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# Influence of silver diamine fluoride on the adhesive properties of interface resin-eroded dentin

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ABSTRACT

*Purpose:* To evaluate the effects of silver diamine fluoride (SDF) on resin-eroded dentin bond strength (μTBS) and the *in-situ* degree of conversion (DC) for universal adhesives, and the chemical/morphological changes induced by SDF.

*Materials and methods:* One hundred ten extracted molars were randomly assigned to 12 experimental groups: (1) SDF (eroded dentin [ED] without SDF, treatment with either 12% SDF [ED + 12%SDF], 38% SDF [ED + 38% SDF] or 38% SDF without iodide potassium [KI] [ED + 38% SDF Without KI]); (2) adhesive systems (Clearfil Universal Bond Quick and Scotchbond Universal Adhesive); (3) adhesive strategies (etch-and-rinse [ER] and self-etch [SE]). After restoration, the specimens were sectioned into resin-dentin sticks and the μTBS was evaluated. Selected sticks were used for DC evaluation via micro-Raman. The dentin-etching patterns and chemical/morphological changes induced by SDF were examined using energy dispersive X-ray spectroscopy (EDX-SEM). Data from the μTBS and DC tests for each adhesive were analyzed using two-way repeated measures ANOVA and Tukey's test ($\alpha = 0.05$).

*Results:* The application of SDF resulted in a higher μTBS compared to without SDF. However, a statistically significant difference was observed only when 38% SDF was applied ($p < 0.001$). The application of SDF did not significantly influence the mean DC for both adhesive systems ($p > 0.61$). EDX-SEM indicated the presence of silver ions in addition to calcium mineral deposition after SDF application. Additionally, the silver crystal deposition was found in the adhesive/hybrid layer, independent of the SDF concentration. No significant differences was observed when 38% SDF was applied with or without KI.

*Conclusion:* The application of SDF to universal adhesives in the ER and SE modes may be a viable alternative to increasing the bonding to eroded dentin without jeopardizing the DC.

## 1. Introduction

Dental erosion is considered an emerging problem in oral health among adults, adolescents, and children [1]. This condition promotes a continuous loss of dental hard substances triggered by nonbacterial acid attacks [2–4]. Dentists frequently encounter established dental erosion that is associated with tooth sensitivity and esthetics impairment that necessitates restorative treatment [5,6]. Unfortunately, the varying characteristics of eroded dentin represent an additional challenge with respect to dental adhesion.

Acid exposure is known to cause enamel surface demineralization. Long-term demineralization leads to erosive wear due to progressive mineral loss [7]. In dentin, the initial dissolution of the mineral exposes the organic matrix [8,9]. The progression of this process is modulated by

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**Table 1**
Adhesive system (batch number), groups, composition, and application modes[a].

| Adhesive system (batch number) | Groups/ composition | Application mode | |
|---|---|---|---|
| | | Etch-and-rinse | Self-etch |
| Clearfil Universal Bond Quick (CUQ); Kuraray (CD0012) | Eroded dentin (ED) | 1. Apply etchant for 15 s.<br>2. Rinse for 15 s and air-dry for 5 s to keep the surface slightly moist.<br>3. Apply adhesive as for the **self-etch mode.** | 1. Apply adhesive to the entire cavity with a microbrush and rub. No waiting time is required.<br>2. Dry by blowing mild air for 5 s until the adhesive does not move.<br>3. Light cure for 10 s at 1200 mW/cm$^2$. |
| | ED + silver diamine fluoride 12% (ED + 12% SDF) (Cariestop Cariostatic, Biodinâmica LTDA, Ibiporã, PR, Brazil) Hydrofluoridric acid, 12% silver nitrate, ammonia hydroxide, and deionized water | 1. Apply etchant for 15 s.<br>2. Rinse for 15 s and air-dry for 5 s to keep the surface slightly moist.<br>3. Apply the SDF solution for 3 min.<br>4. Rinse for 30 s and air-dry for 5 s to keep the surface slightly moist.<br>5. Apply adhesive as for the **self-etch mode (3 to 5 steps).** | 1. Apply the SDF solution for 3 min.<br>2. Rinsing for 30 s with distilled water air-dry for 5 s to keep the surface slightly moist.<br>3. Apply bond to the entire cavity with a microbrush and rub. No waiting time is required.<br>4. Dry by blowing mild air for 5 s until the adhesive does not move.<br>5. Light cure for 10 s at 1200 mW/cm$^2$. |
| | ED + silver diamine fluoride 38% (ED + 38% SDF) (Riva Star, SDI Limited, Victoria, Australia) Step 1 (30–35% silver fluoride and 60% ammonia solution) and step 2 (saturated KI solution) were applied<br>ED + silver diamine fluoride 38% (ED + 38% SDF without KI) (Riva Star, SDI Limited, Victoria, Australia) Only the bottle 1 (30–35% silver fluoride and 60% ammonia solution) was applied | 1. Apply etchant for 15 s.<br>2. Rinse for 15 s and air-dry for 5 s to keep the surface slightly moist.<br>3. Apply the silver capsule with a silver brush.<br>4. Apply a generous amount of solution from the green capsule to the surface until the creamy precipitate turns clear.<br>5. Rinse for 30 s and air-dry for 5 s to keep the surface slightly moist.<br>6. Apply adhesive as for the **self-etch mode (3 to 5 steps).** | 1. Apply the silver capsule with a silver brush.<br>2. Apply a generous amount of solution from the green capsule to the surface until the creamy precipitate turns clear.<br>3. Apply bond to the entire cavity with a microbrush and rub. No waiting time is required.<br>4. Dry by blowing mild air for 5 s until the adhesive does not move.<br>5. Light cure for 10 s at 1200 mW/cm$^2$. |
| Scotchbond Universal (SBU); 3 M Oral Care (638,367) | Eroded dentin (ED) | 1. Apply etchant for 15 s.<br>2. Rinse for 15 s and air-dry for 5 s to keep the surface slightly moist.<br>3. Apply adhesive as for the **self-etch mode**. | 1. Apply the adhesive to the entire preparation and leave undisturbed for 20 s.<br>2. Direct a gentle stream of air over the liquid for approximately 5 s until it no longer moves and the solvent evaporates completely.<br>3. Light cure for 10 s at 1200 mW/cm$^2$. |
| | ED + silver diamine fluoride 12% (ED + 12% SDF) (Cariestop Cariostatic, Biodinâmica LTDA, Ibiporã, PR, Brazil) Hydrofluoridric acid, 12% silver nitrate, ammonia hydroxide, and deionized water | 1. Apply etchant for 15 s.<br>2. Rinse for 15 s and air-dry for 5 s to keep the surface slightly moist.<br>3. Apply the SDF solution for 3 min.<br>4. Rinse for 30 s and air-dry for 5 s to keep the surface slightly moist.<br>5. Apply adhesive as for the **self-etch mode (3 to 5 steps).** | 1. Apply the SDF solution for 3 min.<br>2. Rinsing for 30 s with distilled water and air-dry for 5 s to keep the surface slightly moist.<br>3. Apply the adhesive to the entire preparation and leave undisturbed for 20 s.<br>4. Directed a gentle stream of air over the liquid for approximately 5 s until it no longer moves and the solvent evaporates completely.<br>5. Light cure for 10 s at 1200 mW/cm$^2$. |
| | ED + silver diamine fluoride 38% (ED + 38% SDF) (Riva Start, SDI Limited, Victoria, Australia) Step 1 (30–35% silver fluoride and 60% ammonia solution) and step 2 (saturated KI solution) were applied<br>ED + silver diamine fluoride 38% without KI (ED + 38% SDF without KI) (Riva Star, SDI Limited, Victoria, Australia) Only the bottle 1 (30–35% silver fluoride and 60% ammonia solution) was applied | 1. Apply etchant for 15 s.<br>2. Rinse for 15 s and air-dry for 5 s to keep the surface slightly moist.<br>3. Apply the silver capsule with silver brush.<br>4. Apply a generous amount of solution from the green capsule to the surface until the creamy precipitate turns clear.<br>5. Rinse for 30 s and air-dry for 5 s to keep the surface slightly moist.<br>6. Apply adhesive as for the **self-etch mode (3 to 5 steps)**. | 1. Apply the silver capsule with a silver brush.<br>2. Apply a generous amount of solution from the green capsule to the surface until the creamy precipitate turns clear.<br>3. Apply the adhesive to the entire preparation and leave undisturbed for 20 s.<br>4. Direct a gentle stream of air over the liquid for approximately 5 s until it no longer moves and the solvent evaporates completely.<br>5. Light cure for 10 s at 1200 mW/cm$^2$. |

[a] The materials were applied according to the recommendations of their respective manufacturers, except in the group ED + 38% SDF without KI.

the presence and amount of the demineralized organic matrix [10,11].

Demineralized tissue at the lesion surface interferes with the diffusion of ions outside and inside the demineralized area, thereby reducing the progression of erosion during subsequent acid attacks [10]. However, an increase in the demineralization progression of erosive tooth

**Table 2**
Number of specimens (%) according to fracture mode.

| Groups | Clearfil Universal Bond Quick | | | | | | Scotchbond Universal | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Etch-and-rinse | | | Self-etch | | | Etch-and-rinse | | | Self-etch | | |
| | A/M | C | PF | A/M | C | PF | A/M | C | PF | A/M | C | PF |
| ED | 110 (97) | 0(0) | 3(3) | 105 (95) | 0(0) | 5(5) | 110(96) | 0(0) | 4(4) | 101(97) | 0(0) | 3(3) |
| ED + 12% SDF | 114 (99) | 0(0) | 1(1) | 118 (98) | 1(1) | 1(1) | 110(98) | 2(2) | 0(0) | 113(97) | 2(2) | 1(1) |
| ED + 38% SDF | 117 (99) | 0(0) | 1(1) | 114 (98) | 1(1) | 1(1) | 119(99) | 0(0) | 1(1) | 116(98) | 1(1) | 1(1) |
| ED + 38% SDF | 115 (100) | 0(0) | 0(0) | 120 (98) | 1(1) | 1(1) | 117(98) | 0(0) | 2(2) | 119(99) | 0(0) | 1(1) |

Abbreviations: A/M: adhesive/mixed fracture mode; C: cohesive fracture mode; PF: premature failures.

**Table 3**
Means and standard deviations of μTBS (MPa) for all experimental groups (*).

| Groups | Clearfil Universal Bond Quick | | Scotchbond Universal | |
|---|---|---|---|---|
| | Etch-and-rinse | Self-etch | Etch-and-rinse | Self-etch |
| ED | 33.74 ± 3.6 B | 35.48 ± 2.9 B | 34.90 ± 3.3 b | 36.56 ± 4.1 b |
| ED + 12% SDF | 38.03 ± 3.5 A,B | 39.53 ± 4.2 A,B | 42.45 ± 2.9 a | 39.98 ± 1.7 a,b |
| ED + 38% SDF | 39.68 ± 2.7 A | 41.31 ± 2.0 A | 40.47 ± 4.2 a | 41.08 ± 3.0 a |
| ED + 38% SDF Without KI | 39.38 ± 2.5 A | 40.55 ± 2.9 A | 41.30 ± 2.5 a | 41.57 ± 2.4. a |

(*) Similar capital or lowercase letters means groups statistically similar groups (2-way repeated measures ANOVA and Tukey test; p = 0.0001).

**Table 4**
*In-situ* degree of conversion (DC) values (means ± standard deviations) for all experimental groups (*).

| Groups | Clearfil Universal Bond Quick | | Scotchbond Universal | |
|---|---|---|---|---|
| | Etch-and-rinse | Self-etch | Etch-and-rinse | Self-etch |
| ED | 55.38 ± 4.3 A | 56.14 ± 3.0 A | 60.59 ± 2.3 a | 61.78 ± 2.9 a |
| ED + 12% SDF | 54.30 ± 3.4 A | 53.47 ± 3.7 A | 58.14 ± 2.1 a,b | 59.68 ± 3.6 a |
| ED + 38% SDF | 53.37 ± 3.0 A | 52.61 ± 4.0 A | 57.57 ± 3.8 a,b | 58.29 ± 3.9 a,b |
| ED + 38% SDF Without KI | 54.43 ± 1.9 A | 53.91 ± 3.2 A | 58.31 ± 2.9 a,b | 59.62 ± 3.1 a |

(*) Similar capital or lowercase letters means groups statistically similar groups (2-way repeated measures ANOVA and Tukey test; p = 0.0001).

loss can be accelerated by the presence of pepsin [12], matrix metalloproteinases (MMPs), and cathepsins that are constitutively present in the saliva and dentin [13]. Additionally, a superficial layer of exposed collagen on the demineralized eroded matrix hinders adequate infiltration by resin monomers [6,14,15], impairing the restorative procedure.

Silver diamine fluoride (SDF) is topical silver fluoride solution used in the prevention and treatment of caries lesions [18,21,22]. SDF acted positively influences dentine remineralization and inhibits dentine demineralization [16–18], because it combines the increases dental mineral resistance to acids by the formation of fluor-hydroxyapatite [16], with the antibacterial effect on microorganisms via interaction with silver nitrate [17,18]. Additionally, it has been demonstrated that SDF impedes the degradation of the collagen matrix by inhibiting the matrix metalloproteinases 2, 8, and 9 [19], due to a higher concentration of fluoride and silver ions [20].

Thus, SDF could potentially inhibit the erosive progress, similar to the case when SDF is applied to arrest the progression of caries [18,21,22], and unfortunately, the application of SDF negatively influences the bond strength to caries-affected dentin [23]. However, to the best of our knowledge, there are no studies addressing SDF's effects and concentrations on the bond strength of resin composite on eroded dentin.

Thus, the aim of this study was to evaluate the effects of the two concentrations of SDF on resin-eroded dentin bond strength, the degree of conversion of the adhesive via *in-situ* micro-Raman spectroscopy, and the chemical/morphological changes induced by SDF. The null hypotheses tested were that the use of SDF will not affect (1) μTBS, (2) the DC values of the adhesive system when applied using the etch-and-rinse and self-etch approaches, and (3) the chemical/morphological differences on the eroded dentin surface.

## 2. Method and materials

### 2.1. Selection and preparation of teeth

A total of 124 extracted caries-free human molars were used in this study. The teeth were collected after obtaining informed consent from the patients. The ethics committee of the institution where the research was conducted approved this study under protocol #2.631.289. In this investigation, the molars were disinfected in 1% thymol, stored in distilled water, and used within 6 months of extraction.

The teeth were sectioned parallel to the occlusal surface using a low-speed diamond saw (Isomet, Buehler, Evanston, IL, USA) under cool water to expose the mid-coronal dentin. All specimens received a standardized smear layer by polishing the flat dentin surfaces using 600-grit SiC paper under running water for 60 s.

### 2.2. Experimental design

The teeth were then randomly assigned to 12 experimental groups ($n$ = 80 dentin for microtensile bond strength [μTBS] and *in-situ* degree conversion [DC] assessments, $n$ = 12 for dentin-etching patterns, and $n$ = 32 dentin for adhesive/hybrid layer analysis) as follows: (1) silver diamine fluoride (eroded dentin without SDF treatment [ED], with 12% silver SDF [ED + 12% SD] and 38% SDF [ED + 38% SDF]) and 38% SDF without iodide potassium [KI] [ED + 38% SDF Without KI]); (2) adhesive systems (Clearfil Universal Bond Quick [CUQ], Kuraray Noritake Dental, Tokyo, Japan, and Scotchbond Universal Adhesive [SBU], 3 M Oral Care, St. Paul, MN, USA, also known as Single Bond Universal in some countries); and (3) adhesive strategies (etch-and-rinse [ER] and self-etch [SE]). The materials used, batch numbers, composition, and application modes are described in Table 1.

### 2.3. Sample size calculation

The sample size was determined by considering the microtensile bond strength of the Scotchbond Universal on dentin. The mean and standard deviation of Scotchbond Universal reported in the literature was 53.6 ± 5.0 MPa [15,24–26]. To detect a difference of 10 MPa among the tested groups using a significance level of 5%, a power of 80% and a two-sided test, the minimum sample size was 5 teeth per group.

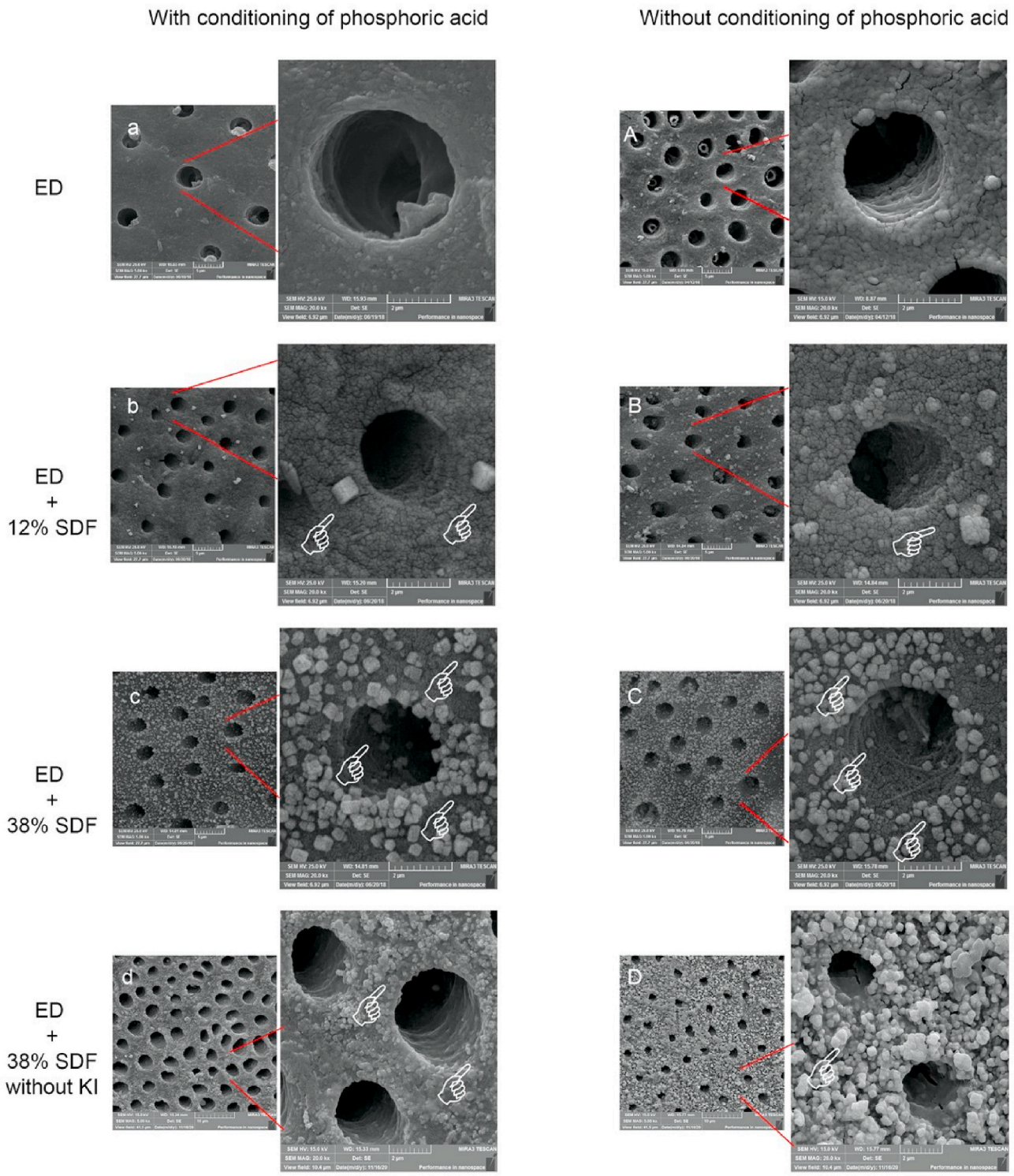


**Fig. 1.** Representative morphology of the eroded dentin and the eroded dentin treated with different silver diamine fluoride solutions, with and without the application of phosphoric acid. The smear layer was completely removed in the eroded dentin to reveal enlarged lumen tubules, particularly after conditioning with phosphoric acid. The SDF solution exhibited silver particles on the surface (white hands in b-d and B-D). This was more evident at a higher concentration of the SDF solution even after etching conditioning with phosphoric acid (white hands in b-d).

### *2.4. pH cycling model*

The specimens were exposed to an erosive cyclic de- and remineralization procedure by immersion in a soft drink (Coca-Cola, pH 2.6) four times daily for 90 s each (10 ml per specimen) for 5 days [13,15,27]. The soft drink was renewed at each erosive challenge and the remineralization solution was replaced daily. After each demineralization, the specimens were rinsed with deionized water (10 s) and immersed in a remineralizing solution (4.08 mM $H_3PO_4$, 20.10 mM KCl, 11.90 mM $Na_2CO_3$, and 1.98 mM $CaCl_2$, pH of 6.7, 10 ml per specimen) for 60 min [15,28]. The pH levels of all solutions were monitored periodically using a pH meter.

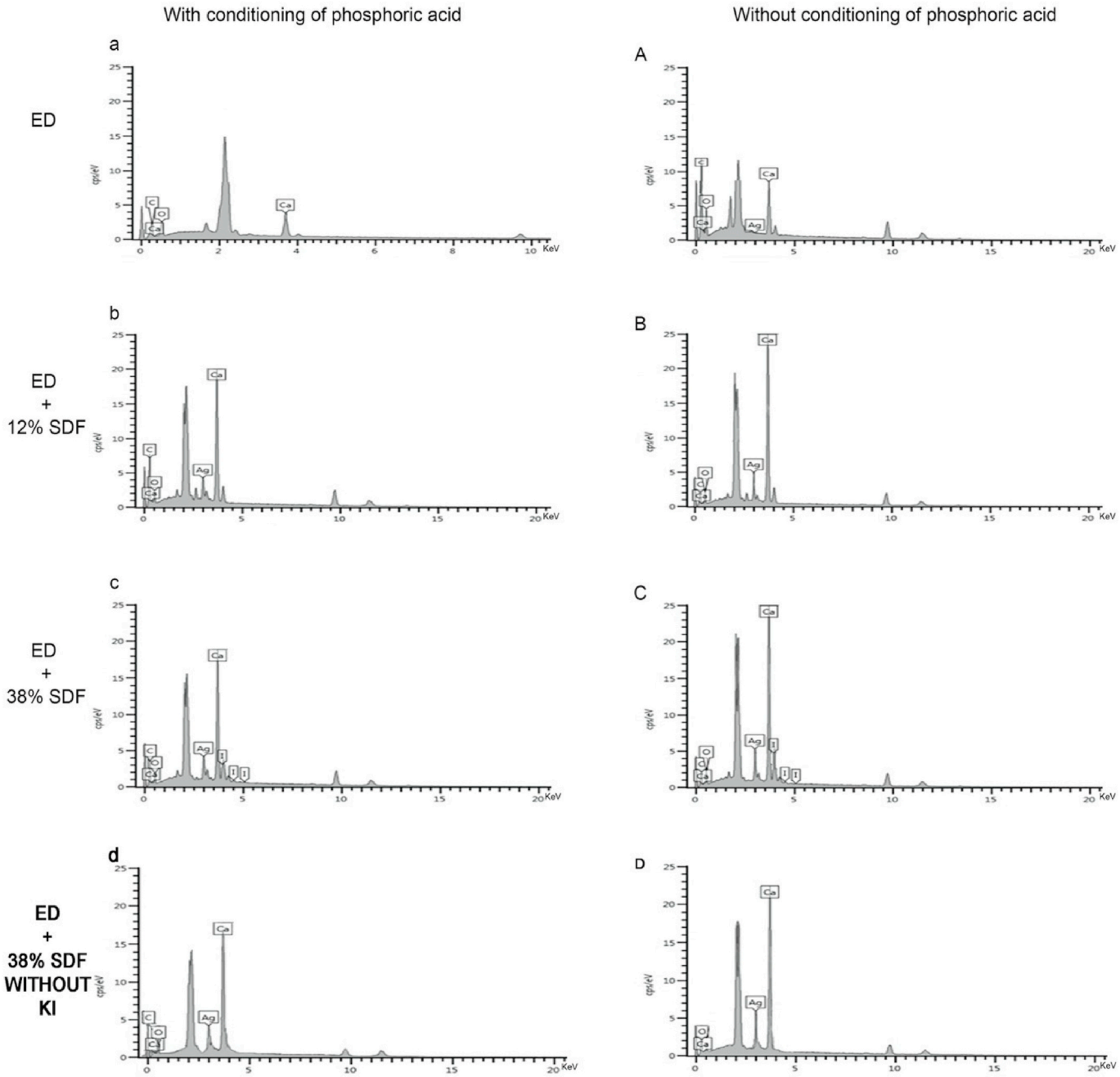


**Fig. 2.** Silver deposition in addition to an increase of calcium content on the surface was confirmed via EDX analysis after SDF treatment in the eroded dentin (spectrum to the left of b-d and B-D). For the group ED + 38% SDF, it is possible to identify the presence of iodide ions on the surface (spectrum to the left of c and C). Observe that, iodide ions were not detected in the group ED + 38% SDF without KI (spectrum to the left of d and D).

### *2.5. Restorative procedures*

After erosive demineralization, the surface of dentin was treated with SDF according to two groups (ED + 12% SDF, ED + 38% SDF and 38% SDF without iodide potassium [ED + 38% SDF Without KI])) as described in Table 1. Afterward, the adhesive systems were applied to all groups according to the step-by-step bonding procedures by following the manufacturer's instructions (Table 1). Composite resin buildup (Opallis, FGM Dental Products, Joinville, SC, Brazil) was achieved incrementally (three increments of 1–2 mm each) and individual light-activation was performed for 40 s each time using an LED light-curing unit set at 1.200 mW/cm$^2$ (Radii, SDI; Bayswater, Victoria, Australia). A single operator performed all bonding procedures.

After storage in distilled water at 37 °C for 24 h, the specimens were sectioned longitudinally in the mesial-to-distal and buccal-to-lingual directions across the bonded interface using a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) to obtain resin-dentin bonded sticks with a cross-sectional area of approximately 0.8 mm$^2$, as measured using digital calipers (Digimatic Caliper, Mitutoyo, Tokyo, Japan). The number of sticks that exhibited premature debonding (D) during specimen preparation process was recorded for each tooth.

Approximately 20–25 resin-dentin bonded sticks were obtained per tooth and they were divided as follows: two sticks from each tooth and from each experimental condition were evaluated for *in situ* degree of conversion (DC) within the adhesive/hybrid layers and the remaining were evaluated for μTBS.

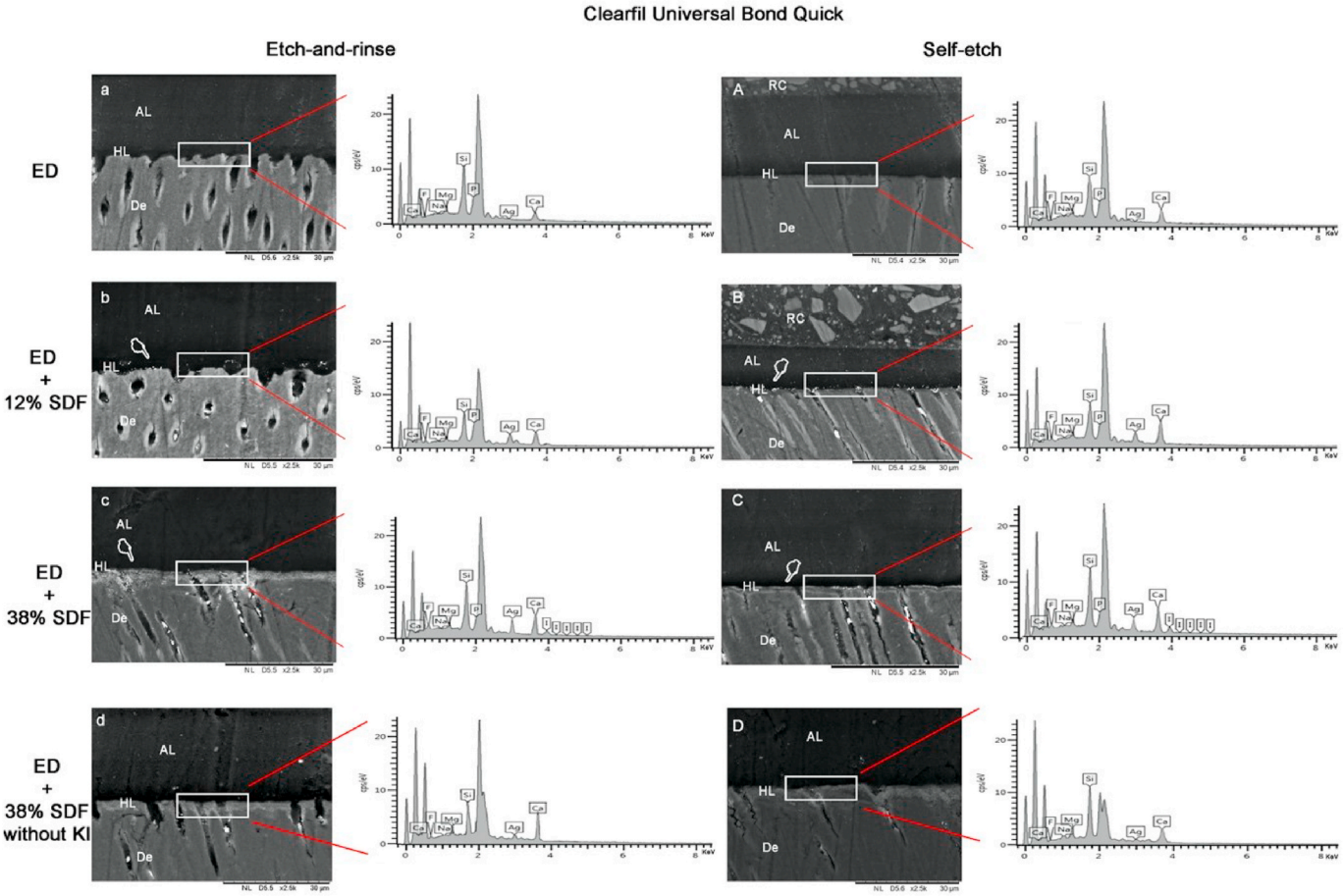


**Fig. 3.** Representative images of adhesive/hybrid layer interfaces produced in eroded dentin and the eroded dentin treated with the two SDF solutions added to Clearfil Universal Bond Quick. Silver traces were only observed for the adhesive interfaces for the ER and SE approaches for the SDF groups (white hands), in addition to an increase of the Ca peak. Additionally, the presence of silver deposition was also observed in dentinal tubules. Iodide ions were only observed for 38% SDF via EDX analysis. On the other hand, iodide ions were not detected in the group ED + 38% SDF without KI. RC = resin composite; AL = adhesive layer; HL = Hybrid layer; De = dentin.

### *2.6. Microtensile bond strength (μTBS)*

The resin-dentin bonded sticks were attached to a Geraldeli's jig [29] using cyanoacrylate adhesive and tested under tension (Kratos Dinamometros, Cotia, SP, Brazil) at a rate of 0.5 mm/min until failure occurred. The μTBS values (MPa) were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure mode of the resin-dentin bonded sticks was classified as cohesive (C, failure exclusively within the dentin or the resin composite) and adhesive (A/M, failure at the resin-dentin interface or with partial cohesive failure of the neighboring substrates). This classification was performed using a stereomicroscope at 100 × magnification (Olympus SZ40, Tokyo, Japan). The specimens with premature failure (PF) were included in the calculation of the mean value of each tooth for statistical analysis.

### *2.7. In-situ degree of conversion (DC) within adhesive/hybrid layers*

The resin-dentin bonded sticks were wet polished using 1500- and 2000-grit silicon-carbide paper. The specimens were ultrasonically cleaned for 10 min and positioned in a micro-Raman instrument (XploRA ONE Raman microscope, HORIBA Scientific, Edison, NJ, USA) that was first calibrated for zero and then for coefficient values using a silicon sample. The samples were analyzed using a 638-nm diode laser through a 100 × air objective. The Raman signal was acquired using 600-line/mm grafting centered between 400 $cm^{-1}$ and 2000 $cm^{-1}$, and the employed parameters were 100 mW, spatial resolution of 3 μm, spectral resolution of 5 $cm^{-1}$, and an accumulation time of 25 s, with 5 co-additions. Spectra were obtained at the dentin-adhesive interface at three random sites per bonded stick within the hybrid layer in the intertubular-infiltrated dentin. Spectra of the uncured adhesives were taken as references. The ratio of the double-bond content of the monomer to the polymer in the adhesive was calculated according to the following formula: DC (%) = (1 - [*R* cured/*R* uncured]) × 100, where *R* is the ratio of the aliphatic and aromatic peak intensities at 1639 $cm^{-1}$ and 1609 $cm^{-1}$ in the cured and uncured adhesives, respectively.

### *2.8. Dentin-etching pattern/energy dispersive X-ray spectroscopy (EDX-SEM)*

For this part of the study, the flat dentin surface of twelve teeth was exposed and polished with 600-grit silicon-carbide paper for 60 s to standardize the smear layer using a water-cooled low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). The roots were then removed 2 mm below the cementoenamel junction. Afterward, each third was transversely sectioned in a buccal-to-lingual direction across the occlusal surface using the same saw to obtain two-thirds of the teeth (*n* = 24 specimens). Prior to erosive demineralization, the proximal and pulpal areas and the sectioned area of each third were covered with two layers of nail varnish to allow erosion only in the occlusal surface.

After erosive cycling, each third was randomly allocated to 8 groups as follows: (1) silver diamine fluoride (eroded dentin without SDF

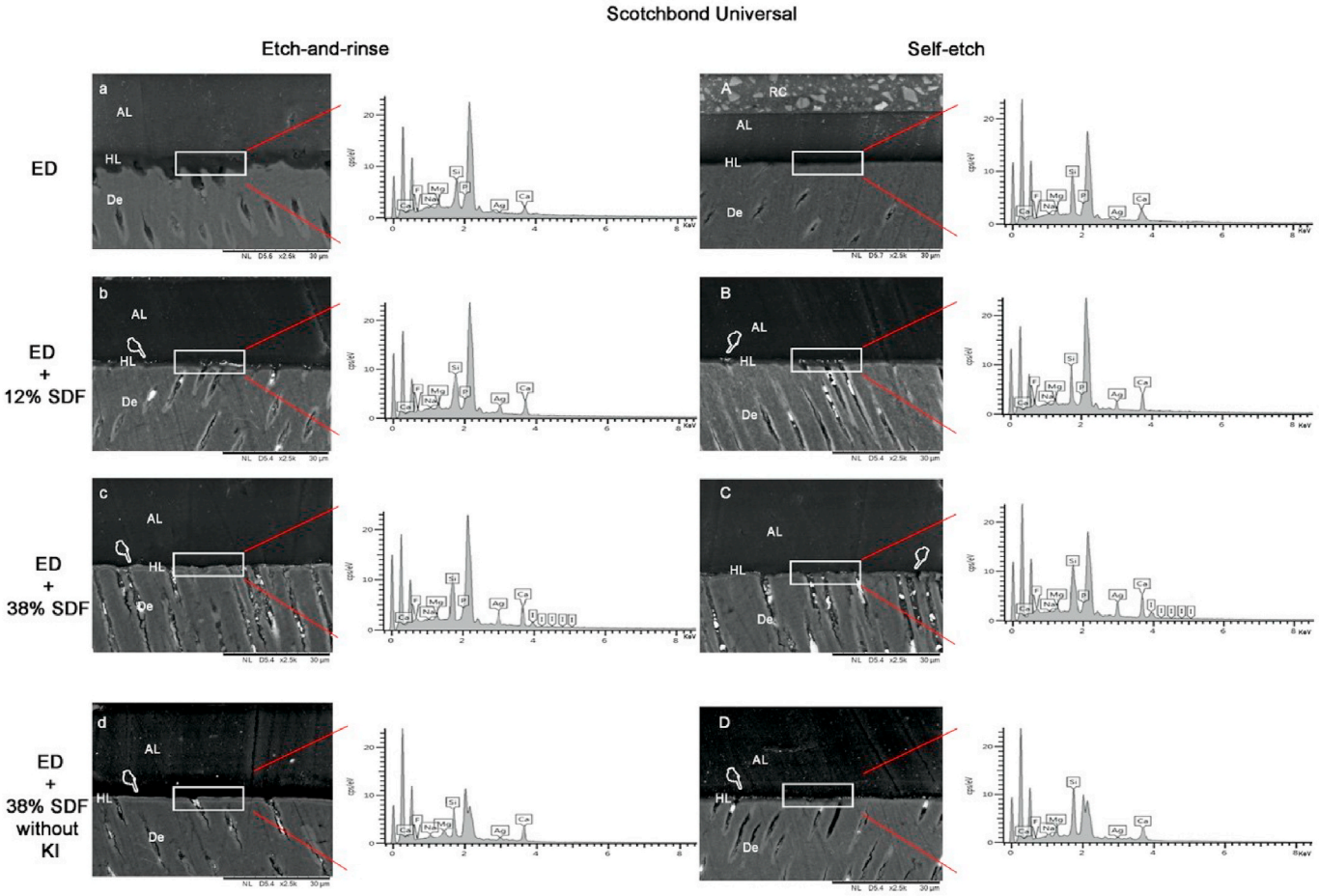


**Fig. 4.** Representative images of adhesive/hybrid layer interfaces produced in eroded dentin and the eroded dentin treated with the two SDF solutions added to Scotchbond Universal Adhesive. Independent of the adhesive strategy used, eroded dentin treated with silver diamine fluoride exhibited an increase of the Ca peak and the presence of silver infiltration (white hands). The presence of iodide ions was observed only in the 38% SDF groups. On the other hand, iodide ions were not detected in the group ED + 38% SDF without KI. RC = resin composite; AL = adhesive layer; HL = Hybrid layer; De = dentin.

treatment [ED], with 12% silver SDF [ED + 12% SDF], 38% SDF [ED + 38% SDF] or 38% SDF without iodide potassium [KI] [ED + 38% SDF Without KI]) and (2) acid application (without or with the application of 37% phosphoric acid [Condac 37%, FGM, Joinville, SC, Brazil]) based on the protocol described in the restorative procedure item (Table 1). Then the surfaces were rinsed with tap water for 30 s, followed by air-drying with an air spray for 5 s, keeping the dentin wet. The specimens were fixed in 2.5% glutaraldehyde in 0.1 M sodium cacodylate buffer at pH 7.4 for 12 h at 4 °C, rinsed with 20 ml of 0.2 M sodium cacodylate buffer at pH 7.4 for 1 h, and dehydrated in ascending grades of ethanol: 25% (20 min), 50% (20 min), 75% (20 min), 95% (30 min), and 100% (60 min) [30].

The specimens were segmented, and sputter coated with gold/palladium in a vacuum evaporator (SCD 050, Balzers, Schaan, Liechtenstein). The entire surface was examined using a scanning electron microscope (MIRA, Tescan Orsay Holding, Warrendale, PA, USA). Three micrographs of representative surface areas were obtained at 5000 × and 20,000 × magnification. Additionally, the specimens were subjected to semi-quantitative chemical microanalysis via EDX-SEM. The analysis was performed along the entire length of the surface.

### 2.9. Adhesive/hybrid layer analysis by energy dispersive X-ray spectroscopy (EDX-SEM)

Two dentin specimens per experimental group were eroded and restored as previously described in the restorative procedure sections (Table 1). After 24 h of water storage, the teeth were longitudinally sectioned across the bonded interface using a low-speed diamond (Isomet, Buehler, Lake Bluff, IL, USA) to obtain four slices for each tooth. All bonded slices were ultrasonically cleaned, air-dried, mounted on stubs, and polished with a wet #600, 1000-, 1500-, 2000- grit SiC paper. The resin-dentin slice interfaces were analyzed using a field emission scanning electron microscope (MIRA, TESCAN ORSAY HOLDING, Warrendale, PA, USA) coupled with an energy-dispersive X-ray spectrometer (EDX). The bonding area was observed, and the analyses focused on the middle of the hybrid layer. Three photomicrographs of representative surface areas were obtained at 2.500× magnification and a semi-quantitative chemical microanalysis was performed via EDX-SEM.

### 2.10. Statistical analysis

The mean μTBS (MPa) and DC (%) values of all bonded sticks from the same tooth were averaged for statistical purposes; thus, the experimental unit in this study was a tooth. . The Kolmogorov–Smirnov test was employed to assess whether the data from these tests followed a normal distribution. The Barlett's test was performed to determine the validity of the assumption of equal variances (data not showed). The μTBS (MPa) and DC (%) data for each adhesive system were subjected to two-way repeated measures ANOVA (silver diamine fluoride vs. adhesive strategies) and Tukey's test with a level of significance of 5%. The dentin-etching pattern assessed by EDX-SEM was only qualitatively evaluated.

## 3. Results

### 3.1. Microtensile bond strength (μTBS)

Approximately 20–25 sticks were obtained per tooth including the premature failures. Most of the specimens (96%–99%) showed adhesive/mixed failures (Table 2). The cross-product interaction, as well as main factors were significant ($p = 0.001$; Table 3). The application of SDF in the eroded dentin resulted in a higher mean μTBS compared to the control for both adhesive systems (Table 3). However, it was statistically significant only when the higher concentration of SDF (ED + 38% SDF and ED + 38% SDF without KI) were applied ($p = 0.001$; Table 3). No significant difference was observed when the adhesive strategies were compared ($p > 0.63$; Table 3).

### 3.2. In-situ degree of conversion (DC) within adhesive/hybrid layers

The cross-product interaction, as well as main factors were not significant ($p > 0.42$; Table 4). The application of SDF in the eroded dentin did not significantly influence the mean DC compared to the controls for both adhesive systems ($p > 0.61$; Table 4). No significant difference was observed when the adhesive strategies were compared ($p > 0.42$; Table 4).

### 3.3. Dentin-etching pattern/energy dispersive X-ray spectroscopy (EDX-SEM)

The representative morphologies of the eroded dentin and the eroded dentin treated with the two SDF solutions are shown in Figs. 1 and 2. The smear layer was completely removed in the eroded dentin and showed a clean dentin surface with a low amount of calcium after the conditioning procedure, compared to the groups without conditioning with phosphoric acid (Fig. 1). After SDF application, silver ion deposition was identified on the surface. These silver ions were found even after phosphoric acid treatment, independent of the SDF concentration. An important characteristic of the eroded dentin surface treatment with SDF is the increase in calcium mineral compared to the surface without SDF application. As expected, only in the SDF 38% with KI group, it was possible to detect iodide deposition of ion on the surface. On the other hand, iodide ions were not detected in the group ED + 38% SDF without KI (Fig. 2).

### 3.4. Adhesive/hybrid layer analysis by energy dispersive X-ray spectroscopy (EDX-SEM)

For both universal adhesives, representative images of the adhesive/hybrid layer interfaces for eroded dentin and eroded dentin treated with the two SDF solutions are shown in Figs. 3 and 4. Independent of the adhesive used and the concentration of SDF, silver deposition is observed on the adhesive interface of the treated eroded dentin. This silver precipitation was observed in the adhesive interfaces, as well as the dentinal tubules, independent of the adhesive strategy. The increase of silver deposition was confirmed for all SDF groups. However, only for 38% SDF with KI, it was possible to confirm the presence of iodide ions. Additionally, an increase in the Ca peak was observed after SDF treatment that was proportional to the concentration, independent of the adhesive used.

## 4. Discussion

In the present study, the application of SDF to eroded dentin increased the bond strength compared to eroded dentin without SDF for both adhesive systems; thus, the first null hypothesis was rejected.

It is known that eroded dentin has a deeper demineralized layer in the presence of a zone of denatured and dense fibrous collagen networks [31], followed by a partially demineralized zone of dentin, and finally sound dentin [32]. The degree of resin infiltration into the exposed collagen fibrils within the demineralized dentin zone has a profound influence on the bond integrity [33–35]. Thus, the authors of the present study hypothesized that the presence of disorganized collagen fibers associated with deep demineralization could influence the infiltration of the resin monomers of the adhesive systems when applied to eroded dentin, resulting in low bond strength values, as previously reported by several authors that evaluated the application of universal adhesives in eroded dentin [6,14,15].

In the present study, SDF concentrations were tested according to the synergistic effect of fluoride and silver present in their composition. It is known that the fluoride component of SDF reacts with calcium phosphate and hydroxyapatite to form fluor-hydroxyapatite and calcium fluoride, which improves the resistance of hard dental tissues [18].

Mei et al. [36] recently demonstrated crystal formation during the remineralization process after the application of SDF of different concentrations. According to the authors, after 24 h, SDF alters the crystal structure of the precipitate minerals and its presence promotes the formation of fluor-hydroxyapatite [16,36]. Furthermore, an important factor in the remineralization by SDF could be the residual mineral crystals of the tooth that serve as nucleation sites for fluor-hydroxyapatite formation [37]. Another factor is the alkaline characteristic of SDF that favors mineral precipitation and could promote the synthesis of fluor-hydroxyapatite [38].

Although the methods used in the present study were not suitable for demonstrating the formation of fluor-hydroxyapatite, according to the EDX-SEM evaluation, an increase in the calcium intensity was observed that was independent of the concentration for the SDF groups, even after acid etching, as well as the silver ions. Thus, we hypothesized that the alkaline property of the SDF associated with the residual mineral crystal of the tooth, in addition to an increase in calcium and silver ions, could promote the deposition of mineral on eroded dentin. However, it is worth mentioning that the time period required for SDF to remineralize dentin is not clear based on the available literature. Therefore, it is more plausible to hypothesized that, the increase of calcium mineral occurred due to the higher amount of calcium in the experimental treatment than related to remineralizing process in the eroded dentin. However, future studies should be conducted to evaluate this issue in the case of eroded dentin.

In addition to promoting dentin remineralization by increasing the mineral density and hardness [19], fluoride present in SDF inhibits matrix metalloproteinases, thereby slowing the degradation of the dentin matrix [39,40]. Additionally, the inhibitory effect of the SDF on the MMPs was reinforced by the high concentration of silver [20]. This is very important because new remineralization cores are created by the outermost collagen network with high calcium and phosphorous. Thus, notably, both complimentary mechanisms (remineralization on the surface and enzymatic inactivation) were associated with the presence of fluoride and silver particles and provided the best adhesive system performance in the eroded dentin without jeopardizing the adhesive polymerization in the resin-eroded dentin interface. Hence, the second null hypothesis was accepted.

However, when a higher concentration of SDF was used, a statistically significant increase in the mean μTBS was observed compared to the control groups. Regarding the effectiveness of SDF, the concentration of the fluoride and silver ions is very important. Silver diamine fluoride 38% contains 44,800 ppm F and 253,870 ppm Ag [41,42] compared to SDF 12% (14,150 ppm F and 80,170 ppm Ag) [22,43,44]. This higher concentration could account for the reduction in the erosion progression by promoting the high remineralization effect, as occurred in the caries lesion [42], resulting in a better performance of the adhesive systems.

Additionally, during the demineralized erosive challenge, a significant loss of mineral was accompanied by a loss in collagen periodicity [45], promoting higher porosity of the collagen matrix, similar to that which occurs in caries [46]. The loss of collagen periodicity in

demineralized dentin may be attributable to enzymatic activity [9] and it is likely that these spaces are occupied by water. Thus, one hypothesis is that the silver particles associated with higher mineral deposition resulting from a higher fluoride concentration, especially in the case of SDF 38%, better occupied the spaces filled with water and contributed to the establishment of a more resistant hybrid layer by increasing the adhesive diffusion [47], thereby preventing micro- and nanoleakage [48].

One of the most common adverse effect of SDF application is the resulting black staining, due to the silver ion deposition [49]. One of the alternatives suggested to minimize this side-effect is to applied a saturated potassium iodide (KI) solution, which can react with residual silver ions, to eliminate the staining effect [16,49]. However, it is not clear if the presence of potassium iodide may affect the bonding properties to dentin. The results of this study showed that the present of potassium iodide in the 38% SDF did not affect none of the properties evaluated when compared to 38% SDF without KI, indicating that the use of KI associated 38% SDF, as recommended by the manufacturer, can be applied in eroded-dentin.

No significant difference was observed for both adhesive systems independent of the adhesive strategy used when the universal adhesive systems were used with the eroded dentin, as previously reported [15,50,51]. According to Siqueira et al. [15], the presence of the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) in both universal adhesive systems resulted in a significant improvement in bonding to the eroded dentin. 10-MDP forms a stable nanolayer together with the deposition of salts of MDP calcium on the adhesive interface [52], increasing the mechanical strength [52,53] and protecting against hydrolysis [54].

However, considering that universal adhesives were developed to simplify both existing adhesive strategies (one-step SE and two-step ER adhesives), these allow the application of each according to the preference of the clinician or clinical demand [55,56]. The results of the present study confirm the applicability of this concept, provided both adhesive strategies exhibit similar results. However, notably, when applied as an etch-and-rinse strategy, it is necessary to use an added step (etching with phosphoric acid), which slightly complicates the procedure. Therefore, in terms of the evaluated universal adhesive, the preference of the clinician should be the self-etch strategy when applied to eroded dentin.

In conclusion, the application of SDF promoted an increase in the bond strength to eroded dentin and is a potential alternative in the restoration of teeth with exposed dentin due to erosion. Additional studies are necessary to determine whether the use of SDF preserves resin-eroded dentin interfaces after long-term water storage.

## 5. Conclusion

The application of silver diamine fluoride to universal adhesives in the etch-and-rinse and self-etch modes may be a viable alternative for increasing bond strength without jeopardizing the degree of conversion in eroded dentin bonding.

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## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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