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## NANO-SILVER FLUORIDE VERSUS SDF/KI ON CARIES-AFFECTED DENTIN: BOND STRENGTH OF A UNIVERSAL ADHESIVE AND A GLASS-HYBRID RESTORATIVE, AND COLOR CHANGE

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| ARTICLE HISTORY                                                                                                                                                                                                                                                                                                                              | ABSTRACT                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |
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| <p>Received on: 23-07-2026<br/>Revised on: 14-08-2026<br/>Accepted on: 19-09-2026</p> <p><b>Keywords:</b> Nano-silver fluoride; silver diamine fluoride; potassium iodide; caries-affected dentin; universal adhesive; glass hybrid; micro-shear bond strength; CIEDE2000.</p> <p><b>*CORRESPONDING AUTHOR</b><br/>Ali Hamzah Al-Hashimi</p> | <p>Silver diamine fluoride (SDF) arrests caries but blackens dentin and may weaken adhesion; potassium iodide (KI) only partly prevents the stain. Nano-silver fluoride (NSF) has been proposed as a non-staining alternative, yet little is known about how it affects bonding to caries-affected dentin (CAD). This in vitro study compared SDF/KI and NSF pretreatment of CAD in terms of micro-shear bond strength (<math>\mu</math>SBS) of a universal adhesive with resin composite (UA) and a glass-hybrid restorative (GH), and of color change. NSF was characterized by XRD, FTIR and FESEM. Dentin discs from human molars were left sound (SD) or pH-cycled for 14 days to produce CAD, which was left untreated or treated with SDF/KI or NSF (<math>n = 10</math> per subgroup). Micro-cylinders of UA or GH were tested after 24 h or after 10,000 thermal cycles, and the color change (<math>\Delta E_{00}</math>) of treated CAD discs was followed for four weeks. Pretreatment, material, aging and their two-way interactions were all significant (three-way ANOVA, <math>p \leq 0.013</math>). With UA at 24 h, NSF (<math>17.68 \pm 2.44</math> MPa) did not differ from untreated CAD (<math>16.00 \pm 2.82</math> MPa), whereas SDF/KI was lower (<math>13.67 \pm 2.53</math> MPa; <math>p = 0.024</math> versus NSF). With GH, the three CAD pretreatments did not differ. Thermocycling reduced all bonds by 10–25%. <math>\Delta E_{00}</math> of SDF/KI rose from 3.70 to 12.94 over four weeks, far beyond the acceptability threshold of 1.8, while NSF stayed between 1.59 and 2.81. NSF therefore preserves bonding to CAD and avoids the progressive darkening seen with SDF/KI.</p> |

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### 1. INTRODUCTION

Selective caries removal, which deliberately leaves caries-affected dentin (CAD) over the pulp, is now recommended for deep lesions because it lowers the risk of pulp exposure [1], as is the conservative management of early childhood caries [2]. This leaves a difficult bonding substrate: CAD is hypomineralized and porous, its tubules are partly blocked by acid-resistant mineral and part of its collagen is denatured, and bond strengths of universal adhesives to CAD are consistently below those to sound dentin, whatever the etching mode [3,4].

Silver diamine fluoride (SDF, 38%) has become a routine agent for arresting such lesions. Silver ions kill cariogenic bacteria and precipitate as silver phosphate and metallic silver in the lesion, fluoride favors less clear: the few available studies deal with sound dentin or glass-ionomer cements on primary teeth [16–18], and none has compared NSF with SDF/KI on CAD using a universal adhesive and a glass hybrid under the same aging

fluorapatite formation, and both inhibit collagenolytic enzymes [5]. Two drawbacks limit its use under restorations. The first is the black stain, which potassium iodide (KI) reduces only partly and only for days to weeks [6–8]. The second is an uncertain effect on adhesion: systematic reviews found no loss of bond strength in some studies and a clear reduction in others, rinsing and the type of restorative being the main sources of heterogeneity [9,10]. Nano-silver fluoride (NSF), a colloid of chitosan-capped silver nanoparticles with sodium fluoride, arrests caries in children at least as well as SDF [11–13] and, because its silver is carried as stabilized metallic particles rather than free ions, causes little or no discoloration [14,15]. Whether NSF also spares the bond to CAD is much

protocol. This study therefore compared NSF and SDF/KI pretreatment of artificial CAD in terms of (i) the micro-shear bond strength ( $\mu$ SBS) and failure mode of a universal adhesive with composite and of a glass hybrid, before and after 10,000 thermal cycles, and (ii) the color change of treated dentin over four weeks. The null hypotheses were that pretreatment would not affect  $\mu$ SBS and that NSF and SDF/KI would not differ in color change.

## 2. MATERIALS AND METHODS

### 2.1. Study design

The bond-strength part followed a  $4 \times 2 \times 2$  factorial design: dentin condition or pretreatment (SD, CAD, SDF/KI, NSF)  $\times$  restorative protocol (UA, GH)  $\times$  aging (24 h, thermocycling), with ten discs per subgroup (160 specimens). A separate set of 30 CAD discs served the color study (control, SDF/KI, NSF;  $n = 10$ ). Figure 1 shows the workflow and Table 1 the materials.

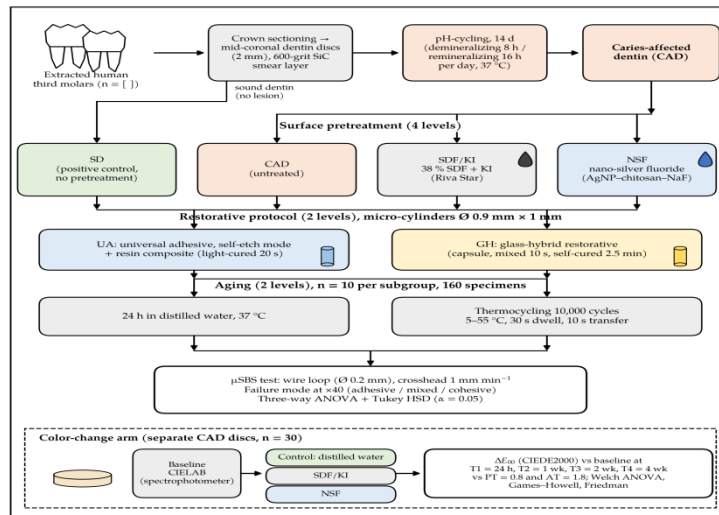


Figure 01: Experimental workflow of the bond-strength arm (upper part) and the color-change arm (dashed box). SD, sound dentin; CAD, caries-affected dentin; SDF/KI, silver diamine fluoride followed by potassium iodide; NSF, nano-silver fluoride; UA, universal adhesive with resin composite; GH, glass-hybrid restorative; TC, thermocycling.

Table 01: Materials used in the study, their main composition and application protocol.

| Material (code)                                     | Product and manufacturer                                                                   | Main composition                                                                                                                                | Application protocol                                                                                                        |
|-----------------------------------------------------|--------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------|
| Silver diamine fluoride + potassium iodide (SDF/KI) | Riva Star, SDI, Bayswater, Australia; lot B2301121                                         | Step 1: 38% w/v $\text{Ag}(\text{NH}_3)_2\text{F}$ (silver $\approx 24\text{--}27\%$ , fluoride $\approx 5\%$ ); Step 2: saturated KI solution  | Step 1 rubbed for 60 s with a microbrush; Step 2 applied until the creamy-white precipitate clears; rinsed 30 s; blot-dried |
| Nano-silver fluoride (NSF)                          | In-house synthesis (Section 2.4)                                                           | Chitosan-capped silver nanoparticles (600 ppm Ag), NaF (44,800 ppm F), chitosan 0.2% w/v                                                        | Two drops rubbed for 60 s, left for 2 min; rinsed 30 s; blot-dried                                                          |
| Universal adhesive (UA)                             | Single Bond Universal, 3M ESPE, St Paul, MN, USA; lot 7910540                              | 10-MDP, HEMA, Bis-GMA, dimethacrylate resins, methacrylate-modified polyalkenoic acid copolymer, silane, ethanol, water, initiators             | Self-etch mode: rubbed 20 s, air-thinned 5 s, light-cured 10 s                                                              |
| Resin composite                                     | Filtek Z350 XT, shade A2, 3M ESPE; lot NA52370                                             | Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA; zirconia/silica nanofillers (78.5 wt%)                                                                  | Packed into the tube; light-cured 20 s                                                                                      |
| Glass-hybrid restorative (GH)                       | EQUIA Forte HT Fil + EQUIA Forte Coat, GC Corporation, Tokyo, Japan; lot 2301121 / 2301204 | Fluoro-alumino-silicate glass, high-reactivity glass, polyacrylic acid, iron oxide; coat: methyl methacrylate, colloidal silica, photoinitiator | Conditioner 10 s, rinse, blot dry; capsule mixed 10 s, injected; self-cured 2.5 min; coat light-cured 20 s                  |
| Dentin conditioner                                  | Cavity Conditioner, GC Corporation; lot 2210083                                            | 20% polyacrylic acid, 3% aluminium chloride                                                                                                     | Applied 10 s, rinsed, blot-dried                                                                                            |

### 2.2. Tooth collection and dentin discs

80 intact human third molars, extracted for surgical reasons, were collected after approval by the Research Ethics Committee, College of Dentistry, University of Baghdad, approval no. 648/2024 and with written informed consent,

cleaned, examined for defects and stored in 0.5% chloramine-T at 4 °C for no longer than three months. Occlusal enamel and roots were removed with a low-speed diamond saw under water cooling (Isomet 4000, Buehler, Lake Bluff, IL, USA), leaving mid-coronal dentin discs  $2.0 \pm 0.1$  mm thick; discs with pulp-horn exposure or residual enamel were discarded. The discs were embedded in acrylic resin in PVC rings and wet-ground with 600-grit silicon carbide paper for 60 s to standardize the smear layer.

### 2.3. Artificial caries-affected dentin

Discs assigned to CAD were pH-cycled for 14 days at 37 °C: 8 h in a demineralizing solution (2.2 mM  $\text{CaCl}_2$ , 2.2 mM  $\text{NaH}_2\text{PO}_4$ , 0.05 M acetic acid, pH 4.4) followed by 16 h in a remineralizing solution (1.5 mM  $\text{CaCl}_2$ , 0.9 mM  $\text{NaH}_2\text{PO}_4$ , 0.15 M KCl, pH 7.0), with both solutions renewed daily. Such a 14-day regime produces a lesion of roughly 40–50  $\mu\text{m}$  with lower mineral content and smaller apatite crystallites than sound dentin, a reasonable stand-in for residual caries-affected dentin [19]. Lesion formation was verified on three extra discs by cross-sectional Knoop microhardness ( $28.4 \pm 3.1$  KHN at 50  $\mu\text{m}$  versus  $62.7 \pm 4.5$  KHN for sound dentin). Discs of the SD group were kept in distilled water at 37 °C for the same 14 days.

### 2.4. Synthesis and characterization of NSF

NSF was prepared by the chitosan-stabilized route. Chitosan (low molecular weight, degree of deacetylation  $\geq 85\%$ , Sigma-Aldrich, St Louis, MO, USA) was dissolved at 0.2% (w/v) in 1% (v/v) acetic acid overnight. Silver nitrate (10 mM final) was added and reduced dropwise with freshly prepared sodium borohydride (20 mM) at 4 °C under vigorous stirring; the mixture turned yellow-brown as the nanoparticles formed. Sodium fluoride was then added to give 5000 ppm fluoride and the pH was adjusted to 7.0 with NaOH. The final colloid contained 600 ppm silver (verified by atomic absorption spectroscopy (Shimadzu AA-7000)) and was kept in amber bottles at 4 °C.

An aliquot was freeze-dried for solid-state characterization. X-ray diffraction (XRD) was recorded on a Shimadzu XRD-6000 diffractometer with Cu K $\alpha$  radiation ( $\lambda = 1.5406$  Å, 40 kV, 30 mA) from 10° to 80° 2 $\theta$  in 0.02° steps, and the crystallite size was estimated from the (111) reflection with the Scherrer equation (Eq. 1), where K = 0.9 and  $\beta$  is the full width at half maximum in radians:

$$D = K\lambda / (\beta \cos \theta) \quad (1)$$

Fourier-transform infrared (FTIR) spectra of the dried NSF and of neat chitosan were collected in KBr discs on a Shimadzu IRAffinity-1 spectrometer (4000–400  $\text{cm}^{-1}$ , 4  $\text{cm}^{-1}$  resolution, 32 scans). Particle morphology and size were examined by field-emission scanning electron microscopy (FESEM, TESCAN MIRA3, 15 kV) on gold-sputtered samples with energy-dispersive X-ray (EDX) analysis; the mean size was obtained from at least 100 particles in ImageJ. The plasmon band was checked by UV–Vis spectroscopy (300–700 nm).

### 2.5. Surface pretreatments

Four groups were formed. SD: sound dentin, no pretreatment. CAD: artificial CAD, no pretreatment. SDF/KI: one drop of 38% SDF (Table 1) was rubbed onto the CAD surface with a microbrush and left for 60 s; the KI solution was then applied until the creamy-white precipitate cleared, and the surface was rinsed with distilled water for 30 s and blot-dried, leaving the dentin visibly moist. NSF: two drops of the colloid were rubbed onto the CAD surface for 60 s and left for 2 min, then rinsed and blot-dried in the same way. Rinsing was included for both agents because leaving the reaction products in place collapses the bond of glass-ionomer cement to carious dentin [20].

### 2.6. Restorative procedures

Two Tygon tubes (internal diameter 0.9 mm, height 1.0 mm) were fixed on each disc. In the UA groups the universal adhesive was applied in self-etch mode: rubbed for 20 s, air-thinned for 5 s and light-cured for 10 s with an LED unit (EliparDeepCure-S, 3M, St Paul, MN, USA,  $\geq 1000$  mW  $\text{cm}^{-2}$ , checked with a radiometer); the tubes were then filled with the nanohybrid composite and light-cured for 20 s. In the GH groups the dentin was conditioned with 20% polyacrylic acid for 10 s, rinsed and blot-dried; the capsule was activated, mixed for 10 s in an amalgamator and injected into the tubes, covered with a Mylar strip for the 2.5-min setting period, and the surface coat was then applied and light-cured for 20 s. After 1 h at 37 °C in 100% humidity the tubes were slit and removed; cylinders with voids or lifted margins were discarded and replaced. The two cylinders on a disc were averaged, so that the disc was the statistical unit.

### 2.7. Aging

Half of the specimens of each subgroup were stored in distilled water at 37 °C for 24 h. The other half were thermocycled for 10,000 cycles between water baths at 5 and 55 °C (dwell 30 s, transfer 10 s; THE-1100, SD Mechatronik, Feldkirchen-Westerham, Germany). This regime approximates about one year of intraoral thermal stress and is far harsher than the 500 cycles of ISO/TS 11405 [21,22].

### 2.8. Micro-shear bond strength and failure mode

Specimens were mounted in a universal testing machine (Instron 3345, Instron Corporation, Norwood, MA, USA) with the dentin surface parallel to the loading direction. A stainless-steel wire ( $\varnothing$  0.2 mm) was looped around the base of each cylinder as close as possible to the interface, and load was applied at 1 mm  $\text{min}^{-1}$  until failure.  $\mu\text{SBS}$  was calculated from the failure load F and the bonded area A (0.636  $\text{mm}^2$ ) according to Eq. 2:

$$\mu\text{SBS (MPa)} = F (\text{N}) / A (\text{mm}^2) \quad (2)$$

The micro-specimen configuration with wire-loop or chisel loading concentrates stress at the interface and is recommended for shear testing of the dentin–composite bond [23]. Fractured surfaces were examined at  $\times 40$  under a stereomicroscope and classified as adhesive (at the dentin–material interface), cohesive (within the material or the dentin) or mixed.

### 2.9. Color change

Thirty CAD discs were prepared as above. Baseline color was recorded in CIELAB coordinates with a dental spectrophotometer (VITA Easyshade V, VITA Zahnfabrik, Bad Säckingen, Germany) in a black box against a neutral grey background (three readings averaged). The discs were then treated with SDF/KI or NSF as in Section 2.5, or with distilled water (control), and stored in distilled water at 37 °C (renewed weekly, with 8 h of ambient laboratory light per day). Measurements were repeated after 24 h (T1), 1 week (T2), 2 weeks (T3) and 4 weeks (T4), and the color difference from baseline was calculated with the CIEDE2000 formula (Eq. 3;  $k_L = k_C = k_H = 1$ ):

$$\Delta E_{00} = [(\Delta L^*/k_L S_L)^2 + (\Delta C^*/k_C S_C)^2 + (\Delta H^*/k_H S_H)^2 + R_T(\Delta C^*/k_C S_C)(\Delta H^*/k_H S_H)]^{1/2} \quad (3)$$

Values were interpreted against the 50:50% perceptibility (PT = 0.8) and acceptability (AT = 1.8) thresholds widely used in dental color research, although recent psychophysical work places both somewhat higher [24,25].

### 2.10. Statistical analysis

Normality and homoscedasticity were checked with the Shapiro–Wilk and Levene tests.  $\mu$ SBS data were analyzed by three-way ANOVA (pretreatment  $\times$  material  $\times$  aging, Type II sums of squares) with partial eta squared ( $\eta_p^2 = SS_{\text{effect}}/(SS_{\text{effect}} + SS_{\text{error}})$ , Eq. 4) as the effect size, followed by Tukey HSD comparisons among the four pretreatments within each material  $\times$  aging combination. Failure-mode distributions were compared with the chi-square test, with Monte Carlo permutation (20,000 resamples) to confirm p-values where expected counts were below five. Because the  $\Delta E_{00}$  variances differed markedly among groups, the groups were compared at each time point with Welch's ANOVA and the Games–Howell test, and the effect of time within each group with the Friedman test followed by the Wilcoxon signed-rank test (T1 versus T4). Analyses were run in Python 3.11 (SciPy 1.17, statsmodels 0.15) at  $\alpha = 0.05$ .

## 3. RESULTS AND DISCUSSION

### 3.1. Characterization of NSF

The colloid was yellow-brown and showed a surface-plasmon band at 418 nm, within the 400–460 nm window usually reported for chitosan-capped silver nanoparticles [26]. The diffractogram of the freeze-dried NSF (Figure 2a) showed reflections at  $2\theta = 38.12, 44.28, 64.42$  and  $77.38^\circ$  that index to the (111), (200), (220) and (311) planes of face-centered cubic silver (JCPDS 04-0783; reference positions  $38.1^\circ, 44.3^\circ, 64.4^\circ$  and  $77.5^\circ$ ) [27]; no reflections of  $\text{Ag}_2\text{O}$  or  $\text{AgCl}$  were detected, and the broad hump near  $20\text{--}25^\circ$  belongs to the amorphous chitosan matrix. Scherrer analysis of the (111) reflection gave a crystallite size of 14.6 nm. The FTIR spectrum (Figure 2b) retained the chitosan bands at  $3425\text{ cm}^{-1}$  (overlapping O–H and N–H stretching),  $2878\text{ cm}^{-1}$  (C–H stretching),  $1648\text{ cm}^{-1}$  (amide I),  $1590\text{ cm}^{-1}$  (N–H bending) and  $1072\text{ cm}^{-1}$  (C–O–C stretching); the N–H band was shifted by  $52\text{ cm}^{-1}$  and weakened relative to neat chitosan, which is the usual sign of amino groups coordinating the silver surface [26,27]. FESEM (Figure 2c) showed spherical, well-dispersed particles with a mean size of  $18.5 \pm 4.2\text{ nm}$  (range 10–32 nm), and EDX confirmed the presence of Ag, F, C, O and N. Small capped particles of this kind explain the antibacterial activity at low silver doses and the low staining that distinguish NSF and related formulations from SDF [15,28].

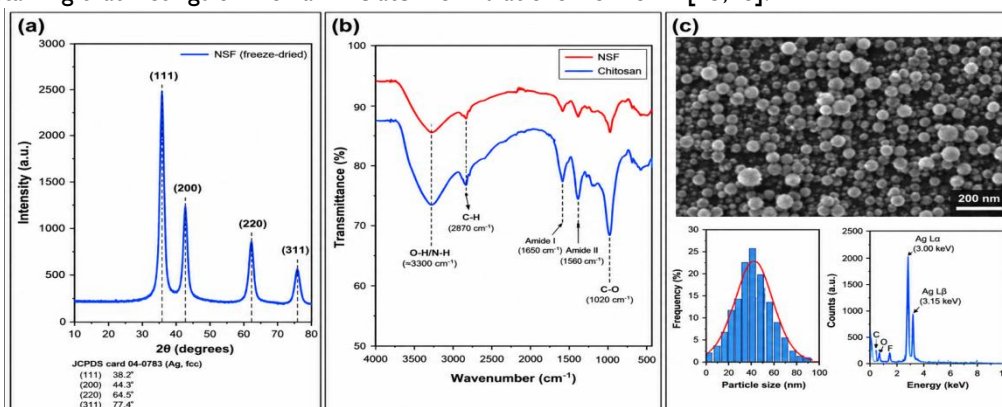


Figure 02: Characterization of the synthesized NSF. (a) XRD pattern of the freeze-dried NSF with the (111), (200), (220) and (311) reflections of face-centered cubic silver (reference positions from JCPDS 04-0783); (b) FTIR spectra of NSF and neat chitosan ( $4000\text{--}400\text{ cm}^{-1}$ ); (c) FESEM micrograph of the NSF particles with the size distribution and EDX spectrum.

### 3.2. Micro-shear bond strength

Table 2 and Figure 3 summarize the  $\mu$ SBS data, and Table 3 gives the three-way ANOVA. The ANOVA assumptions were met within each material  $\times$  aging combination (Shapiro–Wilk  $p \geq 0.06$ ; Levene  $p \geq 0.13$ ). All three main effects and all two-way interactions were significant, whereas the three-way interaction was not ( $p = 0.161$ ). The restorative material was by far the strongest factor ( $F = 827.4$ ,  $\eta_p^2 = 0.85$ ): UA bonds were two to three times higher than GH

bonds in every condition, which reflects the difference between micromechanical resin bonding and the ionic bonding of a low-strength glass-ionomer matrix. Pretreatment came next ( $\eta_p^2 = 0.54$ ) and aging third ( $\eta_p^2 = 0.26$ ). The pretreatment  $\times$  material interaction ( $\eta_p^2 = 0.30$ ) shows that the pretreatments did not act in the same way on the two materials, so post hoc comparisons were made separately for each material and aging level.

Table 03: Three-way ANOVA (Type II) of the micro-shear bond strength data.

| Source of variation                           | Sum of squares | df  | Mean square | F      | p       | $\eta_p^2$ |
|-----------------------------------------------|----------------|-----|-------------|--------|---------|------------|
| Pretreatment                                  | 789.51         | 3   | 263.17      | 57.17  | < 0.001 | 0.544      |
| Restorative material                          | 3808.84        | 1   | 3808.84     | 827.43 | < 0.001 | 0.852      |
| Aging                                         | 238.12         | 1   | 238.12      | 51.73  | < 0.001 | 0.264      |
| Pretreatment $\times$ material                | 280.64         | 3   | 93.55       | 20.32  | < 0.001 | 0.297      |
| Pretreatment $\times$ aging                   | 51.72          | 3   | 17.24       | 3.75   | 0.013   | 0.072      |
| Material $\times$ aging                       | 47.84          | 1   | 47.84       | 10.39  | 0.002   | 0.067      |
| Pretreatment $\times$ material $\times$ aging | 24.07          | 3   | 8.02        | 1.74   | 0.161   | 0.035      |
| Residual                                      | 662.87         | 144 | 4.60        |        |         |            |

Table 02: Micro-shear bond strength (MPa) of the universal adhesive (UA) and the glass hybrid (GH) to the four dentin conditions after 24 h and after 10,000 thermal cycles (TC); n = 10 per subgroup.

| Material | Aging | Pre-treatment | Mean $\pm$ SD    | Min-Max     | Tukey <sup>a</sup> | Change after TC (%) |
|----------|-------|---------------|------------------|-------------|--------------------|---------------------|
| UA       | 24 h  | SD            | 25.57 $\pm$ 3.94 | 19.41–30.09 | A                  | –                   |
| UA       | 24 h  | CAD           | 16.00 $\pm$ 2.82 | 11.32–19.11 | BC                 | –                   |
| UA       | 24 h  | SDF/KI        | 13.67 $\pm$ 2.53 | 8.92–17.06  | C                  | –                   |
| UA       | 24 h  | NSF           | 17.68 $\pm$ 2.44 | 13.89–20.99 | B                  | –                   |
| UA       | TC    | SD            | 19.11 $\pm$ 3.31 | 13.72–24.71 | A                  | –25.3               |
| UA       | TC    | CAD           | 13.49 $\pm$ 2.97 | 8.31–17.04  | B                  | –15.7               |
| UA       | TC    | SDF/KI        | 12.25 $\pm$ 1.93 | 10.25–15.42 | B                  | –10.4               |
| UA       | TC    | NSF           | 13.93 $\pm$ 2.14 | 9.85–16.90  | B                  | –21.2               |
| GH       | 24 h  | SD            | 9.25 $\pm$ 1.28  | 7.01–11.22  | A                  | –                   |
| GH       | 24 h  | CAD           | 6.90 $\pm$ 0.62  | 5.82–7.58   | B                  | –                   |
| GH       | 24 h  | SDF/KI        | 6.40 $\pm$ 1.11  | 4.35–7.95   | B                  | –                   |
| GH       | 24 h  | NSF           | 6.97 $\pm$ 1.47  | 5.19–10.10  | B                  | –                   |
| GH       | TC    | SD            | 7.25 $\pm$ 1.16  | 4.82–9.03   | A                  | –21.6               |
| GH       | TC    | CAD           | 5.24 $\pm$ 0.60  | 4.23–6.16   | B                  | –24.1               |
| GH       | TC    | SDF/KI        | 5.65 $\pm$ 1.15  | 3.96–7.58   | B                  | –11.8               |
| GH       | TC    | NSF           | 6.00 $\pm$ 1.15  | 4.04–7.66   | AB                 | –13.9               |

<sup>a</sup> Different letters within the same material and aging level indicate a significant difference among pretreatments (Tukey HSD,  $p < 0.05$ ). Change after TC is relative to the 24-h mean of the same subgroup.

With UA at 24 h (Figure 3a), sound dentin gave 25.57  $\pm$  3.94 MPa and untreated CAD 16.00  $\pm$  2.82 MPa, a loss of 37% that agrees with the pooled estimates for universal adhesives on CAD [3] and with one-year data on semi-hard CAD [4]. NSF-treated CAD reached 17.68  $\pm$  2.44 MPa, indistinguishable from untreated CAD ( $p = 0.60$ ), whereas SDF/KI-treated CAD fell to 13.67  $\pm$  2.53 MPa, lower than NSF (mean difference 4.0 MPa, 95% CI 0.4–7.6,  $p = 0.024$ ) and numerically 15% below untreated CAD ( $p = 0.32$ ). The first null hypothesis was therefore rejected for the universal adhesive, but only for SDF/KI. The direction of the SDF/KI effect matches reports on self-etch and universal adhesives on sound dentin after Riva Star [29] and on SDF-treated sound and demineralized dentin with etch-and-rinse and self-etch systems, where reductions of about 30% were recorded [30]. The reduction here was milder, probably because the surface was rinsed and blot-dried before bonding, which removes loosely bound silver iodide and unreacted SDF [9,20]. The absence of an NSF effect matches the only comparable study on a universal adhesive, in which 600 ppm NSF left the  $\mu$ TBS to sound dentin unchanged while 1500 ppm began to lower it and left dense nanoparticle deposits on the surface [16]; the concentration used here (600 ppm) lies on the safe side of that boundary.

After 10,000 thermal cycles all UA bonds dropped: by 25% for SD, 16% for CAD, 21% for NSF and only 10% for SDF/KI (Table 2). The three CAD-based groups then converged (13.5, 12.3 and 13.9 MPa; all letter B) and only sound dentin stayed higher. The smaller relative loss in the SDF/KI group is partly a floor effect, but a protective contribution is plausible: silver and fluoride inhibit matrix metalloproteinases and cysteine cathepsins and stiffen the demineralized collagen [5], which should slow the hydrolytic degradation of the interface, and the significant pretreatment  $\times$  aging interaction ( $p = 0.013$ ) fits that reading. Thermocycled interfaces of a universal adhesive on SDF-treated artificial

carious dentin have been examined in detail elsewhere [31]. After aging, then, NSF and SDF/KI ended at the same level, roughly two-thirds to three-quarters of sound dentin.

With the glass hybrid (Figure 3b) the picture was flatter. Sound dentin gave  $9.25 \pm 1.28$  MPa at 24 h, and the three CAD groups lay between 6.4 and 7.0 MPa with no differences among them ( $p \geq 0.70$ ); the SD–CAD gap (25%) was smaller than with UA, a comparison that has also been drawn for other ion-releasing materials on sound and caries-affected dentin [32]. After thermocycling the GH groups lost 12–24%, and NSF ( $6.00 \pm 1.15$  MPa) was the only CAD-based group that no longer differed from sound dentin ( $p = 0.051$ ). SDF/KI did not reduce the GH bond at either time. This agrees with the systematic reviews, in which most glass-ionomer studies found no adverse effect of rinsed SDF [9,10], with the shear data of Rinsathon et al. on carious dentin [20] and with the immediate  $\mu$ SBS of a resin-modified glass ionomer to demineralized primary dentin after SDF [33]; it contrasts with the  $\mu$ TBS reduction reported for EQUIA Forte on SDF-treated demineralized dentin after 10,000 cycles [34], obtained without a KI step and with a different test geometry. For NSF, the neutral effect seen here is more conservative than the two- to threefold increase of GIC and RMGIC bond strength reported on primary carious dentin [17], but agrees with the equivalence of NSF and SDF for a resin-modified glass ionomer on artificial dentin caries before and after 5,000 cycles [18]. Glass-ionomer adhesion relies on carboxylate–calcium bonding; silver deposits may occupy part of the calcium-rich surface, but the polyacrylic-acid conditioner dissolves much of that layer, which probably explains why the glass hybrid was less affected. The GH values (5–9 MPa) are typical of high-viscosity glass ionomers and glass hybrids in micro-shear testing, and the microhardness recovery that the glass hybrid offers on demineralized dentin [35] is a further argument for using it on CAD.

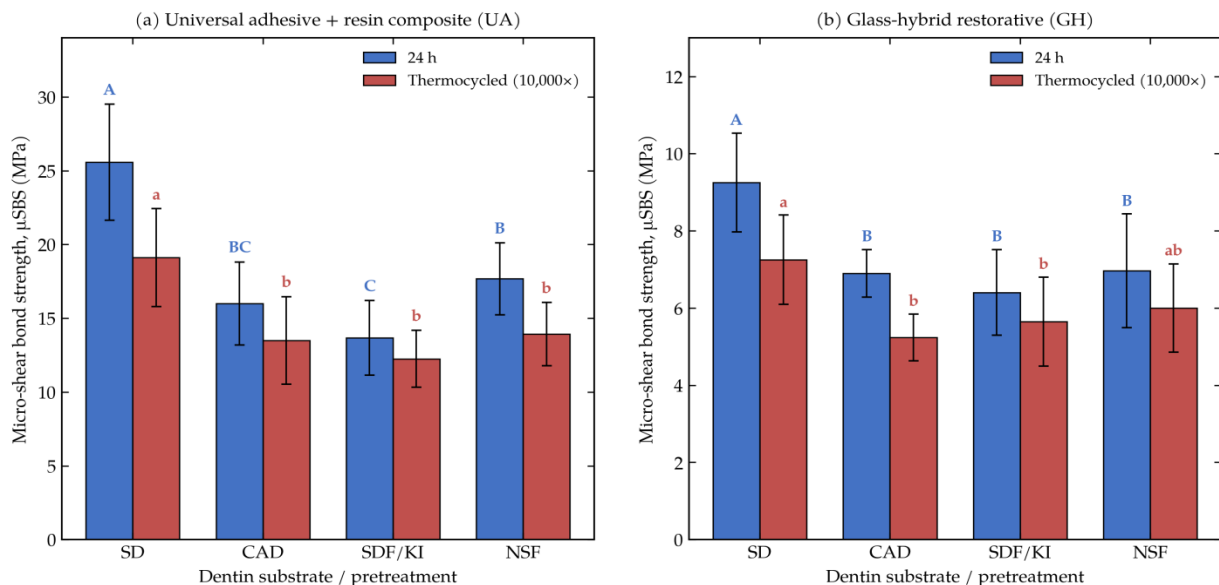


Figure 03: Micro-shear bond strength (mean  $\pm$  SD,  $n = 10$ ) of (a) the universal adhesive with resin composite and (b) the glass-hybrid restorative to sound dentin (SD), untreated caries-affected dentin (CAD), SDF/KI-treated CAD and NSF-treated CAD after 24 h (blue) and after 10,000 thermal cycles (red). Upper-case letters compare the 24-h groups and lower-case letters the thermocycled groups within each panel; different letters indicate a significant difference (Tukey HSD,  $p < 0.05$ ).

### 3.3. Failure modes

Failure modes are given in Table 4 and Figure 4. Within any single material  $\times$  aging combination the distribution did not depend on the pretreatment ( $\chi^2 p = 0.19$ – $0.66$ ; Monte Carlo  $p = 0.21$ – $0.75$ ), so the individual cell counts are best read as trends. Pooled over pretreatment and aging, however, UA and GH differed clearly ( $\chi^2 = 11.5$ ,  $p = 0.003$ ): UA failed mainly adhesively (58%) or in mixed mode (36%), with only 6% cohesive failures, whereas GH showed 36% adhesive, 41% mixed and 23% cohesive failures, the cohesive fractures being concentrated in the sound-dentin groups (60% at 24 h). A cohesive fracture through the glass hybrid means that the interface outperformed the material, as expected when a low-strength cement meets a favorable substrate; on CAD the interface became the weak link again. Pooled over materials, pretreatment also affected the distribution ( $\chi^2 = 20.0$ ,  $p = 0.003$ ): SDF/KI had the highest share of adhesive failures (65%), untreated CAD and NSF were intermediate (50% and 45%) and sound dentin the lowest (28%). Thermocycling shifted failures toward the adhesive mode (40% to 54%), although not significantly ( $p = 0.135$ ), as is usually seen after artificial aging of self-etch interfaces [36]. The micro-specimen geometry itself favors adhesive failures [23], and failure pattern does not necessarily track bond-strength magnitude [37], so these observations support rather than replace the  $\mu$ SBS analysis. What matters for the present question is that NSF did not raise the proportion of adhesive failures relative to untreated CAD, whereas SDF/KI did.

Table 04: Distribution of failure modes (number of specimens, n = 10 per group) after micro-shear testing, examined at  $\times 40$ .

| Material | Aging | Pre-treatment | Adhesive | Mixed | Cohesive | Adhesive (%) |
|----------|-------|---------------|----------|-------|----------|--------------|
| UA       | 24 h  | SD            | 3        | 5     | 2        | 30           |
| UA       | 24 h  | CAD           | 5        | 4     | 1        | 50           |
| UA       | 24 h  | SDF/KI        | 7        | 3     | 0        | 70           |
| UA       | 24 h  | NSF           | 5        | 4     | 1        | 50           |
| UA       | TC    | SD            | 5        | 4     | 1        | 50           |
| UA       | TC    | CAD           | 7        | 3     | 0        | 70           |
| UA       | TC    | SDF/KI        | 8        | 2     | 0        | 80           |
| UA       | TC    | NSF           | 6        | 4     | 0        | 60           |
| GH       | 24 h  | SD            | 1        | 3     | 6        | 10           |
| GH       | 24 h  | CAD           | 3        | 5     | 2        | 30           |
| GH       | 24 h  | SDF/KI        | 5        | 4     | 1        | 50           |
| GH       | 24 h  | NSF           | 3        | 5     | 2        | 30           |
| GH       | TC    | SD            | 2        | 4     | 4        | 20           |
| GH       | TC    | CAD           | 5        | 4     | 1        | 50           |
| GH       | TC    | SDF/KI        | 6        | 3     | 1        | 60           |
| GH       | TC    | NSF           | 4        | 5     | 1        | 40           |

Chi-square within each material  $\times$  aging combination: UA 24 h  $p = 0.66$ ; UA TC  $p = 0.59$ ; GH 24 h  $p = 0.19$ ; GH TC  $p = 0.38$ . Pooled comparisons: UA versus GH  $\chi^2 = 11.5$ ,  $p = 0.003$ ; among pretreatments  $\chi^2 = 20.0$ ,  $p = 0.003$ ; 24 h versus TC  $\chi^2 = 4.0$ ,  $p = 0.135$ .

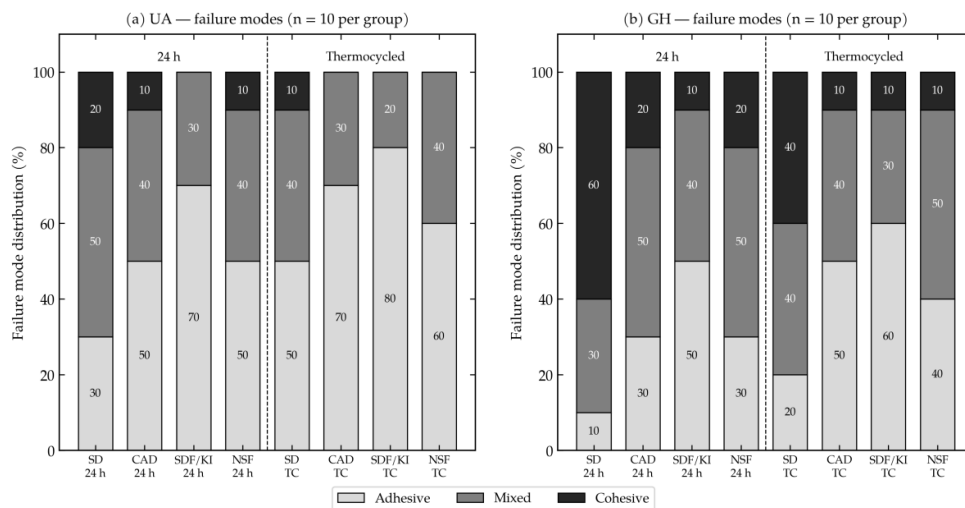


Figure 04: Failure-mode distribution (%) of (a) the universal adhesive groups and (b) the glass-hybrid groups after 24 h and after thermocycling (TC).

### 3.4. Color change

Table 5 and Figure 5 show the color data. Because the variances differed markedly among groups (Levene  $p \leq 0.027$  at every time point), Welch's ANOVA was used; it was significant from T1 to T4 ( $F = 62.6$ – $157.1$ , all  $p < 0.001$ ), and Games–Howell comparisons separated all three groups at every time point ( $p < 0.004$ ). The control discs changed by  $0.92 \pm 0.14$  at 24 h and  $1.45 \pm 0.25$  at four weeks, above the perceptibility threshold but never above the acceptability threshold; this drift was significant over time (Friedman  $p < 0.001$ ), reflects the slow rehydration and mineral exchange of pH-cycled dentin in water, and is the background against which the silver agents must be judged. NSF-treated dentin changed by  $1.59 \pm 0.42$  at 24 h,  $2.13 \pm 0.67$  at one week and  $2.81 \pm 0.84$  at four weeks; the difference from the control never exceeded 1.4 units, and although the share of NSF discs above the acceptability threshold rose from 40% at 24 h to 90–100% from two weeks onward, the values stayed within what recent psychophysical work regards as acceptable to a mixed population of observers ( $AT_{00} \approx 2.7$ ) [24]. SDF/KI-treated dentin behaved quite differently:  $\Delta E_{00}$  was already  $3.70 \pm 0.81$  at 24 h, doubled by one week ( $7.42 \pm 1.11$ ) and reached  $12.94 \pm 2.78$  at four weeks, seven times the acceptability threshold, with every disc above the threshold from the first day. The second null hypothesis was therefore rejected.

Table 05: Color change ( $\Delta E_{00}$ , mean  $\pm$  SD,  $n = 10$ ) of caries-affected dentin discs relative to baseline, and its interpretation against the perceptibility (PT = 0.8) and acceptability (AT = 1.8) thresholds.

| Group                                      | T1 (24 h)         | T2 (1 week)       | T3 (2 weeks)       | T4 (4 weeks)       | Friedman $\chi^2$ (p) |
|--------------------------------------------|-------------------|-------------------|--------------------|--------------------|-----------------------|
| Control (distilled water)                  | $0.92 \pm 0.14^a$ | $1.16 \pm 0.14^a$ | $1.33 \pm 0.20^a$  | $1.45 \pm 0.25^a$  | 24.5 (< 0.001)        |
| NSF                                        | $1.59 \pm 0.42^b$ | $2.13 \pm 0.67^b$ | $2.57 \pm 0.82^b$  | $2.81 \pm 0.84^b$  | 26.0 (< 0.001)        |
| SDF/KI                                     | $3.70 \pm 0.81^c$ | $7.42 \pm 1.11^c$ | $10.03 \pm 2.15^c$ | $12.94 \pm 2.78^c$ | 30.0 (< 0.001)        |
| Welch F (p)                                | 62.6 (< 0.001)    | 157.1 (< 0.001)   | 86.6 (< 0.001)     | 90.6 (< 0.001)     |                       |
| Discs above AT (%): control / NSF / SDF/KI | 0 / 40 / 100      | 0 / 50 / 100      | 0 / 100 / 100      | 0 / 90 / 100       |                       |

Different superscript letters within a column indicate a significant difference (Games–Howell,  $p < 0.05$ ). Interpretation: control, perceptible but acceptable at all times; NSF, perceptible, at or slightly above the acceptability threshold; SDF/KI, unacceptable at all times.

The progressive darkening of SDF/KI is well documented. The KI step converts free silver ions into silver iodide, which is initially white, but the lightening is dose-dependent and short-lived; Detsomboonrat et al. showed that the anti-staining benefit of all KI concentrations had largely converged by 7–14 days [7], and the clinical evidence on any long-term benefit of KI is inconsistent [6,8]. Silver iodide itself is photosensitive, and residual silver in the lesion continues to be reduced to metallic silver on exposure to light [5]. The rate observed here, about 0.6  $\Delta E_{00}$  units per day over the first week, is compatible with the very large color changes reported for SDF on carious primary dentin ( $\Delta E = 17.2$ ), which KI masked only transiently under accelerated aging [38]. NSF, by contrast, carries its silver as stabilized metallic nanoparticles capped by chitosan; there is little free  $Ag^+$  to be reduced, and the particles are too small to scatter visible light appreciably. The NSF values found here ( $\Delta E_{00}$  1.6–2.8 over four weeks) are close to the  $\Delta E$  of 1.0–1.5 measured for 600 and 1500 ppm NSF [14] and to the  $\Delta E_{00}$  of 3.3 recorded under a resin-modified glass ionomer after NSF versus 5.0 after SDF [18], and echo the near-absence of staining of other fluoridated silver nanoparticle formulations [15]. The difference matters clinically: in the trial of Quritum et al., 97% of parents were satisfied with the appearance of NSF-treated teeth against 76% for SDF, and caries arrest was actually higher with NSF [12]. Resin composite masks the stain of SDF/KI-treated dentin reasonably well [39], but darkened margins and a black substrate showing through a glass hybrid remain a concern, and NSF avoids the problem at its source.

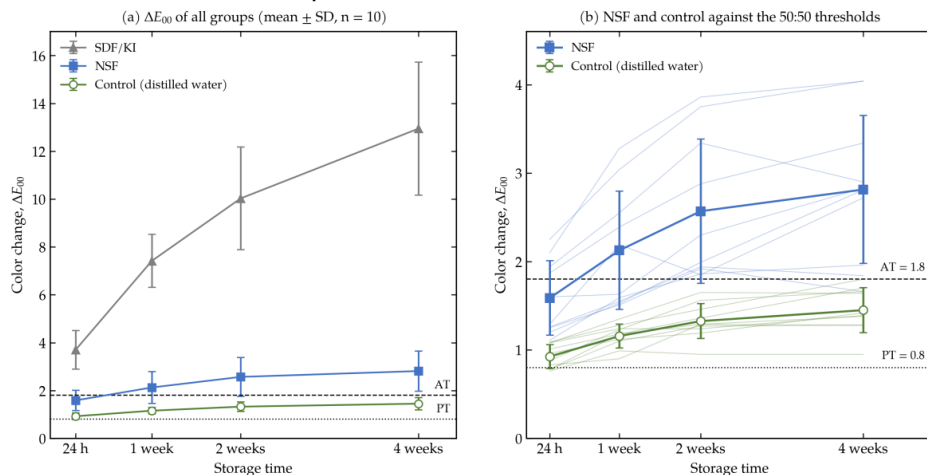


Figure 05: Color change ( $\Delta E_{00}$ ) of caries-affected dentin discs treated with distilled water (control), NSF or SDF/KI over four weeks of storage: (a) all groups (mean  $\pm$  SD,  $n = 10$ ); (b) NSF and control with the individual specimens (thin lines) against the 50:50% perceptibility (PT, dotted) and acceptability (AT, dashed) thresholds.

### 3.5. Interfacial mechanism, comparison with previous work and limitations

Figure 6 summarizes the interpretation. SDF/KI leaves a continuous layer of silver phosphate, silver iodide and metallic silver that plugs the tubule entrances and coats the demineralized collagen; a mild self-etch adhesive cannot dissolve this layer, so resin infiltration is limited and the hybrid layer is thin and defective, which lowers  $\mu$ SBS and shifts failures toward the adhesive mode. The glass hybrid is less affected because its conditioner and acidic matrix partly remove the deposits and because its bond depends on ionic exchange rather than deep infiltration. NSF deposits discrete chitosan-capped nanoparticles that leave most tubules patent, so the adhesive can infiltrate around them while fluoride and silver still supply the remineralizing action examined for silver nanoparticles added to sodium fluoride on root dentin [40] and the antibacterial action expected of silver nanoparticles in dentistry [41]. Table 6 places the present values beside the

closest studies of the last six years; every comparison points the same way, and the SDF/KI effect on resin bonding falls between the mild reductions reported after rinsing and the losses of about 30% reported without it.

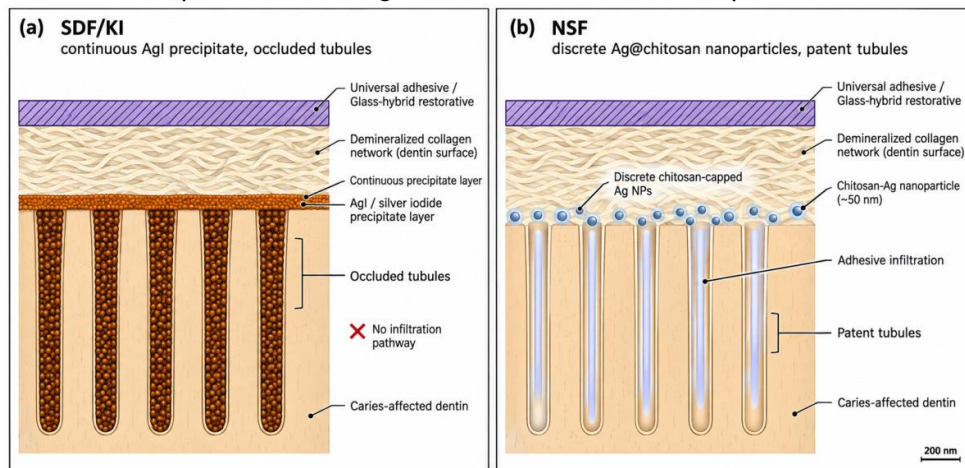


Figure 06: Proposed structure of the interface (schematic, not a micrograph) between a restorative and caries-affected dentin pretreated with (a) SDF/KI, which leaves a continuous precipitate layer and occluded tubules, and (b) NSF, which leaves discrete chitosan-capped silver nanoparticles and patent tubules that allow infiltration.

Table 06: Comparison of the present results with previous studies (2020–2026) on bonding to silver-treated dentin and on the color change of silver-treated dentin.

| Study                             | Substrate and restorative                                                                | Main finding                                                                                                               | Relation to the present study                                                                                    |
|-----------------------------------|------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------|
| Shirani et al. 2025 [30]          | Sound and demineralized dentin; composite with etch-and-rinse or self-etch adhesive; SDF | SBS fell from 26.6 to 17.8 MPa (sound) and from 15.1 to 9.8 MPa (demineralized) with OptiBond FL; similar with Clearfil SE | Same direction; the reduction here was milder (13.7 vs 16.0 MPa), most likely because of the KI step and rinsing |
| Matijević et al. 2026 [29]        | Sound dentin; self-etch and universal adhesive; Riva Star (SDF/KI)                       | SDF/KI reduced SBS of both adhesives at 1, 3 and 6 months ( $p < 0.001$ )                                                  | Agrees with the lower UA bond after SDF/KI                                                                       |
| Silva et al. 2026 [16]            | Sound dentin; universal adhesive; NSF 600 and 1500 ppm                                   | $\mu$ TBS 33.0 (control), 35.6 (600 ppm) and 21.4 MPa (1500 ppm); no significant difference                                | Agrees with the absence of an NSF effect at 600 ppm                                                              |
| Das et al. 2024 [17]              | Primary carious dentin; GIC and RMGIC; NSF                                               | $\mu$ TBS rose from 6.8 to 11.6 MPa (GIC) and from 10.5 to 22.0 MPa (RMGIC) after NSF                                      | More favorable than the neutral GH result here                                                                   |
| Mubarak et al. 2026 [18]          | Artificial dentin caries (primary molars); RMGIC; NSF vs SDF                             | SBS 8.6 (NSF) vs 7.8 MPa (SDF) before and 6.0 vs 6.1 MPa after 5,000 cycles; $\Delta E_{00}$ 3.3 (NSF) vs 5.0 (SDF)        | Same equivalence of the two agents for a glass-ionomer material; same color advantage of NSF                     |
| Rinsathon et al. 2023 [20]        | Artificial carious dentin; GIC; SDF with or without rinsing                              | SBS 2.6 MPa with rinsing vs 0.35 MPa without rinsing (control 2.5 MPa)                                                     | Supports the rinsing step adopted here                                                                           |
| Abdelshafi et al. 2025 [34]       | Artificially demineralized dentin; alkasite, RMGIC and EQUIA Forte; SDF; 10,000 cycles   | SDF reduced $\mu$ TBS of all three materials; EQUIA Forte lowest                                                           | Differs from the neutral GH result here (no KI, no conditioner, $\mu$ TBS geometry)                              |
| AbedinzadeZare et al. 2026 [33]   | Demineralized primary dentin; RMGIC; SDF vs ammonia-free AgF                             | $\mu$ SBS 6.9 (control), 6.7 (SDF) and 3.7 MPa (AgF)                                                                       | Agrees with the absence of an SDF/KI effect on the glass hybrid                                                  |
| Espindola-Castro et al. 2020 [14] | Coronal dentin; NSF 600/1500 ppm vs SDF-based agents; 4 weeks                            | NSF $\Delta E \approx 1.0$ – $1.5$ , far below the SDF agents ( $p < 0.001$ )                                              | Agrees with the low $\Delta E_{00}$ of NSF (2.8 at 4 weeks)                                                      |
| Hamdy et al. 2021 [38]            | Carious primary dentin; SDF, then KI, GIC or composite                                   | SDF gave $\Delta E = 17.2$ ; KI masked the stain only transiently under aging                                              | Agrees with the progressive darkening of SDF/KI ( $\Delta E_{00}$ 3.7 $\rightarrow$ 12.9)                        |
| Present study                     | Artificial CAD (pH-                                                                      | UA 24 h: CAD 16.0, SDF/KI 13.7,                                                                                            | –                                                                                                                |

|  |                                                                                         |                                                                                                         |  |
|--|-----------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------|--|
|  | cycling); universal adhesive + composite and glass hybrid; SDF/KI vs NSF; 10,000 cycles | NSF 17.7 MPa; GH 24 h: 6.9, 6.4, 7.0 MPa; $\Delta E_{00}$ at 4 weeks: SDF/KI 12.9, NSF 2.8, control 1.5 |  |
|--|-----------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------|--|

Some limitations should be kept in mind. pH-cycled CAD reproduces the mineral loss of natural lesions but not the bacterial invasion and collagen changes of a biofilm-generated lesion [19,42]; one universal adhesive and one glass hybrid were tested; aging was limited to thermocycling; and color was measured on bare dentin rather than through a restoration. The sample size ( $n = 10$ ) was adequate for the large effects observed but modest for the failure-mode comparisons. In the mouth, saliva, occlusal loading and the pulp would modify both bonding and color, so clinical confirmation is needed in the primary and permanent teeth for which selective caries removal is recommended [1,2].

#### 4. CONCLUSION

Within the limits of this in vitro study, nano-silver fluoride and SDF/KI behaved differently on caries-affected dentin. NSF did not reduce the micro-shear bond strength of a universal adhesive used in self-etch mode or of a glass-hybrid restorative, either at 24 h or after 10,000 thermal cycles, and it did not change the failure pattern relative to untreated CAD. SDF/KI lowered the 24-h bond of the universal adhesive by about 23% relative to NSF and raised the share of adhesive failures, although the difference disappeared after thermocycling; it had no measurable effect on the glass hybrid. The restorative material was the dominant source of variance, and aging cut all bonds by 10–25%. The two agents differed most clearly in color: SDF/KI-treated dentin darkened steadily to a  $\Delta E_{00}$  of about 13 at four weeks, whereas NSF stayed close to the acceptability threshold. NSF therefore appears to be a suitable non-staining pretreatment when residual caries-affected dentin is to be restored with an adhesive composite or a glass hybrid, pending confirmation on natural lesions and in clinical trials.

#### 5. AUTHOR CONTRIBUTIONS

Noor A. Al-Tamimi: conceptualization, methodology, investigation, writing – original draft. Zainab K. Mahmood: investigation, formal analysis, visualization. Haider M. Al-Saadi: supervision, validation, writing – review and editing. All authors read and approved the final manuscript.

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This research received no external funding.

#### 7. CONFLICTS OF INTEREST

The author declares no conflict of interest.

#### 8. DATA AVAILABILITY

The data supporting the findings of this study are available from the corresponding author on reasonable request.

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