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Original Article

# Influence of silver fluoride plus potassium iodine on adhesive properties of primary carious dentin-resin interface: An in vitro study

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ABSTRACT

This paper evaluated the influence of different protocols of silver fluoride (SF) pretreatment of artificial carious lesions on the adhesive interface of composite resin restorations and remineralization of deciduous dentin compared to silver diamine fluoride (SDF). *S*ixty-four deciduous molar teeth were randomly divided into 8 groups (n = 8) according to the restoration time (immediately – IM; 30 days after SDF/SF treatment – 30 D) and treatment before restoration (SDF 38 %; SDF 38 % + potassium iodide – KI; SF 38 %; SF 38 % +KI). After SDF/SF application, teeth in the IM group were restored with self-etch universal adhesive system/composite resin. Samples in the 30D groups were stored in artificial saliva (37 °C) for 30 days before receiving the same restoring protocol. Beams were obtained from all groups and subjected to bond strength tests (μTBS), ultrastructural qualitative analysis (FEG) and mineral analysis (SEM/EDX; Micro-Raman spectroscopy). The μTBS data were subjected to three-factor ANOVA and multiple comparisons (Holm-Sidak method). Bond strength values (MPa) for IM groups were 16.9 ± 2.7 (SDF); 17.6 ± 3.5 (SDF + KI); 16.8 ± 5.5 (SF); 18.4 ± 4.1 (SF + KI); and 14.9 ± 4.2 (SDF); 16.0 ± 5.4 (SDF + KI); 14.1 ± 3.6(SF); 16.4 ± 5.4 (SF + KI) for 30D groups. Bond strength wasn't influenced by the moment of restoration (IM or 30D); the use of KI didn't alter adhesion characteristics; SDF/SF solutions resulted in similar adhesive strength; calcium and phosphate expressions were identified at the interfaces on IM and 30D moments. However, 30D presented qualitative increase in these ions, compatible with remineralization. It was concluded that the adhesion of composite resin restorations in artificial caries lesions of deciduous teeth treated with SDF (38 %) and SF (38 %) had similar effects in vitro; the use of KI or the moment when restorations were accomplished did not influence the adhesion and all tested protocols promoted remineralization of carious dentin.

## 1. Introduction

Silver diamine fluoride (SDF) is a noninvasive cariostatic agent and different systematic reviews reported its effectiveness in inactivating dentin carious lesions in deciduous teeth (Gao et al., 2016, Chibinski et al., 2017, Jabin et al., 2020).

In pediatric dentistry, SDF is commonly used as an interim treatment to quickly halt the progression of caries lesions. However, there are situations where the lesions may reactivate after SDF treatment (Mei et al., 2020). It occurs due to factors such as the shape and the localization of the cavity in the mouth (Fung et al., 2018), which hinder biofilm control. Therefore, dental restoration after SDF application may be a biological necessity.

Previous studies have documented the influence of SDF on the adhesive interface of immediate restorations with glass ionomer cement (Fröhlich et al., 2020, Jiang et al., 2020, Uchil et al., 2020), and adhesive systems (Koizumi et al., 2016, Puwanawiroj et al., 2018). However, recently, the association between SDF and potassium iodide (KI) was introduced for clinical use (Riva Star, SDI) aiming to minimize SDF staining. A second version of this product, which eliminates ammonium from the cariostatic solution, composed of silver fluoride (SF), has also become available (Riva Star Aqua, SDI). Nevertheless, very little is

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known about the effect of these associations on adhesion of composite resin restorations, especially on deciduous teeth and an investigation about the influence of SF or SF plus KI is needed.

Therefore, the objective of this research is to investigate the influence of different protocols of silver fluoride (SF) pretreatment of artificial carious lesions on the adhesive interface of composite resin restorations and remineralization of deciduous dentin compared to SDF. This investigation was conducted in the context of the immediate restoration or restoration thirty days after the application of the product on deciduous teeth with artificial caries lesions. To the best of our knowledge, this is the first study to test this possibility.

## 2. Materials and methods

This study was approved by the Ethics and Research Committee of the State University of Ponta Grossa (# 4.424.292). It followed the CRIS guidelines (*Cheklist for Reporting In vitro Studies*) [17] and addressed the following research question: How does silver fluoride pretreatment influence the adhesive properties and the mineral content of carious primary dentin-composite resin interface compared to silver diamine fluoride?

### 2.1. Sample size calculation

The bond strength for Single Bond Universal in deciduous dentin was used for sample size calculation (https://www.sealedenvelope.com). According to the literature, the mean bond strength of Single Bond Universal is 19.5 ± 4.95 MPa (Sun et al., 2020). To detect a difference of 10 MPa among the test groups (Siqueira et al., 2020), with a significance level of 5 %, a power of 80 % and a two-sided test, the sample size required was 8 teeth per group.

### 2.2. Tooth selection and preparation

Sixty-four human deciduous second molars obtained from the human tooth bank of the State University of Ponta Grossa were used. Teeth with carious lesions, enamel development defects or previous restorations were excluded.

The teeth were disinfected (0.5 % chloramine) and stored in distilled water at 4 °C until use. The occlusal surfaces were cut (Isomet 1000, Buehler Ltd., Illinois, USA) under irrigation obtaining dentin plane surfaces, which were polished with silicon carbide sandpaper (#600; 60 s) to standardize the smear layer. Subsequently, teeth were cleaned in ultrasonic bath for 2 min and stored in distilled water.

### 2.3. Artificial caries induction

Artificial carious lesion induction using pH cycling was performed using two solutions (Lenzi et al., 2015): (1) demineralizing solution (2.2 mm $CaCl_2$, 2.2 mm $NaH_2PO_2$, 50 mm acetic acid - adjusted pH 4.8), and (2) remineralizing solution (1.5 mm $CaCl_2$, 0.9 mm $NaH_2PO_4$, 0.15 mm Kcal - adjusted pH 7.0). Teeth were placed in individual containers and sealed for the demineralization-remineralization (DE-RE) process. A 2 ml demineralizing solution (pH 4.8) was added to each container and kept in contact with the teeth for 8 h. Afterward, the teeth were washed with distilled water, and the 2 ml of remineralizing solution (pH 7.0) was added, after which remained in contact with the teeth for 16 h.

For each cycle, the DE/RE solutions were changed, and the teeth were washed with distilled water. The caries induction protocol lasted for 14 days, starting with the demineralizing solution and ending with the remineralizing one. Finally, the teeth were cleaned in an ultrasonic bath (30 s), and stored in distilled water (4 °C) until the application of SDF or SF.

The pH-cycling method simulated a substrate that resembles affected caries dentine layer, resulting in a moderate demineralization of intertubular dentine and extensive opening of the dentinal tubules (Marquezan et al., 2009).

### 2.4. Experimental design

After artificial caries induction, the teeth were randomly divided into eight groups (n = 8) (Fig. 1) according to the different protocols for SDF application and restoration times (IM and 30D). The teeth were numbered from 1 to 64 and randomized using a computer-generated numbers table in blocks of four (sealedenvelop.com). The study groups formed were: 38 % SDF (IM and 30D), 38 % SDF + KI (IM and 30D), 38 % a SF (IM and 30D), and 38 % SF + KI (IM and 30D). After SDF or SF application, teeth from IM groups were restored. The teeth in the 30D groups were stored in artificial saliva[1] (12.9 mm Kcal, 1.9 mm KSCN, 2.4 mm $Na_2SO_4.10\,H_2O$, 3.3 mm $NH_4Cl$, 1.5 mm $CaCl_2{\cdot}2H_2O$, 7.5 mm $NaHCO_3$, 0.02 mm $ZnCl_2$, pH 7.4) at 37 °C for 30 days, and subsequently restored.

### 2.5. Application of silver fluoride solutions

The SDF and SF solutions are commercially presented in two opaque flasks: one containing 38 % SDF or 38 % SF (step 1) and the other containing a concentrated KI solution (step 2). In the groups of teeth treated with 38 % SDF/SF + KI, steps 1 and 2 were used; in the groups treated without KI, step 2 was not used. SDF and SF solutions were applied in all groups without prior removal of carious tissue. The product compositions and application modes are detailed in Table 1.

### 2.6. Restorative procedures

The restorations were performed at two times: immediately (IM) and 30 days (30D) after the SDF/SF application. Regardless of when the restoration procedure was performed, the protocol/materials used were the same for all groups (Table 1).

The restorations were carried out using Single Bond Universal adhesive system (3 M ESPE, MN, USA) and the composite resin Filtek Z-250 XT™ (3 M ESPE, MN, USA). The composite resin was applied in three increments of 2 mm each, photoactivated for 40 s at 1000 mW/cm$^2$, with the LED curing light (VALO, Ultradent Products, UT, USA). A single operator performed all the restorative procedures and prepared the beams.

After 20 h of storage in a dark container with deionized water (37 °C), the teeth were taken to a cutting machine (Isomet 1000, Buehler Ltd., Illinois, USA). Using a diamond disk (15 HC-4in, Buehler Ltd., Illinois, USA), under irrigation at 300 rpm, the teeth were cut perpendicularly in the 'x' and 'y' axes, and beams were obtained with a cross-section of 0.8 ± 0.1 mm$^2$. The cross-sections were measured with a digital for micro tensile calculation (Digimatic Caliper, Mitutoyo, Kanagawa, Tokyo, Japan). The number of beams prematurely lost was also recorded.

For each study group, eight teeth were prepared (n = 8). In each experimental group, two beams were randomly separated for microscopic ultrastructural qualitative analysis via FEG and EDS, and other two beams for mineral analysis via micro-Raman spectroscopy, while the remaining beams were reserved for micro tensile bond strength test. The test was performed by a single, trained operator, who was blinded to the study group and did not participate in other phases of the study.

### 2.7. Microtensile bond strength test

Each beam was affixed to Geraldelli's device with gel cyanoacrylate glue, which was attached to the universal test machine (Kratos Dinamômetros, SP, Brazil), ensuring that the tensions occurred perpendicularly to the bond interface, at a rate of 0.5 mm/min, resulting in the bond strength values in MPa. The test was carried out 24 h after the completion of the restorations in groups IM and 30D. Using a stereoscopic magnifier at 100x magnification (Olympus SZ40, Shinjuku-ku,

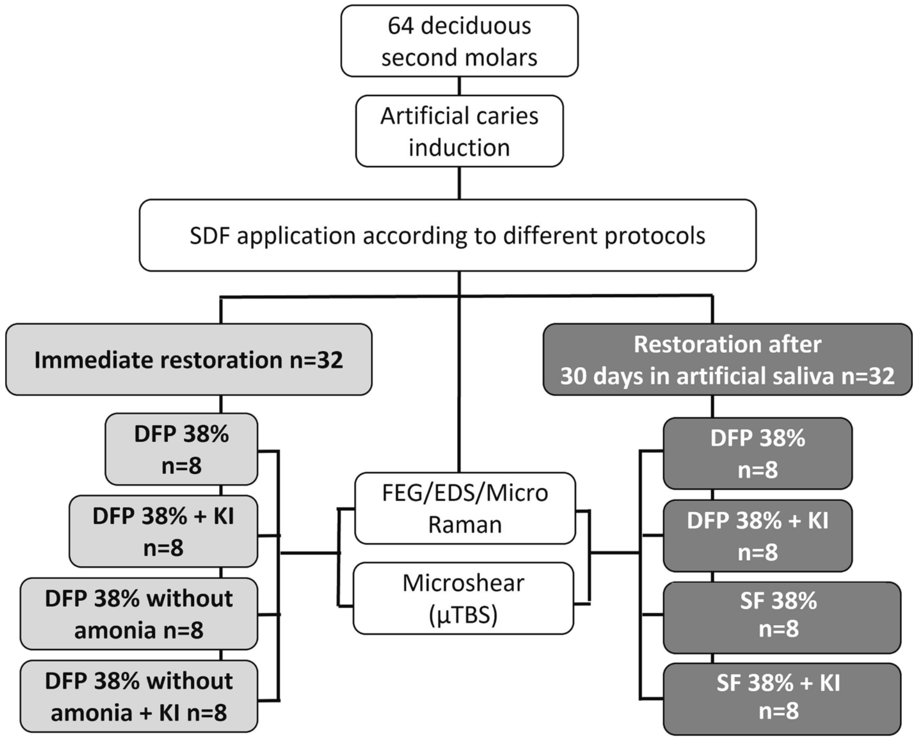


**Fig. 1.** Experimental design flow chart.

Tokyo, Japan), the fracture mode of each stick was classified according to its type as follows: cohesive in dentin (CD); cohesive in composite resin (CR); adhesive/mixed (A/M) or premature fracture.

### *2.8. FEG/EDX analysis*

Two beams from each study group were used for these analyses. To fix the organic material present in the dentin, the beams were immersed in the fixing substance (225 ml 0.1 mol/ L-1 Tris + 25 ml glutaraldehyde 25 %) for 12 h at 4° C. Subsequently, they were immersed in the 0.2 mol/ L Tris solution for 20 min, and washed with distilled water three times. Then, they were dehydrated with alcohols at ascending concentrations: 25 % (20 min), 50 % (20 min), 75 % (20 min), 95 % (30 min), 100 % (60 min). The final dehydration step was carried out by placing the samples in contact with colloidal silica for 24 h. After the fixation and dehydration, the beams were glued to the stubs using a cyanoacrylate-based glue, and metalized in 33 carbon/gold (MED 010, Balzers Union Balzers, Liechtenstein).

A 15 mm field emission scanning electronic microscope (VEGA SB; Tescan, Warrendale, PA, USA) was used in retro-scattered electron mode. Photomicrographs were obtained after visualizing the samples at 1000x magnification, employing 15 kV acceleration voltages. Three photomicrographs were taken for each sample along the adhesion interface. Additionally, the samples were subjected to semiquantitative chemical microanalysis, evaluating the mineral content of calcium, phosphorus and silver with EDX.

### *2.9. Micro-Raman spectroscopy*

The beams were subjected micro-Raman spectroscopy to verify the mineral composition. After being polished and washed in ultrasonic bath, two samples from each group were separated and analyzed. With a spectrophotometer (BruckerOptik GmBH, Ettlingen, Baden-Wurttenberg, Germany), the phosphate (961 $cm^{-1}$) and carbonate (1072 $cm^{-1}$) spectra at the adhesive interface were analyzed at three random sites per beam. Before the analysis, the equipment was calibrated to redefine zero. The parameters used were: 4 $cm^{-1}$ spectral resolution, 7 mm opening for the laser beam with a 1064 nm focused line. The power used was 0.2 Mw, with 5 co-additions.

### *2.10. Data analysis*

The results obtained from the bond strength test underwent normality verification (Kolmogorov-Smirnov test) and were analyzed using the 3-factor ANOVA test and Tukey's post-test, considering the following variables: material (SDF or SF), presence of KI (with or without application) and time (IM and 30D), with a pre-set statistical significance level of 0.05. Next, multiple comparisons were performed using the Holm-Sidak method to analyze specific factors. The software used was SigmaPlot for Windows version 12.0. The ultrastructural and mineral analyses of the samples are presented with a qualitative description of the data observed.

## 3. Results

### *3.1. Microtensile bond strength*

Approximately 10 to 12 composite resin-dentin beams were obtained per tooth, including those that fractured prematurely. The fracture patterns are shown in Table 2. In all the groups, adhesive/mixed fracture prevailed, with a greater percentage occurring in the groups tested after 30 days. The highest premature fracture indices occurred in the SDF groups after 30D, and SF + KI in the immediate time.

The inferential statistical analysis did not reveal significant interactions between factors ($p = 0.943$) (Table 3), which ruled out comparisons between groups. Therefore, multiple comparisons were carried out. Differences were observed in the KI and restoration time factors. Higher bond strength values were observed with the application of KI ($p = 0.0001$) and in restorations performed at IM period ($p < 0.0001$). The absence of ammonium in the SF solution did not affect adhesion (Table 4).

**Table 1**
Composition, presentation and mode of application of the SDF solutions used.

| Product | Lot | | Composition | Application |
|---|---|---|---|---|
| **Riva Star** (SDI, Bayswater, Victoria, Australia) | 200821 | **Flask 1** | Hydrofluoric acid, silver nitrate, ammonium hydroxide, and deionized water | 1. Flask agitation to homogenize the solution;<br>2. Tooth surface drying;<br>3. SDF application with active microbrush for 1 min;<br>4. Surface washing for 30 s[39] |
| | 200821 | **Flask 2** | KI supersaturated solution | 1. Flask agitation to homogenize the KI solution;<br>2. Application with active microbrush, with 3 replications, until the solution stopped presenting a white-milkish coloration;<br>3. Surface washing for 30 s[39] |
| **Riva Star Aqua** (SDI, Bayswater, Victoria, Australia) | 20081204 | **Flask 1** | Hydrofluoric acid, silver nitrate, and deionized water | 1. Flask agitation to homogenize the solution<br>2. Tooth surface drying;<br>3. SDF application with active microbrush for 1 min;<br>4. Surface washing for 30 s[39] |
| | 20081204 | **Flask 2** | KI supersaturated solution | 1. Flask agitation to homogenize the KI solution;<br>2. Application with active microbrush, with 3 replications, until the solution stopped presenting a white-milkish coloration;<br>3. Surface washing for 30 s[39] |
| Single Bond Universal- 3M ESPE; St Paul, MN, USA | 2035300661 | -hydroxyethyl Methacrylate2, Bisphenol A diglycidyl ether dimethacrylate (BisGMA), Decamethylene dimethacrylate, ethanol, silane treated silica, water, 1,10-Decanodyol phosphate methacrylate, acrylic copolymer, and itaconic acid, Camphorquinone, N, N-Dimethylbenzocaine | 1. Adhesive active application all over the surface with a microbrush for 20 s<br>2. Gentle air jet for 5 s until the adhesive no longer moves.<br>3. Light curing for 10 s at 1200Mw/cm | Single Bond Universal- 3 M ESPE; St Paul, MN, USA |
| Composite resin Filtek Z250 XT- 3M ESPE; St Paul, MN, USA | 1829800312 | Silane treated ceramic, bisphenol A diglycidyl ether dimethacrylate (BIS-GMA), bisphenol A polyethylene glycol diether dimethacrylate (BIS-EMA), silane treated silica, diurethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEG-DMA), polyethylene glycol 600 dimethacrylate (PEGDMA) | 1. 2 mm increments (3)<br>2. Photoactivation for 40 s at 1000 mW/cm$^2$. | Composite resin Filtek Z250 XT- 3 M ESPE; St Paul, MN, USA |

**Table 2**
Absolute and relative frequencies of beams' fracture patterns according to SDF application protocol and moment of the restoration.

| SDF Protocol | Moment of restoration | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Immediate | | | | | After 30 days | | | | |
| | A/M | CD | CR | PF | Total | A/M | CD | CR | PF | Total |
| SDF | 72(87.8 %) | 3(3.9 %) | 2(2.4 %) | 5(6.5 %) | 82 | 77(92.3 %) | 0 (0.0 %) | 0 (0.0 %) | 6 (7.2 %) | 83 |
| SDF + KI | 69(81.2 %) | 7(9 %) | 5(5.9 %) | 4(5.1 %) | 85 | 78(91.8 %) | 0 (0.0 %) | 2(2.4 %) | 5(5.9 %) | 85 |
| SF | 74 %(89.2 %) | 5(6.4 %) | 0 (0.0 %) | 4(5.1 %) | 83 | 78 %(94 %) | 0 (0.0 %) | 0 (0.0 %) | 5(6 %) | 83 |
| SF + KI | 73(84.9 %) | 7(9.1 %) | 0 (0.0 %) | 6(7.8 %%) | 86 | 77 %(90.6 %) | 1(1.2 %) | 2(2.4 %) | 5(5.9 %) | 85 |

* A/M (adhesive/mixed), CD (cohesive in dentin), CR (cohesive in resin), and PF (premature fracture).

**Table 3**
Bond strength values (MPa) according to SDF application protocols and moment of restoration.

| SDF Protocol | Moment of restoration | |
|---|---|---|
| | **Immediate** | **After 30 days** |
| SDF | 16.9 ± 2.7 | 14.9 ± 4.2 |
| SDF + KI | 17.6 ± 3.5 | 16.0 ± 5.4 |
| SF | 16.8 ± 5.5 | 14.1 ± 3.6 |
| SF + KI | 18.4 ± 4.1 | 16.4 ± 5.4 |

### 3.2. FEG/EDX analysis

The SEM images of the dentin bonding showed that the adhesive layers of the groups treated with different protocols maintained a constant thickness, regardless of the SDF/SF protocol and the restoration time. All groups exhibited silver deposition on the dentin and the application of KI seemed to intensify the presence of silver on the hybrid layer and dentin, at both moments (IM and 30D); the slightest presence of silver deposits were observed on the specimens treated with SF (Fig. 2).

All groups increased the intensity of phosphorus (P) and calcium (Ca) peaks in dentin after 30 days. Silver was observed in the SDF and SF groups, irrespective of the moment when the restorative procedure was accomplished. However, the silver peak intensity was most evident and

**Table 4**
Bond strength values (MPa) according to the different factors.

| Factors | | | Mean differences (IC 95 %) | p value |
|---|---|---|---|---|
| Material | SDF | 16.37 ± 4.09 | −0.04 (-0.96 a 0.88) | $p > 0.05$ |
| | SF | 16.40 ± 4.95 | | |
| KI | with application | 17.65 ± 4.70 | 1.98 (-0.31 a 4.27) | $p = 0.001$ |
| | without application | 15.67 ± 4.29 | | |
| Restoration time | Immediate | 17.41 ± 4.12 | 2.1 (1.20 a 3.00) | $p < 0.001$ |
| | After 30 days | 15.31 ± 4.75 | | |

stable in the groups where the KI solution was applied (Fig. 3).

EDX was also used to map the distribution of the mineral elements on the surface of the specimens at the adhesive interface. This qualitative analysis revealed the presence of silver (shown in green in Fig. 4) on the adhesive interface in all the study groups. However, a slight reduction in silver was observed in the hybrid layer when restoration was carried out after 30 days, irrespective of the SDF/SF protocol used. Calcium (shown in purple in Fig. 4) and phosphorus (shown in yellow in Fig. 4) exhibited increased expression in the 30D groups, regardless of the SDF protocol applied (Fig. 4).

### *3.3. Micro-Raman spectroscopy*

These spectra were obtained from the chemical profiles of all the experimental groups. The most intense peak, observed at 961 $cm^{-1}$ was ascribed to the dentin phosphate ($PO4^{-3}$) (red arrows). The groups restored after 30 days exhibited phosphate peaks with greater intensity and width than did those from the immediate groups. The SF + KI (30D) and the SDF + KI (30D) groups had the highest phosphate peaks, respectively (Fig. 5).

Another mineral peak characteristic of dentin is ascribed to carbonate ($CO3^{-2}$) at 1072 $cm^{-1}$ (green arrows). The most evident and intense carbonate peak was observed in the SF + KI (30D) group (Fig. 5).

## 4. Discussion

This study demonstrated that the use of SF on artificial carious lesions resulted in adhesive patterns to deciduous demineralized dentin similar to those observed when using SDF, irrespective of its association with KI. Such results were observed in the restorations carried out in the same session or 30 days after SDF/SF application; evidence of remineralization of the carious dentin was also observed. To date, only one clinical study has compared SDF and SF in deciduous teeth, and no differences in the rates of arrested carious lesions has been found (Turton et al., 2020). Therefore, if further studies confirm these findings, the SF solution might become the first choice for the treatment of pediatric patients. The main advantage of using ammonium-free solutions is that this product does not have the potential to cause mucosa ulcerations and has a milder odor.

Regarding KI use, it seems to minimize staining after SDF application (Garg et al., 2019, Siqueira et al., 2020, Haiat et al., 2021), but the results regarding adhesion are conflicting and the data refer only to permanent dentin. Some studies have demonstrated that the dentin pretreatment with SDF + KI did not affect the bond strength (Selvaraj et al., 2016, Siqueira et al., 2020); others showed the opposite (Kassebaum et al., 2017, Cifuentes-Jimenez et al., 2021). In deciduous dentin, the available studies have analyzed bond strength to GIC (Puwanawiroj et al., 2018, Turton et al., 2020), and the use of KI did not interfere on it.

In our study, higher bond strength values were observed when the restorations were carried out immediately after SF/SDF associated with

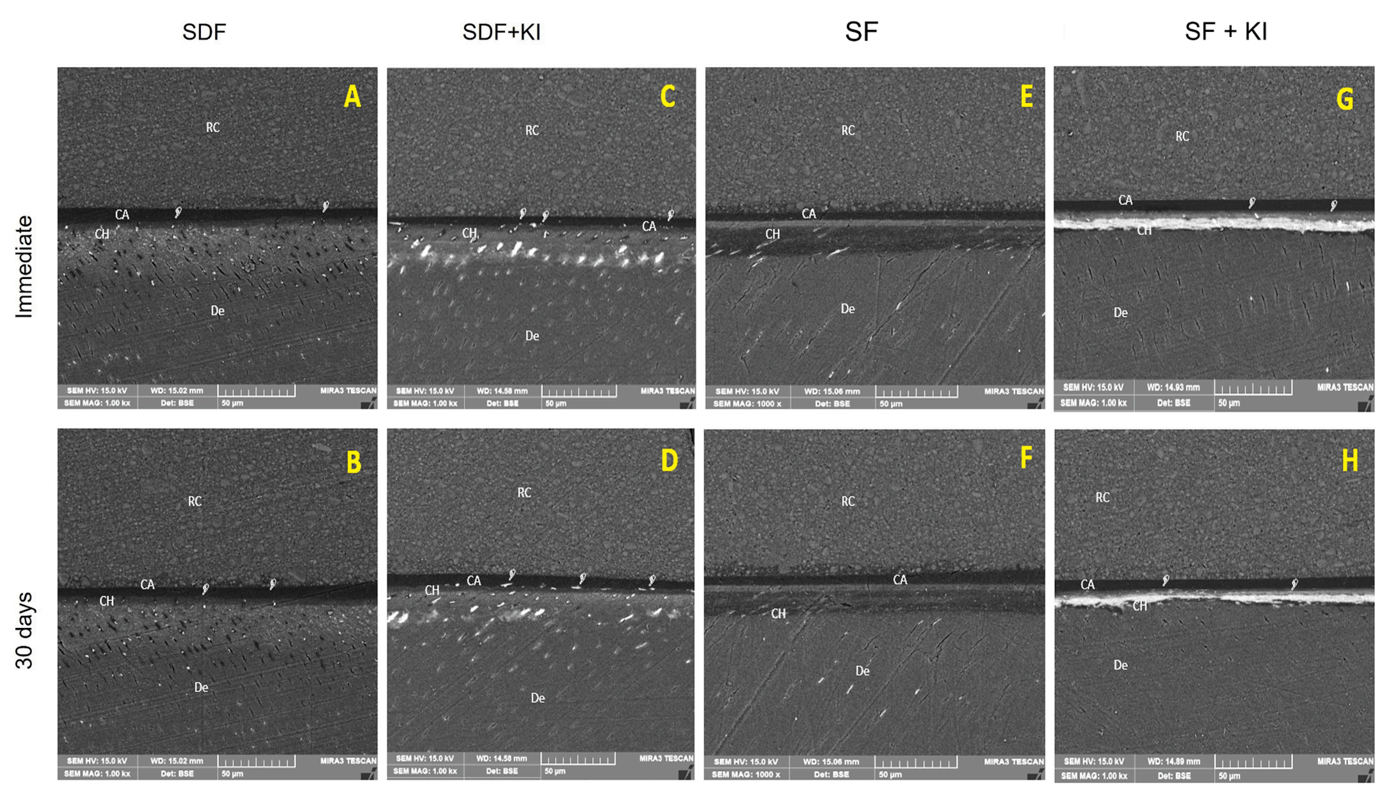


**Fig. 2.** FEG images of the denting bond interface in each experimental condition. The association SDF + KI resulted in greater silver deposit in the hybrid layer and dentin (hands in C, D, G, and H), but the presence of silver was also observed with the use of SDF alone (hands in A and B). (RE = resin; CH = hybrid layer; DE = dentin; CA = adhesive layer).

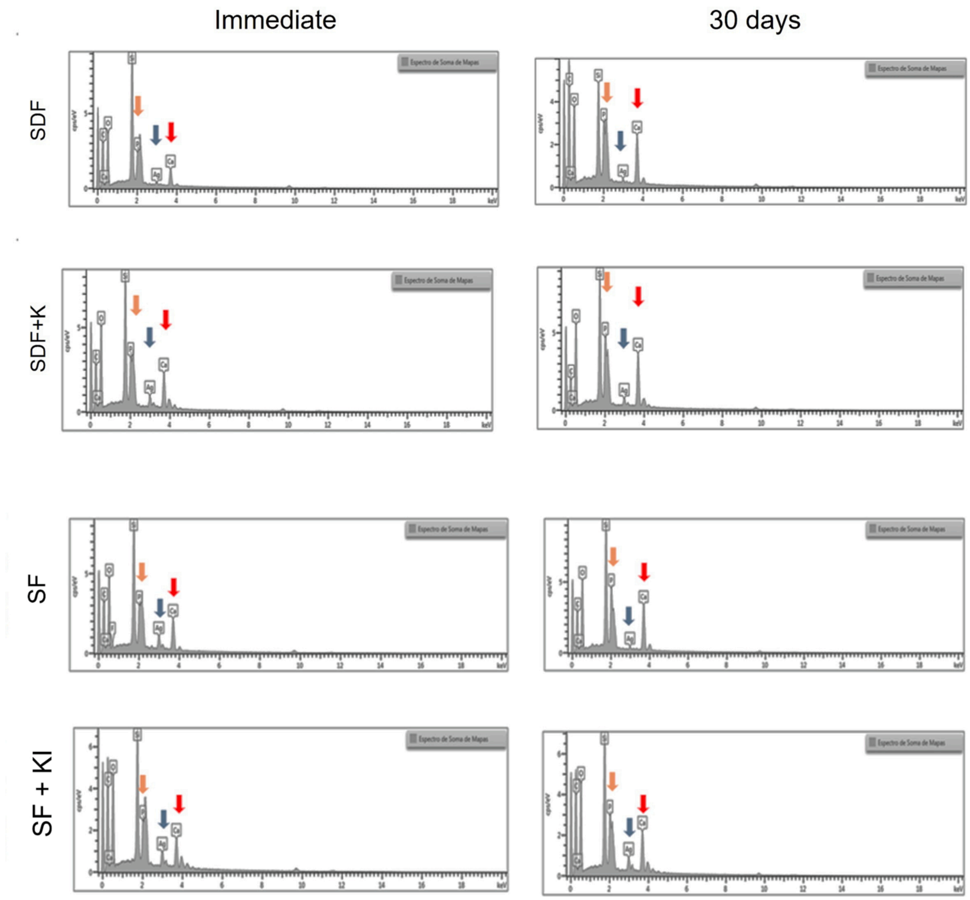


**Fig. 3.** Element distribution analysis using EDX according to SDF application protocols and moment of restoration (calcium = purple arrow; silver = green arrow; phosphorus = yellow arrow).

KI application. However, the numeric difference between the protocols with and without KI was between 1 and 2 MPa. The discussion should analyze whether this small increase, even being statistically significant, would justify adding another step in the SF/SDF application protocol in children. Therefore, considering the small variation in adhesion values obtained and previous findings in the literature, we believe that the different protocols tested might yield similar results over time. However, this statement must be confirmed, both in in vitro research, which is already being sought by our research team, and in randomized clinical trials, so that the clinical significance of different protocols can be better understood.

There are no studies in the literature analyzing the moment when the composite resin restoration is carried out, consequently the literature offers no data to compare with our results. The bond strength values obtained were greater when the restoration was carried out in the same session after SDF/SF application. The numeric difference was also small (±2 MPa) and it is not likely to result in a significant clinical effect, but this fact also needs to be confirmed by further research.

One characteristic of the adhesive interface when SDF/SF is used is the presence of silver deposits in the hybrid layer and subjacent dentin, as verified in the FEG images. The presence of silver was documented in cavities of deciduous dentin carious lesions after SDF application (Li et al., 2019). Since silver has an inhibitory effect on the collagen matrix degradation due to the inhibition of metalloproteinases (MMPs) (Mei et al., 2013), the longevity of the adhesive interface may be improved. Although silver deposits were present in all study groups, they were more evident in the SF/SDF + KI groups at both restoration times (IM and 30D). The reaction that occurs in the tooth after SDF/SF + KI application results in the formation of tripotassium phosphate and silver iodide (AgI), a subproduct that is very stable, insoluble in water and acids, and has strong antimicrobial action. We hypothesized that AgI was the compound detected in the samples treated with SDF + KI.

Micro-Raman spectroscopy revealed that the phosphate peaks in the 30D groups were more intense than those in the IM groups, and that the carbonate peaks remained constant. The phosphate spectral position classifies it as a crystal, while its peak width represents the degree of mineral crystallinity (Hass et al., 2021). In other words, increased height and width of phosphate peak indicates remineralized samples.

In fact, our FEG/EDX results confirmed these findings, revealing higher mineral levels in 30D groups, especially those associated with KI. The SDF/SF solutions provided an alkaline environment that promoted physicochemical interactions between the phosphate groups and the dentin collagen (Mei et al., 2014). Once the phosphate groups bond to the collagen, they contribute to the formation of calcium ion bonding sites, facilitating mineral nucleation (Ng et al., 2020) and dentin mineralization, as verified in this study using two distinct methods (micro-Raman spectroscopy and EDX).

Considering that the samples were stored in artificial saliva for 30 days before the restorative procedure, we could assume that the increased expression of these ions was related to the incorporation of artificial saliva ions. During this 30-day period, no cariogenic challenge or pH drop occurred in the medium, which may be considered one limitation of our study. However, the ability of SDF/SF to remineralize carious dentin has been widely studied. In a clinical setting, after the pH cycling, the existing mineral residue might favor heterogeneous integration of crystals and act as a reservoir of calcium and phosphorus ions, which promote the precipitation of other crystalline phases on the dentin surface (Zhao et al., 2018, Zhao et al., 2020).

Another limitation is the fact that our specimens were deciduous dentin with artificially induced carious lesions, and lacked the

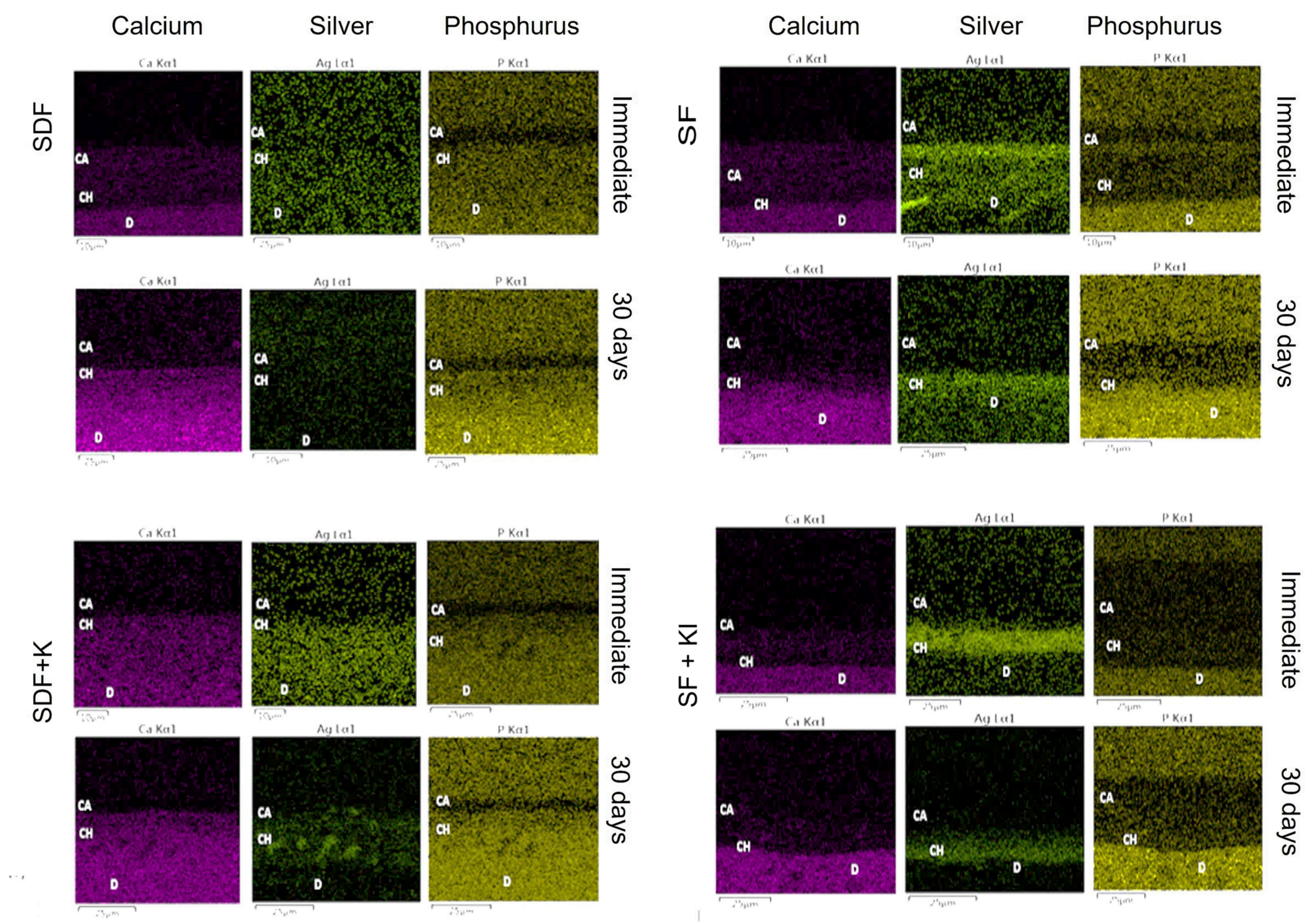


**Fig. 4.** EDX analysis of the calcium, phosphorus and silver ions distribution in adhesive interface according to SDF application protocols and moment of restoration.

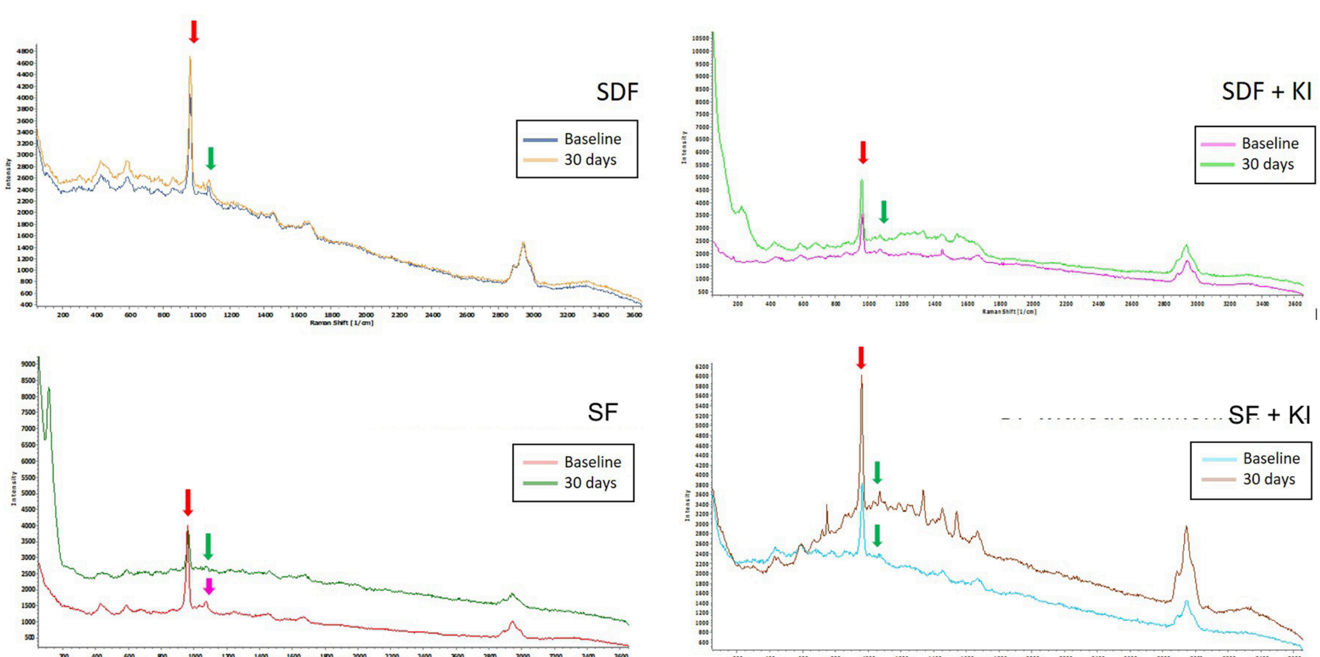


**Fig. 5.** Micro-Raman spectroscopy of experimental groups according to the moment of the restoration. Red arrows represent the phosphate peaks; green arrows the carbonate peaks.

microbiological component. However, this is a validated method that has been widely used in in vitro research, and it mimics the selective removal of carious tissue (Marquezan et al., 2009), offering greater standardization than the use of natural carious lesions. Also, it must be noted that we still do not have long term data and all the comments reflected our observations without the aging of the adhesive interface. On the other hand, the use of different methodologies to corroborate the findings is a strength of our study, that associate qualitative and quantitative findings to analyze the bonding/dentin interface.

The data shown in this study, even though they were being obtained in laboratory, point out some paths. The bond strength of the restorative procedure and the remineralization of the carious dentin were not affected by the KI use or by the presence or absence of ammonium in the cariostatic solution. Therefore, if the staining becomes a relevant issue for parents/patients, the use of KI may improve this, with some additional time being added to the protocol. Also, we showed that SF can be used to prevent mucosa ulcerations. The final decision depends on the professional's scientifically based option after discussing the possibilities of treatments with the parents. However, this alternative must be tested in a clinical setting, where all the different variables that interfere with restoration longevity will be in action before any new clinical protocol is adopted.

Taken together, these findings led us to conclude that the adhesion of composite resin restorations to artificial caries lesions on deciduous teeth treated with SDF (38 %) or SF (38 %) had similar performances in vitro; the use of KI or the moment when the restorations were completed did not influence the adhesion and all the tested protocols promoted the remineralization of carious dentin.

## 5. Ethical approval.

A document from the Ethical Committee was included in the submission site.

## 6. Authors' contributions

N.L. and V.M wrote the main manuscript text. N.L. and V.M prepared the samples and ran the tests. N.L.; C.M.C.L.; A.F.M.C. and A.C.R.C performed the statistical analysis. N.L.; A.F.M.C. and A. C. R. C. interpreted the data. N.L.; C.M.C.L. and A.C.R.C wrote the manuscript. All authors revised the manuscript.

## 7. Funding.

No funding was related to this research.

## 8. Availability of data and materials

Any datasets used will be provided by the corresponding author upon request.

## 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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