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Adhesion of Resin Composite to Hydrofluoric Acid-exposed Enamel and Dentin in Repair Protocols

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Clinical Relevance

When dental tissues are to be repaired next to ceramic, enamel or dentin should be etched with phosphoric acid before ceramic is etched with hydrofluoric acid gel.

SUMMARY

Intraoral repairs of ceramic fixed-dental-prostheses (FDP) often include cervical recessions that require pretreatment of the exposed tooth surfaces either before or after the ceramic is conditioned with hydrofluoric (HF) acid gel. The sequence of repair protocol may cross-contaminate the exposed etched enamel or dentin surfaces during the application or rinsing process and thereby affect the adhesion. This study evaluated the influence of HF acid gel with two concentrations on bond strengths of composite to enamel and dentin. Human third molars (N=100, n=10 per group) with similar sizes were selected and randomly divided into 10 groups. Flat surfaces of enamel and dentin were created by wet ground finishing. Before or after the enamel (E) or dentin (D) was conditioned with phosphoric acid (P), substrate surfaces were conditioned with either 9.5% HF ($HF_{9.5}$) or 5% HF (HF_5). Subsequently, a bonding agent (B) was applied. The experimental groups by conditioning sequence were as follows where the first letter of the group abbreviation represents the substrate (E or D) followed by the acid type and concentration: group 1 ($EPHF_{9.5}$), group 2 ($EPHF_5$), group 3 ($EHF_{9.5}P$), group 4 (EHF_5P), group 5 ($DPHF_{9.5}$), group 6 ($DPHF_5$), group 7 ($DHF_{9.5}P$), and group 8 (DHF_5P). Group 9 (EPB) and group 10 (DPB) acted as the control groups. Repair resin was adhered incrementally onto the conditioned enamel and dentin in polyethyl-

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ene molds. Each layer was photo-polymerized for 40 seconds. All specimens were thermocycled (×1000, 5°-55°C) and subjected to shear test (universal testing machine, 1 mm/min). Specimens that debonded during thermocycling were considered as 0 MPa. The bond strength data were analyzed using Kruskal-Wallis test and failure types using the chi-square test (α=0.05). Overall, the bond results (MPa) were lower on dentin than on enamel ($p<0.01$). EPB (25.6 ± 6.6) and DPB (20.2 ± 4.9) control groups showed significantly higher results than those of other groups ($p<0.05$). While higher mean bond strengths were obtained in group 1 ($EPHF_{9.5}$) (11.5 ± 2.1) and group 2 ($EPHF_5$) (7.3 ± 0.6), lower results were obtained when HF acid gels were applied prior to phosphoric acid ($EHF_{9.5}P$: 5.0 ± 1.1, EHF_5P: 3.6 ± 0.1) ($p<0.05$). On dentin, the results were the lowest in group 8 (DHF_5P: 1.5 ± 1.6), being significantly lower than those of group 5 ($DPHF_{9.5}$) ($p<0.05$). Scanning electron microscope (SEM) images revealed predominantly mixed failures with less than half of the composite left on both enamel and dentin surfaces (64 out of 80) ($p<0.05$), indicating that in general, adhesion was not ideal. Contamination of the enamel or dentin surfaces with HF acid gel impairs the bond strength of composites. Considering both the bond strength results and failure types, when dental tissues are to be repaired next to ceramic, application of phosphoric acid before HF acid gel application can be recommended. HF acid gel concentration did not influence the results except on enamel.

INTRODUCTION

In extensive fixed-dental-prostheses (FDP), technical and biological problems reduce the survival rates of such restorations.[1,2] One potential technical problem is chipping or fracture of the veneering ceramics. Ceramic fractures may result from a wide range of factors such as improper framework design, micro-defects in ceramics, inadequate framework support, excessive thickness of veneering ceramic, poor abutment preparation, technical errors, incompatible thermal coefficients between the metal or core ceramic and the veneering ceramic, parafunctional habits, trauma, or occlusal prematurity.[3,4] In such situations, it may be desirable to repair the failed ceramic part of the FDP intraorally since prosthesis removal could possibly destroy the entire restoration or damage the abutment teeth.[5,6] Repairs could increase the clinical longevity of failed restorations and offer the dentist and the patient a cost-effective alternative when compared to replacement options.[5]

In principle, repair procedures for composites,[7] metals, and ceramics[8-12] rely on cleaning and roughening the surface and subsequent application of a coupling agent to make covalent bonds with the methacrylate groups of the adhesive promoter or the resin-based composites (hereon: composite).

The repair of ceramic restorations could be accomplished by either employing surface conditioning methods and adhesive promoters intraorally or indirectly by removing the restoration and baking a new layer of ceramic in the laboratory.[6,13] Intraoral repair methods for ceramics are based on micromechanical retention and chemical adhesion. These can be accomplished by using hydrofluoric (HF) acid gel followed by the application of a silane coupling agent. Such direct ceramic repair systems show excellent durability.[8-10,12,14-20] Since HF is a hazardous compound to use *in vivo*, air-abrasion methods using alumina-coated silica particles in combination with a silane coupling agent (ie, CoJet) have been suggested for conditioning ceramic surfaces,[21] but this application may damage the ceramic glaze.[22] Unfortunately, especially after the long-term service life of FDP, cervical recession may be encountered that needs to be covered during intraoral repairs. Depending on the patient's hygiene conditions, when such cervical recessions are not restored, the exposed dental surfaces may yield to caries or periodontal problems.[23] Moreover, due to recession, microleakage, caries, or discolorations may take place at the crown margins.[24] These may cause esthetic problems particularly in the anterior region. Gingival recessions or apical margins of the restorations could be covered using coronally positioned flap techniques.[25,26] However, maintenance of the achieved situation is not always predictable, and some patients may not be willing to undergo surgical procedures.

The exact conditioning protocol for complex substrates in such a situation, namely dental tissues being next to ceramic, is not known to date. Theoretically, repairs at cervical recessions would require cervical tooth surface conditioning either before HF acid gel application or after. Although HF acid gel application is crucial in conditioning glass ceramics, this process may contaminate the exposed etched enamel or dentin surfaces during etching procedures or rinsing.[27] When fluoride reacts with Ca in dentinal tubules, CaF_2 is formed, and this yields to tubular occlusion.[28] Hence, impaired bond strength of composites to dentin could be expected. Also, concentration of the etching gels varies, and this may influence the results.

The objectives of this study therefore, were to evaluate the bond strengths of a composite to enamel and dentin using different adhesion protocols, employing HF acid gel at two concentrations either before or after application of phosphoric acid, and to assess the failure types. The null hypothesis tested was that the application of HF acid gel before etching enamel and dentin with phosphoric acid would not affect the shear bond strength.

MATERIALS AND METHODS

The product names, manufacturers, chemical compositions, and batch numbers of the materials used in this study are listed in Table 1.

Specimen Preparation

Eighty human third molar teeth (N=100, n=10 per group) with similar sizes stored in distilled water with 0.1% thymol solution at room temperature were selected from a pool of recently extracted teeth. The teeth were stored up to a maximum of 5 months prior to the experiments.

The roots were sectioned under cooling, and the crowns were embedded in metal rings using autopolymerized polymethylmethacrylate (AutoPlast, Candulor, Switzerland) with the adhesion surface exposed. The specimens were stored in distilled water for up to 1 week until the experiments. They were then randomly divided into two groups. In half of the groups, flat enamel surfaces were achieved after wet ground finishing using 1200 grit silicone carbide abrasive under water cooling (Struers A/S, Rodovre, Denmark) (groups 1 to 4); in the remaining groups, dentin was exposed in the buccolingual direction (groups 5 to 8) with fine diamond burs (Swiss Dental Products, Intensiv SA, Grancia, Switzerland; batch 2309) under controlled conditions and then wet ground finished as enamel surfaces. The enamel and dentin surfaces were further confirmed under stereomicroscope (Lucia, Nikon Corporation, Tokyo, Japan) prior to bonding procedures.

Surface Conditioning Methods

Experimental groups, conditioning sequences, and group abbreviations are presented in Figure 1.

Table 1: *The Product Names, Manufacturers, Compositions, and Batch Numbers of the Materials Used in This Study*

Product name	Manufacturer	Chemical Composition	Batch Number
PMMA	AutoPlast, Candulor AG, Altstätten, Switzerland	Polymethylmethacrylate	F42028
Etching gel	Ivoclar Vivadent, Schaan, Liechtenstein	35% Orthophosphoric acid	6HG
IPS Empress HF gel	Ivoclar Vivadent, Schaan, Liechtenstein	5% Hydrofluoric acid	J12407
Neutralizer for IPS ceramic etching gel	Ivoclar Vivadent, Schaan, Liechtenstein	Calcium carbonate and sodium bicarbonate	J16639
Ultradent Porcelain Etch	Ultradent Products Inc, South Jordan, UT, USA	9.5% Hydrofluoric acid	165092
Ultradent EtchArrest	Ultradent Products Inc, South Jordan, UT, USA	Calcium carbonate and sodium bicarbonate	P129
ExciTE	Ivoclar Vivadent, Schaan, Liechtenstein	Phosphoric acid acrylate, HEMA, dimethacrylate, silicon dioxide, initiators and stabilizers	J01968
Tetric EvoCeram	Ivoclar Vivadent, Schaan, Liechtenstein	Dimethacrylates (17%-18%), fillers (82%-83%), additives, catalysts, stabilizers	260950

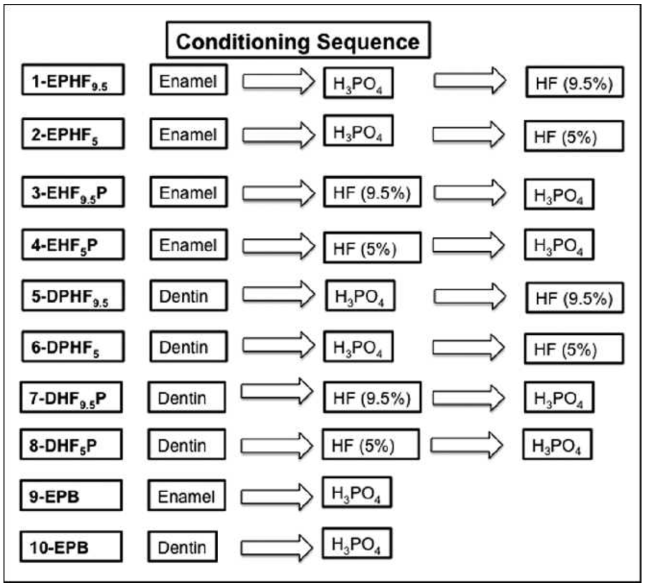

Figure 1. *Experimental groups, group abbreviations, and the conditioning sequences on enamel and dentin.*

Groups 1 to 4: Enamel surfaces were etched with 35% H_3PO_4 for 30 seconds, rinsed with copious water for 30 seconds, and gently dried with oil-free air until a frosty surface was achieved. The HF acid gel (either 9.5% or 5%) was applied for 20 seconds and rinsed with water for 60 seconds. They were then neutralized with the diluted solution of neutralizing powder ($CaCO_3$ and Na_2CO_3)[29] and washed thoroughly for 20 seconds[27] with water and then air-dried. Subsequently, the adhesive resin (ExciTE) was applied with a microbrush and photo-polymerized from a distance of 2 mm for 10 seconds using an LED photo-polymerization unit (Elipar Freelight 2, 3M ESPE, Seefeld, Germany) with a light intensity of 1000 mW/cm^2.

Groups 5 to 8: In these groups, the same procedures were applied as described for groups 1 to 4 but this time on dentin.

Group 9 (control-enamel): Enamel surfaces were etched only with 35% H_3PO_4 for 30 seconds, rinsed with copious water for 30 seconds, and gently dried. The adhesive resin was applied as described above.

Group 10 (control-dentin): Dentin surfaces were etched with only 35% H_3PO_4 for 15 seconds, rinsed with copious water for 15 seconds, and gently dried. The adhesive resin was applied.

After conditioning the enamel and dentin surfaces, nano-hybrid composite (Tetric EvoCeram), representing the repair composite, was packed into the polyethylene molds (inner diameter: 3.5 mm, height: 4 mm) with a hand instrument and photo-polymerized incrementally in layers of not more than 2 mm. After polymerization, the polyethylene molds were gently removed from the test specimens. All specimens were thermocycled 1000 times (5°-55°C, dwell time: 30 seconds, transfer time from one bath to the other: 5 seconds) (Willytec, Gräfelfing, Germany).[30]

Testing Procedure and Failure Analysis

Specimens were mounted in the jig of the universal testing machine (Autograph Model AG-50kNG, Shimadzu, Japan) specific for the shear test. The force was applied to the tooth/composite interface using a shearing blade with a 45° bevel at its tip until failure occurred. The load was applied at a crosshead speed of 1 mm/min, and the stress-strain curve was analyzed with the software program.

Cold field emission scanning electron microscope (SEM) (JSM 5200, Kyoto, Japan) images were made at 25 kV at a magnification of 600×. The debonded enamel/dentin surfaces were first sputter-coated with a 3-nm thick layer of gold (80%)/palladium (20%) prior to examination. The failure types were defined as either "adhesive" with no composite left on the enamel/dentin (score 0) or "mixed" with less than half of the composite left on the surface (score 1).

Statistical Analysis

Statistical analysis was performed using SPSS 11.0 software for Windows (SPSS Inc, Chicago, IL, USA). The data were found not to be normally distributed with unequal variance (Kolmogorov-Smirnov and Shapiro-Wilk, α=0.05). Accordingly, Kruskal-Wallis nonparametric analysis was carried out to determine the significant differences between groups. Differences between failure types were analyzed using a chi-square test. p values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

Specimens debonded during thermocycling were considered as 0 MPa. Mean bond strength (MPa) results and significant differences between the experimental groups are presented in Table 2. EPB (25.6 ± 6.6) and DPB (20.2 ± 4.9) control groups showed significantly higher results than those of other groups ($p<0.05$). In groups representing repair protocols, on enamel, while the higher mean bond strengths were obtained in group 1 ($EPHF_{9.5}$) (11.5 ± 2.1) and group 2 ($EPHF_5$) (7.3 ± 0.6), significantly lower results were obtained when HF acid gels were

Table 2: *The Mean Shear Bond Strength Values (MPa) (±Standard Deviations) for the Experimental Groups*

Groups[a]	**Mean (±SD)**	**Homogeneous Groups[b]**
1 ($EPHF_{9.5}$)	11.5 ± 2.1	A
2 ($EPHF_5$)	7.3 ± 0.6	B
3 ($EHF_{9.5}P$)	5.0 ± 1.1	C
4 (EHF_5P)	3.6 ± 0.1	C D
5 ($DPHF_{9.5}$)	3.5 ± 1.4	C D
6 ($DPHF_5$)	2.2 ± 1.2	D E
7 ($DHF_{9.5}P$)	1.9 ± 1.4	D E
8 (DHF_5P)	1.5 ± 1.6	E
9 (EPB)	25.6 ± 6.6	F
10 (DPB)	20.2 ± 4.9	G

[a] *For group descriptions, see Figure 1.*
[b] *The same letters in the same column indicate no significant differences; α=0.05.*

applied prior to phosphoric acid ($EHF_{9.5}P$: 5.0 ± 1.1, EHF_5P: 3.6 ± 0.1) ($p<0.05$). On dentin, the results were the lowest in group 8 (DHF_5P: 1.5 ± 1.6) showing significantly lower mean values than those of group 5 ($DPHF_{9.5}$) ($p<0.05$) (Figure 2).

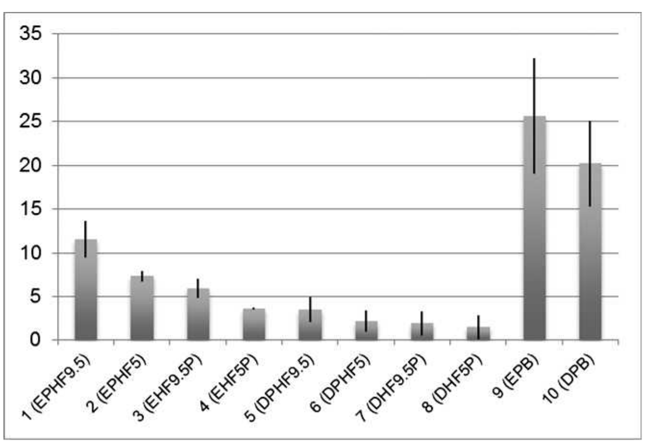

Figure 2. *Shear mean bond strength values (MPa) for enamel and dentin after conditioning sequences.*

Table 3: *Distribution and Frequency of Failure Types Per Experimental Group Analyzed After Bond Strength Test: Score 0 - Adhesive: No Composite Left on the Enamel/Dentin; Score 1 - Mixed: Less Than Half of the Composite Left on the Surface*

Groups[a]	**Dislodged[b]**	**Score 0[c]**	**Score 1[c]**
1 ($EPHF_{9.5}$)	0	6^b	4^A
2 ($EPHF_5$)	0	10^a	0^B
3 ($EHF_{9.5}P$)	5	5^b	0^B
4 (EHF_5P)	3	7^a	0^B
5 ($DPHF_{9.5}$)	0	8^a	2^B
6 ($DPHF_5$)	0	10^a	0^B
7 ($DHF_{9.5}P$)	0	10^a	0^B
8 (DHF_5P)	2	8^a	0^B
9 (EPB)	0	1^c	9^C
10 (DPB)	0	4^b	6^A

[a] *For group descriptions, see Figure 1.*
[b] *Debonding during thermocycling.*
[c] *Same superscript letters indicate no significant differences in each column ($p>0.05$).*

After evaluating all SEM images, all failure types were noted as interfacial failures between the resin and the enamel/dentin substrate. Failure analysis revealed predominantly adhesive failures (score 0: 64 out of 80) and a few mixed failures (score 1: 6 out of 80) both on enamel and dentin surfaces (Table 3). Score 0 was significantly more frequently observed in Groups 2, 4, 5, 6, 7, and 8 than in groups 1, 3, 9, and 10 ($p<0.05$). The highest incidence of score 1 was in the control enamel group (group 9) ($p<0.05$).

Representative SEM images of failure types from enamel and dentin groups are presented in Figure 3a-d.

DISCUSSION

This study tried to simulate the repair protocols where exposed tooth surfaces (enamel or dentin) were present next to ceramic restorations. The aim was to suggest the most reliable repair sequence

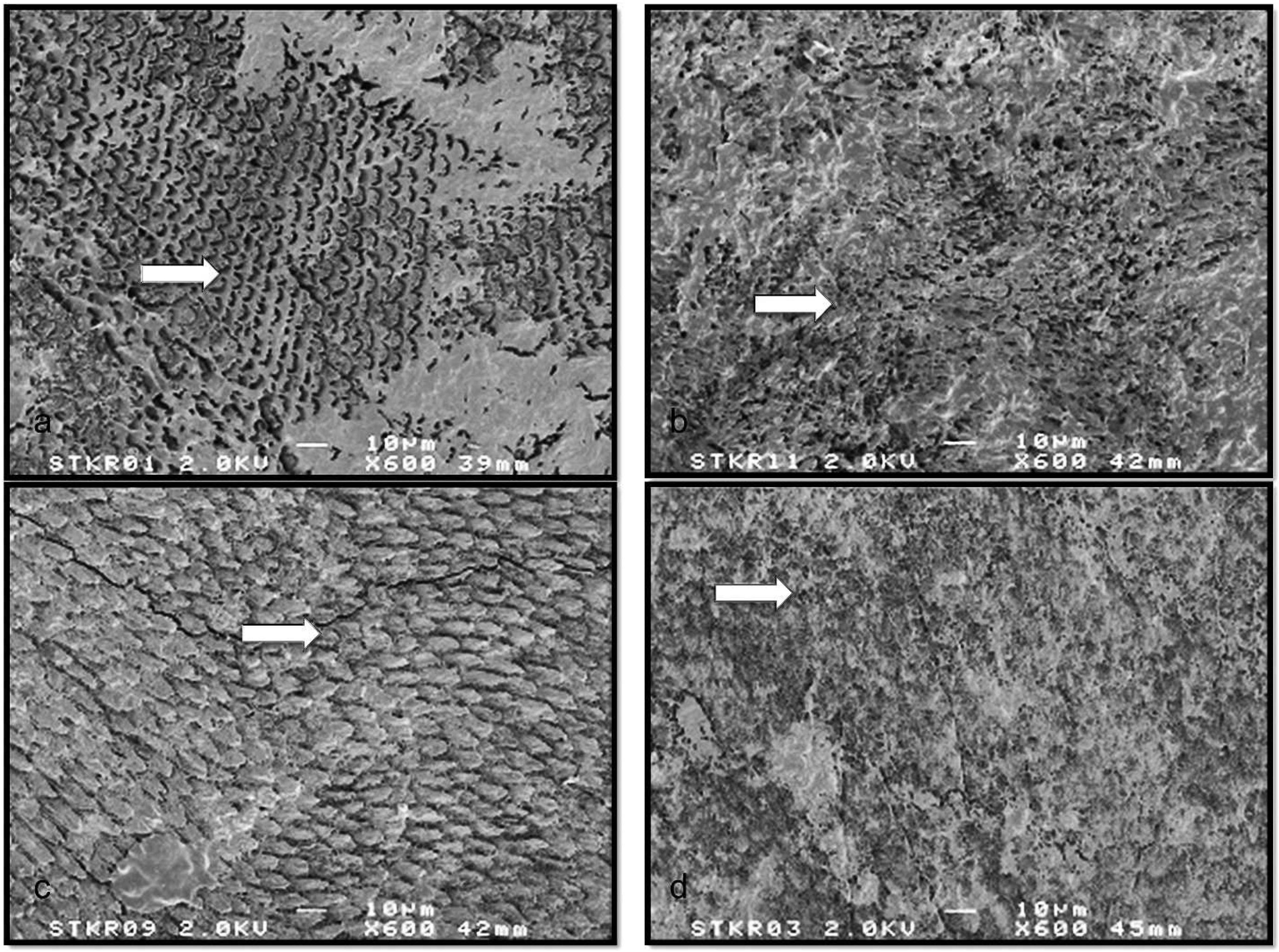

Figure 3. *SEM images of representative specimens of mixed failures with visible areas of etched enamel and dentin surfaces. Left panel from enamel (a,c), right panel from dentin surfaces (b,d). Note the absence of resin covering enamel or dentin tubules indicating unfavorable adhesion (indicated by arrow).*

even in the case of possible contamination of the enamel and dentin with HF acid gel which was evident in previous morphologic studies.[27,28]

Regardless of the repair protocol tested, the mean bond strength results were higher on enamel than on dentin. The bond strength of composite to etched enamel in this study was not reached by any of the other conditioning sequences tested. Hence, the obtained results clearly indicated the inhibiting effect of HF acid gel on bond strength when it is applied either before or after conditioning enamel. Since higher results were obtained in group 1, where enamel was conditioned first with 35% H_3PO_4, this sequence could result in clinically more durable repairs. In repair situations where enamel is present near a ceramic inlay, onlay, overlay or laminate chipping or fracture occasions, this sequence could be recommended.

Unfortunately, such repair actions are usually required in the case of recessions that are observed at the cemento-enamel junction or even on dentin or cement surfaces. Therefore, enamel may not always be available in the areas to be repaired. The results of this study with all repair protocols were inferior in the dentin groups (groups 5 to 8) compared to the results obtained from enamel groups (groups 1 to 4). Although adhesion of composites was not studied, in a previous study where dentin surfaces were characterized after HF acid gel application, dense amorphous precipitates of fluoride were observed on the peritubular zone.[27] Aggressive demineralization of dentin by strong acids causes a collapse, probably denaturing the collagen covering the surface of the dentin and internal walls of the tubules.

Investigators speculated that aggressive demineralization of dentin might result in the production of a deep demineralized layer that is inaccessible to complete resin infiltration during the priming step.[28] This phenomenon could be the reason for low results in groups 7 and 8 where HF acid application was conducted first. In groups 5 and 6, application of 35% H_3PO_4 first on dentin did not differ, both being less than the controls. Nevertheless, HF acid gel application before etching enamel and dentin with phosphoric acid tends to decrease the results without always being statistically significant in the dentin group. Since the data were not normally distributed, the hypothesis could not be accepted.

The obtained results should also be coupled with the failure types. The majority of the observed failure types were adhesive between the enamel/dentin and the composite (score 0: 64 out of 80) indicating unfavorable adhesion. Furthermore, in some groups, especially when HF acid was applied prior to enamel etching, incidental debondings were observed during thermocyling. In order to represent worst-case scenarios, these debondings were considered as 0 MPa.

It can be expected that during rinsing, the contamination of HF gel would be less severe than during acid etching. In that respect, in this study two HF acid gels were studied with two concentrations, namely Ultradent Porcelain Etch gel (9.5%) and IPS Empress HF gel (5%). The results showed only significant differences in the first two enamel groups (groups 1 and 2). Concentration, however, seems to be effective only on enamel. Based on the results of this present study, since viscosity properties are not measured, it cannot be identified whether HF acid gel viscosity affects the results. It can, however, be stated that micromechanical retention obtained by phosphoric acid was possibly dominating over the contamination effect of subsequent steps of the repair protocol. During rinsing of the acid, probably diluted HF would be in contact with the exposed dental tissues, and this may impair the results less than those obtained in this study. The results of this study add to the findings of the previous studies where structural changes and obliterations were noted on dentin after HF gel application.[27,28] Yet, the results of this study could be used to make the same clinical recommendations on the sequence of repair protocols, accepting the fact that dentin obliteration was expected when HF gel is in contact with dentin.

When adhesion protocols involve several steps, the possibility of such cross-contamination or operator factor may come into play. To reduce such problems while avoiding inadequate adhesion, a two-step etch and rinse bonding system was used in this study. The results of this study should also be compared with a three-step etch-and-rinse adhesive system. This may then contaminate the ceramic surface with the primer. Similarly, further aging protocols on dentin need to be investigated.[31] Nonetheless, since there are no real guidelines regarding when to repair or replace such restorations in case repairs need to be undertaken at the margins of ceramic FDPs, impaired adhesion could be expected, and this needs to be communicated with the patient. The obtained results were also lower than the bond strength data reported for composite-ceramic adhesion.[8-10,16,20]

Furthermore, in this study, the dentin was exposed in the buccolingual direction in order to obtain flat surfaces with sufficient bonding surface area. In fact, cervical dentin may represent a more realistic situation, but occlusion of tubules by mineral deposits as a result of physiologic or pathologic sclerosis should be taken into consideration.[32] When metal surface is also exposed in the fracture, they need to be air-abraded with alumina[33] or silica-coated alumina and then silanized.[22] The ceramic part may require cleaning with phosphoric acid.[33] Although the effect of alumina abrasion on metal or phosphoric acid application on ceramic was reported to result in low repair bond strengths,[33] such applications may change the results obtained in this study.

CONCLUSIONS

From this study, the following could be concluded:

1) Contamination of the enamel or dentin surfaces with HF acid gel impairs the repair bond strength of composites since the results were significantly lower than those of the control groups.
2) Starting the repair action with enamel/dentin conditioning with phosphoric acid etching diminishes the bond strength of repair composite due to possible contamination and the inhibiting effect of HF acid gel.

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