Immediate Repair Bond Strength of Fiber-reinforced Composite after Saliva or Water Contamination

Jasmina Bijelic-Donova[a] / Andrew Flett[b] / Lippo V.J. Lassila[c] / Pekka K. Vallittu[d]

Purpose: This in vitro study aimed to evaluate the shear bond strength (SBS) of particulate filler composite (PFC) to saliva- or water-contaminated fiber-reinforced composite (FRC).

Materials and Methods: One type of FRC substrate with semi-interpenetrating polymer matrix (semi-IPN) (everStick C&B) was used in this investigation. A microhybrid PFC (Filtek Z250) substrate served as control. Freshly cured PFC and FRC substrates were first subjected to different contamination and surface cleaning treatments, then the microhybrid PFC restorative material (Filtek Z250) was built up on the substrates in 2-mm increments and light cured. Uncontaminated and saliva- or water-contaminated substrate surfaces were either left untreated or were cleaned via phosphoric acid etching or water spray accompanied with or without adhesive composite application prior applying the adherent PFC material. SBS was evaluated after thermocycling the specimens (6000 cycles, 5°C and 55°C).

Results: Three-way ANOVA showed that both the surface contamination and the surface treatment signficantly affected the bond strength ($p < 0.05$). Saliva contamination reduced the SBS more than did the water contamination. SBS loss after saliva contamination was 73.7% and 31.3% for PFC and FRC, respectively. After water contamination, SBS loss was 17.2% and 13.3% for PFC and FRC, respectively. The type of surface treatment was significant for PFC ($p < 0.05$), but not for FRC ($p = 0.572$).

Conclusion: Upon contamination of freshly cured PFC or semi-IPN FRC, surfaces should be re-prepared via phosphoric acid etching, water cleaning, drying, and application of adhesive composite in order to recover optimal bond strength.

Keywords: saliva contamination, water contamination, composite, fiber-reinforced composite, shear bond strength, composite repair.

J Adhes Dent 2018; 20: 1–8.
doi: 10.3290/j.jad.a40515

Submitted for publication: 26.01.18; accepted for publication: 06.04.18

Moisture contamination of freshly cured particulate filler composite (PFC) adversely affects the bond strength of incrementally placed direct adhesive restorations.[4,5,9] Studies have shown that moisture (saliva, water) contamination reduces the high energy state of freshly etched enamel, resulting in lower bond strength.[23] Likewise, moisture contamination of a freshly cured PFC increment impairs the oxygen inhibition layer (OIL) which mediates the bond between successively placed composite increments.[24] All types of contaminants, eg, saliva,[5,9] blood,[6] and chemical compounds from gloves,[16] can affect the bonding properties between the substrate and the adherent material. Some crucial parameters in restoring contaminated PFC restoration are the type of decontamination method used, and whether or not adhesive is used as an intermediate agent between the old and new composite increment.[5,6,9] Simple drying of the saliva or blood contaminant before applying the next composite increment is not sufficient to restore the interfacial bond strength, and has poor long-term stability.[5,6,9] The minimum suggested by some authors should be washing of the contaminated surface and application of an adhesive agent in order to recover the bond strength.[4-6,9] Ideally, the contaminated surface should be abraded and retreated with adhesive, or then etched and retreated with silane and/or adhesive in order to gain stable bonding between the composite increments.[7,9] Among

---

[a] *Assistant Professor, Department of Prosthetic Dentistry and Stomatognathic Physiology, and Turku Clinical Biomaterials Centre-TCBC, Institute of Dentistry, University of Turku, Turku, Finland. Idea, test group design, hypothesis, performed the testing, wrote the manuscript.*

[b] *Consultant Orthodontist, Queen's Medical Centre, Nottingham University Hospital NHS Trust, Nottingham, UK. Test group design, specimen preparation, proofread the manuscript.*

[c] *Laboratory Head, Turku Clinical Biomaterials Centre-TCBC, Institute of Dentistry, University of Turku, Turku, Finland. Statistical evaluation, contributed to discussion.*

[d] *Professor and Chair of Biomaterials Science, Director of Turku Clinical Biomaterials Centre-TCBC, Institute of Dentistry, University of Turku, Turku, Finland, and City of Turku Welfare Division, Oral Health Care, Turku, Finland. Idea, study design, supervised the work, contributed significantly to analyzing the results and to the discussion.*

Correspondence: *Dr. Jasmina Bijelic-Donova, Department of Prosthetic Dentistry and Stomatognathic Physiology, and Turku Clinical Biomaterials Centre (TCBC), Itäinen pitkäkatu 4B, 20520 Turku, Finland. Tel: +358-2-333-8203; e-mail: jabije@utu.fi*

Table 1 Materials used in the study

Name	everStick C&B	Filtek Z250	Scotchbond Universal Etchant	Adper Scotchbond Adhesive
Code	FRC	PFC	Etchant	Adhesive
Manufacturer	Stick Tech, GC; Tokyo, Japan	3M Oral Care; St Paul, MN, USA	3M Oral Care	3M Oral Care
Type of matrix	semi-IPN	CLP		CLP
Resin matrix	PMMA Bis-GMA	Bis-GMA Bis-EMA UDMA, TEG-DMA		Bis-GMA HEMA
Type of fiber	Continuous unidirectional E-glass			
Fiber volume fraction (vol%)	~ 65			
Lot	2050711-ES-134 2050105-ES-118 2050617-ES-130	20050315 (5CN) 20050311 (5PK)	571374	20040730

FRC: fiber-reinforced composite; PFC: particulate filler composite; semi-IPN: semi-interpenetrating polymer network; CLP: cross-linked polymer; PMMA: polymethylmetacrylate; bis-GMA: bisphenol-A-glycidyl dimethacrylate; bis-EMA: bisphenol-A-dyethoxy dimethacrylate; UDMA: urethane dimethacrylate; TEG-DMA: triethylene-glycol dimethacrylate; HEMA: hydroxyethylmethacrylate.

the direct restorative materials, PFCs are considerably more sensitive to moisture contamination in comparison to glass-ionomer cements (GIC) and resin-modified GICs (RMGIC).[19] Namely, it has been shown that the interfacial bond strength of uncontaminated PFCs is greater than uncontaminated GIC or RMGIC, but upon moisture contamination both GIC and RMGIC have better bonding properties than PFCs.[19]

Repair of aged fiber-reinforced composite (FRC) has also been the focus of some studies.[8,11,13,14,22,28] It was shown that repair bond strength improves if the FRC resin matrix is a semi-interpenetrating network (semi-IPN), which is due to the secondary IPN bonding mechanism enabled by the IPN's ability to swell.[8,13,14,26] Semi-IPN is a multiphase polymer matrix comprising linear (thermoplastic) polymethylmethacrylate (PMMA) and cross-linked (thermoset) dimethacrylate monomers. Semi-IPN polymer is formed upon polymerization of the multiphase polymer matrix. Semi-IPNs have advanced handling and bonding properties in comparison to purely cross-linked systems due to the PMMA chains; these are dissolvable with monomers that have solubility parameters similar to their own.[14,15,26,29] Practically, these are low-viscosity monomers containing bisphenol-A-glycidyl dimethacrylate hydroxyethylmethacrylate (bis-GMA-HEMA) or bisphenol-A-glycidyl dimethacrylate triethylene-glycol dimethacrylate (bis-GMA-TEG-DMA), which simultaneously wet the substrate and swell the PMMA layer.[14] The benefit of this feature particularly lies in repairs. Namely, enhanced repair has been observed for the semi-IPN matrix, because it permitted interdiffusion of fresh monomers into the polymerized polymer.[8] This kind of infiltration does not happen[26] or is very restricted in cross-linked matrices.[29] Consequently, reparability of cross-linked (thermoset) matrices is poor.

Immediate repair following momentary FRC surface contamination has yet to be conducted. Specifically, there is some concern regarding the bonding of the protective PFC layer to the underlying FRC substrate upon momentary contamination of the fibers' surface, as well as in terms of the best method of immediate repair treatment upon momentary fiber contamination. According to the authors' knowledge, this issue has been addressed. Consequently, the aim of this study was to investigate the effect of saliva and water contamination of semi-IPN-based FRC (everStick C&B) on the bonding properties of PFC used as a veneering material to cover the FRC substrate. The rationale for using only semi-IPN FRC is the poor reparability potential of cross-linked matrices, as mentioned above. Currently, there is only one type of semi-IPN FRC on the market (everStick fiber family, Stick Tech, GC; Tokyo, Japan).

The null hypothesis was that the shear bond strength of immediately repaired semi-IPN FRC is not affected by the contamination and surface cleaning treatment prior to repair.

MATERIALS AND METHODS

Specimen Preparation

Two substrate types (FRC and PFC) and one adherent material (PFC) were used in the present investigation. The substrate materials were unidirectional E-glass FRC (everStick C&B, Stick Tech, GC) and PFC (Filtek Z250, 3M Oral Care; St Paul, MN, USA). The adherent material in both groups was PFC (Filtek Z250).

Table 2 Overview of the protocols used in the present study

Contamination conditions*	
1. No contamination (control)	The bonding site of the PFC and FRC substrate was not contaminated.
2. Saliva contamination	The bonding site of the PFC and FRC substrate was contaminated with saliva for 30 s. Saliva was collected from a single individual at the same time that the specimens were prepared. Saliva was applied to the bonding site with a microbrush.
3. Water contamination	The bonding site of the PFC and FRC substrate was contaminated with water for 30 s. The water was applied as a spray from a distance of 10 cm.
Decontamination protocols/surface cleaning treatments**	
1. No cleaning	The adherent material (Filtek Z250) was placed directly on the contaminated or non-contaminated FRC (everStick C&B) or PFC (Filtek Z250) substrate. Saliva and water were dried with oil-free compressed air as part of the protocol.
2. Water spray and adhesive composite	The saliva- or water-contaminated, or previously non-contaminated, bonding site was cleaned with water spray for 20 s, dried for 20 s with oil-free compressed air, and treated with an adhesive composite (Adper Scotchbond Adhesive) applied with microbrush and subsequently light-polymerized for 20 s.
3. Phosphoric acid etching, water spray and adhesive composite	The saliva- or water-contaminated, or previously non-contaminated, bonding site was etched with 37% phosphoric acid (Adper Scotchbond Etchant) for 20 s, washed and dried for 20 s each, and treated with an adhesive composite (Adper Scotchbond Adhesive) as previously described.

*The bonding sites (FRC and PFC substrate surfaces) were subjected to one of three contamination conditions.
**Immediately after contamination, each group received one of three decontamination protocols/surface cleaning treatments.

Table 3 Grouping of the specimens and group codes

Group description	No contamination (N)	Saliva contamination (for 30 s) (S)	Water contamination (for 30 s) (W)
No cleaning (N), control	NN	SN	WN
Water spray (20 s) (W) Drying (20 s) (D) Adhesive composite and light polymerization (20 s) (A)	NWDA	SWDA	WWDA
Etching (20 s) (E) Water spray (20 s) (W) Drying (20 s) (D) Adhesive composite and light polymerization (20 s) (A)	NEWDA	SEWDA	WEWDA

Each contamination and decontamination protocol was performed for both PFC (Filtek Z250) and FRC (EverStick C&B) groups (9 groups per material, n = 14/group, in total 126 specimens per material category).

The elected FRC was preimpregnated with semi-IPN resin, comprising bis-GMA and PMMA. The control was a cross-linked thermoset, bis-GMA-based microhybrid PFC. Materials, manufacturers, and lot numbers are listed in Table 1.

One hundred twenty-six (126) specimens were prepared on FRC substrates. These substrates were fabricated by placing three layers of semi-IPN FRC material into the retentive cavity (8 x 6 x 4 mm) prepared in an acrylic resin cylinder. A Mylar strip was used to flatten the fibers, and the substrate was first light-polymerized through the Mylar strip for 3 s (Optilux-501, Kerr; Orange, CA, USA) at 800 mW/cm^2 light intensity. Upon removal of the Mylar strip, the substrate was additionally light polymerized for 40 s. It has been shown that a Mylar strip does not affect the bond strength between composite increments,[27] and that replenishment of oxygen during polymerization produces an OIL.[10]

For the control groups, 126 specimens were prepared on PFC substrates. For this purpose, Filtek Z250 restorative material was first placed into retentive holes of 5 mm diameter and 3 mm depth prepared in acrylic resin cylinder, and then light polymerized through the Mylar strip for 3 s. Next, the Mylar strip was removed and the substrate was additionally light polymerized for another 40 s.

Contamination and Surface Cleaning Treatments

Specimens (126 per material category) were divided into 9 groups (n = 14) according to the contamination and surface cleaning treatment method. The protocol is described in Table 2, whereas the groups and codes are shown in Table 3.

Once the substrates were contaminated and cleaned, the PFC adherent material (Filtek Z250) was immediately built up on the bonding site, each time after contamination and surface cleaning. The PFC was applied in 2-mm increments using a translucent polyethylene mold (inner diameter 3.6 mm, surface area 40.7 mm^2), and each layer was light polymerized for 40 s. The specimens were stored in water at room temperature for 48 h until all groups were prepared, and then thermocycled for 6000 cycles between 5 (± 2)°C and 55 (± 2)°C with a dwell time of 30 s and a transfer time of 5 s.

Shear Bond Test

The shear bond strength test was performed 24 h after thermocycling, using a universal testing machine (Lloyd Instruments; Fareham, UK) at room temperature (23 ± 1°C), and the results were recorded using PC software (Nexygen, Lloyd Instruments). The specimens were secured in a mounting jig (Bencor Multi-T shear assembly, Danville Engineering; San Ramon, CA, USA), with the shearing rod against and parallel to the bonding sites.[13] A circular-edged blade created the load positioned over the FRC-PFC or PFC-PFC interface, at a crosshead speed of 1.0 mm/min until fracture. In FRC groups, the fibers were orientated parallel to the shear force.[13,22] The shear load at failure was recorded in N (Newtons) and converted to MPa (Megapascals) as a function of the bonded area automatically by the software. Shear bond strength (SBS) is presented in MPa.

Fracture surfaces of the specimens were analyzed visually after shear bond testing and divided into adhesive, cohesive, and mixed (adhesive and cohesive) failures.

The surfaces of uncontaminated, water-sprayed and phosphoric-acid etched FRC specimens (n = 3/group) were analyzed using SEM (JSM 5500, JEOL; Tokyo, Japan). The specimens were gold sputter-coated before the SEM examination.

Statistical Analysis

The data were statistically analyzed with SPSS v 19 (SPSS, IBM; Armonk, NY, USA). Three-way ANOVA followed by Tukey's post-hoc test at a significance level of $p = 0.05$ was performed in order to analyse the contamination and surface cleaning treatment effect on the SBS. Material, contamination, and surface cleaning treatment were the independent variables, and shear bond strength was the dependent variable.

RESULTS

Shear bond strength results with different contamination and surface cleaning treatments are shown in Figs 1 and 2. The particulate filler composite revealed higher original shear bond strengths than did FRC, whereas FRC was less sensitive to contamination.

Three-way ANOVA revealed that contamination and surface cleaning treatment had a significant effect ($p = 0.007$) on SBS for the PFC, but not for the FRC ($p = 0.582$). However, interaction between the factors "contamination" and "surface cleaning treatment" existed ($p < 0.05$). In other words, the effect of the contaminant on the SBS was modified by the type of surface treatment. Interestingly, however, the surface cleaning treatment effect was not statistically significant for the FRC ($p = 0.572$), but it was for the PFC ($p < 0.05$). In other words, it was easier to obtain clean FRC surfaces regardless of the surface cleaning treatment applied.

Visual analysis of fracture surfaces for FRC substructures showed fiber breakage along the FRC surface in all specimens (100%) in the control group (NN). The fracture did not penetrate into the surface but instead continued along the surface, tearing the fibers from the surface. This was classified as cohesive fracture. Similar findings were found for the other FRC specimens as well, except the saliva contaminated groups, which showed adhesive failures (100%). PFC specimens failed adhesively (100%). The PFC control group NN (uncontaminated and uncleaned group, NN) included cohesive failures, whereas the NEWDA group (surface cleaned with phosphoric acid etching followed by adhesive application on the uncontaminated surface) and WEWDA group (surface cleaned with phosphoric acid etching followed by adhesive application on water-contaminated substrate) exhibited some mixed failures.

SEM (Figs 3a and 3b) investigation of uncontaminated fiber surface showed that water spray and phosphoric acid etching produced only localized fiber surface exposure.

DISCUSSION

FRC dental appliances (fixed dental prostheses, post-cores, periodontal and orthodontic splints) consist of a fiber framework, which is the internal (FRC) substrate, and an external layer of particulate filler composite that protects the fibers. Usually, the fiber framework is covered by directly layering veneering particulate filler composite in several increments. In clinical situations, this direct intraoral technique includes several steps, thus increasing the chances of contamination of the fiber framework by saliva or the moisture of the oral cavity. In order to simulate this clinical situation, the present study examined the effect of saliva- and water-contaminated fiber surface on the bonding properties to the adjacent veneering composite. Contaminated particulate filler composite surface was used as the control. In addition, the reliability of two clinically easily performed cleaning methods was assessed. Other surface cleaning techniques that require further armamentarium were not employed, because it has been reported that similar results are obtained regardless of the surface cleaning method used prior to immediate repair.[18] For instance, it has been demonstrated that grinding alone or grinding followed by phosphoric acid etching provide no advantages over etching alone.[7] Grinding surface treatment prior to repair has been recommended only

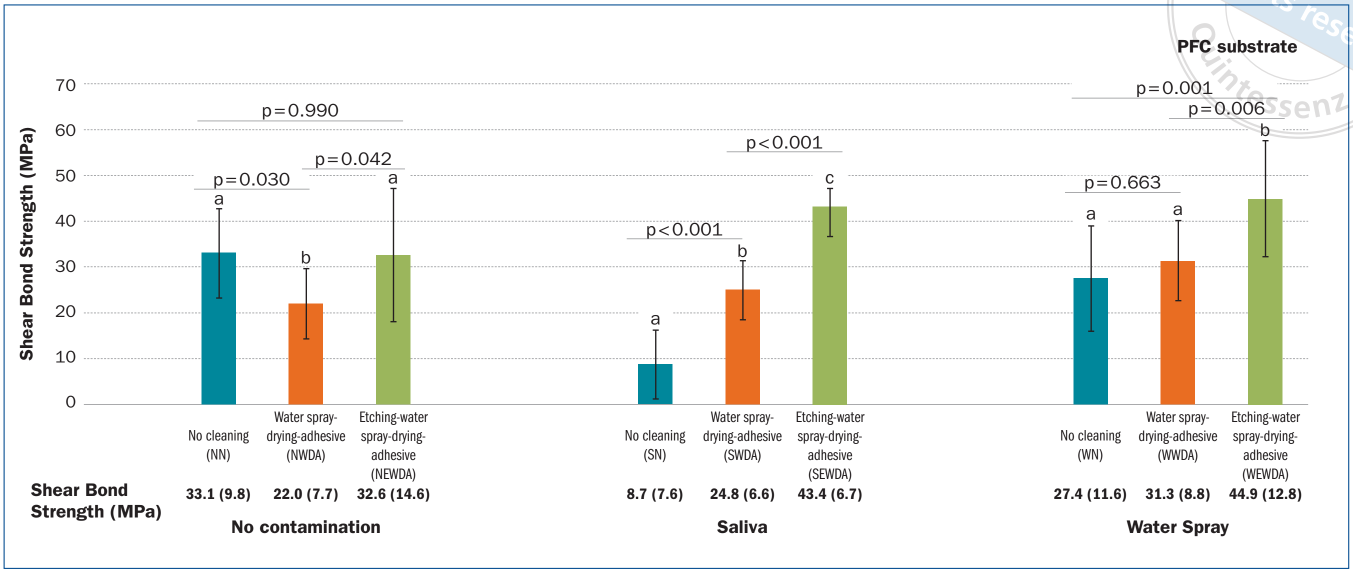

Fig 1 Shear bond strengths between contaminated or uncontaminated PFC substrate and veneering composite (PFC). ANOVA and Levene's test of equality were conducted to determine the homogeneity between the groups. Same letters above columns indicate groups that were statistically similar ($p > 0.05$). Analysis was conducted within the contaminant category.

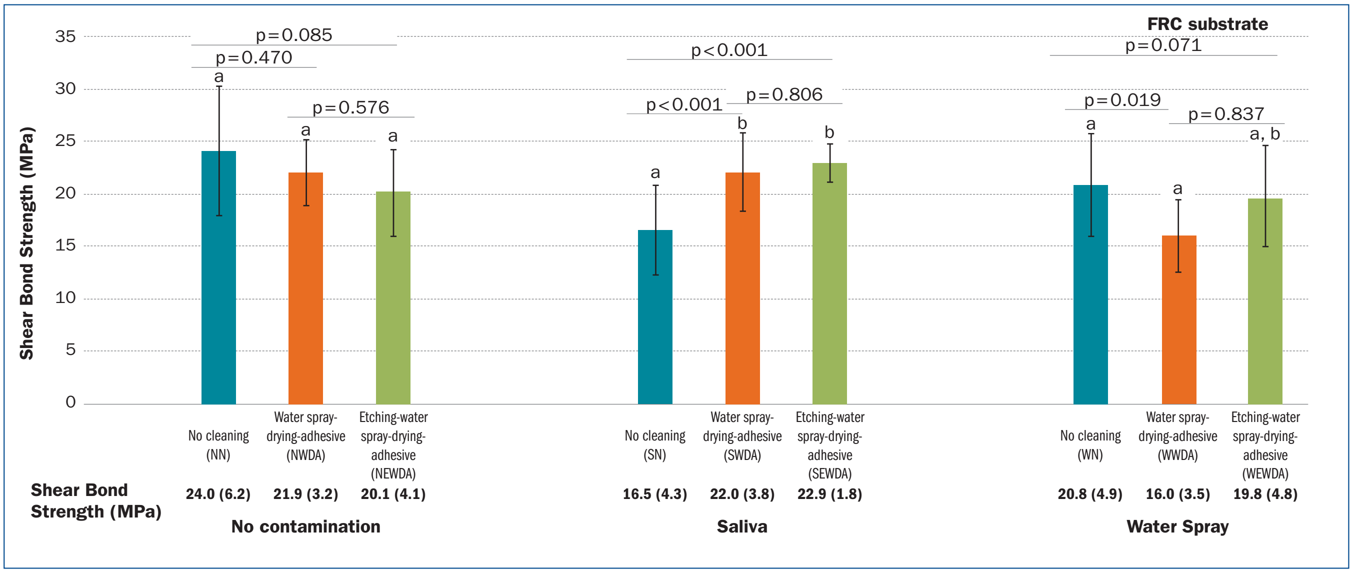

Fig 2 Shear bond strengths between contaminated or uncontaminated FRC substrate and veneering composite (PFC). ANOVA and Levene's test of equality were conducted to determine the homogeneity between the groups. Same letters above columns indicate groups that were statistically similar ($p > 0.05$). Analysis was conducted within the contaminant category.

if the formation of an oxygen inhibition layer (OIL) is prevented.[7] These findings allow the assumption that in immediate repairs, it is of paramount importance to remove the contaminant by any means and to re-establish the OIL.[5-7]

Contaminated, freshly cured FRC substrate should be distinguished from exposed, aged FRC surfaces needing repair: a freshly polymerized FRC layer that has been accidentally contaminated in the oral cavity is not aged, still not saturated with water, or previously stressed in a moist environment. In other words, the contamination is momentary. OIL becomes impaired by the contamination, but the free radical activity continues.[24] On the other hand, aged FRC substrate needing repair has an OIL free surface, is saturated with water, and no longer shows free radical activity. These differences compel the use of different methods of FRC substrate treatment before layering the new veneering composite layer.

Semi-IPN FRC material was used in the present investigation. An EverStick fiber bundle contains densely packed,

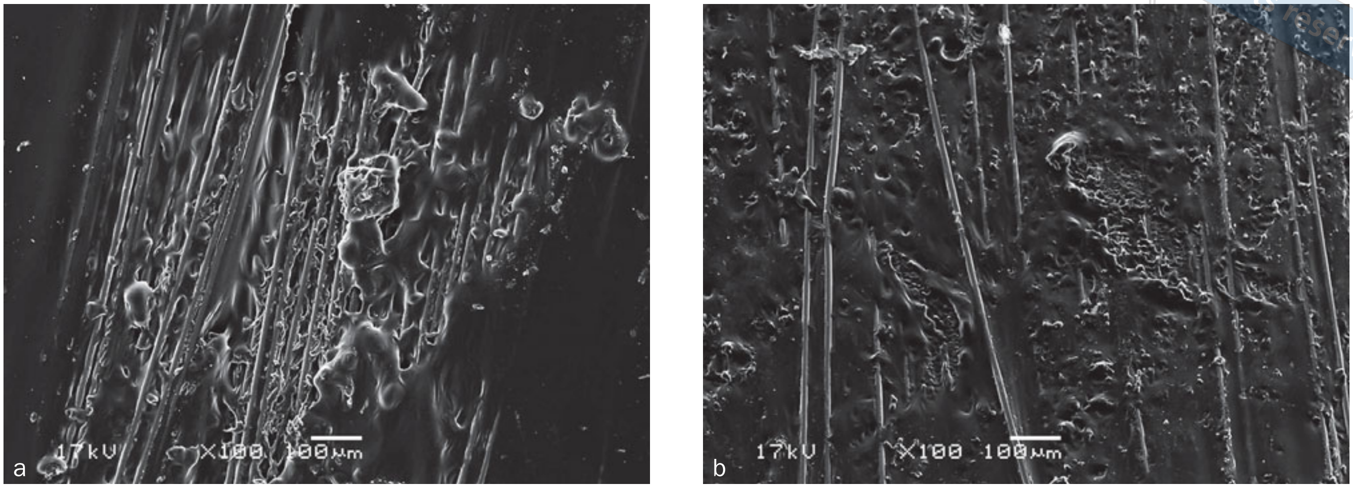

Fig 3 SEM micrograph (100X) shows localized fiber exposure areas after water spraying (A) and phosphoric acid (B) treatment of uncontaminated FRC substrate.

silanated E-glass fibers in a gel matrix. The fiber bundle is non-prepolymerized, and is thus flexible, its gel matrix light-curable. The matrix is multiphase in nature, comprising PMMA (polymethylmetacrylate) chains in a bis-GMA (bisphenol A-glycidyl dimethacrylate) matrix, which is additionally encapsulated by a PMMA layer. This outmost PMMA layer improves the handling and bonding properties of the FRC materials. Once the linear phase (PMMA) has been swollen by the monomers of the newly applied resin, two types of bonding mechanisms occur on the joint side. One is the adhesive bond mediated by monomer diffusion (of the newly applied resin) into the underlying FRC substrate. The second is mechanical, by way of creating micromechanical interlocking upon polymerizing the newly applied resin to the swollen linear phase layer.[14] This is the main mechanism in the repair of aged semi-IPN FRC substrates. Freshly cured FRC layers, however, still possess free radical activity, and this could be utilized in the cases when freshly cured fiber necessitates immediate repair, eg, due to contamination. However, as the activity of the free radicals within the OIL declines with time, it is recommended to complete the composite addition/repair within 30 min of OIL formation.[3] Therefore, in this investigation, contaminants (saliva, water) were applied after the substrates were polymerized, and repairs were performed immediately. Both, but particularly the saliva contamination, reduced the interfacial bond strength for both substrate types (PFC, FRC; SBS loss 73.7% and 31.3%, respectively).

Contaminated surfaces were either cleaned merely with water or etched with phosphoric acid (35%) and cleaned with water spray. Water spray alone is not sufficient to remove the OIL,[2,5] and in water-spray groups, the OIL was probably only dispersed. Consequently, in these groups, the OIL mediated adhesion due to the double bonds preserved in it was still possible (Figs 1 and 2, group codes WN). On the other hand, cleaning with phosphoric acid etching was followed by an adhesive resin application in each group. This procedure allowed bond strength recovery close to the cohesive strength of the material (Figs 1 and 2, group codes NEWDA, SEWDA, and WEWDA). Consequently, it could be anticipated that etching with phosphoric acid was an effective saliva-cleaning method. This effect may be due to denaturing of the salivary proteins, which lose the ability to adhere to the composite surface and then are easily removed through washing and drying. The bond strength recovery thus could have been a result of both contaminant elimination (by phosphoric acid etching) and re-establishing the OIL (new inhibition layer formation after polymerizing the thinly applied adhesive layer). Phosphoric acid etching of the contaminated surface is considered to improve the wettability of the adhesive, which in turn facilitates chemical adhesion between the newly applied composite and the cleaned substrate.[7] Actually, chemical activation in immediate repair seems to be more important than mechanical treatment of the contaminated surface, because curing an adhesive in air over a cleaned surface (renewing the OIL) has been proven to be crucial in recovering the bond strength.[5-7] The finding of the present study concurs with earlier studies, suggesting that regardless of the decontamination protocol used to clean the contaminated surface,[7] re-establishing the OIL could be the key element in immediate repairs.[5-7] The critical time for this could be 30 min.[3]

The original bond strength was higher for the PFC-PFC than for the FRC-PFC (Figs 1 and 2, group codes NN), which could be attributed to the identical chemical composition of the substrate and the adherent material. Nonetheless, PFC was clearly more sensitive to saliva contamination than FRC (SBS loss 73.7% and 31.3% for PFC and FRC, respectively). The high sensitivity of PFC to various contaminants (saliva, water, gloves) is well documented, and findings of the present study are in agreement with earlier reports.[4,5,9,24] On the other hand, studies on FRC contamination and immediate repair are lacking. The present study showed that momentary water contact did not statistically significantly affect

FRC-PFC interface bonding (Fig 2, code WN; SBS loss 13.3%), whereas momentary saliva contact was detrimental to the bond strength (Fig 2, code SN; SBS loss 31.3%). This was confirmed by the failure mode analysis, which showed adhesive failures for all saliva-contaminated substructures. On the other hand, predominantly cohesive failures were observed for all intact specimens and for the water contaminated, etched surfaces. Cohesive failure is a sign for good adhesion between FRC and PFC, which could be due to the resin matrix composition of the semi-IPN FRC, longitudinal fiber orientation, and compatibility of the adhesive used with semi-IPN (everStick). The multipurpose adhesive contains bis-GMA/HEMA.[14] This finding is in accordance with that of other studies.[13] However, adhesive failures occurred frequently, particularly in the saliva-contaminated groups and in the groups with PFC substrate. The adhesive failures could have been a result of a combination of factors, the contaminant type (saliva), aging and stressing the substrate-adherent joint by thermocycling, not treating the substrates mechanically, nor chemically activating them in some groups (the groups with no adhesives), and not leaving the adhesive on the semi-IPN FRC substructure long enough to dissolve the PMMA layer (for the groups with FRC substrate).

As the FRC-PFC bond was not significantly disturbed by momentary water contamination, it might be assumed that sufficient bond strength could have been obtained simply by blowing off the excess water and drying the FRC substrate before applying the intermediate adhesive and subsequent composite layer, as happened after washing off the saliva contaminant from the FRC surface. This, however, was not the case (Fig 2, code WWDA). One possible explanation could be the formation of a double inhibition layer. Namely, a sticky OIL probably remained dispersed on the surface after water spray washing (ie, it was not removed),[2,5] and the newly applied intermediate adhesive layer was polymerized on the top of this dispersed OIL. This may have aided formation of a (second) OIL on the top of the first (dispersed) OIL, resulting thus in a double, ie, thick OIL. The newly developed thick OIL may have led to a significant decrease of the shear bond strength and adhesive failures. The detrimental effect of a thick OIL has also been reported in an earlier study.[3] The fact that bond recovery was achieved by blowing away the excess water and drying the FRC substrate, but without adhesive treatment (Fig 2, code WN), reinforces the finding that a thin OIL mediates adhesion. Interestingly, the same bond strength reduction was not observed for dried, water contaminated PFC surfaces with cured adhesive (Fig 1, code WWDA). This could be related to the surface topography and wettability characteristics and their effect on the OIL thickness. PFC's surface is smoother than FRC's,[12] whereas FRC's is a fibrous material, with a higher ability to retain the OIL in surface irregularities, resulting in thicker OIL. Although same adhesive with a surface free-energy parameter of 64.0 mN/m[25] was used to wet both substrates (PFC, FRC), the surface roughness of the substrate could have modified the wettability. A rougher surface (FRC) would absorb the adhesive (thicker layer), whereas a smoother surface (PFC) would spread the adhesive and result in a thinner OIL. Furthermore, the E-glass fiber orientation can influence the surface wettability because of the roughness of the solid substrate and fiber exposure.[1] As a consequence, taking all factors together (substrate surface roughness, its effect on wettability, and the adhesive layer's OIL thickness), the thinner inhibition layer that developed on the PFC surface[2] most likely allowed complete interdiffusion of the freshly overlaid composite through the oxygen inhibition zone, contributing to the shear bond strength. This observation is in agreement with a previously described phenomenon.[20] Although the OIL thickness of everStick in not known, based on earlier investigations on semi-IPN FRCs (short and unidirectional), it could be assumed that it is the range of 25 μm.[2,29] For comparison, the OIL thickness of PFC (Z250) is 14.5 μm.[2]

The secondary IPN for immediate semi-IPN FRC repairs (when immediately repairing or adding veneering composite to a freshly cured semi-IPN FRC surface that has been contaminated [saliva, water] and cleaned) is not possible, because the time elapsed between the addition of the adhesive and curing is relatively short, thus reducing the chances for interdiffusion; this phenomenon typical is for semi-IPNs, which necessitates a minimum of 3 to 5 min.[11,14,15] On the other hand, polymerizing the adhesive in presence of air enables the formation of new OIL. Therefore, the bond between the substrate and adherent is due to the unreacted double bonds in the adhesive layer's OIL. It should be mentioned that not all adhesives are compatible with semi-IPN FRCs;[13] a bis-GMA and HEMA (hydroxyethylmethacrylate) based adhesive, as used in the present investigation, should be preferred because it matches the semi-IPN FRC resin matrix network.[14,15]

In summary, the present study shows that for freshly polymerized semi-IPN fiber that has been contaminated and cleaned, the adhesion to adjacent veneering composite (immediate repair) is based on the OIL-aided adhesive bond. Although surface treatments did not appear to change the surface morphology, some localized surface exposure of fibers was observed upon water spray and acid etching cleaning (Figs 3a and 3b). This might have provided some mechanical interlocking, which could explain the cohesive breaks in the groups with water-contaminated FRC substructure. The rough, fibrous fiber surface may have contributed to that. For comparison, the corresponding secondary IPN mechanisms of semi-IPN fibers (when repairing old semi-FRC or cementing a semi-FRC dental appliance) are adhesive, due to monomer diffusion into the aged fiber substrate and micromechanical interlocking created upon polymerizing the newly applied resin layer to the swollen linear phase layer.[14]

One limitation of the present study was that only two surface cleaning treatments were used. However, as mentioned earlier, other surface cleaning methods have not been proven to significantly contribute to the immediate repair bond.[7,18] Hence, this limitation of the current study design could be considered as relative. Furthermore, only one FRC type was tested and other resin matrices (cross-linked) were not included. Also, adhesives containing silane were not used. Further studies are needed to analyze the surface morphology changes upon various surface contaminants (saliva, blood) and surface treatment techniques (bur grinding, sandblasting) used to clean them. Finally, only clinical studies can truly show the longevity and the typical failures in freshly cured and immediately repaired semi-IPN-based FRC materials.

CONCLUSION

The null hypothesis was rejected. First, PFC is significantly more sensitive to contaminants than is semi-IPN-based FRC. Both saliva and water contaminants alter the bond strength, but phosphoric acid etching of the contaminated surface recovers the bond strength. Second, as a consequence, the best course of treatment upon saliva or water contamination of a PFC surface is phosphoric acid etching of the contaminated surface before intermediate adhesive and subsequent composite layer application, which should be cured separately. This is in agreement with previous reports.[4,9,17,21] Upon momentary water contamination of semi-IPN FRC substructure, merely drying the surface before applying the veneering material sufficiently recovers the bond strength. Adhesive resin application in this case is not necessary, as it does not provide any advantage. If a semi-IPN FRC surface is saliva contaminated, then phosphoric acid etching and subsequent washing of the etchant should be used prior to repair, and after drying, the surface treatment should be completed by adhesive and composite application cured separately. The adhesive layer acting as intermediate between the substrate (FRC, PFC) and the adherent (PFC) should be polymerized, in order to wet the cleaned substrate surface and develop a new OIL.

REFERENCES

1. Abdulmajeed AA, Lassila LV, Vallittu PK, Närhi TO. The effect of exposed glass fibers and particles of bioactive glass on the surface wettability of composite implants. Int J Biomater 2011;2011:607971.
2. Bijelic-Donova J, Garoushi S, Lassila LV, Vallittu PK. Oxygen inhibition layer of composite resins: effect of layer thickness and surface layer treatment on the interlayer bond strength. Eur J Oral Sci 2015;123:53-60.
3. Boyer DB, Chan KC, Torney DL. The strength of multilayer and repaired composite resin. J Prosthet Dent 1978;39:63–67.
4. Chiba K, Hosoda H, Fusayama T. The addition of an adhesive composite resin to the same material: bond strength and clinical techniques. J Prosthet Dent 1989;61:660–675.
5. Eiriksson SO, Pereira PN, Je Swift EJ, Heymann HO, Sigurdsson A. Effect of saliva contamination on resin-resin bond strength. Dent Mater 2004;20:37–44.
6. Eiriksson SO, Pereira PN, Swift EJ, Heymann HO, Sigurdsson A. Effect of blood contamination on resin-resin bond strength. Dent Mater 2004;20:184–190.
7. El-Askary FS, Fawzy AS, Abd Elmohsen HM. Tensile bond strength on immediately repaired anterior microfine hybrid restorative composite using nontrimmed hourglass specimens. J Adhes Dent 2009;11:41–47.
8. Frese C, Decke C, Rebholz J, Stucke K, Staehle HJ, Wolff D. Original and repair bond strength of fiber-reinforced composites in vitro. Dent Mater 2014;30:456–462.
9. Furuse AY, da Cunha LF, Benetti AR, Mondelli J. Bond strength of resin-resin interfaces contaminated with saliva and submitted to different surface treatments. J Appl Oral Sci 2004;15:501–505.
10. Gauthier MA, Stangel I, Ellis TH, Zhu XX. Oxygen inhibition in dental resins. J Dent Res 2005;84:725–729.
11. Kallio TT, Lastumäki TM, Vallittu PK. Effect of resin application time on bond strength of polymer substrate repaired with particulate filler composite. J Mater Sci Mater Med 2003;14:999–1004.
12. Lassila LV, Garoushi S, Tanner J, Vallittu PK, Söderling E. Adherence of Streptococcus mutans to fiber-reinforced filling composite and conventional restorative materials. Open Dent J 2009;3:227–232.
13. Lassila LVJ, Tezvergil A, Dyer SR, Vallittu PK. The bond strength of particulate-filler composite to differently orientated fiber-reinfroced composite substrate. J Prosthodont 2007;16:10–17.
14. Lastumäki TM, Kallio TT, Vallittu PK. The bond strength of light-curing composite resin to finally polymerized and aged glass fiber-reinforced composite substrate. Biomaterials 2002;23:4533–4539.
15. Mannocci F, Sherriff M, Watson TF, Vallittu PK. Penetration of bonding resins into fibre-reinforced composite posts: a confocal microscopic study. Int Endod J 2005;38:46–51.
16. Martins NM, Schmitt GU, Oliveira HL, Madruga MM, Moraes RR, Cenci MS. Contamination of composite resin by glove powder and saliva contaminants: impact on mechanical properties and incremental layer debonding. Oper Dent 2015;40:396–402.
17. Perriard J, Lorente MC, Scherrer S, Belser UC, Wiskott HW. The effect of water storage, elapsed time and contaminants on the bond strength and interfacial polymerization of a nanohybrid composite. J Adhes Dent 2009;11:469–478.
18. Rinastiti M, Özcan M, Siswomihardjo W, Busscher HJ. Effects of surface conditioning on repair bond strengths of non-aged and aged microhybrid, nanohybrid, and nanofilled composite resins. Clin Oral Investig 2011;15:625–633.
19. Smimazu K, Karibe H, Ogata K. Effect of artificial saliva contamination on adhesion of dental restorative materials. Dent Mater J 2014;33:545–550.
20. Suh BI. Oxygen-inhibited layer in adhesion dentistry. J Esthet Restor Dent 2004;16:316–323.
21. Tantbirojn D, Fernando C, Versluis A. Failure strengths of composite additions and repairs. Oper Dent 2015;40:364–371.
22. Tezvergil A, Lassila L, Yli-Urpo A, Vallittu PK. Repair bond strength of restorative resin composite applied to fiber-reinforced composite substrate. Acta Odontol Scand 2004;62:51–60.
23. Townsend RD, Dunn WJ. The effect of saliva contamination on enamel and dentin using a self-etching adhesive. J Am Dent Assoc 2004;135:895–901.
24. Truffier-Boutry D, Place E, Devaux J, Leloup G. Interfacial layer characterization in dental composite. J Oral Rehabil 2003;30:74–77.
25. Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, Miyazaki M. Bonding performance and interfacial characteristics of short fiber-reinforced composite in comparison with other composite restoratives. Eur J Oral Sci 2016;124:301–308.
26. Vallittu PK. Interpenetrating polymer networks (IPNs) in dental polymers and composites. J Adhes Sci Technol 2009;23:961–972.
27. von Beetzen M, Li J, Nicander I, Sundström F. Factors influencing shear strength of incrementally cured composite resins. Acta Odontol Scand 1996;54:275–278.
28. Wolff D, Coupek M, Erber R, Krueger J, Stahle HJ, Frese C. Effect of aqueous storage on original and repair bond strength and residual monomer release of fiber-reinforced composites. J Adhes Dent 2016;18:535–543.
29. Wolff D, Geiger S, Ding P, Staehle HJ, Frese C. Analysis of the interdiffusion of resin monomers into pre-polymerized fiber-reinforced composites. Dent Mater 2012;28:541-547.

Clinical relevance: Upon saliva or water contamination of freshly cured semi-IPN based fiber-reinforced composite, surfaces should be re-prepared via phosphoric acid etching, water rinsing, drying and application of adhesive, in order to recover the bond strength. The same surface treatment is also recommended for momentarily contaminated particulate filler composite.