SHEAR BOND STRENGTH OF SELF ADHERING FLOWABLE COMPOSITES AFTER PRELIMINARY ACID ETCHING OF DENTIN

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ABSTRACT

INTRODUCTION: Many advances in flowable composites have been developed in the last few years, including the introduction of self-adhering flowable composites (SAFCs) that do not require the application of adhesive systems.

OBJECTIVES: The objective of this study was to evaluate the influence of preliminary etching with 37% phosphoric acid on the shear bond strengths of two SAFCs (Vertise Flow and Fusio Liquid Dentin) to dentin.

MATERIALS AND METHODS: Sixty sound human permanent molars were collected and prepared to expose the mid-coronal dentin. Dentin specimens were randomly divided into six groups (n=10) according to the material applied: (1) Vertise Flow (VF); (2) Fusio Liquid Dentin (FLD); (3) Gel Etchant/Vertise Flow (GE/VF); (4) Gel Etchant/Fusio Liquid Dentin (GE/FLD); (5) Optibond all-in-one/Premise Flowable (Opti-A/PF); (6) Gel Etchant/ Optibond all-in-one/Premise Flowable (GE/Opti-A/PF). Specimens were subjected to shear bond strength (SBS) test. Failure modes were evaluated under stereomicroscope and assessed using Adhesive Remnant Index (ARI) scores. Data were collected and statistically analyzed using ANOVA F-test and Chi-square test ($p<0.05$).

RESULTS: Group 6 (GE/Opti-A/PF) recorded the highest mean SBS (16.39 ± 2.418 MPa), while Group 1 (VF) recorded the lowest mean SBS (4.32 ± 0.993 MPa). The one-step self-etch adhesive Optibond all-in-one with and without a separate etching step had significantly higher mean SBS than those of the two SAFCs. Preliminary etching of dentin significantly increased the SBS of only Vertise Flow.

CONCLUSIONS: The bonding performance of the 1-step self-etch adhesive (SEA) (Opti-A) was considerably better than those of the two SAFCs. Preliminary acid etching of dentin enhances the bonding performance of VF.

KEYWORDS: Self adhering, flowable composites, shear bond strength, all-in-one adhesives, preliminary acid etching.

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INTRODUCTION

Self-etch approach has not been introduced until late 1990's in the form of 2-components adhesive systems that do not require a separate etching step (1). Instead, they contain acidic monomers that can simultaneously condition and prime tooth structure (2, 3, 4). For further simplification, one-bottle self-etch adhesives have then been manufactured to combine bonding agent application with the conditioning and priming steps (2).

Later, acidic functional monomers were incorporated into flowable composite resins in an attempt to combine both self-adhesion property with flowability into a single resin material, without the need of a separate bonding agent. (5, 6). The first self-adhering flowable composite (SAFC) has been introduced in 2009 (5, 7) to greatly simplify the operative procedure, and to considerably reduce time consumption and handling errors (8). SAFCs are indicated as pits and fissure sealants, liners beneath posterior composite restorations, restorative material in small classes I, II, and III restorations, for porcelain repair, and in blocking undercuts (9, 10).

In order to achieve optimal bond, SAFCs should overcome the hydrophobic-hydrophilic mismatch between the composite restoration and the tooth structure by containing acidic functional monomers as glycerol phosphate dimethacrylate (GPDM) or 4-methacryloyloxyethyl trimellitic acid (4-MET) (10, 11). Their primary bonding mechanism according to their manufacturers depends on the chemical interaction between the functional groups in their acidic monomers (as phosphate functional group in GPDM monomer, or carboxylic group in 4-MET monomer) and the hydroxyl apatite crystals of the tooth structure. The secondary bonding mechanism is micromechanical, which is achieved by the slight conditioning effect gained by their low pH (10, 11) which increases to almost neutral after light curing (11). In addition to acidic monomers, manufacturers incorporate 2-hydroxyethyl methacrylate (HEMA) into the formulae of SAFCs (12) to increase monomer penetration and wetting of dentin (13).

Since etching with 37% phosphoric acid demineralizes the tooth surface (14) and obtains patency of the dentinal tubules (15), it might affect the interaction of the functional monomer with the dental substrate as well as the micromechanical retention of the adhesive material. Therefore, it would be of interest to test the effect of preliminary acid etching on the bonding performance of self-adhering flowable composites. The null hypothesis of the present study was that acid etching of dentin with 37% phosphoric acid does not affect the bond strength of two SAFCs.

MATERIALS AND METHODS

Sixty freshly extracted sound human permanent molars free of caries, cracks, restorations and dental anomalies were collected from the out-patient clinic of the Oral Surgery Department, Faculty of Dentistry, Alexandria University. Teeth were extracted due to periodontal or orthodontic problems based on periodontist or orthodontist professional opinions. They were stored in 0.5% Chloramine T aqueous solution at 4°C till use (16). The composites employed in the current study were two self-adhering flowable composites Vertise Flow (Kerr Orange, CA, USA) and Fusio Liquid Dentin (Pentron Clinical, CT, USA), in addition to flowable composite Premise Flowable (Kerr Orange, CA, USA). Moreover, one-step self-etch adhesive

Optibond all-in-one (Kerr Orange, CA, USA) and 37.5% phosphoric acid Gel Etchant (Kerr Orange, CA, USA) were also employed in the study (table 1).

At time of usage, the occlusal enamel and superficial dentin of teeth was completely removed using a model trimmer under running water to create flat surface of mid coronal dentin. Then each tooth was embedded into auto-polymerizing acrylic resin with its long axis perpendicular to the base of the acrylic block. The dentin surface of each specimen was then polished with wet 600 grit silicon carbide paper disc to produce a uniform smear layer prior to bonding (12). The sixty dentin specimens were then randomly divided into six groups with ten teeth per group (n=10) according to the materials applied on the dentin surface.

Group 1: Vertise Flow (VF).
Group 2: Fusio Liquid Dentin (FLD).
Group 3: 37.5% phosphoric acid followed by Vertise Flow (GE/VF).
Group 4: 37.5% phosphoric acid followed by Fusio Liquid Dentin (GE/FLD).
Group 5: Optibond all-in-one followed by Premise Flowable (Opti-A/PF).
Group 6: 37.5% phosphoric acid followed by Optibond all-in-one and Premise Flowable (GE/Opti-A/PF).

In order to standardize the working area, a thin walled plastic mold 3mm in diameter and 2mm in height was secured at the center of dentin surface of each specimen by means of a double face adhesive tape (16). Within the plastic mold, flowable composites were applied and cured by a light emitting diode (LED) curing unit (Elipar S10; 3M ESPE, St. Paul, MN, USA) with a light intensity of 1200 mW/cm2, and with curing time of 10 or 20s according to the manufacturers (table 1).

Table 1: Types, compositions, and application procedures of the materials used.

Materials	Type	Composition	Application procedures
Vertise Flow (VF)	Light cured, self adhering flowable composite	GPDM, HEMA, prepolymerized filler, 1-µm barium glass filler, nano-sized colloidal silica, nano-sized ytterbium fluoride (filler content 70 % wt)	Dispense a thin layer (<0.5mm) of VF onto the dentin substrate and brush with moderate pressure for 15-20s and light cure for 20s. Then apply a 2nd increment of thickness 1.5 mm and light cure for 20s
Fusio Liquid Dentin (FLD)	Light cured, self adhering flowable composite	UDMA, TEGDMA, HEMA, 4-MET, nano-sized amorphous silica, silane treated barium glass, minor additives, photo-curing system (filler content 65% wt)	Inject 1mm increment of FLD and agitate with needle tip for 20s to condition the tooth prior to 10s light curing. Syringe a second increment of 1mm thickness and light cure for 10s
Premise Flowable (PF)	Light cured, flowable composite	BIS-EMA, TEGDMA, Prepolymerized fillers, Barium glass, Silica filler, Light cure initiators and stabilizers, Organophosphate dispersant (filler content 72.5% wt)	Apply PF in an increment of 2mm thickness and light cure for 20s
Optibond All-In-One (Opti-A)	Light cured, single component, single step self-etch adhesive	GPDM, mono- and di-functional methacrylate co-monomers, water, acetone, ethanol, CQ photo-initiator, nano-silica sodium hexafluorosilicate (filler content 7% wt)	Apply two coats with scrubbing motion for 20s each, then air dry with medium force for 5s, then light cure for 10s
Gel Etchant (GE)	37.5% phosphoric acid	Water, 37.5% orto-phosphoric acid, silica thickener	Etch for 15s and rinse thoroughly for 15s, then gently air dry for 5s
Composition as provided by respective manufacturer: Bis-EMA: Ethoxylatedbisphenol-A-glycol dimethacrylate; CQ: Camphorquinone; GPDM: Glycerol Phosphate Dimethacrylate; HEMA: Hydroxyethylmethacrylate; TEGDMA: Triethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate; 4-MET: 4-Methacryloxyethyltrimetellitic acid.			

The bonded specimens were then stored in distilled water at 37˚C for 24 hours (17), and were then subjected to thermocycling for 500 cycles between 5ºC to 55ºC with 30 seconds dwell time to simulate changes in temperature in the oral cavity (18). The shear bond strength test was carried out using a universal testing machine (Comten Industries Inc, St. Petersburg, Florida, USA). Each specimen was oriented so that shear load was directed parallel to the bonded interface through a knife- edge blade at cross head of speed 1 mm/min until failure occurs. The load at failure was recorded in Newton (N). Shear bond strength was then calculated in magapascals (MPa) by dividing the failure load in Newtons (N) by the bonded surface area (in mm^2).

The bond failure sites of the fractured specimens were then examined optically using a stereomicroscope (Nikon-Japan) at x 30 magnification, and each specimen was given a score according to the Adhesive Remnant Index (ARI) with a score scale from 0 to 3 (19). As the ARI score increases, as better bonding performance is indicated as follows (19):

- Score 0: No composite is left on dentin.
- Score 1: Less than half of the composite is left on dentin.
- Score 2: More than half of the composite is left on dentin.
- Score 3: The entire composite is left on dentin.

All data were collected and statistically analyzed by Statistical Package for Social Science version 20.0 (SPSS Inc., Chicago IL). Statistical significance was judged at the 5% level. Quantitative data of shear bond strength values were analyzed using ANOVA F-test, followed by Post Hoc test (Scheffe) for pair wise comparison between groups. Qualitative data of ARI scores were described using number and percent. Comparison between different groups regarding categorical variables was tested using Chi-square test.

RESULTS

For the shear bond strength test, the highest mean SBS was recorded for Group 6 (Gel Etchant/Optibond all-in-one) with mean 16.39 ± 2.418 MPa, whereas the lowest mean SBS was recorded for Group 1 (Vertise Flow) with mean 4.32 ± 0.993 MPa (table 2, figure 1).

Table 2: Descriptive statistics of shear bond strength to dentin (in MPa) for the six studied groups and statistical significant difference between groups.

	Gp. 1 (VF)[a]	Gp. 2 (FLD)[b]	Gp. 3 (GE/VF)[c]	Gp. 4 (GE/FLD)[b]	Gp. 5 (Opti-A/PF)[c]	Gp. 6 (GE/Opti-A/PF)[c]
Min.	2.86	5.20	11.90	5.30	11.80	12.20
Max.	6.12	8.60	18.60	9.00	17.10	19.10
Mean	4.320	6.960	15.830	7.120	14.860	16.390
S.D.	0.993	1.129	2.449	1.155	1.803	2.418
ANOVA	91.97					
Significance ($p<0.05$)	0.0001					

Different letters indicate statistical significant difference regarding SBS values

SD: standard deviation

Anova F-test proved significance in SBS values among groups (p= 0.0001). Post Hoc test (Scheffe) for pair-wise comparison between groups showed that the mean SBS of the 1-step self-etch adhesive (SEA) Optibond all-in-one (Group 5) was significantly higher than those of the two SAFCs when used without etching (Group 1 and Group 2). However, the mean SBS of Opti-A was not significantly different from that of VF when the latter was preceded with a pre-etching step (Group 3). Among the two SAFCs, the mean SBS of Fusio Liquid Dentin was significantly higher than that of Vertise Flow (p= 0.002). Post Hoc test (Scheffe) revealed that preliminary acid etching step resulted in significant increase in the SBS of only Vertise Flow (p= 0.0001).

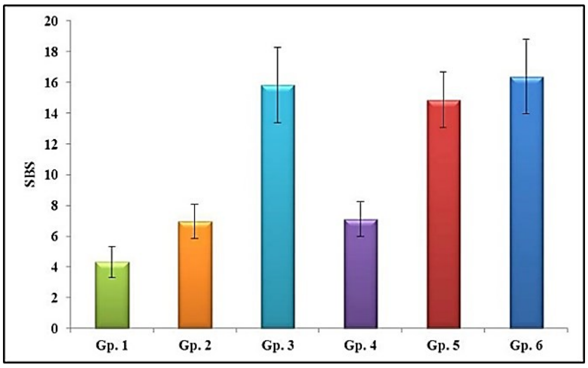

Figure 1: Bar chart representing shear bond strengths (in MPa) of all the applied materials in the six groups.

For the Adhesive Remnant Index (ARI) scores, the highest scores were obtained by Group 6 (GE/Opti-A) with 10% score 0, 40% score 1, 40% score 2, and 10% score 3. On the other hand, the lowest ARI scores were obtained by Group 1 (VF) and Group 2 (FLD) with 80% score 0 and 20% score 1 (table 3, figure 2).

Chi-square test proved significance in ARI scores among groups (p=0.0001). ARI score 0 was predominant in Group 1(VF), Group 2 (FLD) and Group 4 (GE/FLD). ARI scores 1 and 2 were predominant in Group 3(GE/VF), Group 5 (Opti-A/PF) and Group 6 (GE/Opti-A/PF). Score 3 was obtained in 10% of specimens of Groups 5 and 6.

Table 3: Descriptive statistics of Adhesive Remnant Index (ARI) scores for the six studied groups and statistical significant difference between groups.

Adhesive Remnant Index (ARI) Scores		Group						Total
		Gp. 1 (VF)[a]	Gp. 2 (FLD)[a]	Gp. 3 (GE/VF)[b]	Gp. 4 (GE/FLD)[a]	Gp. 5 (Opti-A/PF)[b]	Gp. 6 (GE/Opti-A/PF)[b]	
0	No.	8	8	2	7	2	1	28
	%	80.0%	80.0%	20.0%	70.0%	20.0%	10.0%	46.7%
1	No.	2	2	4	3	4	4	19
	%	20.0%	20.0%	40.0%	30.0%	40.0%	40.0%	31.7%
2	No.	0	0	4	0	3	4	11
	%	.0%	.0%	40.0%	.0%	30.0%	40.0%	18.3%
3	No.	0	0	0	0	1	1	2
	%	.0%	.0%	.0%	.0%	10.0%	10.0%	3.3%
Total	No.	10	10	10	10	10	10	60
	%	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
Chi Square (X2)		42.085						
Significance (p<0.05)		0.0001						

Different letters indicate statistical significant difference regarding ARI scores.

DISCUSSION

In the present study, the bonding performance of two self-adhering flowable composites with and without a preliminary etching step was compared with that of a one-bottle self -etch adhesive when applied on dentin.

When the shear bond strengths of the materials were tested without preliminary acid etching, it was found that both SAFCs Vertise Flow and Fusio Liquid Dentin recorded significantly lower SBS than the 1-step SEA Optibond all-in-one. The poor bonding performance of Vertise Flow coincide with the results of Bektas et al (17) and Tuloglu et al. (20) who related the low bond strength of the material to the incorporation of acidic functional monomer into the flowable composite, causing incomplete infiltration of the adhesive monomer into dentin surface. Bektas et al. (17) suggested that the reduced bond strengths of the SAFCs were due to their higher filler contents with respect to the adhesive bonding agent. Fu et al. (21) also recorded significantly lower bond strengths of both VF and FLD than that of a 1-step self-etch adhesive. According to Miyazaki et al. (22), the increased filler loading in the adhesive resin material might decrease the wetting of dentin surface because of the higher viscosity of filled resins.

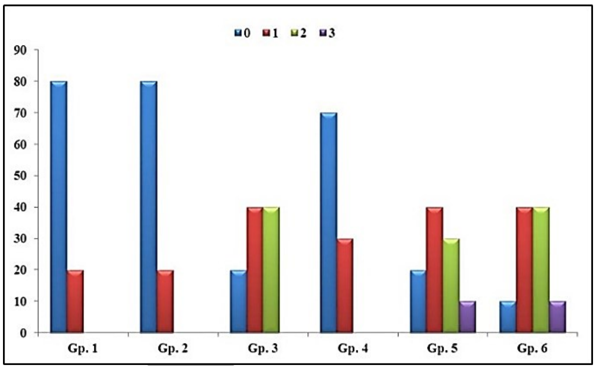

Figure 2: Bar chart representing Adhesive Remnant Index (ARI) scores in the six studied groups

Although both SAFCs tested contain HEMA to increase their wettability, they do not contain solvents, which may have led to the decrease in their penetration capacity into the dentin surface, and consequently the decrease in their shear bond strengths (16). On the other hand, Optibond all-in-one SEA used in the current study contains three types of solvents; water, acetone and ethanol (23). Solvents are essential in the increase of wettability of the adhesive material, enhance penetration of the functional monomers, and permit closer adhesive-substrate chemical interaction with dentin hydroxyapaptite (24, 25, 26), leading to the increase of the bond strength.

In the current study, the self-adhering flowable composites recorded low mean SBS values when used without preliminary etching (4.3 MPa for VF and 6.9 MPa for FLD). These low values coincide with the results reported by Juloski et al. (27), Xu et al. (28), and Vichi et al. (16). In these three studies, the mean SBS of Vertise Flow [or Dyad Flow in the study of Xu et al. (28)] to dentin recorded less than 4 MPa. Juloski et al. (27) pointed out that in spite of brushing a first thin increment of VF with moderate pressure for 20 s as recommended by the manufacturer, this action did not enhance the penetration of the relatively viscous material into the dentin surface.

Comparable bond strength values have also been shown in previous studies performed on self-adhering resin cements, which also incorporate acidic functional monomers to be applied directly on to the tooth surface (29, 30, 31). De Munck et al. (32) used SEM that showed remnants of smear layer, insufficient penetration of the self- adhering resin cement, and lack of hybrid layer formation at the interface. In Farrokh et al. (30) study, the mean SBS of three self -adhering resin cements to dentin were found to be less than 4 MPa. They related this finding to the lack of penetration of the materials around the collagen fibers. Hatter et al. (31) reported that the mean SBS of three self- adhering resin cements to dentin were less than 6 MPa. They related their low SBS values to their inability to dissolve the smear layer and their superficial interaction with the tooth structure.

Among the two SAFCs used in the current study, the mean SBS of Vertise Flow was significantly lower than that of Fusio Liquid Dentin. This might be attributed to the fact that the two SAFCs incorporate different acidic functional monomers (GPDM in Vertise Flow, and 4-MET in Fusio

Liquid Dentin) (10, 11). Similar results were also recorded by Fu et al. (21) and Poitevin et al. (33), who explained that the GPDM monomer used with Vertise Flow might probably etch rather than bond chemically to tooth structure, and pointed out that a self-adhering flowable composite should possess some chemical adhesion capacity as it cannot penetrate deeply. On the contrary, 4-MET incorporated in Fusio Liquid Dentin could have some chemical bonding potential to hydroxyl apatite crystals (34) which might be higher than that of GPDM and may explain why Fusio Liquid dentin had higher bond strength than Vertise Flow (33).

The present study showed that preliminary acid etching of dentin resulted in a significant increase in the shear bond strength of only Vertise Flow, with which the null hypothesis was rejected. The action of acid etching in obtaining patency of the dentinal tubules (15), providing clear exposure of the collagen matrix and increasing microporosities (35) could have cleared the pathway for deeper penetration of Vertise Flow with improvement of the micromechanical retention. These findings come in line with Xu et al. (28), Poitevin et al. (33), and Altunsoy et al. (12) who also recorded significant increase in the bond strength of Vertise Flow with preliminary acid etching of dentin.

On the other hand, the current study did not show significant increase in the bond strength of Fusio Liquid Dentin by the pre-etching step. It might be expected that the chemical retention between 4- MET monomer in FLD and hydroxyl apatite crystals might have been reduced due to the loss of minerals following acid etching. However, this loss might have been partially compensated by slight increase in the micromechanical retention achieved by acid etching step.

It was shown in our study that acid etching did not significantly influence the bond strength of Optibond all-in-one. As previously pointed out, the GPDM functional monomer incorporated in Opti-A might possess more etching capacity rather than chemical potential with hydroxyl apatite crystals (33). Consequently further loss of minerals by acid etching might not have influenced the SBS of the bonding agent. This result was contradictory to those of Van Landuyt et al. (36), Xu et al. (28), and Sabatini (37) who showed significant decrease in the bond strength of 1-step self-etch adhesives to dentin after acid etching. On the contrary, Taschner et al. (38) demonstrated significantly increased bond strength of two 1-step SEAs when applied on acid etched dentin.

It should be noted that these studies used one bottle SEAs other than Optibond all-in-one, and a pre-etching step with acid could affect various types of SEAs differently according to the difference in acidic monomers or adhesive technologies they incorporate. Taschner et al. (38) explained that they polished dentin surface with 180-grit SiC which could have produced a thick smear layer that may have decreased the penetration of the SEA they tested. When this smear layer was totally removed by acid etching, the bond strength of the SEA significantly increased. On the other side, Sabatini (37) showed that acid etching of dentin significantly decreased the bond strength of the SEA they used in their study. They suggested that the functional monomer incorporated in the bonding agent they used might have been more dependent on residual hydroxyapatite crystals.

The failure mode in the current study was assessed by Adhesive Remnant Index (ARI) using stereomicroscopic observation. The ARI score 0 was predominant in specimens of Vertise Flow (Group 1), Fusio Liquid Dentin (Group 2), and Fusio Liquid Dentin following preliminary acid etching (Group 4). Although FLD had significantly higher mean SBS than VF, score 0 was predominant with both self-adhering flowable composites, probably due to the increased viscosity of the two materials which might have resulted in their lack of penetration into the dentin surface. Therefore, shear stresses that were directed towards the interface during the shear bond strength test produced complete separation of the SAFCs in most specimens without leaving any remnants on the dentin surface.

ARI Scores 1 and 2 were predominant in specimens of Vertise Flow following a preliminary etching step (Group 3), and in specimens of Optibond all-in-one used with and without the pre-etching step (Groups 6 and 5 respectively). Generally, it was observed that the higher the bond strength, the lower the percentage of score 0 (with the exception of ARI scores of VF and FLD).

The limitations of the current study were the inability to simulate clinical loading forces and chemical attacks by acids and enzymes that may cause degradation in the bonding interface of a restored tooth. Temperature changes in the oral cavity were simulated in the current study through subjecting specimens to thermocycling. Stewardson et al. (39) claimed that 500 cycles would correspond to the number of cycles occurring in less than two months in the oral cavity. However, the International Standards Organization (ISO) in 1994 considered a protocol of 500 cycles between water baths held at 5ºC and 55ºC with a dwell time in each bath ≥20 s appropriate in simulating the aging of biomaterials in the oral cavity (40).

CONCLUSIONS

The objective of the present study was to test the effect of preliminary etching of dentin with 37% phosphoric acid on the shear bond strengths of two self-adhering flowable composites. Within the limitations of this study, it was concluded that the bonding performance of the 1-step self-etch adhesive (Optibond all-in-one) is better than that of the two self-adhering flowable composites (Vertise Flow and Fusio Liquid Dentin) when applied on dentin, with better bonding performance of Fusio Liquid Dentin than Vetise Flow. A preliminary etching step with 37% phosphoric acid for 15 seconds enhances the bond strength of Vertise Flow to dentin. Therefore, according to the findings of the current study, it is recommended to acid etch dentin prior to the clinical application of Vertise Flow.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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