CAN THE SELF-ADHESIVE FLOWABLE COMPOSITE BE SECURELY BONDED TO PRIMARY TOOTH ENAMEL?

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This study evaluated the shear bond strength of self-adhesive flowable composite when used with acid etching or a self-etch adhesive system. Buccal enamel surfaces on 80 extracted human primary incisors were used and randomly assigned into four groups (n=20); group 1: Vertise™ Flow; group 2: Vertise Flow with acid etching; group 3: Vertise Flow with a self-etching bonding agent; group 4: Premise Flowable™ with a total-etch bonding agent as the control group. After 5000 rounds of thermocycling, the bond strength test was performed using a universal testing machine at a crosshead speed of 0.5 mm/min. The fracture analyzes of samples were evaluated using a light stereomicroscope. The results obtained were analyzed via analysis of variance (ANOVA) and Tukey's tests. Statistically significant differences were observed among all groups (P<0.001). Group 1 generated a lower mean shear bond strength (2.63±1.08 MPa) than those of the other groups [group 2 (7.52±2.14 MPa), group 3 (5.12±2.93 MPa), and group 4 (14.18±2.93 MPa)]. Adhesive failure was the most common failure mode in Vertise Flow groups. Vertise Flow used with a self-etching agent or acid etching exhibited in lower shear bond strength than the control group.

Keywords: *Dental materials, Primary teeth, Self-adhesive flowable composite, Shear bond strength, Vertise™ flow*

1. Introduction

The science of adhesive dentistry is in continuous development. Most dentists are still using the conventional etch-and-rinse adhesive approach developed by Buonocore [1,2]. However, the self-etch adhesives substantially reduces application time as well as technique sensitivity because they do not require a rinse phase [3]. This approach to the tooth surface includes the simultaneous infiltration of resin monomers into demineralized areas created by the acid in the enamel and the chemical interaction of functional monomers contained in the adhesive with residual hydroxyapatite crystals [3,4].

First generation flowable composites (FCs) which for especially use in Class V restorations were introduced in 1996. FCs have been used as a restorative material in young patients for many indications. These indications are available in low-stress applications and in situations where access is difficult or good penetration is required, and the indications that can be highlighted for these materials can be listed as follows: applications in minimally invasive occlusal restorations, pit and fissure sealant applications, splinting fractured and mobile teeth, bonding process of brackets and space maintainers to the teeth surface, and as liner or base materials under composite resin restorations in extensive Class I or II restorations [5,6].

The seek for faster and simpler restorative procedures promote the effectiveness of treatment in dentistry and may be important also in the paediatric dentistry [7]. Lately, modern self-adhesive FC resin systems have been introduced to the restorative dentistry. New self-adhesive composite resin systems can be bonded directly enamel or dentin, eliminating intermediate steps such as etching, rinsing, and bonding [8,9]. There is some disagreement in the literature about the bonding efficacy of self-etch systems to the sound enamel [10]. Since the FCs have become an integral part of the numerous clinical applications, dentists should have adequate comparative information so that they can select the flowable materials with the most appropriate properties for any restorative process [11].

Previous studies reported that the bond strength of resin restorations to primary teeth is lower compared to permanent teeth due to different physiological, morphological, and chemical properties between primary and permanent teeth [12-14]. Although the bonding strengths of self-adhesive flowable composite (SAFC) to permanent teeth, when applied with different bonding protocols, are evaluated in the literature [13,15-17], there are limited- data available related to the bond strengths of SAFCs in the primary teeth. The objective of this in vitro study was to investigate the shear bond strength (SBS) of SAFC to primary teeth enamel with different bonding protocols. The null hypothesis tested is that SBS of VF groups to enamel, do not differ significantly from the control group.

2. Materials and Methods

One SAFC (Vertise™ Flowable; Kerr Dental, Orange, CA, USA) and one conventional FC (Premise™ Flowable; Kerr Dental, Orange, CA, USA) were examined in this study. Their compositions and the application instructions of the using materials in the study are listed in Table 1. A power analysis set by using G*Power statistical

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Table 1

Compositions and the application modes of the materials used in the study.

Material	Composition [lot number]	Application Mode
Gel Etchhant	37.5% ortho-phosphoric acid, silica thickener. [3353342]	Apply for 15 seconds; rinse with water for 15 seconds; gently air dry for few seconds.
OptiBond FL	Primer: HEMA, GPDM, PAMM, ethanol, water, photo initiator. [3457744] Adhesive: TEGDMA, UDMA, GPDM, HEMA, Bis-GMA, filler, photo initiator. [3461592]	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	GPDM, HEMA, self-etching adhesive monomer, co-monomers including mono and di-functional methacrylate monomers, water, acetone, ethanol, camphorquinone-based photo-initiator system three nano sized fillers, fluoride-releasing fillers, sodium hexafluorosilicate and ytterbium fluoride. [2894473]	Apply first application with scrubbing motion for 20 seconds. Apply second application with scrubbing motion for 20 seconds. Air dry gently, then air dry with medium force for 5 seconds. Light-cure for 10 seconds
Premise flowable	Matrix: Bis-EMA, TEGDMA, initiators, and stabilizers. Fillers: 84% by weight. Prepolymerized filler (30 to 50 µm), barium glass (0.4 µm), and silica nanoparticles (0.02 µm). [3044072]	Apply 2 mm-thick layer; light cure for 20 seconds.
Vertise Flow	Matrix: GPDM, HEMA, methacrylate co-monomers. Fillers: 70% by weight. Prepolymerized fillers (20 µm); nano-sized ytterbium fluoride (40 nm); barium glass filler (0.7–1.0 µm); colloidal nanosillica (10–40 nm). [3566527]	Dispense first layer less than 0.5 mm thick; brush with moderate pressure for 15-20 seconds; light cure for 20 seconds; apply additional layer 1.5 mm-thick; light cure for 20 seconds.

Abbreviations: HEMA, 2-hydroxyethyl-methacrylate; GPDM, glycerol-phosphate dimethacrylate; PAMM, phthalic acid monoethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate

package, Version 3.1.3 (Franz Faul Universität Kiel, Germany), according to an equal ratio between groups and a sample size of 80 teeth would supply over 85% (actual power =0.8641) power to determine significant differences with 0.35 effect size and at the α=0.05 significance level.

Eighty freshly extracted caries-free primary human incisors without cracks, fractures, and hypoplasia were used in this study. After approval from the institutional ethics review board (20/22-15), the extracted teeth were stored in 0.5% Chloramine T solution at 4°C until they were used in the experiment. Surface debris and contaminants were cleaned with a scaler, and then the specimens were polished with pumice to attain a flat surface. The surface of the teeth was rinsed and then dried with an air-water syringe. The root fragments of the teeth were removed with water-cooled high-speed diamond burs and then coronal fragments were embedded in an acrylic cylinder with leaving the buccal enamel surfaces facing up. The teeth were randomly divided into four groups in this study (n=20): Group 1: Vertise Flow (VF) without acid etching; Group 2: VF with acid etching; Group 3: VF with a self-etching bonding agent; Group 4: Premise Flowable with total-etch bonding agent as control group. The detailed bonding protocols used in the three experimental groups and the control group were applied according to the manufacturers' instructions:

Group 1: Vertise™ Flow (VF).

A cylindrical-shaped plastic matrix (Ultradent, South Jordan, UT, USA) with an internal diameter of approximately 3 mm and a height of 2-mm was placed on the buccal enamel surface of each specimen. VF was applied to matrices in a thin layer (<0.5mm). At first, the implemented thin layer of material was brushed with moderate pressure for 15–20 s. It has been stated by the manufacturer that the brushing action is crucial to the effectiveness of the VF bonding mechanism. Additional material was applied in increments of less than 2 mm and than light cured each increment for 20 s using a light-emitting diode (LED) curing light (Elipar Free Light 2, 3M ESPE Dental Products, St. Paul, MN, USA).

Group 2: Acid etch in combination with VF.

The teeth were initially etched with 37.5% phosphoric acid (Kerr Gel Etchant, Orange, CA, USA) during 30 s, rinsed with a water spray for 10 s and air dried for 5 s. The VF was placed and light-cured as same as group 1.

Group 3: Self-etch adhesive Optibond™ All-in-one in combination with VF

Optibond™ All-in-one self etch adhesive system (Kerr, Orange, CA, USA) was applied to the enamel surface with an applicator for 20 s, the surface was air dried for 5 s and light curing was performed using a LED curing light for 10 s. The VF was placed and light-cured as in group 1.

Group 4: Total-etch adhesive OptiBond FL in combination with the flowable composite Premise flowable as control.

The teeth were conditioned with 37,5% phosphoric acid for 30 s at first, rinsed with a water

spray for 10 s and then air dried for 5 s. OptiBond ™ FL Primer (Kerr, Orange, CA, USA) was applied to enamel surfaces in a thin layer with a soft brushing motion using the applicator and then air dried for 5 s. OptiBond ™ FL Adhesive (Kerr, Orange, CA, USA) was applied to the enamel surface by soft brushing movements with an applicator for 15 seconds and then polymerized using an LED light source for 10 s according to manufacturer's instructions. Premise Flowable was applied to the buccal enamel surfaces, not exceeding a maximum filling level of 2 mm and then polymerized with LED for 20 s. After sample preparation completed, all specimens were stored in distilled water at 37°C for 24 h and then were submitted to a thermal cycling procedure (5000 cycles, 5–55°C) with a duration of 30 s at each temperature.

2.1. Shear Bond Strength Test

Following thermocycling, the specimens were subjected to shear loading in a direction parallel to the composite-tooth interface at a speed of 0,5 mm/min using the universal testing machine (Hounsfield Test Equipment, Salford, Lancashire, UK). Shear force was performed until failure occurred and to state recorded values in MegaPascals (MPa), the maximum fracture load recorded in Newton (N) was divided by the cross-sectional area of bonded interface (mm^2).

2.2. Fracture analysis

Failure modes analysis were specified by examination of all debonded surface by a single operator with a stereomicroscope (SZ 40, Olympus, Tokyo, Japan) at ×20 magnification. Fracture modes were assessed as an adhesive failure if less than 20% adhesive remained on the dental substrate (enamel), as a cohesive failure if more than 80% adhesive remained on the dental substrate (enamel), and as a mixed failure if adhesive and cohesive fractures occurred simultaneously. Samples were randomly selected from each fracture mode for SEM observation at ×300 magnification.

2.3. Statistical analysis

In all the analyses, the level of significance was set at $p < 0.05$ and calculations were conducted with the Statistical Package for Social Sciences version 20.0 (SPSS Inc, Chicago, IL). Bond strength values were analyzed by the Levene test ($p<0.05$) and the Shapiro-Wilk test ($p <0.05$) for normality of data distribution and homogeneity of group variances. Parametric tests were used because the test results showed normal distribution and homogeneous group variances. One-way analysis of variance (ANOVA) was applied to statistically compare the SBS data of each group, and then the Tukey test was used for post hoc comparisons. Statistically significant differences between the

Table 2

Shear bond strengths: descriptive statistics and intergroup comparison.

Groups	N	Mean	SD	Min-Max	ANOVA	Post-Hoc
SAFC without acid etching	20	2.63	1.08	0-4.12		D
SAFC with acid etching	20	7.52	2.14	2.32-9.87	p<0.001	B
SAFC with self etching	20	5.12	1.30	2.31-8.78	F=148.652	C
Control with Premise Flowable	20	14.18	2.93	9.54-18.46		A

N sample size, SD standard deviation, Min minimum, Max maximum. Groups with different letters differ significantly from each other.

Table 3

Patterns of shear bond failure. Fracture modes after testing shear bond strength

Groups	N	Adhesive	Cohesive	Mixed	P
SAFC without acid etching (A)	20	20 (100%)	0	0	
SAFC with acid etching (B)	20	12 (60%)	2 (10%)	6 (30%)	
SAFC with self etching (C)	20	16 (80%)	0	4 (20%)	p<0.001
Control with Premise Flowable (D)	20	5 (25%)	8 (40%)	7 (35%)	

Groups with different letters differ significantly from each other.

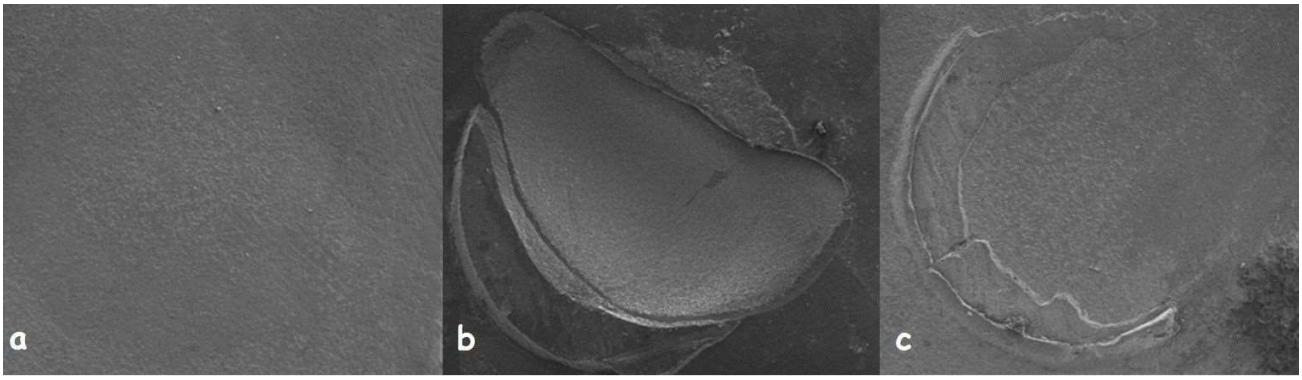

Fig. 1 - Representative SEM micrographs of fracture modes (×200 magnification): (a) adhesive failure, (b) cohesive failure, (c) mixed failure.

failure modes of the subgroups were evaluated using χ2-test ($p < 0.05$).

3. Results and Discussion

Table 2 shows the mean and standard deviations of SBS value, and statistical comparison of groups. The highest mean SBS value was recorded for group 4 in primary teeth (14.18±2.93 MPa), whereas the lowest value was recorded for group 1 in primary teeth (2.63±1.08 MPa), and group 2 had a mean SBS (7.52±2.14 MPa) that was higher than that of groups 3 (5.12±2.93 MPa). Statistically significant differences were detected among all groups in the mean SBS values according to the ANOVA test ($p<0.001$, $F=148.652$).

The distribution of fracture patterns for the analyzed specimens is shown in Table 3. The Chi-square test detected statistically significant differences in failure modes among analyzed groups ($p<0.001$). According to the failure mode analysis, significantly more cohesive fractures were detected in the control group compared to all SAFC groups. After shear loading in all the SAFC groups (group 1-3), adhesive fracture modes (fracture site at the composite-tooth interface) were observed more frequently than cohesive (fracture site in the composite material) and mixed fracture modes. Cohesive and mixed fracture modes were not observed in any of the Group 1 samples. SEM images of each fracture mode sample at ×300 magnification are shown in Figure 1.

Bonding of conventional composite resin materials to dental hard tissues often requires a separate conditioning application with an adhesive system, and clinicians frequently encounter problems due to this technique-sensitive procedure [7]. VF is a self-adhesive, light-cured FC resin with 70% filler loading which including adhesive monomer, glycerol phosphate dimethacrylate (GPDM), added by the manufacturer to control the bonding mechanism of the material [11]. GPDM is defined as a functional monomer that is responsible for chemical interaction to the calcium ions in the tooth structure. Also, VF bonds to tooth surfaces through a micromechanical etching capacity by the low pH (1.9) of the resin material, which is similar to that of numerous self-etching materials [18]. Thus, the present study aimed to investigate the bond strength of SAFC that developed to eliminate separate bonding protocol and extended chair time in the restorative treatment. Among the fillers of VF, nano-sized ytterbium fluoride provides radiopacity to the material, prepolymerized fillers also reduce microleakage and provide improved polishability with nanoparticles [19].

The etch-and-rinse adhesive OptiBond FL applied in combination with the FC Premise flowable was chosen to represent the control group when assessing the performance of new-generation adhesives, since this approach has been successfully bonded to enamel in previous studies [1,20,21]. It has been found that the interaction of SAFCs with dental hard tissues such as enamel and dentin is significantly different from each other due to their composition contain different functional monomers [22]. We used OptiBond All-in-One that same adhesive technology is incorporated into the VF in the present study because materials produced by the same manufacturer was preferred to reduce the undesirable effects caused by unexamined interaction between different substances [23].

Newly developed dental materials may be stronger and bond better to the tooth substrate than currently available materials, and various laboratory tests have been presented assessing differences in material's adhesive properties [24]. The SBS test is the widely accepted laboratory test used in assessing the bonding performance of restorative materials and testing in shear mode has become comparatively simple and reproducible [18,25]. In this study, the SBS test method was selected to compare the adhesive properties of self-adhesive flowable resin composites and conventional FC on primary teeth enamel surfaces.

Thermocycling is a widely used standard procedure to imitate the physiological aging that occurs in dental materials in clinical applications Özcan [26] reported that thermocycling represents a more challenging situation for the composite resin under investigation since it is more effective than other aging methods in the degradation of composite resins [27]. A literature review performed

by Gale and Darvell [26] concluded that approximately 10,000 cycles caused one year of physiological aging in the oral environment, however, another review concluded that the choice related the number of thermocycling decided by authors was varied and not clearly explained [28]. Many authors have preferred the guideline that 5,000 cycles between 5 and 55°C with a duration of 30 s correspond to one year of physiological aging for restorative materials in the oral environment similar to our study [29-31].

The finding presented in this in-vitro study showed that the bond strength of all experimental groups in primary teeth enamel was significantly lower than that in the control group ($P<0.05$). Therefore, the null hypothesis was rejected. These findings are in line with previous studies that investigate the effectiveness of self-etch adhesive systems for bonding resin composite to permanent teeth enamel compared to total-etch adhesives [1,10,20,32]. The lower bond strength of experimental groups may be attributed to the lower etching pattern on the enamel surface of self-etch adhesives and subsequently reduced micro-mechanical retention [33]. Better micromechanical retention in adhesive systems is necessary to resist acute debonding forces, such as those acting in bond strength testing [20].

The results also showed that initially enamel etching enhanced the SBS of VF. While one investigation reported that the bond strength was not affected by acid etching [34], a majority of previous studies reported that preliminary acid etching produced higher enamel bond strength in self-etch adhesives [10,31,32]. Differences in the pattern of acid etching may influence the bonding performance of an adhesive system because the enamel bond strength of the adhesive system principally relies on the micromechanical infiltration of a low-viscosity monomer resin into micro-porosities on the etched enamel surface [35]. A possible explanation for enhanced bond strength achieved by VF on etched enamel could be that firstly acid etching application adequately increases the surface energy of enamel and thus providing significantly more micro-retention areas for adhesion.

Thermal cycling procedure may have caused lower bond strength values associated with VF containing a self-etching adhesive. Generally, flowable materials that contain higher matrix content, present higher water absorption than conventional composites and those interactions might affect their long-term performance. The degradation of the interface components by hydrolysis resulting from the thermocycling process may cause a decrease in the bonding efficiency [23]. In addition, the mechanical properties of the restorative material may be weakened due to water infiltration into the polymer matrix [36]. Although the SBS of the self-adhesive system were not decreased after thermal cycling in Yuasa et al's study [37], the mean bond strength values of VF found in the present study are lower than mean values observed in previous studies [1,38] which did not prefer the thermocycling method. Wei et al. concluded that VF showed remarkable hygroscopic changes during water absorption/ desorption cycles [39]. Therefore, it seems reasonable to consider the thermocycling decreases bond strength in groups without prior acid etching, which is also in line with the findings of other working groups [15,17].

The adhesive type failure mode was mostly observed for all SAFC groups, although a relatively high frequency of cohesive failures in enamel was recorded for the control group. Regarding failure modes, it has been noted that the higher the bond strength, the higher the rate of cohesive failure [40]. Such finding is in agreement with this study that the highest mean bond strength values were measured in the control group. As a matter of fact, mostly cohesive failure within enamel did not occur in the specimens of the SAFCs group in which lower bond strengths to enamel are noted.

Simplification of bonding application steps is defined as the current trend in the development of adhesive systems, although the efficiency and comfort of restorative treatment should be developed without significantly reducing the quality and durability of the adhesion between tooth and resin [41]. Regarding the bonding strength to enamel in the literature, it has been reported that the minimal mean SBS values between 5.9-7.8 MPa are appropriate in clinical bonding applications [42]. Considering the mean SBS values in this study, groups 2 and 4 showed clinically acceptable results.

It should also be noted that mean SBS values in this study was obtained by means of in vitro experiments performed in the laboratory and in vitro tests might not completely mimic the oral environment [43]. Thus such results can differ significantly. In addition to this, it has been notified that the age features of the enamel, the mineral densities of the tooth, and the tooth enamel morphology influence the durability of the bonded interfaces [44]. One limitation of this study is the teeth with enamel hypoplasia were removed and did not use in this study, however, teeth of different morphology may be encountered in clinical applications. Besides, patient-related variables such as tooth brushing, bruxism, and eating habits can also influence restorative application outcomes. Further in vitro and clinical studies are needed to specify the long-term success of the SAFC tested in the present study.

4. Conclusions

Within the limitations of the our study, it may be concluded that: the new SAFC Vertise Flow did not achieve bond strengths to enamel comparable

to those of a conventional FC Premise flowable with total-etch adhesive tested as a control. However, Vertise Flow measured relatively low SBS on enamel, and phosphoric acid treatment of the substrate before applying Vertise Flow significantly changed their adhesion potential to the enamel.

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