the GPDM functional monomer may "condition" hydroxyapatite rather than establish a direct bond [11]. Furthermore, based on previous studies, these composite resins have a higher modulus of elasticity, hardness [12], and degree of conversion [13] than other conventional low-viscosity composite resins. Additionally, these composite resins exhibited greater hygroscopic expansion [14] and water absorption [15] when compared to other low-viscosity composite resins, after 150 days of immersion in water.

The most reliable method for adhesion, known as the gold standard, involves a three-step process: total etching, washing, and drying of the substrate [16]. This procedure, in contrast to self-etching, involves a step in which phosphoric acid is used to create microporosities in the hydroxyapatite of the enamel. When the resin is applied to this previously acid-treated enamel, it fits perfectly into these porosities, promoting optimal adhesion to the enamel surface. This results in an effective sealing of the restoration margins, filling any space between the restoration and the tooth, thus preventing bacterial infiltration [2].

Taking into account the crucial importance of effective adhesion for composite resin

restorations and the recent introduction of low-viscosity self-adhesive composite resins, this study set out to evaluate the micro-shear bond strength of low-viscosity self-adhesive composite resin. This resin was applied directly to the cavity and compared with a conventional low-viscosity composite resin. The latter was applied after acid etching, washing, drying, and applying the conventional adhesive to the enamel. The null hypothesis tested was that there was no significant difference in micro-shear bond strength between the low-viscosity composite resins evaluated on enamel. This study was particularly relevant given the limited data available on the performance of these resins and the existing controversy regarding their adhesion to tooth structure.

2. MATERIALS AND METHODS

The sample size calculation was conducted using GPower software, version 3.1.9.2, developed by the University of Düsseldorf, based on probability distributions associated with the Mann-Whitney test for comparing two groups. With an effect size of 0.8, a Type 1 error (α) of 0.05, and a power analysis (β error) of 0.8, a total of 15 specimens per group were determined as necessary for this study. For specimen selection and preparation, ten clinically healthy bovine lower incisors, devoid of caries, cracks, or

enamel fractures, were chosen. They underwent cleaning with periodontal curettes and a sodium bicarbonate jet, then were stored in a supersaturated 0.1% thymol solution at 4°C in a refrigerator until their utilization. Initially, the roots were separated from the crowns using a 0.10x22mm double-sided diamond blade (KG Sorensen - Cotia- SP - Brazil). Subsequently, each crown was immersed in polyvinyl chloride (PVC) cylinders (15.0mm high and 25.0 mm in diameter) filled with acrylic resin (Clássico Jet, colorless), leaving the vestibular surface exposed. The buccal surfaces of the crowns were then worn down and polished with a polishing machine (Arotec - Aropol 2V200 - Cotia - SP - Brazil) at a speed of 300 rpm to expose and level the enamel. To expose the enamel, sandpaper disks were used in decreasing order of grit: 320, 600, 1,200, and 1,500 μ m (3M Water Sheet Sandpaper), applying each grit for 5 minutes.

All crowns were cleaned with pumice stone (Maquira Dental Group, Sao Jose dos Pinhais, PR, Brazil) and water. The specimens were divided into two experimental groups, each with 5 teeth (n=5), according to the low-viscosity composite resin and substrate treatment (Fig. 1). The enamel-OF group was considered the control group.

Table 1. The composition and manufacturer of the materials were evaluated

Material	Composition	Lot	Manufacturer
Opallis Flow, color A3 (Universal)	72 % silanized inorganic filler made up of microparticles of barium aluminum silicate and nanoparticulate silicon dioxide with a particle size in the range of 0.05 to 5.0 microns. Methacrylic monomers such as TEGDMA, Bis(EMA), Bis(GMA), camphor quinone, co-initiators, preservatives, and pigments.	030822	FGM
Amber sticker	Active Ingredients: MDP (10-methacryloyloxydecyl dihydrogen phosphate), methacrylate monomers, photoinitiators, co-initiators, and stabilizers. Inactive Ingredients: Inert filler (silica nanoparticles) and vehicle (ethanol).		FGM
Yflow SA, color A3	Methacrylate monomers, acid monomers, inorganic fillers, pigments, initiators, and stabilizers.	00010973	Yller Biomaterials SA
Phosphoric Acid (Condac 37)	37% phosphoric acid, colloidal silica, surfactant, and colorant.		FGM

Source: Author

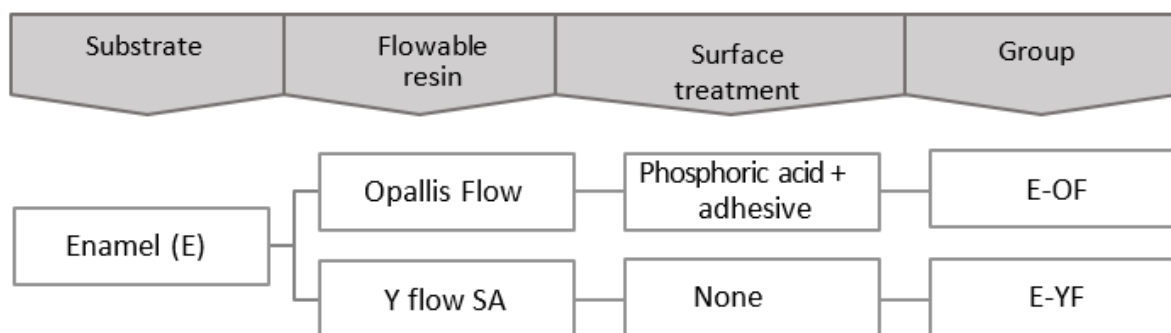


Fig. 1. Experimental groups according to substrate, resin, and surface treatment

All groups received low-viscosity composite resin cylinders, meticulously molded using a Tygon matrix (Tygontubing, TYG-030, Saint-Gobain Performance Plastic, Miami Lakes, FL, USA) with an internal diameter of 3 mm and a height of 2 mm. The resin was carefully dispensed directly into the matrix with the applicator tip of the product syringe in a single increment and photoactivated for 40 seconds with a light-emitting diode device with a power density of 500 mW / cm², which was checked before use with a radiometer. The test specimens (SP) of the E-OF group were made with Opallis Flow low viscosity composite resin, universal shade A3 (FGM, Joinville, SC, Brazil), while the E-YF group used Y flow SA resin, shade A3 (Yllor, Biomaterials AS, Pelotas, RS, Brazil). Before the low-viscosity composite resin was applied, the crowns in the E-OF group underwent surface treatment in the adhesion areas with phosphoric acid gel for 15 seconds. After this, they were washed and dried with absorbent paper, ensuring that the surface was not dehydrated. Using a micro-applicator (Cavibrush, FGM, Joinville, SC, Brazil), the Ambar adhesive, an etch-and-rinse adhesive (FGM, Joinville, SC, Brazil), was applied by rubbing the first drop of the product for 10 seconds. A new layer of adhesive was then applied to the same surface for a further 10 seconds. After applying the adhesive, an air jet was applied for 10 seconds to promote evaporation of the solvent and increase adhesion. The adhesive was then light-cured for 10 seconds before the low-viscosity resin was applied. The adhesive areas of the E-YF group received no prior treatment. After a simple

cleaning with pumice stone and water, the resin was applied directly to the surface.

The excess resin was removed using a scalpel blade. Three specimens were prepared for each bovine crown, allowing for a duplicate test to be conducted, totaling 15 specimens in each group. This was done to eliminate possible biases, and the results of this test were duly recorded.

Before the study commenced, a single operator was thoroughly trained and calibrated to perform all experimental procedures. Calibration encompassed tooth selection, familiarization with composite resin application procedures, specimen preparation, micro-shear bond strength testing, and fracture analysis. Additionally, the operator was instructed to strictly adhere to established protocols to minimize any experimental biases.

After 7 days of storage in distilled water at a constant temperature of 37°C, the PCs were subjected to the micro-shear bond strength test. This test was carried out on a universal mechanical testing machine (EMIC - São José dos Pinhais - PR- Brazil), equipped with a 50 kg load cell.

Shear loading was applied to the base of the cylinders using a 0.2 mm orthodontic wire at a controlled speed of 0.5 mm/min. This process continued until the bond was broken (as shown in Fig. 2A e B). Subsequently, the micro-shear bond strength was carefully calculated and expressed in terms of Newton (N).

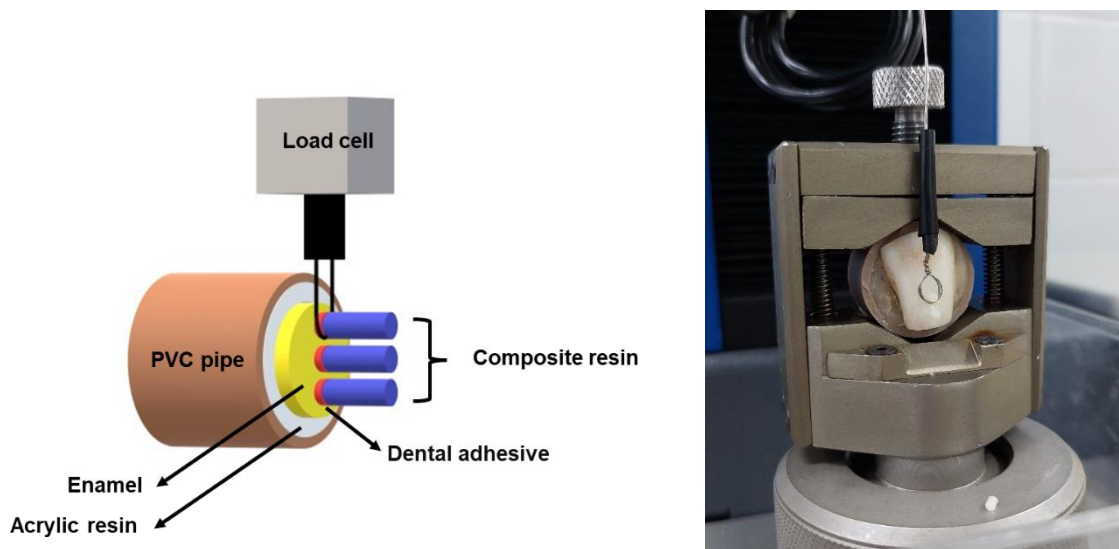


Fig. 2. A. Schematic view of the shear strength test. B. In view of the micro-shear bond strength testing. * In the E-YF group, adhesive application was not performed

Statistical analyses were conducted using Jamovi software (Jamovi, Version 2.3, Computer Software, <https://www.jamovi.org>). To check whether the data for the quantitative dependent variables (i.e. bond strength) followed a normal distribution, the Shapiro-Wilk test was used as the first measure. As the data did not fit the normality curve, the Mann-Whitney U non-parametric analysis of variance was used to determine whether there were significant differences between the groups ($p < 0.001$). This analysis was complemented by the Dwass-Steel-Critchlow-Fligner (DSCF) test to carry out multiple comparisons ($p < 0.001$).

Fractures were assessed in all samples using a stereoscopic magnifying glass at 40X magnification. The fractures were categorized as adhesive, enamel cohesive, dentin cohesive, cement cohesive, or mixed. The results of this classification were then subjected to detailed analysis using descriptive statistics.

3. RESULTS

The median values, together with the first and third quartiles of the micro-shear bond strength of the resins tested, are detailed in Table 2.

Table 2. The median, first quartile, and third quartile of the bond strength of the resin cements evaluated (N) in enamel and dentin

Evaluation	Group	First Quartile	Third Quartile	Median
7 days	E-OF	22.7	35.5	26.8 ^a
	E-YF	1.90	8.64	4.11 ^b

In the column, different letters show that is a statistically significant difference ($p < 0.001$).

Source: Author

The analysis carried out using the Shapiro-Wilk test suggested that the "Opallis Flow" sample, with a p-value of 0.3147, can follow a normal distribution since the p-value is greater than 0.05. On the other hand, the "Y flow SA" sample, with a p-value of 0.0103, does not fit into a normal distribution, as the p-value is less than 0.05.

The Mann-Whitney U test revealed a statistically significant difference between the E-OF and E-YF groups ($p < 0.001$) (Fig. 3).

This resulted in the rejection of the null hypothesis and indicated the existence of significant differences in the values of the groups, even without a normal distribution of the data.

Finally, the Dwass-Steel-Critchlow-Fligner (DSCF) test confirmed the existence of significant differences between the E-OF and E-YF groups. As shown in Table 2, the conventional flowable composite resin (E-OF group) showed a significantly higher micro-shear bond strength than the self-adhesive flowable composite resin (E-YF).

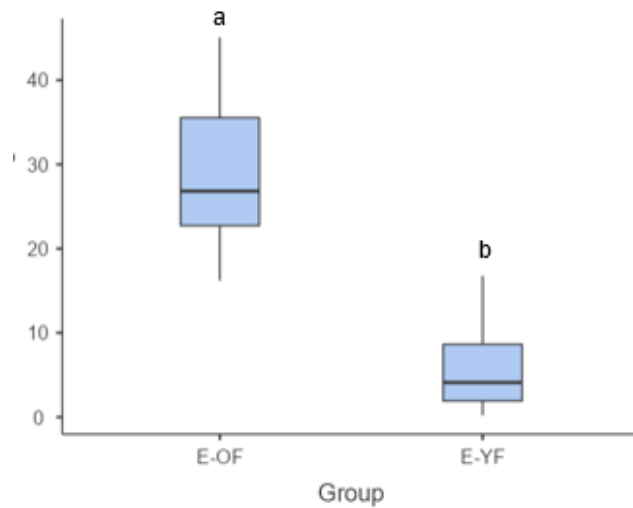
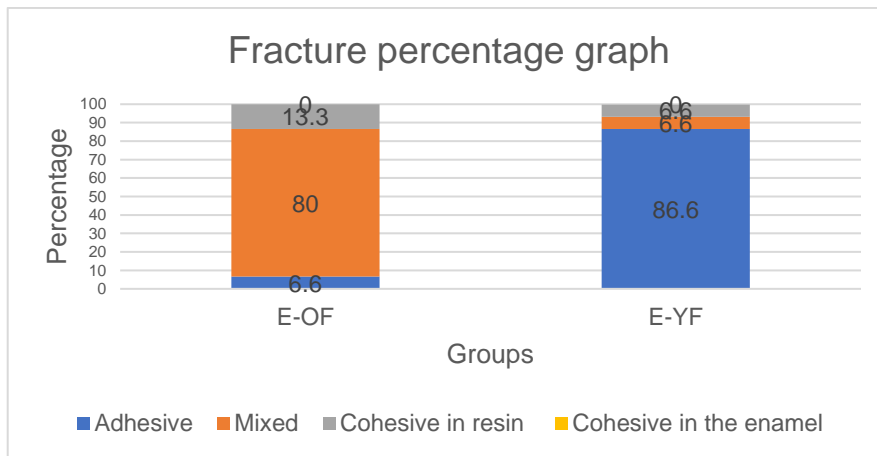


Fig. 3. Box plot of bond strength results. The box represents the dispersion of the data between the first and third quartiles. The central horizontal line represents the median. In each box, vertical lines extend to the minimum and maximum values obtained and different letters indicate that there is a statistically significant difference

Source: Jamovi software



Graph 1. Percentage of fracture modes in the groups

Source: Author

It was observed that the majority of fractures in the conventional flowable composite resin group were mixed fractures, representing 80% of the total in the E-OF group. On the other hand, in the self-adhesive flowable composite resin group, adhesive fractures predominated, accounting for 86.6% of the total in the E-YF group (as shown in Graph 1).

4. DISCUSSION

Given the wide range of clinical applications for which flowable composite resins are used, dentists need to have sufficient comparative information to enable them to select the material with the most appropriate properties for any

specific use [4,5,11,17]. This *In vitro* study yielded significant findings regarding the micro-shear bond strength of self-adhesive low-viscosity composite resins compared to conventional ones. The results of this research pointed to the rejection of the null hypothesis, indicating that conventional low-viscosity composite resins when combined with dentin adhesives, showed higher micro-shear bond strength values than self-adhesive low-viscosity composite resins. These results are consistent with several previous studies cited [2,10,17–21].

However, it is important to note that some studies have found no significant differences between these two types of low-viscosity

composite resins [4,7,22–25]. This suggests that the results may vary depending on the specific characteristics of the self-adhesive composite resins and the substrate used.

Studies show that conventional flowable composite resin, when combined with some type of dentin adhesive - be it total acid etching [2,10,18–21], self-etching [20] or universal - demonstrates superior bond strength values compared to self-etching flowable composite resins [17,18,20]. These results were consistent both in laboratory studies with thermocycler and non-thermocyclergroups [19] and in clinical studies. This is due to acid etching with 37% phosphoric acid and the application of the adhesive system before resin insertion, which promotes effective adhesion [2,10,18,20,21,26].

This process involves the elimination of the smear layer [10,26] and the creation of microporosities in the hydroxyapatite of the enamel [2], followed by the formation of a hybrid layer [26] and resin tags [2, 26, 27], where adhesion occurs exclusively by mechanical retention [2]. Materials recently launched on the market, such as Y flow AS self-adhesive flowable composite resin (Yllor, Biomaterials), use methacrylate monomers in their chemical composition to establish self-adhesion. The polymerization of these materials occurs through covalent bonding, forming linear polymers, which can result in shrinkage of the material [16]. Failures in these restorations can be related to factors such as shrinkage and polymerization stress, which can cause microcracks in the enamel, sensitivity, microleakage, and caries, as well as maladaptation and marginal pigmentation [11].

In general, the results suggest that self-adhesive fluid resins interact with the smear layer of enamel. However, the acidity of these resins is not high enough to alter this layer and allow the resin to penetrate the enamel substrate [26]. The bond strength of these resins is significantly lower [10, 19, 21, 28, 29] in both deciduous and permanent teeth [28]. These results may be related to the high viscosity and low wettability of these restorative materials [20,26–28], since they do not diffuse properly to demineralize the substrate [19], thus reducing the adhesion potential [20,27].

Evaluation of the fracture mode also revealed these differences between the two types of flowable composite resins. In the E-YF group,

which used the self-adhesive resin, adhesive fractures were predominant, accounting for 86.6% of the total. These fractures, which occur at the interface between the adhesive and the substrate, suggest a possible fragility in the bond between the self-adhesive resin and the substrate [30]. In contrast, in the E-OF group, which used conventional resin, mixed fractures were more frequent, accounting for 80% of the total. These fractures indicate failures in both the adhesive interface and the materials (enamel or resin), suggesting a greater resistance to conventional resin at the adhesive interface [30]. These findings may influence the choice of material in clinical applications. For example, if the strength of the adhesive interface is crucial, conventional flowable composite resin may be the most suitable option.

The interest in studying self-adhesive fluid composite resin arises from the fact that it is an innovative material that does not require pre-treatment of the dental substrate or dentin adhesives [4, 11, 20]. Self-adhesive resins were developed to simplify the direct restoration process, reduce the complexity of the technique, and reduce post-operative sensitivity. In addition, they facilitate application and guarantee a reduction in clinical time [11]. Self-adhesive resin composites can be an alternative to glass ionomer-based materials in the restorations of primary teeth [20]. In addition, as it is a resin composite, it has less porosity, resulting in better aesthetics and reduced plaque accumulation [20,26].

It has been observed that the best adhesive strength and margin sealing results were achieved when a dentin adhesive system was applied before the self-adhesive flowable composite resin [29]. Three-step total acid etching adhesives are more effective in this context than self-etching adhesives [19,21]. This is because three-step adhesives remove the demineralization product from the tooth substrate with phosphoric acid, which is not the case with self-etching adhesives [28]. However, the addition of additional operative steps can increase the complexity of the operative technique, counteracting the intended simplicity of self-adhesive resins [20].

Although self-adhesive flowable composite resins have the potential to simplify the process of direct restoration and reduce clinical time, the results of this study indicate that there are still challenges in terms of bond strength when compared to conventional low-viscosity resins.

The main limitations of the study include its laboratory setting, the limited number of samples, and the evaluation of bond strength over a short period of time. Therefore, further long-term laboratory and clinical studies are needed to fully assess the clinical advantages and limitations of these innovative materials in the market.

5. CONCLUSION

Based on this *In vitro* study carried out, it can be concluded that the self-adhesive fluid resin showed lower bond strength values to enamel than the conventional fluid resin.

CONCENT AND ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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