Adhesive application before hydrofluoric acid etching during repair procedure in dentistry?

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Abstract: Purpose To evaluate the influence of hydrofluoric acid (HF) contamination on the microtensile bond strength (TBS) during dental repair procedures before and after application of different dentin adhesives (Optibond FL and Optibond XTR). Materials and methods Thirty-five human molars were ground down into the dentin and were randomly divided into seven groups (G1-G7; n = 5), G1 and G2 being the control groups. Only in the test groups (G3-G7) samples were subjected to HF (9.5%) contamination. Two adhesive systems, Optibond FL (G1, G3, G4, G6) and Optibond XTR (G2, G5, G7) were used. In G3-G5 the adhesive was applied before and after contamination, the test groups G6 and G7 were treated with a single adhesive application after contamination. After composite build-up, samples were stored in water (7 d) and TBS was determined. Data were evaluated using Wilcoxon test (p < 0.05). Results Control group G2 showed significantly higher TBS than G1. The HF-contamination did not result in a significant reduction of TBS. The TBS of the test groups treated with OptiBond XTR (G5, G7) were significantly higher than the test groups treated with OptiBond FL (G3, G4, G6) following the same procedure. Conclusions HF-contamination of dentin or the adhesive layer does not significantly impair the bond strength, if the adhesive is subsequently re-applied.

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Adhesive application before hydrofluoric acid etching during repair procedure in dentistry?

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**Purpose:** To evaluate the influence of hydrofluoric acid (HF) contamination on the microtensile bond strength ($\mu$TBS) during dental repair procedures before and after application of different dentin adhesives (OptiBond FL and OptiBond XTR).

**Materials and methods:** Thirty-five human molars were ground down into the dentin and were randomly divided into seven groups (G1-G7; n = 5), G1 and G2 being the control groups. Only in the test groups (G3-G7) samples were subjected to HF (9.5%) contamination. Two adhesive systems, OptiBond FL (G1, G3, G4, G6) and OptiBond XTR (G2, G5, G7) were used. In G3-G5 the adhesive was applied before and after contamination, the test groups G6 and G7 were treated with a single adhesive application after contamination. After composite build-up, samples were stored in water (7 d) and $\mu$TBS was determined. Data were evaluated using Wilcoxon test (p < 0.05).

**Results:** Control group G2 showed significantly higher $\mu$TBS than G1. The HF-contamination did not result in a significant reduction of $\mu$TBS. The $\mu$TBS of the test groups treated with OptiBond XTR (G5, G7) were significantly higher than the test groups treated with OptiBond FL (G3, G4, G6) following the same procedure.

**Conclusions:** HF-contamination of dentin or the adhesive layer does not significantly impair the bond strength, if the adhesive is subsequently re-applied.

1. Introduction

Clinical complications in form of fractures are still occurring in all ceramic systems. With increasing popularity of all-ceramic restorations the number of failures has increased, as these appear to be more susceptible to fractures of the veneering porcelain [1]. The functional and aesthetic rehabilitation by means of a repair filling is a time-saving, cost-effective and above all, tooth substance-conserving alternative to the replacement of the whole prosthetic restoration [2]. Repair fillings should also be mentioned in the context of the treatment of secondary caries in the area of restorative margins or corrections of visible crown margins caused by cervical recession, for example.

Intraoral repair is based on cleaning, establishing a microretentive etching surface by roughening and a subsequent chemical adhesion [3–6]. Various repair protocols that have been evaluated do not show significant differences in bond strength [5]. These include air-particle abrasion with aluminium oxide powder (50 μm Al$_2$O$_3$) and tribochemical-pretreatment with alumina-coated silica particles (30 μm SiO$_2$) (ie. CoJet) [7]. As this may impair the glaze of the ceramic [3], another conditioning procedure for porcelain restorations (and composite) is recommended by using 6–9.5% HF resulting in a partial dissolution of the glass phase [4,8].

Contamination of HF on soft tissue reportedly have serious consequences. Because of its solubility in water at every concentration and the ability to dehydrate substances, it can quickly penetrate into skin and other soft tissues and cause a burn reaction [9]. To prevent this, rubber dam and proper suction technique is required for intraoral use. In contrast, the inadvertent cross-contamination on dental tissue being next to ceramics cannot always be avoided in clinical circumstances due to limiting factors such as applicator type and defect size [10]. Thus, depending on the size of the dental tissue involved, the influence of HF on dentin and enamel may have a considerable impact on the repair fillings.

Using the scanning electron microscope, it was shown that HF does not completely remove the dentin smear layer and leaves an amorphous precipitate of fluoride [11] which prevents penetration of the adhesive. Resin-infiltration is hindered and an adequate resin-impregnated (hybrid) layer can hardly be formed [10].

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This seems to impair the repair bond strength on dentin. However, there are different results as to whether the sequence of phosphoric and HF-etching has an influence on the bond strength [6,12]. In these studies, adhesives were applied on dentin, which has been contaminated by HF-etching, with no special pretreatment of the dentin before HF-contamination.

There is no data available in literature about HF-contamination of dentin that has already been treated with an adhesive. The adhesive may protect the dentin preventing dissolution of inorganic substances and the resulting sealing of the dentin surface and disturbed hybridization of demineralized dentin with bonding agents. So, the purpose of this study was to evaluate the influence of HF-contamination on dentin that is already treated with an etch-and-rinse or a self-etch adhesive. The null hypothesis tested was that contamination of HF (9.5%) after adhesive application on dentin would not affect the μTBS.

2. Materials and Methods

2.1. Specimen preparation

For this in vitro study, 35 extracted non-carious human molars were selected. They were collected as by-products of regular dental treatments and after cleaning stored in tap water until use. Patients gave their informed consent that the teeth can be used in further studies. All patients gave their informed consent that the teeth can be used in further studies. All teeth were irreversible anonymised. Therefore research with these teeth is no more covered by the human research regulations (Article 2, Paragraph 2, Section c of Federal Act on Research involving Human Beings) and no approval from the ethic committee is required. The root tips were fixed centrally on a specimen holder for electron microscopy (Stemi 1000, Carl Zeiss, Feldbach, Switzerland) to check for remnants of enamel or exposure of pulp tissue.

The teeth were randomly allocated to seven groups (n = 5): two control groups (G1, G2) and five test groups (G3-G7).

2.2. Bonding procedure

After random allocation to the seven groups, the teeth were treated as follows:

- G1 (Optibond FL/control 1): Application of the three-step adhesive OptiBond FL (etch-and-rinse system) as per manufacturer’s instructions. Therefore the etchant (37.5 % phosphoric acid) (Lot 5062962, Syringe Kerr Gel Etchant, Kerr, Scafati, Italy) was applied 15 s on the surface, rinsed for 15 s with water until the etchant was completely removed and air dried for 3 s with an air syringe. Primer (OptiBond FL Prime, Lot 5055655, Kerr) was applied with light brushing motion for 15 s followed by air drying for 5 s. The surface was then coated with a thin layer of adhesive which was air thinned for 3 s and light cured for 20 s using the Bluephase G2 light-curing unit (Ivoclar Vivadent AG, Liechtenstein).
- G2 (OptiBond XTR/control 2): Application of the two-step self-etch adhesive OptiBond XTR as per manufacture’s instruction. For this purpose, the Primer was applied using scrubbing motion and aired thin with medium air pressure. The adhesive was then applied with light brushing movements and dried using a gentle air stream followed by strong air for at least 5 s. The surface was light cured for 10 s with the same light-curing unit as above.
- G3 (OptiBond FL/HF/OptiBond FL): After application and light-curing of the three-step etch-and-rinse adhesive OptiBond FL as described above, the samples were contaminated with buffered HF (9.5%) (Porcelain Etch, Ultradent Products Inc., Cologne, Germany) for 60 s. A second application of the three-step etch-and-rinse adhesive OptiBond FL followed.
- G4 (OptiBond FL/HF/OptiBond FL without etchant): After application and light-curing of the three-step etch-and-rinse adhesive OptiBond FL as described above, the samples were treated with buffered HF (9.5%). As in G3 a second application of OptiBond FL followed, but phosphoric etching was omitted.
- G5 (OptiBond XTR/HF/OptiBond XTR): The samples were treated with Opti-Bond XTR as described above, followed by application of 9.5% HF and a second application of Opti Bond XTR.
- G6 (HF/OptiBond FL): Application of HF (9.5%) on the dentine surface for 60 s and a single application of three-step etch-and-rinse OptiBond FL including the phosphoric acid etching as in G3.
- G7 (HF/OptiBond XTR): Application of HF (9.5%) on the dentin surface for 60 s, followed by Optibond XTR.

Following the treatment as described above, build-ups with a nanofilled composite (Filtek Supreme XTE, shade A2B, Lot N549719, 3 M ESPE; St Paul, MN, USA) of 4–5 mm height were made. The composite was applied in three increments, max. 1.5 mm each, to cover the whole dentin surface and each increment was light-cured (20 s) using the Bluephase G2 light-curing unit.

The experimental design of the study is illustrated in Table 1.

2.3. μTBS

The samples were cut longitudinally in two directions using a water-cooled diamond saw (Struers Accutom-50, Struers, Ballerup, Denmark) with a diamond disc (M1D10, Struers; size: 102 mm x 0.3 mm x 12.7 mm) in nine rectangular sticks. They were cut parallel to the surface with a slow running saw (Isomet, Buehler, Illinois, USA) in sticks with a length of 8–9 mm. The sticks had cross-sectional bonding areas between 0.713 mm² and 0.864 mm², which was measured and recorded with a digital caliper (Kisling; Zurich, Switzerland). The specimens were fixed at both ends on a sandblasted μTBS jig with superglue and loaded under tension until failure in a universal testing machine (Zwick Roell Z2010; Ulm, Germany). A load cell of 200 N (KAF-TC; AST; Dresden, Germany) and a crosshead speed of 1 mm/min was used. The tensile bond strength in MPa resulted from the quotient of the load at failure (N) and the respective bonding area (mm²) [13].

2.4. Failure analysis

Failure modes were evaluated using a dual-head stereo zoom microscope (Wild; Heerbrugg, Switzerland) at 25X magnification. Four modes of failure were determined and classified either as adhesive or cohesive (in dentin or composite or mixed).
Data were entered in Microsoft Excel (Microsoft Office Professional Plus 2010, Micro-soft; Redmond, WA, USA) and analysed using SPSS (IBM SPSS Statistics for Windows, Version 21.0; Armonk, NY, USA).

The μTBS of specimens that failed prior to testing (pre-test failures) was set at zero MPa.

The Wilcoxon test was used as a non-parametric test for dependent data. P-values < 0.05 were considered to be statistically significant. The relative frequency of the fracture analysis was given as a percentage.

3. Results

3.1. Microtensile bond strength

Figure 1 illustrates the median and interquartile range of the μTBS (MPa) in G1-G7.

The μTBS in control group G2 (Optibond XTR; self-etch adhesive system) was significantly higher than those of control group G1 (Optibond FL; etch-and-rinse system) (p = 0.0079).

In groups G3-5 where HF was applied between two applications of the adhesive, G5 (Optibond XTR) showed significantly better μTBS than G3 and G4 (Optibond FL). In group G4, separate phosphoric acid etching after HF-application was omitted, but no significant difference was observed between the μTBS of groups G3 and G4 (p > 0.05).

In groups G6 and G7, HF was applied prior to a single application of the corresponding adhesive and μTBS of G7 (Optibond XTR) was significantly higher than that of G6 (Optibond FL).

However, within the Optibond FL test series (groups G1, G3, G4 and G6) and the Optibond XTR test series (G2, G5 and G7) no significant difference in μTBS was observed (p > 0.05).

3.2. Failure mode analysis

Failure mode distributions and number of pre-test failures are shown in Table 2. In all groups except G5 and G7, the most frequent failure mode was adhesive. In groups G5 and G7, a similarly high proportion of fractures in the mixed zone (adhesive and composite) could be observed in addition to those in the adhesive. The highest proportion of dentin fractures (27.3%) was also found in G7 compared to the other groups.

4. Discussion

The present study imitated repair fillings, where exposed dentin was next to ceramic restorations. The aim of the study was to analyse the μTBS when adhesive is applied to dentin prior to a HF-contamination. This would allow for the adjustment of dental repair protocols if necessary.

The adhesive was applied prior to HF-contamination to investigate whether the adhesive seals the dentin and thus protects it against HF attack. HF seems to dissolve inorganic substances of dentin because of its acidic pH value. Free calcium ions (Ca²⁺) react with fluoride (F⁻) and precipitate as CaF₂ crystals under acidic conditions [14,15]. Energy dispersive X-ray (EDX) analysis showed that treatments of dentin using HF increased the fluoride content within the treated dentin surface. Contact of dentin with fluorides seems to affect the dissolution properties and hamper the etching effect of H₃PO₄. Examination with an electron microscope also showed that a HF-treatment after H₃PO₄-etching also produces a precipitation of CaF₂ and other salts resulting in a sealing of the dentin surface and closing of the dentinal tubules openings [10,11]. In the present study it was assumed that application of an adhesive on dentin prior to a HF-attack prevents dissolution of inorganic substances and the resulting tubule obliteration and subsequently allows adequate hybridization between dentin and bonding resin.

Optibond FL is considered the gold standard of bonding agents as it has demonstrated the best bonding properties on dentin when different etch-and-rinse adhesives were evaluated and thus, it was selected for use in the present study [16]. When selecting a self-etch adhesive, a product from the same manufacturer (Optibond XTR) was chosen. Etching of ceramic surfaces with 6–9.5% HF is recommended. In the present study, 9.5% buffered HF was used. However, Saracoglu et al [6]. Showed that different HF acid gel concentrations do not result in significant differences in bond strength on dentin. To evaluate the adhesion of restorations to dentin in the present study, μTBS tests were chosen as used in previous studies [17,18]. As a limitation of the present study, it should be mentioned that no thermocycling was performed. The test set-up was used for the initial detection of possible effects of HF on dentin. To investigate whether HF-contamination causes any long-term adhesion impairments, thermocycling should be considered for future studies.

This investigation did not observe significant differences in bond strengths when HF was applied on dentin before or after application of the respective adhesive. Thus, no adjustments need to be made to dental repair protocols. The present study was also not able to confirm that contamination with HF impairs the bond strength to dentin at all, as within the groups of Optibond FL and Optibond XTR no significant differences in μTBS could be found.

Significant differences in bond strength were only observed between the two different adhesive systems tested. The self-etch adhesive, Optibond FL, showed significantly higher bond strengths than the tested etch-and-rinse adhesive Optibond XTR, both with and without prior contamination. Due to the demand for less technique-sensitive adhesives with fewer application steps and associated increased time efficiency, as well as the reduction of postoperative sensitivities, self-etch adhesive systems have become increasingly important over the past twenty years [19]. Optibond XTR is a two-step self-etch adhesive, which consists of a mild primer containing acidic monomers (pH ≈ 2) and an adhesive. Mild primers partially dissolve the smear layer. A proportion of hydroxyapatite remains on the collagen fibrils and is then available for chemical interaction with the carboxyl and phosphorus groups of the functional monomers of the adhesive. The amide groups of the monomers and the carboxyl groups of the collagen can form a further chemical bond to the dentin. Through these features, a mild self-etch adhesive may achieve very good bond strength values in dentin [20,21]. The results of this study confirm the recommendations found in literature that self-etch adhesive systems should be used on dentin due to the better bond strength values.

Currently only limited data is available regarding bond strength to dentin which has been contaminated with HF. Two recent studies found that contamination of dentin with HF is detrimental for adhesion to dentin [6,12]. However, due to different experimental setups and test procedures used, studies are difficult to compare. Due to methodological differences at different departments, only statistical ranking within one study and no absolute MPa values may be compared. However, neither of the aforementioned studies dealt with self-etch adhesives and applied an adhesive on the dentin before HF-contamination. In the present study, no significant decrease in μTBS was observed when dentin was subjected to HF contamination prior to adhesive application. This contrary finding might be attributed to the fact that the adhesives used in this experiment
were “actively” applied (rubbing the adhesives with a microbrush during application). This might lead to a mechanical manipulation of the fluoride precipitates formed during the contamination of the dentin with the HF and thus leads to partial dislocation of these precipitates, leaving collagen uncovered. Furthermore, one could postulate that the acidic pH of the self-etch adhesive primer additionally helps to disrupt the fluoride precipitates. To explore this hypothesis, SEM-pictures were performed for selected settings: Dentin surface after HF contamination (Figure 2); dentin surface after HF contamination and application of the primer without scrubbing motion (Figure 3); dentin surface after HF contamination and application of the primer with scrubbing motion (Figure 4).

The pictures can be found in the attachments. For the samples with dentin surface after HF contamination and application of the primer with scrubbing motion an exposure of collagen matrix could be observed. We assume that this may be attributed to the aforementioned manipulation of the fluoride precipitates formed during the HF contamination.

Further studies are needed to verify this assumption for different self-etch adhesives.

5. Conclusion

No significant differences in microtensile bond strength of the adhesive systems tested were documented depending on whether hydrofluoric acid was applied before or after application of the respective adhesive. Subsequently no adjustment of established repair protocols is required.

Appendices A. Tables

Table 1
Sample allocation and experimental procedure

| Group 1 (n=5) | Group 2 (n=5) | Group 3 (n=5) | Group 4 (n=5) | Group 5 (n=5) | Group 6 (n=5) | Group 7 (n=5) |
|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
| Optibond FL (with phosphoric acid etching) | Optibond XTR | Optibond FL (with phosphoric acid etching) | Optibond FL (with phosphoric acid etching) | Optibond XTR | Optibond FL (with phosphoric acid etching) | Optibond XTR |
| Application following the manufacturer’s instructions |
| Application (60 s) of buffered 9.5 % HF (Ultradent® Porcelain Etch) |
| Application following the manufacturer’s instructions |
| Optibond FL (with phosphoric acid etching) | Optibond FL (without phosphoric acid etching) | Optibond FL (with phosphoric acid etching) | Optibond XTR |
| Composite build-up (Filtek supreme XTE) |
| Water storage (1 week) |
| Microtensile bond strength test, failure analysis |

Table 2
Percentage distribution of fractures in the different groups and number of pre-test failures

| Group | Adhesive application steps | Number of pre-test failures | Adhesive | Mixed | Dentin | Composite |
|-------|----------------------------|----------------------------|----------|-------|--------|-----------|
| G1    | Optibond FL                | 8                          | 64.9 %   | 29.7 %| 2.7 %  | 2.7 %     |
| G2    | Optibond XTR               | 0                          | 44.4 %   | 17.8 %| 15.6 % | 22.2 %    |
| G3    | Optibond FL + HF + Optibond FL | 1                      | 65.9 %   | 29.6 %| 4.6 %  | 0.0 %     |
| G4    | Optibond FL + HF + Optibond FL without phosphoric acid etching | 3                          | 79.6 %   | 20.5 %| 0.0 %  | 0.0 %     |
| G5    |                            | 0                          | 34.0 %   | 38.3 %| 12.8 % | 14.9 %    |

(continued on next page)
Table 2 (continued)

| Group | Adhesive application steps | Number of pre-test failures | Adhesive | Mixed | Dentin | Composite |
|-------|----------------------------|----------------------------|----------|-------|--------|-----------|
| Optibond XTR + HF + Optibond XTR |
| G6    | HF + Optibond FL           | 3                          | 52.4 %   | 31.0 %| 14.3 % | 2.4 %     |
| G7    | HF + Optibond XTR          | 2                          | 29.6 %   | 31.8 %| 27.3 % | 13.6 %    |

B. Figures

Figure 1. μTBS in MPa (median and interquartile range) for G1-G7.
G1: Optibond FL
G2: Optibond XTR
G3: Optibond FL + HF + Optibond FL
G4: Optibond FL + HF + Optibond FL without phosphoric acid etching
G5: Optibond XTR + HF + Optibond XTR
G6: HF + Optibond FL
G7: HF + Optibond XTR
Values which did not differ at the 5 % level from each other, are designated with the same capital letters.

Figure 2. SEM-picture: Dentin surface treated with 9.5% HF for 60 s.
Samples were sputter-coated with a gold layer of 5 nm. Images were made at 10 kV and 200 pA with a WD of 10.4 mm and a magnification of 4000.
Figure 3. SEM-picture: Dentin surface after HF contamination for 60 s and application of the primer without scrubbing motion.

Figure 4. SEM-picture: Dentin surface after HF contamination for 60 s and application of the primer with scrubbing motion. A partial exposure of collagen matrix can be observed.

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