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Repair Surface Conditioning Measures Affect Enamel and Dentin Bond Strength

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Clinical Relevance

Contamination of enamel and dentin with repair surface conditioning measures should be avoided.

SUMMARY

Objectives: To analyze whether the contamination with different repair conditioning measures impairs the adhesive performance of a universal adhesive applied in etch-and-rinse mode (ER) or self-etch mode (SE).

Methods and Materials: Bovine enamel and dentin surfaces (each subgroup n=16) were bonded with a universal adhesive in ER or SE after contamination with different repair conditioning measures (sandblasting, silica coating, hydrofluoric acid etching, self-etching ceramic primer). In half of the groups, sandblasting, silica coating, and hydrofluoric acid etching was followed by the use of a universal primer. If the universal adhesive was applied in ER, contamination was performed either before or after phosphoric acid etching. If the universal adhesive was applied in SE, bonding was performed after contamination. In the control groups, no contamination was simulated. Shear bond strength (SBS) and failure modes of composite buildups were determined after thermal cycling (10,000 cycles, 5°C-55°C). Statistical analysis was performed using analyses of variance, Weibull statistics, and χ^2 tests ($p<0.05$).

Results: In ER, sandblasting and silica coating significantly reduced SBS (control: enamel = 25.7±4.2 MPa; dentin = 22.0±5.3 MPa) only when performed after phosphoric acid etching. Contamination with hydrofluoric acid impaired SBS on enamel but not on dentin. The self-etching ceramic primer reduced SBS, but not significantly. The contamination with the universal primer had no significant effect. In SE, all repair conditioning measures except the universal primer reduced SBS (control: enamel = 20.3±5.5 MPa; dentin = 23.0±4.0 MPa).

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Conclusion: Contamination of enamel and dentin by repair conditioning measures potentially affects bond strength.

INTRODUCTION

Repair of partially defective dental restorations is becoming increasingly accepted by dentists and patients as it comes along with substantial advantages compared with complete replacement, for example, preservation of tooth structure, fewer pulpal complications, and reduced treatment time and costs.[1]

Optimal adhesion between the repair material (in most cases composite) and the original restoration makes complex conditioning of the repair surface necessary. Depending on the kind of restorative material, pretreatment of the repair surface to adhere to composite includes mechanical roughening, silica coating, sandblasting (composite, metal, zirconia), or hydrofluoric acid etching (glass ceramic), followed by the use of primers and adhesives containing silane/10-methacryloyloxydecyl dihydrogen phosphate (10-MDP).[2,3] However, in many clinical situations, the defective part of the restoration is surrounded by dental hard tissue, which makes the additional conditioning of enamel and/or dentin necessary. Especially in small cavities, contamination of enamel or dentin by repair conditioning measures is often unavoidable and might affect adhesion of composite to these dental hard tissues.

Several studies have shown that hydrofluoric acid application on dentin creates an amorphous layer of fluoride, rendering it difficult for adhesives to penetrate.[4-7] Moreover, as hydrofluoric acid contamination also has the potential to cause chemical burn of surrounding soft tissues, alternative conditioning methods for intraoral repair of glass ceramic restorations are necessary. Recently, a self-etching glass ceramic primer containing ammonium polyfluoride and silane methacrylate was introduced to the market to replace conditioning with hydrofluoric acid and silanization. Although this product is not yet approved for intraoral use, it may be of interest if possible hazardous effects on hard and soft tissues can be avoided. So far, bonding performance of self-etching ceramic primer on lithium disilicate and feldspathic ceramics was shown to be limited compared with hydrofluoric acid etching.[8,9] Nevertheless, bonding performance might still be acceptable for repaired restorations, especially if possible side effects are reduced. However, no information on contamination effects of the self-etching glass ceramic primer on dental hard tissues is so far available.

Besides hydrofluoric acid etching, potential side effects of silica coating and aluminum oxide sandblasting were investigated but showed conflicting results, depending on the substrate (enamel or dentin) and kind of abrasion particle.[10,11]

However, potential detrimental effects of different repair conditioning measures and sequences have not been systematically analyzed, especially with regard to the bond strength of etch-and-rinse or self-etch adhesives. Therefore, in this study we aimed to analyze the shear bond strength of a universal adhesive applied in etch-and-rinse mode (ER) or self-etch mode (SE) on enamel and dentin after contamination with different repair surface conditioning measures and sequences. The null hypothesis tested was that the bond strength of the universal adhesive is not affected by the various repair conditioning measures.

METHODS AND MATERIALS

Specimen Preparation

Cylindrical enamel and dentin specimens (5.7 mm in diameter) were prepared from the crowns or roots of freshly extracted, nondamaged bovine incisors. The specimens were then embedded in chemically cured acrylic resin (Paladur, Kulzer, Hanau, Germany) and ground flat using sandpaper (WS flex 18C, grits 500 und 800, Hermes, Hamburg, Germany) while being water-cooled (RotoPol-35 and PdM-Force-20, Struers, Willich, Germany). Specimens were randomly divided into 25 groups of enamel or dentin specimens (n=16 for shear bond strength analysis, n=2 for scanning electron microscopy [SEM] analysis).

Simulated Contamination and Bonding Procedures

The composition of the universal adhesive and the materials used in this study is presented in Table 1; surface contamination measures/sequences are shown in Tables 2 through 5.

The universal adhesive (Adhese Universal, Ivoclar Vivadent, Schaan, Liechtenstein) was either applied in ER (groups 1-16) or SE (groups 17-25) on enamel and dentin specimens.

If used in ER, 37% phosphoric acid was applied for 30 seconds (enamel) or 15 seconds (dentin) to the surface, rinsed with water for 30 seconds or 15 seconds (respectively) and gently air-dried. In the control group (group 1), the adhesive was applied without any simulated contamination and light-cured (20 seconds, B.A. Optima 10 LED, B.A. International, Hamburg, Germany). Contamination by sandblast-

Table 1: *Composition of Materials Used as Described in Manufacturers' Safety Data Sheets*

Product	Manufacturer	Lot No	Composition	Application Mode
Adhese Universal	Ivoclar Vivadent, Schaan, Liechtenstein	W91987, X38352	HEMA (10%-25%), Bis-GMA (10%-25%), ethanol (10%-25%), 1,10-decandiol dimethacrylate (≥2.5%-<10%), methacrylated phosphoric acid ester (≥2.5%-<10%), campherquinone (1%-<2.5%), DMAEMA (≥1%-<2.5%)	20 s (scrubbing into the tooth surface using a disposable microbrush), 20 s light-curing
Filtek Supreme XTE	3M ESPE, Seefeld, Germany	N779140	Silane treated ceramic (60%-80%), silane treated silica (1%-10%), UDMA (1%-10%), bisphenol A polyethylene glycol diether dimethacrylate (1%-10%), Bis-GMA (1%-10%), silane treated zirconia (1%-5%), polyethylene glycol dimethacrylate (<5%), triethylene glycol dimethacrylate (<1%)	2-mm-thick increments, each 20 s light-cured
CoJet sand (silica-coated aluminum oxide)	3M ESPE, Seefeld, Germany	630975, 4236465	Aluminum oxide (>95%), synthetic amorphous silica (<5%)	4 s, 10-mm distance, 45°, 2-3 bar air pressure
Super Etch (phosphoric acid gel)	SDI, Bayswater, Australia	171138	Phosphoric acid (37%)	30 s (enamel) or 15 s (dentin), 30 s or 15 s (respectively) water-rinsing, gentle air-drying
Airsonic Alu-Oxyd (aluminum oxide)	Hager & Werken, Duisburg, Germany	N/A	N/A	4 s, 10-mm distance, 45°, 2-3 bar air pressure
Porcelain Etch (9% buffered hydrofluoric acid gel)	Ultradent Products, Cologne, Germany	BFTKV	Hydrofluoric acid (<10%)	90 s, water-rinsing (15 s)
Monobond Etch&Prime	Ivoclar Vivadent, Schaan, Liechtenstein	X30890	Butanol (20%-<25%), tetrybutylammonium dihydrogen trifluoride (≤10%), methacrylated phosphoric acid ester (2.5%-<10%), bis(triethoxysilyl)ethane (1%-<2.5%)	20 s, after 40 s water-rinsing (15 s), gentle air-drying
Monobond Plus	Ivoclar Vivadent, Schaan, Liechtenstein	X11257	Ethanol (50%-100%), methacrylated phosphoric acid ester (1%-<2.5%)	20 s gentle air-blowing after 60 s

Abbreviations: Bis-GMA indicates bisphenol A diglycidyl ether dimethacrylate; DMAEMA, 2-dimethylaminoethyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; N/A, not available; UDAM, diurethane dimethacrylate.

ing, silica coating, hydrofluoric acid etching, or the self-etching ceramic primer was performed either before (groups 10-16) or after (groups 3-9) phosphoric acid etching. In half of the groups, surfaces were also exposed to a universal primer.

If used in SE, the adhesive was applied for 20 seconds (enamel and dentin) before light-curing for 20 seconds (B.A. Optima 10 LED): In the respective control group (group 17), no contamination of the surfaces was performed. In the test groups, sandblasting, silica coating, hydrofluoric acid etching, or the application of the self-etching primer was performed before the adhesive was applied. In half of the specimens in each group, surfaces were also contaminated with a universal primer.

The following surface contamination measures were applied either alone or in combination:

1) Air-abrasion with aluminum oxide (50 μm; Hager & Werken, Duisburg, Germany) for 4 seconds at a distance of 10 mm (45°) and 2-3 bar air pressure. Remnants were air blown.
2) Silica coating: Surfaces were silica coated (30 μm; CoJet, 3M ESPE, Seefeld, Germany) for 4 seconds at a distance of 10 mm (45°) and 2-3 bar air pressure. Loose particles were air blown.
3) Hydrofluoric acid (9% buffered hydrofluoric acid gel; Porcelain Etch, Ultradent Products, Cologne, Germany) was applied for 90 seconds. Samples were subsequently rinsed with water for 60 seconds.
4) The self-etching ceramic primer (Monobond Etch&Prime, Ivoclar Vivadent) was applied with a microbrush for 20 seconds followed by a 40-second contact time. Specimens were subsequently rinsed with water for 15 seconds.
5) Universal primer (Monobond Plus, Ivoclar Vivadent) was applied using a microbrush and allowed to evaporate for 60 seconds as recommended by the manufacturer. Afterward, samples were gently air blown.

After the universal adhesive was applied, the repair composite (Filtek Supreme XTE, 3M ESPE; shade A2) was adhered onto the specimen surface using acrylic hollow cylinders (inner diameter: 3 mm, height: 4 mm). The composite was packed against the surface in a 2-mm-thick increment and light-cured for 20 seconds by applying the curing unit directly onto the acrylic cylinder. Light irradiance (>800 mW/cm^2) was verified every 30 specimens using a radiometer (Cure Rite Model 644726, Dentsply Caulk, Milford, DE, USA). Contamination and bonding procedures were carried out by one operator (LP) throughout all experiments.

All specimens were then submitted to a thermal cycling procedure (10,000 cycles, between 5°C and 55°C, dwell time: 20 seconds, transfer time: 10 seconds) before shear bond testing.

Shear Bond Measurements and Failure Analysis

Shear bond strength was tested with a universal testing machine (Materialprüfmaschine 1446, Zwick, Ulm, Germany). A shear force was applied to the adhesive interface through a chisel-shaped loading device at a crosshead speed of 1 mm/min parallel to the surface of the specimens. Load at fracture was recorded, and shear bond strength (σ) was calculated by software (TestXpert 11.02, Zwick) using the load at failure F (N) and the adhesive area A (mm^2): $\sigma = F/A$.

The debonded area was examined for failure mode analysis with a stereomicroscope at 16× magnification (Stemi SV 11, Zeiss, Oberkochen, Germany). Failure mode was considered as adhesive if it occurred at the interface; as cohesive if at least parts of either enamel, dentin, or composite were affected; or as mixed when both adhesive and cohesive failures occurred.

SEM Analysis

Two specimens of each group were desiccated for 2 weeks in silica gel, sputter-coated with carbon, and analyzed by SEM (Ultra plus FE-SEM, Zeiss) at 10 kV.

Statistical Analysis

Statistical analyses were performed using the software SPSS Statistics for Macintosh (version 25.0.0.1, IBM, Armonk, NY, USA).

Separately for enamel and dentin, three-way analysis of variance (ANOVA) (ER without groups 9 and 16) and two-way ANOVA (SE without group 25) were performed. Factors were the sequence in which phosphoric acid etching was performed (first or second step, only in ER), the kind of contamination (sandblasting, silica coating, hydrofluoric acid etching, control), and the use of a universal primer. Moreover, one-way ANOVAs followed by Scheffe post hoc tests were applied to compare all groups, including those where the self-etching ceramic primer was used.

Additionally, characteristic bond strength σ_0 and Weibull modulus m (Weibull distribution parameters) were assessed using the maximum likelihood estimates and 95% confidence intervals in MATLAB (version R2018b, 9.5.0.1049112, The MathWorks, Natick, MA, USA).

For both substrates and each adhesive mode, effects of different repair surface treatments on failure modes were analyzed using χ^2 tests adjusted according to Bonferroni. The overall level of significance was set at $\alpha = 0.05$.

RESULTS

When the universal adhesive was applied in ER, shear bond strength values in enamel were significantly affected by the sequence of phosphoric acid etching ($p < 0.001$, after contamination > before contamination) and the kind of contamination ($p < 0.001$, control > sandblasting = silica coating > hydrofluoric acid) but not by the contamination with the universal primer ($p = 0.056$). The interaction between factors was not significant ($p > 0.169$), except for the interaction between "sequence of phosphoric acid etching" and "kind of contamination" ($p < 0.001$). Between-group comparisons showed that groups 3-8, 14, and 15 led to significantly lower bond strength than the control. Shear bond strength, Weibull parameters, and failure modes of enamel specimens conditioned with the universal adhesive in ER are presented in Table 2.

If applied in SE, shear bond strength values on enamel were significantly affected by the kind of contamination ($p < 0.001$, control > sandblasting = silica coating > hydrofluoric acid) and by the use of a universal primer ($p = 0.028$). The interaction between factors was not significant ($p = 0.389$). Between-group comparisons showed that all repair contamination measures (except application of the universal primer, group 2) led to significantly reduced bond strength values. Shear bond strength, Weibull parameters, and failure modes of enamel specimens conditioned with the universal adhesive in SE are presented in Table 3.

On dentin, the shear bond strength of the universal adhesive applied in ER was significantly affected by the sequence of phosphoric acid etching ($p < 0.001$, after contamination > before contamina-

Table 2: *Shear Bond Strength, Weibull Parameters, and Failure Modes of Enamel Specimens in Etch-and-rinse Mode*[a]

Group	Surface Contamination/Conditioning				Bond Strength (MPa)	Weibull Parameters [95% CI]		Failure Mode (%)		
	1. Step	**2. Step**	**3. Step**	**4. Step**	**Mean±SD**	**σ_0 (MPa)**	***m***	**Adhesive**	**Mixed**	**Cohesive**
1	Phosphoric acid	None	– (Control)	Universal adhesive	25.7±4.2 EF	27.4 [25.5, 29.5]	7.2 [4.9, 10.7]	6.7	93.3	0.0
2			Universal primer		27.3±3.6 F	28.6 [27.4, 29.9]	11.6 [7.8, 17.3]	37.5	50.0	12.5
3		Sandblasting	–		15.8±4.3 ABC	17.4 [15.3, 19.8]	4.0 [2.8, 5.8]	75.0	25.0	0.0
4			Universal primer		16.0±1.7 ABC	16.8 [15.9, 17.6]	10.5 [7.2, 15.2]	56.3	43.8	0.0
5		Silica coating	–		11.3±3.6 A	12.5 [10.8, 14.6]	3.4 [2.4; 4.9]	50.0	50.0	0.0
6			Universal primer		12.6±2.3 A	13.6 [12.5, 14.7]	6.4 [4.3, 9.4]	31.3	68.8	0.0
7		Hydrofluoric acid	–		13.9±5.6 AB	15.6 [13.0, 18.7]	2.8 [1.9, 4.2]	25.0	68.8	6.3
8			Universal primer		14.3±4.4 AB	15.9 [14.0, 18.1]	4.0 [2.6, 6.0]	25.0	75.0	0.0
9		Self-etching ceramic primer			21.6±4.1 BCDEF	23.1 [21.5, 24.9]	6.8 [4.6, 10.2]	50.0	50.0	0.0
10	Sandblasting	Phosphoric acid	–		23.8±4.4 CDEF	25.4 [23.8, 27.0]	8.2 [5.4, 12.5]	43.8	43.8	12.5
11			Universal primer		23.9±5.7 CDEF	25.6 [23.5, 27.9]	5.9 [3.9, 8.9]	50.0	50.0	0.0
12	Silica coating		–		24.8±3.9 EF	26.4 [24.7, 28.2]	7.8 [5.3, 11.4]	0.0	92.9	7.1
13			Universal primer		24.5±3.7 DEF	26.2 [24.2, 28.2]	6.8 [4.8, 9.7]	12.5	87.5	0.0
14	Hydrofluoric acid		–		12.0±5.5 A	13.6 [11.0, 16.7]	2.5 [1.6, 3.7]	37.5	62.5	0.0
15			Universal primer		16.5±6.1 ABCD	18.5 [15.7, 21.6]	3.2 [2.1, 4.9]	25.0	68.8	6.3
16	Self-etching ceramic primer		–		18.9±6.4 ABCDE	20.9 [18.2, 23.9]	3.7 [2.4, 5.8]	46.7	46.7	6.7

[a] *Shear bond strength (mean±standard deviation [SD], characteristic strength* σ_0 *and Weibull modulus* m *with their 95% confidence intervals [CIs]) and failure modes of enamel specimens with the universal adhesive applied in etch-and-rinse mode. Different letters indicate significant differences between the groups as assessed by one-way analysis of variance followed by Scheffe post hoc tests.*

tion) and the kind of contamination ($p<0.001$, control = hydrofluoric acid > sandblasting > silica coating) but not by the contamination with the universal primer ($p=0.923$). The interactions between factors were significant ($p<0.043$), except for the interaction between "sequence of phosphoric acid etching" and "kind of contamination" ($p=0.268$). Between-group comparisons revealed that bond strength was significantly reduced in groups 3-6 and 13 compared with the respective control. Shear bond strength, Weibull parameters, and failure modes of enamel specimens conditioned with the universal adhesive in ER are presented in Table 4.

If applied in SE, shear bond strength was affected by the kind of contamination ($p<0.001$, control > hydrofluoric acid > sandblasting > silica coating) and the use of the universal primer ($p=0.001$); the interaction between factors was not significant ($p=0.210$). Between-group comparisons showed that all contamination measures, except for the universal primer (group 18) and hydrofluoric acid/universal primer (group 24), led to significantly reduced bond strength. Shear bond strength, Weibull parameters, and failure modes of enamel specimens conditioned with the universal adhesive in SE are presented in Table 5.

Table 3: *Shear Bond Strength, Weibull Parameters, and Failure Modes of Enamel Specimens in Self-etch Mode* [a]

Group	Surface Contamination/Conditioning			Bond Strength (MPa)	Weibull Parameters [95% CI]		Failure Mode (%)		
	1. Step	**2. Step**	**3. Step**	**Mean±SD**	**σ_0 (MPa)**	***m***	**Adhesive**	**Mixed**	**Cohesive**
17	None	– (Control)	Universal adhesive	20.3±5.5 C	22.3 [19.9, 24.9]	4.5 [3.1, 6.7]	31.3	43.8	25.0
18		Universal primer		23.7±4.3 C	25.3 [23.6, 27.2]	7.3 [4.9, 10.9]	37.5	43.8	18.8
19	Sandblasting	–		11.4±5.3 B	12.8 [10.3, 15.9]	2.4 [1.6, 3.5]	73.3	26.7	0.0
20		Universal primer		13.6±3.9 B	14.9 [13.1, 16.9]	4.1 [2.8, 6.0]	60.0	40.0	0.0
21	Silica coating	–		12.8±3.8 B	14.1 [12.6, 15.8]	4.5 [2.9, 7.0]	64.3	35.7	0.0
22		Universal primer		13.8±3.4 B	15.1 [13.6, 16.7]	4.9 [3.3, 7.3]	80.0	20.0	0.0
23	Hydrofluoric acid	–		2.3±2.9 A	2.1 [1.2, 3.8]	0.9 [0.6, 1.3]	68.8	31.3	0.0
24		Universal primer		2.3±3.3 A	1.6 [0.6, 3.9]	0.5 [0.4, 0.8]	87.5	12.5	0.0
25	Self-etching ceramic primer			2.3±2.2 A	1.6 [0.5, 5.2]	0.4 [0.3, 0.7]	100.0	0.0	0.0

[a] *Shear bond strength (mean±standard deviation [SD], characteristic strength* σ_0 *and Weibull modulus* m *with their 95% confidence intervals [CIs]) and failure modes of enamel specimens with the universal adhesive applied in self-etch mode. Different letters indicate significant differences between groups as assessed by one-way analysis of variance followed by Scheffe post hoc tests.*

Table 4: *Shear Bond Strength, Weibull Parameters, and Failure Modes of Dentin Specimens in Etch-and-rinse Mode* [a]

Group	Surface Contamination/Conditioning				Bond Strength (MPa)	Weibull Parameters [95% CI]		Failure Mode (%)		
	1. Step	2. Step	3. Step	4. Step	Mean±SD	σ_0 (MPa)	*m*	Adhesive	Mixed	Cohesive
1	Phosphoric acid	None	– (Control)	Universal adhesive	22.0±5.3 E	24.0 [21.7, 26.6]	5.0 [3.4, 7.5]	53.3	46.7	0.0
2			Universal primer		21.2±3.8 E	22.7 [21.1, 24.4]	7.1 [4.8, 10.5]	25.0	75.0	0.0
3		Sandblasting	–		12.3±4.6 ABC	13.7 [11.6, 16.2]	3.0 [2.0, 4.5]	62.5	37.5	0.0
4			Universal primer		13.7±3.2 ABCD	14.9 [13.6, 16.3]	5.5 [3.7, 8.4]	68.8	31.3	0.0
5		Silica coating	–		9.6±3.0 AB	10.6 [9.3, 12.1]	3.9 [2.6, 5.8]	68.8	31.3	0.0
6			Universal primer		8.7±3.8 A	9.5 [7.4, 12.1]	2.0 [1.3, 3.1]	56.3	43.8	0.0
7		Hydrofluoric acid	–		15.6±5.3 ABCDE	17.0 [14.6, 19.8]	3.3 [2.1, 5.0]	43.8	56.3	0.0
8			Universal primer		22.4±1.8 E	23.1 [22.4, 23.9]	15.9 [10.8, 23.6]	31.3	68.8	0.0
9		Self-etching ceramic primer			20.5±2.3 DE	21.5 [20.5, 22.5]	11.0 [7.5, 16.0]	43.8	56.3	0.0
10	Sandblasting	Phosphoric acid	–		16.2±4.4 BCDE	17.7 [15.9, 19.8]	4.7 [3.1, 7.1]	62.5	37.5	0.0
11			Universal primer		17.6±3.1 CDE	18.7 [17.6, 19.8]	8.7 [5.8, 13.3]	64.3	35.7	0.0
12	Silica coating		–		17.9±5.3 CDE	19.7 [17.4, 22.4]	4.0 [2.7, 5.9]	28.6	71.4	0.0
13			Universal primer		10.0±4.2 AB	11.2 [9.2, 13.6]	2.7 [1.8, 4.0]	50.0	50.0	0.0
14	Hydrofluoric acid		–		20.7±3.2 DE	22.0 [20.6, 23.5]	7.7 [5.3, 11.3]	33.3	66.7	0.0
15			Universal primer		22.0±5.3 E	23.9 [21.7, 26.4]	5.2 [3.5, 7.7]	18.8	81.3	0.0
16	Self-etching ceramic primer		–		17.6±4.5 CDE	19.3 [17.2, 21.6]	4.5 [3.1, 6.6]	87.5	12.5	0.0

[a] *Shear bond strength (mean± standard deviation [SD], characteristic strength σ_0 and Weibull modulus m with their 95% confidence intervals [CIs]) and failure modes of dentin specimens with the universal adhesive applied in etch-and-rinse mode. Different letters indicate significant differences between groups as assessed by one-way analysis of variance followed by Scheffe post hoc tests.*

Highest Weibull parameters (characteristic strength σ_0 and Weibull modulus m) were reached in the control groups or when solely the universal primer was applied, irrespective of the adhesive strategy and the substrate. The only exception was found in the ER groups on dentin, where group 8 (phosphoric acid etching followed by hydrofluoric acid and the universal primer) obtained the highest Weibull modulus. For both enamel and dentin in ER, the majority of the groups reached higher charac-

Table 5: *Shear Bond Strength, Weibull Parameters, and Failure Modes of Dentin Specimens in Self-etch Mode* [a]

Group	Surface Contamination/Conditioning			Bond Strength (MPa)	Weibull Parameters [95% CI]		Failure Mode (%)		
	1. Step	2. Step	3. Step	Mean±SD	σ_0 (MPa)	*m*	Adhesive	Mixed	Cohesive
17	None	– (Control)	Universal adhesive	23.0±4.0 DE	24.6 [23.0, 26.3]	7.6 [5.1, 11.5]	14.3	64.3	21.4
18		Universal primer		24.9±4.6 E	26.7 [24.5, 29.2]	5.9 [4.1, 8.3]	0.0	28.6	71.4
19	Sandblasting	–		10.3±4.6 AB	10.1 [5.8, 17.7]	0.9 [0.5, 1.4]	86.7	13.3	0.0
20		Universal primer		12.3±2.3 ABC	13.2 [12.1, 14.5]	5.7 [4.0, 8.2]	53.3	46.7	0.0
21	Silica coating	–		7.3±4.0 A	8.2 [6.3, 10.7]	2.0 [1.3, 2.9]	85.7	14.3	0.0
22		Universal primer		8.1±4.9 AB	9.0 [6.6, 12.3]	1.6 [1.1, 2.5]	92.3	7.7	0.0
23	Hydrofluoric acid	–		13.1±5.1 BC	14.5 [12.2, 17.2]	2.9 [1.9, 4.5]	53.3	46.7	0.0
24		Universal primer		18.3±4.1 CD	19.9 [17.9, 22.1]	5.0 [3.4, 7.2]	46.2	53.8	0.0
25	Self-etching ceramic primer			9.6±4.1 AB	10.8 [8.9, 13.2]	2.6 [1.8, 3.8]	100.0	0.0	0.0

[a] *Shear bond strength (mean± standard deviation [SD] characteristic strength σ_0 and Weibull modulus m with their 95% confidence intervals [CIs]) and failure modes of dentin specimens with the universal adhesive applied in self-etch mode. Different letters indicate significant differences between groups as assessed by one-way analysis of variance followed by Scheffe post hoc tests.*

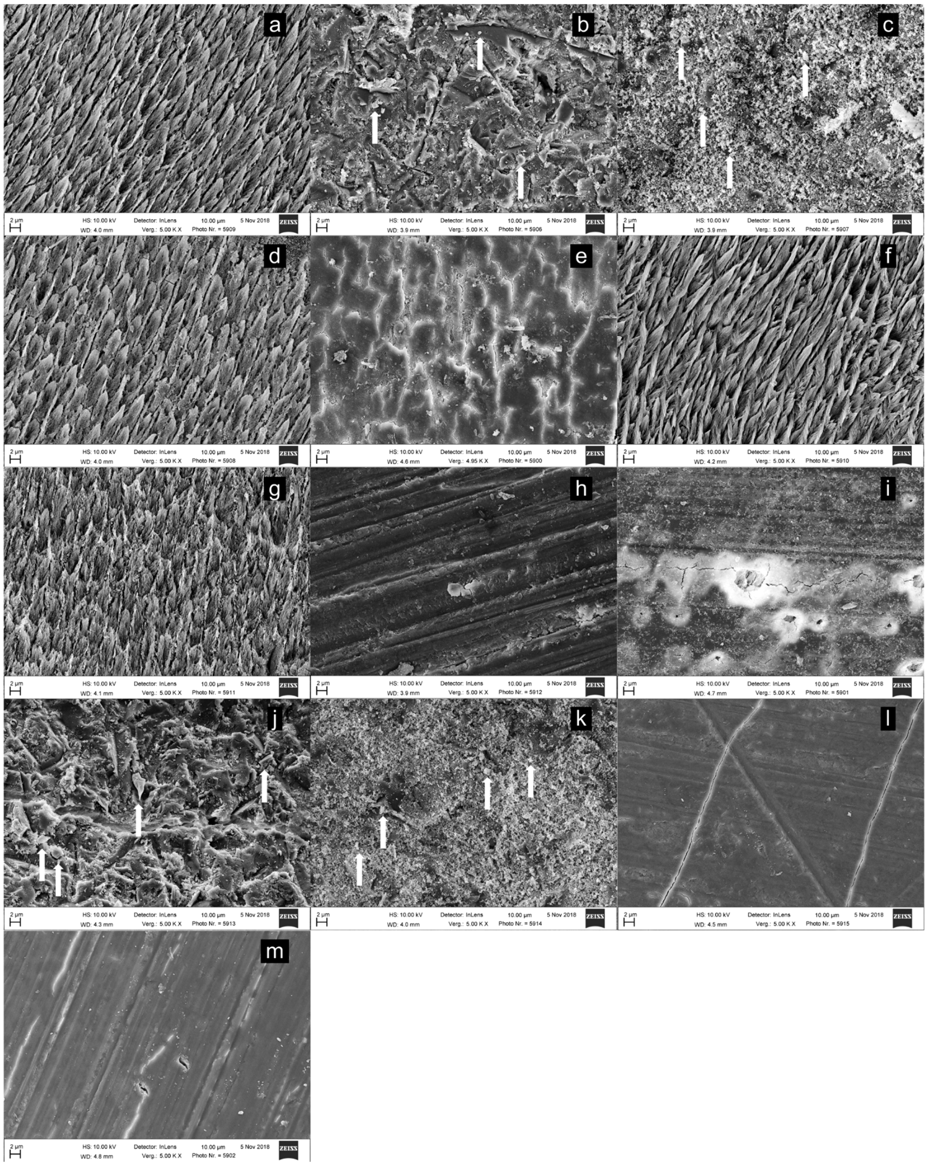

Figure 1. *Representative scanning electron micrographs of enamel surfaces treated with sandblasting, silica coating, hydrofluoric acid etching, or the self-etching ceramic primer either alone (contamination before application of the adhesive in self-etch mode) or before/after phosphoric acid etching (contamination before application in etch-and-rinse mode). Note that contamination with the universal primer is not shown as it was not visible. Original magnification ×5000. (a): Phosphoric acid (control etch-and-rinse, group 1). (b): Phosphoric acid → sandblasting (group 3). (c): Phosphoric acid → silica coating (group 5). (d): Phosphoric acid → hydrofluoric acid (group 7). (e): Phosphoric acid → self-etching ceramic primer (group 9). (f): Sandblasting → phosphoric acid (group 10). (g): Silica coating → phosphoric acid (group 12). (h): Hydrofluoric acid → phosphoric acid (group 14). (i): Self-etching ceramic primer → phosphoric acid (group 16). (j): Sandblasting (group 19). (k): Silica coating (group 21). (l): Hydrofluoric acid (group 23). (m): Self-etching ceramic primer (group 25). Abrasive remnants are marked by ↑.*

teristic bond strengths when contamination was followed by phosphoric acid etching compared with phosphoric acid etching before contamination.

For both substrates and both adhesive application modes, the distribution of failure types differed significantly between different repair contaminations ($p \leq 0.003$).

SEM Images

SEM images are presented in Figures 1 (enamel) and 2 (dentin). Contamination of phosphoric acid etched enamel (Figure 1a) with sandblasting (Figure 1b) or silica coating (Figure 1c) resulted in removal of the etching pattern. When phosphoric acid etching was performed after sandblasting (Figure 1f) or silica coating (Figure 1g), the surface presented an etching pattern not different from the control (Figure 1a). If sandblasting or silica coating, respectively, was not followed by phosphoric acid etching (ie, the universal adhesive was applied in SE), then the surface was distinctly roughened and remnants of abrasive particles could be seen (Figure 1j,k).

Hydrofluoric acid contamination of etched enamel resulted in an etching pattern with few visible surface precipitates (Figure 1d), while hydrofluoric acid etching of native enamel resulted in an amorphous surface layer (Figure 1h,l), which could not be removed by phosphoric acid etching (Figure 1h). Surface precipitation was also visible after

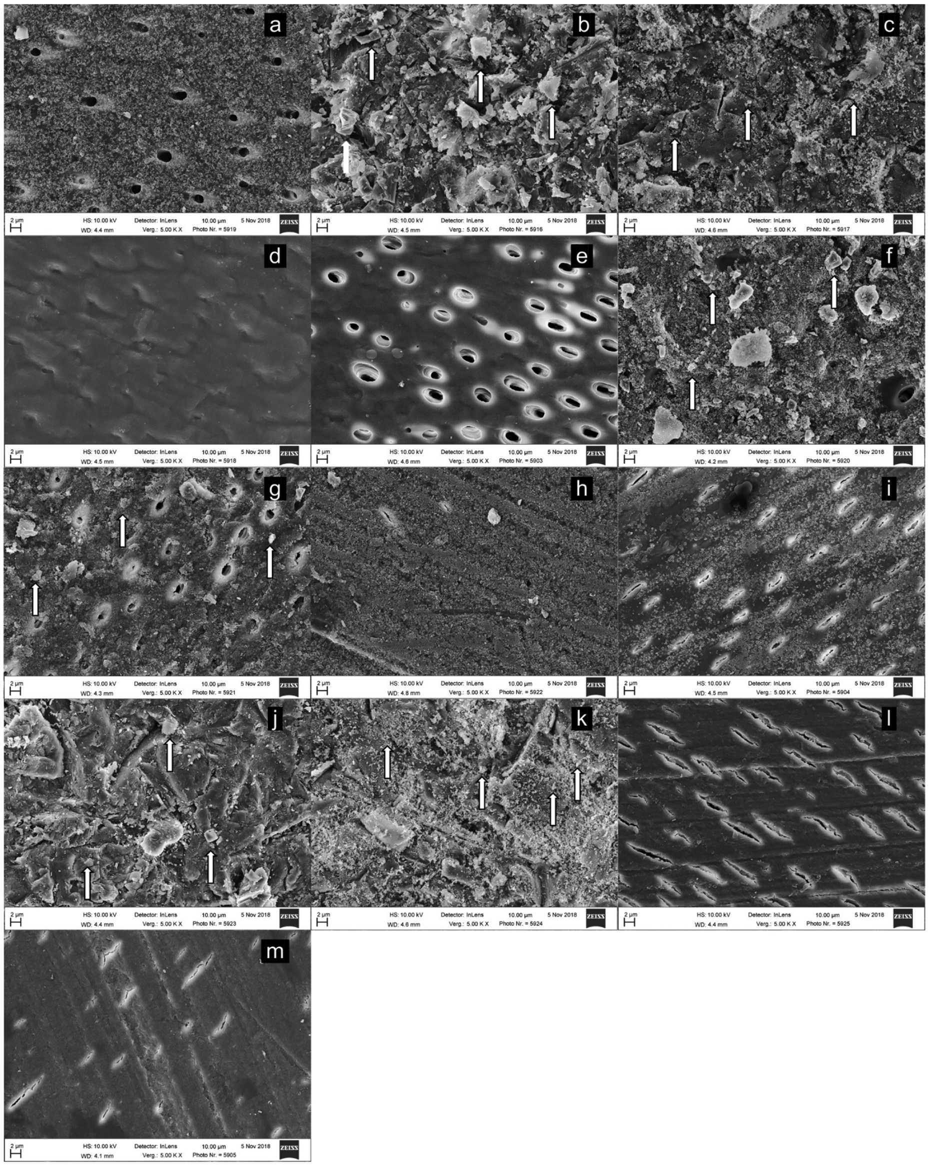

Figure 2. *Representative scanning electron micrographs of dentin surfaces treated with sandblasting, silica coating, hydrofluoric acid etching, or the self-etching ceramic primer either alone (contamination before application of the adhesive in self-etch mode) or before/after phosphoric acid etching (contamination before application in etch-and-rinse mode). Note that contamination with the universal primer is not shown as it was not visible. Original magnification ×5000. (a): Phosphoric acid (control etch-and-rinse, group 1). (b): Phosphoric acid → sandblasting (group 3). (c): Phosphoric acid → silica coating (group 5). (d): Phosphoric acid → hydrofluoric acid (group 7). (e): Phosphoric acid → self-etching ceramic primer (group 9). (f): Sandblasting → phosphoric acid (group 10). (g): Silica coating → phosphoric acid (group 12). (h): Hydrofluoric acid → phosphoric acid (group 14). (i): Self-etching ceramic primer → phosphoric acid (group 16). (j): Sandblasting (group 19). (k): Silica coating (group 21). (l): Hydrofluoric acid (group 23). (m): Self-etching ceramic primer (group 25). Abrasive remnants are marked by ↑.*

contamination with the self-etching ceramic primer (Figure 1e,i,m). Depending on the sequence of phosphoric acid etching, the surface precipitates either covered the enamel etching pattern (Figure 1e) or could not be removed by phosphoric acid etching (Figure 1i).

When etched dentin (Figure 2a) was contaminated by sandblasting (Figure 2b) or silica coating (Figure 2c), the surface was damaged and cracked, the exposed collagen network was removed, and dentinal tubules were no longer visible. When phosphoric acid etching was performed after sandblasting (Figure 2f) or silica coating (Figure 2g), the surface became partly etched and some tubules were visible. If sandblasting or silica coating was not followed by phosphoric acid etching (ie, if the universal adhesive was applied in SE), the dentin surface was damaged (Figure 2j,k). Hydrofluoric acid contamination of etched dentin resulted in an amorphous surface precipitation (Figure 2d,h,l) that also partly covered the dentinal tubules. Slight surface precipitation was also visible after contamination with the self-etching ceramic primer (Figure 2e,i,m).

DISCUSSION

This study aimed to simulate potential contamination of dental hard tissues with different repair conditioning measures. Due to the large number of specimens, bovine specimens rather than human teeth were used. A recent systematic review found that bovine teeth can be a suitable alternative for human enamel and dentin substrates in shear bond strength tests as no significant differences have been detected in shear bond strength between human and bovine enamel and dentin.[12] Adhesion was tested using a universal adhesive containing 10-MDP, but without a silane component. A universal adhesive was chosen not only as it can be applied using different etching strategies (selective enamel etch, ER, and SE) but also as universal adhesives can be used for conditioning of restorative materials when restorations need to be repaired. The functional monomer 10-MDP can bond to zirconia and nonprecious metals by ionic and hydrogen interactions of the phosphate group with the oxide layer of the material[13] while silane-containing adhesives bond to silica-based materials or silica fillers by forming siloxane linkages.[14] Nevertheless, for repair conditioning of glass ceramics, a separate pretreatment step with a silane-containing solution or a universal primer is recommended to reduce the possibility of hydrolytic degradation of the ceramic-resin bonding.[15,16] As opposed to the universal adhesive, the universal primer includes not only phosphoric acid methacrylate but also silane methacrylate (capable of bonding to silicate surfaces) and disulfide methacrylate (capable of bonding to precious metals).

Besides the universal primer, the contamination effects of different repair measures to increase surface roughness of a restorative material by physical (sandblasting, silica coating) or chemical (hydrofluoric acid etching, self-etching ceramic primer) measures were tested. An increased surface roughness might allow for better wettability by the adhesive. In addition to surface roughening, silica coating might further improve repair bond strength due to the incorporation of silica particles. Thereby, formation of Si-O-Si-bonds to silane-containing adhesives or universal primers becomes possible.[17]

For all simulated repair surface conditioning measures, the duration of contamination was based on the duration of application recommended for conventional use of the respective products. Under clinical conditions, contact time to dental hard tissues might be shorter, when unintended contamination is noticed and stopped.

For the ER, air abrasion and silica coating reduced enamel and dentin bond strength and increased the amount of adhesive failures when contamination was performed on etched surfaces. This observation is in line with previous studies showing reduced bond strength of etched enamel and dentin after silica coating.[18,19] The SEM images indicate that the enamel etching pattern and the exposed collagen network of dentin was removed by sandblasting. Conversely, when phosphoric acid etching was performed after sandblasting contamination, bond strength values were not reduced and enamel and dentin specimens presented an etched surface. Previous studies indicated that sandblasting of dental hard tissues does not affect bond strength or margin quality adversely if sandblasted surfaces are cleaned with phosphoric acid[10,11] or water spray before adhesive application.[20] For the SE, sandblasting contamination impaired bond strength values and increased the amount of adhesive failures, probably as the surface was not washed/cleaned, and loose abrasion particles were only air blown.

Hydrofluoric acid contamination of enamel resulted in significantly lower bond strength values, independently of the application sequence and the adhesive strategy. Saracoglu and others[4] demonstrated that 5% and 9.5% hydrofluoric acid inhibited enamel bond strength independently whether applied before or after phosphoric acid etching. However, in contrast to the present study, the etch-and-rinse adhesive performed slightly better if hydrofluoric acid was applied after phosphoric acid etching instead of before.[4] Interestingly, in the present study, enamel specimens presented clearly different surface characteristics (Figure 1d,h). While the sequence phosphoric acid etching/hydrofluoric acid contamination resulted in an etching pattern with only a few visible surface precipitates, the reverse order led to an amorphous surface without any signs of etching. Hydrofluoric acid is known to form dense amorphous fluoride precipitates on the tooth surface, which most probably cannot be removed by further phosphoric acid etching. However, irrespective of the sequence of phosphoric acid etching, hydrofluoric acid contamination significantly reduced enamel bond strength.

Shear bond strength measurement on hydrofluoric acid–exposed dentin yielded ambiguous results. Previous studies found that contact to hydrofluoric acid significantly impaired dentin bond strength, independent of the etching sequence.[4,5] Nevertheless, ultramorphologic analyses showed distinct differences between the etching sequences: If dentin was first etched with phosphoric acid, a collagen-rich layer was exposed, which probably collapsed when further etched with hydrofluoric acid. As a result, a thinner hybrid layer was formed and mineral precipitates were detected at the bottom of the hybrid layer. If hydrofluoric acid etching was performed first, calcium fluoride precipitates were formed that hampered phosphoric acid etching. Underneath the precipitates, a collagen-rich layer was formed, which could not be infiltrated by resin.[5] In the present study, hydrofluoric acid contamination led to precipitates that were covering the surface (Figure 2d) or partial occlusion of dentinal tubules (Figure 2l). Phosphoric acid etching of hydrofluoric acid contaminated dentin was unable to remove the surface precipitates (Figure 2h). Nevertheless, bond strength was significantly impaired only when the adhesive was applied in SE or when phosphoric acid etched dentin was contaminated (group 7). It remains unclear why the sequence hydrofluoric acid contamination/phosphoric acid etching led to only slightly reduced bond strength values, even after thermocycling. Similar results were obtained with the self-etching ceramic primer. On both enamel and dentin, the primer led to surface precipitates (Figure 1e,i,m, Figure 2e,i,m) that impaired bond strength significantly if the universal adhesive was applied in SE (Figures 1m and 2m). Etched enamel became visible underneath the precipitates (Figure 1i). Dentinal tubules were partly occluded by the precipitation (Figure 2e,i,m), but the precipitation seemed to be less severe than after hydrofluoric acid contamination. Further studies have to be performed to fully elucidate how the self-etching ceramic primer affects enamel and dentin adhesion.

The universal primer did not affect bond strength adversely. Previous studies showed that the contamination of enamel[19] or dentin[21] with silane solutions did not reduce bond strength of subsequently applied adhesives. Potentially, the universal primer increased the wettability of the contaminated enamel or dentin surface under certain conditions, leading to slightly higher bond strength values.

The brittleness of the materials included in this study makes their failure difficult to predict and their bond strength less reliable. The Weibull statistic takes into account the variable existence of strength-controlling flaws within brittle materials by providing information about the variability of their bond strengths. A high Weibull modulus reflects a narrow variability of bond strengths and consequently a high reliability on the characteristic strength. Materials or methods with a high Weibull modulus are likely to be less technique-sensitive than those with a low Weibull modulus and should therefore be preferred.[22] Irrespective of the substrate or the adhesive strategy, our results showed the highest Weibull moduli and characteristic strengths in the control groups or when only the universal primer was applied, with the exception of group 8. Thus, contamination of enamel or dentin by repair conditioning measures seemed to result in a less reliable compound between repair restoration and tooth structure and should therefore ideally be avoided.

The present study only assessed the effect of repair surface conditioning measures on enamel and dentin shear bond strengths. However, in a clinical setting mixed surfaces with both tooth and restoration surfaces require optimal repair pretreatment. The potential interference of tooth surface conditioning measures (ie, phosphoric acid etching) on repair bond strength was not assessed in the present study. In particular, zirconia and nonprecious metals should not be treated with phosphoric acid to avoid the formation of a stable phosphate layer inhibiting the adhesion of primers containing phosphoric acid methacrylate (eg, 10-MDP).[23]

Based on these results, the null hypothesis that repair conditioning measures do not affect bond strength of a universal adhesive to enamel or dentin has to be rejected. Within the limitations of this study, the following clinical recommendations for repairing restorations next to enamel and dentin substrates may be suggested: 1) contamination of enamel/dentin by repair conditioning measures should be avoided; 2) if enamel/dentin is contaminated, use of phosphoric acid (ER) before applying a universal adhesive may minimize the effects of the contamination; and 3) contamination of enamel/dentin with a universal primer alone may not significantly affect the bond strength of dentin or enamel to resin composite when using a universal adhesive.

These recommendations are in line with a recently published policy statement on the repair of restorations published by the FDI World Dental Federation: When tooth substance is next to a restoration in need of repair, physical repair surface conditioning measures should be performed before phosphoric acid etching of the tooth substance.[24] However, it has also been suggested that repairs be performed in a two-step technique if tooth substance is present: In the first step, only the tooth surface is covered using composite. Afterward, physical/chemical repair surface conditioning measures are applied to both the composite and the adjacent restoration.[25] Given the results of the present study, this technique seems rational as it combines optimal repair bonding procedures for both tooth surfaces and restorations.

Note

This study was previously presented at the 9th CONSEURO, June 14-15, 2019, Berlin, Germany.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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