

# Shear-Bond Strength of a New Self-Adhering Flowable Restorative Material to Dentin of Primary Molars

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**Objective:** The aim of this study was to evaluate the shear bond strength to dentin of primary molars of a new self-adhering flowable resin composite with two ionomer-based cements and one flowable resin composite in combination with two different adhesive systems. **Study design:** Fifty primary molars were grinded on the occlusal surface to obtain flat dentin substrate and randomly divided into 5 groups (n=10): OFL: Phosphoric Acid/Optibond FL/Premise Flow; OAO: Optibond All-In-One/Premise Flowable; II: Polyacrylic Acid/Fuji II; IX: Polyacrylic Acid/Fuji IX; V: Vertise Flow. Cylinders (3mm diameter – 5mm height) of restorative material were built-up in three increments over the dentin surfaces. A shear load was applied until failure. Bond strength values were statistically analysed with Kruskal-Wallis ANOVA followed by Dunn's test ( $P < .05$ ). Differences in failure mode distribution were assessed with Chi-square ( $P < .05$ ). **Results:** OFL and OAO recorded significantly higher bond strengths than the other groups. Adhesive failures were evident in all groups except OFL and OAO, in which also cohesive failures in dentin were observed. **Conclusions:** Vertise Flow established on primary dentin bond strengths values similar to those of glass ionomer cements routinely used for restorations of primary teeth. The combination of flowable resin with etch-and-rinse or all-in-one adhesives obtained higher bond strength values, thus involving a more complex handling.

**Keywords:** Pediatric Dentistry, Dental Bonding, Composite resin, Glass Ionomer Cements

## INTRODUCTION

Preservation of primary teeth is important for the management of the developing dentition and in nurturing a positive attitude in children toward dental health.<sup>1</sup> Restorations of primary

teeth need to be durable as they are usually performed in early ages.<sup>2</sup> Such restorations are usually performed using composite resin (CR)<sup>3</sup> or glass ionomer cement (GIC). For the clinical success of CR, an effective bond between dental materials and tooth substrates is critical. The adhesive procedure preliminary to CR application involves conditioning/etching, priming, and bonding of the substrate. These three functions can be implemented separately (4th generation dental adhesives), combined<sup>4</sup> into two steps (5th and 6th generation dental adhesives) or rather in one step (7th generation). Fourth and fifth generation adhesives are based on the etch-and-rinse approach, implying complete removal of the smear layer and demineralization of the subsurface intact dentin via acid etching. Conversely, in the self-etch approach (6th and 7th generation), the self-conditioning primer or primer-adhesive is applied to smear layer-covered dentin, and acidic resin monomers simultaneously demineralize and infiltrate the substrate.<sup>5</sup> Since the introduction of 5th generation dentin adhesives, it has been shown that dentists prefer simpler adhesive systems.<sup>6</sup> In addition, the overall reduction in the application steps is expected to reduce the probability of handling mistakes.<sup>7</sup> This aspect is particularly relevant in pediatric dentistry, where less time-consuming procedures are desirable,<sup>3</sup> especially with uncooperative children. The simplification enabled by self-etch adhesives, not requiring acid conditioning and water rinsing steps,<sup>7,8</sup> is therefore a clinical advantage for the restoration of primary teeth. The other material conventionally used in pediatric restorative dentistry, the GIC, was firstly introduced in 1972 as the product of the reaction between ion-leachable glass and an aqueous solution of polyacrylic acid.<sup>9</sup> Later, resin monomers were added to conventional GICs<sup>10</sup> to obtain resin-modified glass ionomer cements (RMGICs), with improved handling characteristics and color matching.<sup>11</sup> Ionomer-based materials are appreciated for their chemical adhesion to

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**Table 1.** Composition, batch numbers and the application modes of the materials used in the study.

Materials	MANUFACTURER	Composition	Batch	Application mode
Gel Etchant	<i>Kerr, Orange, CA, USA</i>	37,5% orto-phosphoric acid, silica thickener	# 3213200	Apply for 15 seconds; rinse with water for 5 seconds; gently air dry for 5 seconds.
OptiBond FL	<i>Kerr, Orange, CA, USA</i>	<i>Kerr Gel Etchant: 37. 5% H<sub>3</sub>PO<sub>4</sub> (pH 0.1)</i> <i>Primer: HEMA, GPDM, MMEP, water, ethanol, CQ, BHT (pH 1.9)</i> <i>Adhesive: Bis-GMA, HEMA, GDMA, CQ, ODMAB, Filler (fumed SiO<sub>2</sub>, barium aluminoborosilicate, Na<sub>2</sub>SiF<sub>6</sub>), coupling factor A174 (approximately 48 wt.% filled) (pH 6.9)</i>	# 3220025	Apply Primer with light scrubbing motion for 15 seconds; gently air dry 5 seconds; apply Adhesive; light application of air; light cure for 20 seconds.
OptiBond All-In-One Unidose	<i>Kerr, Orange, CA, USA</i>	GPDM, GDM, HEMA, Bis-GMA, water, ethanol, acetone, silica filler, CQ, sodium hexafluorosilicate (pH=2. 5)	# LB01754	Apply two consecutively layers with scrubbing for 20 s; gently air-dried for 5 s; light-cured for 10 s.
Premise flowable	<i>Kerr, Orange, CA, USA</i>	PPF, barium glass, silica filler, ethoxylated bis-DMA, TEGDMA, light-cure initiators and stabilizers, organophosphate dispersant	# 3221664	Apply 2 mm thick layer; light cure for 20 seconds
Cavity Conditioner	<i>GC, Tokyo, Japan</i>	20% polyacrylic acid , 3% Aluminium chloride hexahydrate (pH 1. 2)	# 0906101	Apply for 10 seconds using a cotton pellet.
Fuji II LC	<i>GC, Tokyo, Japan</i>	<i>Powder: fluoro-alumino-silicate glass;</i> <i>Liquid: polyalkenoic acid, HEMA, dimethacrylate camphorquinone, water (pH 1. 3)</i>	# 0803121	Vibrating the capsule 10 s; apply one layer; light-cure for 20 s.
Fuji IX	<i>GC, Tokyo, Japan</i>	<i>Powder: Fluoro-alumino-silicate glass, polyacrylic acid power</i> <i>Liquid: Polyacrylic acid, polybasic carboxylic acid, distilled water (pH 1. 9)</i>	# 0807101	Vibrated for 10 seconds; apply one layer; auto-cure for 5 min
Vertise Flow	<i>Kerr, Orange, CA, USA</i>	GPDM; Prepolymerized filler, 1-micron barium glass filler, nano-sized colloidal silica, nano-sized Ytterbium fluoride.	# 3172311	Apply first layer of 0,5 mm with light scrubbing motion for 20 s; light-cure for 40 s; consecutively applying maximum 2 mm thickness layer singularly cured for 40 s.

Abbreviations: HEMA (hydroxyethylmethacrylate), GPDM (glyceroldimethacrylate dihydrogen phosphate), PAMM (phthalic acid monoethyl methacrylate), TEGDMA (triethylene glycol dimethacrylate), UDMA (urethane dimethacrylate), bis-GMA (bisphenol-glycidyl methacrylate), PPF (prepolymerized filler), bis-DMA (bis-phenol-A-dimethacrylate), CQ (camphorquinone)

tooth structure, for their fluoride release and for being less sensitive to moisture contamination than CRs. Such properties are particularly useful in pediatric dentistry.

A recent innovation in adhesive dentistry is the introduction of a self-adhering flowable composite resin that does not require preliminary treatment of the dental substrate. This simplification in the restorative procedure can be particularly relevant in pediatric dentistry.

Currently, data on the bonding potential of the new self-adhering composite resin is reduced. Most of the information so far available on the bonding mechanism and performance of this new material is that provided by the manufacturer. Therefore, independent studies are required to thoroughly investigate its bonding potential. For laboratory assessment of bond strength, shear, tensile, and micro-tensile test are the most commonly used methods.<sup>6</sup>

As no information are at present available on the bonding performances of this newly introduced self-adhering flowable composite on primary teeth, the aim of this study was to evaluate using a shear bond strength test, the adhesive potential on primary dentin of this

material in comparison with bonding/CRs and GICs.

The tested null hypothesis was that no statistically significant differences in shear bond strength to primary dentin exist among a new self-adhering flowable composite, ionomer-based restorative materials, etch-and-rinse three-step adhesives, and all-in-one adhesives. The latter adhesives were used in combination with the proprietary flowable resin composite.

**MATERIALS AND METHOD**

Fifty human sound primary molars were collected for the study at natural exfoliation time. Teeth were used for the study after having obtained the written informed consent of the donors’ parents. The samples were thoroughly cleaned and stored in 1% Chloramine T solution at 4°C to prevent bacterial growth until used for investigation. Teeth were randomly divided into five groups (n=10). A dental student that was blinded to the objective of the study placed to his liking the teeth in 9 different containers. Each container provided the 10 specimens for each group.

**Table 2.** Descriptive statistics of Shear Bond Strength test values and statistical significance of between-groups differences (Kruskal-Wallis Non-Parametric and Dunn’s Multiple Range test). In the significance column different letters label statistically significant between-group differences ( $p < 0.05$ ).

Group	Material	N	Mean (MPa)	sd (MPa)	Median (MPa)	25%-75%	Significance
A	Optibond All-in-One/Premise Flow	10	16,59	1,77	16,84	15,61-18,08	a
B	Phosphoric Acid/Optibond FL/Premise Flow	10	16,02	4,80	17,95	15,48-18,41	a
C	Polyacrylic Acid/Fuji II	10	5,91	3,15	5,81	4,86-6,93	b
D	Polyacrylic Acid/Fuji IX	10	6,04	3,76	4,85	4,0-6,82	b
E	Vertise Flow	10	4,31	2,66	3,51	3,29-6,27	b

**Group A:** 37% Phosphoric Acid (Gel Etchant Kerr, Orange, CA, USA) / Optibond FL (Kerr, Orange, CA, USA) / Premise Flowable (Kerr, Orange, CA, USA)

**Group B:** Optibond All-In-One Unidose (Kerr, Orange, CA, USA) / Premise Flowable

**Group C:** Fuji II LC Capsule (GC Corp. , Tokyo, Japan)

**Group D:** Fuji IX GP Fast Capsule (GC Corp. , Tokyo, Japan)

**Group E:** Vertise Flow (Kerr, Orange, CA, USA).

Table 1 provides a detailed list and procedure details of the materials used in the different Groups. Routine prophylactic procedure was carried out with a rubber cup and an aqueous slurry of pumice for all the teeth before the bonding procedure was performed. Each tooth was then embedded with self-cure acrylic resin (Palavit 55 VS, Haereus Kulzer, Hanau, Germany) in a cylindrical steel mould. On teeth used for dentin bond strength testing, a flat dentin surface was created by grinding the occlusal surface with a 320-grit silicon carbide abrasive paper (South Bay Technology Inc., San Clemente, CA, USA; batch # 21157), using the Labpol 8-12 polisher (Benetec Limited, Leicester, UK) under water-cooling for 30 s at 150 rpm. The teeth were ground at the occlusal surface to such a depth as to expose a flat dentin surface large enough to accommodate the 3 mm diameter cylindrical mould used for build-up of restorative material. The materials were handled strictly according to manufacturers’ instructions for all groups. Light curings were performed with a conventional quartz–tungsten–halogen light (VIP; Bisco, Schaumburg, IL, USA; output 600 mW/cm<sup>2</sup>). Capsule vibrations were assessed with RotoMix (2850 rpm, 3M ESPE, Seefeld, Germany). All the bonded specimens were then stored in an incubator for 24 h at 37°C and 100% humidity before testing.

**Shear Bond Strength Test**

Shear bond strength test was performed according to ISO/TS 11405:2003.<sup>12</sup> Each bonded specimen was positioned in a universal testing machine (Triax Controls, Milano, Italy). A shear load was applied with a steel blade in a direction parallel to the bonded interface at a crosshead speed of 1 mm/min until failure occurred. The load at failure was recorded in Newtons (N) using a 25 Kg loading cell. Bond strength was calculated in MegaPascals (MPa) by dividing the load at failure by the adhesive surface area (mm<sup>2</sup>). For this purpose the diameter of the cylindrical build-up of restorative material had been measured prior to loading using a digital caliper (Orteam s. r. l, Milan, Italy) with an accuracy of 0.01 mm.

The failure mode of each specimen was independently assessed by two experienced investigators using a stereomicroscope (Nikon

SMZ645, Tokyo, Japan) at 40x magnification and classified according to the following criteria:

1. Adhesive failure at the interface between dental substrate and adhesive material.
2. Cohesive failure within dental substrates.
3. Mixed failure: adhesive and cohesive fractures occurred simultaneously.

Differences in scoring between the evaluators were discussed until a final score was reached on consensus.

**Statistical Analysis**

As the bond strength data were not normally distributed (Kolmogorov-Smirnov test), the Kruskal-Wallis Analysis of Variance (ANOVA) was applied to assess the statistical significance of between-group differences. The Dunns’ Multiple Range test was applied for post hoc comparison.

Differences among Groups in the distribution of failure modes were assessed with the chi-square test. The level of significance was set at  $\alpha = 0,05$  for all the analyses.

**RESULTS**

Descriptive statistics of shear bond strength data are reported in Table 2, along with the statistical significance of between-group differences. Table 3 illustrates the distribution of failure modes in the experimental groups, along with the statistical significance of between-group differences.

**Shear Bond Strength**

Statistically significant differences in bond strength to primary dentin emerged among the tested materials ( $P < .001$ ). The etch-and-rinse and the self-etch adhesive used in combination with the proprietary flowable composite measured significantly higher bond strength values than all the other tested materials. The bond strengths achieved by the self-adhering flowable composite were statistically comparable to those yielded by the conventional glass-ionomer and the resin-modified glass ionomer restorative materials.

Statistically significant differences among the tested materials also emerged in the distribution of failure modes ( $P = .002$ ). Only the adhesives used in combination with the proprietary flowable composite (Optibond FL and Optibond All-in-One) produced cohesive failures within dentin. With ionomer-based cements and the new self-adhesive flowable composite exclusively adhesive failures were recorded.

**Table 3.** Failure modes (A= adhesive failure; C= Cohesive failure). Groups A-B show nearly half of cohesive failure within the dentin substrate. Vertise Flow self adhering flowable resin and GICs show 100% adhesive failures. Statistical significance of between-group differences in failure modes distribution was assessed with the chi-square test. In the significance column different letters label statistically significant between-group differences ( $p < 0.05$ ).

Group	Failure Mode	N	A	C	Significance
A	Phosphoric Acid/Optibond FL/Premise Flow	10	60%	40%	a
B	Optibond All-in-One/Premise Flow	10	50%	50%	a
C	Polyacrylic Acid/Fuji II	10	100%	0%	b
D	Polyacrylic Acid/Fuji IX	10	100%	0%	b
E	Vertise Flow	10	100%	0%	b

## DISCUSSION

As statistically significant differences emerged among the tested groups in both shear bond strength to dentin and failure modes distribution, the null hypothesis formulated has to be rejected.

The two restorative systems commonly used for the restoration of primary teeth, the combination of Bonding/Composite Resin and the Glass Ionomer Cements, differ considerably in the adhesion path to the dental substrates. For the combination bonding/resin composite (B/CR), the fundamental principle of adhesion is the process of exchanging inorganic tooth material with synthetic resin<sup>13-16</sup>. This process requires two steps, the first consisting of removing calcium phosphates, by which microporosities are exposed on the dentin surface<sup>16</sup>, and the second consisting of infiltration and polymerization of resin in the produced micro-porosities. Even if diffusion is a key mechanism for micro-mechanical interlocking<sup>16</sup>, chemical interaction between functional monomers and tooth substrate components has a fundamental importance, especially when self-etching adhesives or self-adhesive flowables are used. GICs adhere to dentin through chemical interaction and micro-mechanical interlocking<sup>17</sup>. Polyalkenoic acid from the cement is buffered by phosphate ions within the tooth and calcium and phosphate ions are dispersed from the hydroxyapatite into the unset cement<sup>18,19</sup>. Ions are also able to disperse from the cement into the dentin, resulting in an intermediate layer between the unset GIC and hydroxyapatite referred to as the "ion exchange layer"<sup>20</sup>. Ferrari and Davidson<sup>21</sup> have interpreted this as a hybrid layer of approximately 6  $\mu\text{m}$  in thickness. The bonding mechanism of Vertise Flow, the new self-adhering composite resin tested in the present study as an alternative to the two formerly described restorative systems, is twofold. Primarily it is a chemical bond between the phosphate functional group of GPDM (glycerol phosphate dimethacrylate) and calcium ions of the tooth. Secondly, a micromechanical bond results from an interpenetrating network between polymerized monomers of Vertise Flow and the partially demineralized smear layer. Vertise Flow has a pH of 1.9, similar to that of several self-etching materials<sup>22</sup>, and is claimed to be effective in demineralizing the substrate. Subsequently, an inter-diffusion zone is created as a result of monomers penetration among partially demineralized collagen fibers<sup>22</sup>. After polymerization, the pH is claimed to rise and to get close to neutrality.

In the present study, the B/CR groups achieved the highest bond strength values on primary dentin. This is not surprising, as higher bond strength values are systematically reported for B/CR systems when compared with GICs<sup>23</sup>. No statistically significant differences were found between the two B/CR groups (etch-and-rinse and all-in-one), in agreement with the study of Krifka *et al.*<sup>24</sup> To

explain this findings, that differ from the usually reported higher bond strength values for etch-and-rinse systems when compared with all-in-one, the peculiarity of the primary dentin has to be taken into account. Generally adhesive systems achieve lower bond strength to primary than to permanent dentin<sup>25</sup>. This difference has been related to the amount of mineral components in primary teeth<sup>6</sup>, as well as to morphological and structural differences<sup>24,26</sup>. Courson *et al.*<sup>27</sup>, through neutron activation analysis and dispersive spectroscopy, reported that calcium and phosphorous concentrations in peritubular and intertubular dentin of primary teeth are lower than in permanent teeth, with possible effects on bond strength to primary dentin. Furthermore, due to the lower thickness of primary dentin, even a limited tissue removal is likely to expose dense and wider dentin tubules in proximity of the pulp<sup>28</sup>. It has been reported that higher tubule density, larger tubule diameters, and higher prevalence of microcanals in primary dentin as compared with permanent teeth will interfere with the adhesion process yielding lower bond strength values<sup>29</sup>. Even the etching process behaves differently in primary teeth in comparison with permanent teeth. Burrow *et al.*<sup>23</sup> reported that primary teeth were more demineralized than the permanent counterpart. Uekusa *et al.*<sup>25</sup> observed that the peritubular dentin was demineralized more rapidly during acid treatment. They also noted that acids used to condition the dentin surface removed smear layer more rapidly from primary teeth than from permanent teeth. Thus, they concluded that either shorter conditioning time or the use of weaker acid solutions should be considered for primary teeth. Araujo and Garcia Godoy<sup>30</sup> suggested that a reduced demineralization for dentin of primary teeth could lead to a thinner hybrid layer, and be sufficient for a complete penetration of the resin material. Asakawa *et al.*<sup>31</sup> in a study limited to all-in-one adhesives on primary teeth, showed that demineralization, diffusion, and embedding of the bonding-system around the dentinal collagen was the same as in permanent teeth. These observations lead to the idea that a less acidic material, like an all-in-one adhesive, could obtain similar values in primary as well as in permanent teeth, while a more aggressive acidic material, like PA used in etch-and-rinse system, could be aggressive for primary dentin, limiting the effectiveness of the bonding process.

In the present investigation, the bond strength achieved by GICs was modest and significantly lower than that of B/CR combinations. This is in agreement with the data usually reported for such materials<sup>32</sup>. However, the fluoride release, the self-adhesive capability, and their easy of use still make GICs useful in the restoration of primary teeth<sup>33</sup>.

The self-adhering restorative material tested, the Vertise Flow, achieved bond strength comparable to that of GICs, yet lower than



that of B/CR systems. This finding is somehow unexpected, as the self-adhering property of the material and the low acidity should be a favourable assumption for bond strength values on primary teeth. A possible explanation for this result could be found in the wettability properties of the material. The adhesion to the tooth substrate, whichever material is used, is in fact influenced by the wettability of the material itself<sup>34</sup>. Favorable wettability of the dental surface by a bonding solution entails a close adhesive-dentin interaction, which contributes to enhanced dental adhesion, both for etch-and-rinse and for self-etch adhesives.<sup>35</sup> Chemical interaction is promoted for self-adhesives restorative material by close contact between substrate and adhesive material and is favored by wettability of the latter.<sup>36</sup> Self-adhesive restorative materials, like Vertise Flow, are more viscous and consequently have lower wettability,<sup>37</sup> that could limit the bonding potential. Nevertheless, Vertise Flow can still be considered as an alternative to GICs for the restoration of primary teeth. The bond strength values achieved by the two materials in the present study are comparable, and both materials can be considered to have a simplified handling. Vertise Flow does not provide the advantage of fluoride release, but an higher wear resistance than GICs could be expected, due to its filler content. This aspect is particularly important when restorations have to be performed on early ages in teeth that will be exposed for some years to functional load. Moreover, as Vertise is a Flowable composite resin, a less porous surface than with GICs, can be obtained. With the benefits of improved aesthetics and lower plaque accumulation. All these aspects could be further investigated with specific *in vitro* protocols, as well as with *in vivo* studies.

Within the limitation of this *in vitro* study, the finding that the new self-adhering flowable resin composite Vertise Flow achieved bond strength values similar to GIC, encourages the use of Vertise Flow for primary teeth restorations. Further *in vitro* studies involving aging methods, marginal seal assessment, as well as clinical trials should be performed to further investigate the performance of this material when used in pediatric dentistry.

## CONCLUSION

When tested on primary dentin, the etch-and-rinse adhesive/flowable resin composite combinations showed higher bond strength values. However, a more complex handling was involved for this procedure. The new self-adhesive flowable composite Vertise Flow achieved bond strength values which were similar to those of glass ionomer-based restorative materials that have been routinely used for primary teeth restorations.

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