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# Influence of Silver Diamine Fluoride and Potassium Iodine Pretreatment on Bond Durability, Nanoleakage, and Interfacial Integrity of Universal Adhesives to Caries-Affected Dentin

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## Abstract

Although silver diamine fluoride (SDF) has been widely applied to arrest dentinal caries, the precipitates generated after application may negatively affect the ability of the fluoride to bond to the tooth. The use of potassium iodide (KI) may be useful in minimizing SDF discoloration, while its effects on the longevity of universal adhesives are not yet known. This in vitro study aimed to assess the immediate and aged bond strength, nanoleakage, and failure-mode distribution of two universal adhesives bonded to caries-affected dentin with or without SDF followed by KI. The total of 120 extracted permanent teeth were used to create standardized artificial caries-affected dentin. The specimens were randomly divided into three dentin pretreatment groups: the control group (C), the 38% SDF group (A), and the 38% SDF followed by KI group (B). Each group was subdivided based on the type of universal adhesive used (Scotchbond Universal Plus vs. Clearfil Universal Bond Quick). The specimens were either examined immediately after composite build-up or after 10,000 thermal cycles plus six months of water storage. The microtensile bond strength (μTBS), nanoleakage and failure-mode distribution were evaluated. Linear mixed-effects models were used to control for clustering of beams within teeth and $p < 0.05$ was used as the threshold for significance. The SDF significantly reduced immediate and aged μTBS and increased nanoleakage ($p < 0.001$) compared to untreated caries-affected dentin. The application of KI after SDF significantly increased bond strength and decreased nanoleakage compared with SDF alone ($p < 0.05$); however, neither outcome returned to the values observed in the untreated control groups. The bond strength was significantly diminished and the nanoleakage was significantly increased in all groups after artificial aging, the most in the SDF-only groups. The number of adhesive failures was also higher after SDF treatment, especially after aging. The overall performance of Clearfil Universal Bond Quick was just slightly better than Scotchbond Universal Plus**.** Pretreatment with SDF had a negative impact on the durability of the universal adhesive bonding to caries-affected dentin. These negative effects were partially mitigated but not fully restored by applying KI immediately after SDF, and the bonding performance was not restored to the level of that in the untreated dentin. Therefore, adequate surface management should be considered prior to the placement of adhesive composite restorations after SDF.

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## Introduction

Dental caries is one of the most prevalent chronic oral diseases and is caused by an imbalance between the demineralization and remineralization occurring at the tooth-biofilm interface. Progress in caries management has shifted towards biologically based and minimally invasive treatment strategies, over time, rather than complete removal of all discolored and softened dentin. The strategies are designed to inhibit the growth of the bacteria, maintain mineralizable dental tissues, maintain dental pulp vitality and avoid unnecessary structural weakening of the tooth. Thus, selective removal of carious tissue and appropriate restoration with a seal, has emerged as a key approach to managing deep carious lesions [1].
Caries-affected dentin is structurally and chemically different from sound dentin. It is partially demineralized but partially contaminated by bacteria and still has collagen that can be remineralized. However, because of its reduced mineral density, higher water content, different tubular morphology and more exposed collagen it can make adhesive bonding more difficult. Such alterations might affect resin infiltration, affect the development of a homogeneous hybrid zone and make the resin–dentin interface more vulnerable to hydrolytic and enzymatic degradation [2]. Thus, bonding properties of sound dentin may not apply to carious dentin.
Silver diamine fluoride (SDF) has garnered significant attention as a minimally invasive treatment for dentinal caries prevention and arrest. Available commercially, 38% SDF is a solution of silver and fluoride ions that is alkaline and based on ammonia. Silver ions are having wide antimicrobial effect, while fluoride can remineralize and make the dental hard tissues more resistant to further acid attack. Alternatively, SDF has been shown to inhibit collagen-degrading enzymes, such as matrix metalloproteinases and cysteine cathepsins, which also decreases the exposed dentinal organic matrix degradation [3].
The clinical benefits of SDF are its easy application, low cost, non-invasive nature and capacity to arrest carious lesions without significant tissue removal. Its properties are especially useful for young children, elderly, patients with special healthcare needs and others without easy access to traditional restorative care. However, the development of dark silver-deposits in carious dentin is the main problem with it in terms of aesthetics. Black staining that is linked to SDF could affect the likelihood of successful acceptance by patients and caregivers, particularly if the lesion is in a visible location [4].
The use of potassium iodide immediately after SDF has been proposed to minimize this discoloration. Potassium iodide reacts with free silver ions, first to give silver iodide as a cream-colored precipitate. This reaction can lessen the amount of the remaining unreacted silver which can then be oxidized and darkened. The color masking effect, however, can be time-dependent and may not be a permanent effect, as silver iodide can undergo photochemical decomposition over time. Furthermore, the precipitates formed by the SDF/KI reaction might stay on the dentin surface and could interact with the underlying substrate and interfere with the bonding of the adhesives to the dentin [5].
Generating compounds which may reinforce the surface or create a physical barrier to the penetration of the adhesive. Thus, the effect of SDF on bonding is a complex phenomenon and may be influenced by SDF concentration, SDF application duration, SDF washing protocol, dentin condition, restorative material, adhesive formulation, and bonding approaches [6,7].
Studies published so far have yielded conflicting results about the effect of SDF on resin bonding. Studies revealed that SDF decreased bond strength due to extensive surface precipitation and ineffective infiltration of adhesive. Other studies have found no significant adverse effect, or even reported beneficial improvement in bonding, especially after rinsing, conditioning, or restoration of the treated surface with bonding materials that can chemically interact with mineralized dentin. The conflicting results might be attributed to the differences in substrates tested (sound, artificially demineralized, naturally carious dentin, primary dentin, and permanent dentin); differences in specimen preparation and aging methods [8,9].
The effect of KI on bonding following SDF treatment is also unknown. KI could help bind or transform the surplus of the silver ions and create a better adhesive surface. On the other hand, silver iodide precipitates can block the dentinal surface and have an adverse effect on micromechanical retention. Farahat et al. (2022) found that SDF/KI treatment decreased the shear bond strength of an etch and rinse adhesive, and bonding performance of a universal adhesive was less influenced by SDF/KI treatment [8]. Other studies have reported, however, that bonding did not significantly decrease with KI or that KI resulted in higher bond than SDF alone [10,11]. The differences suggest that the behavior of SDF/KI can't be extrapolated to any other adhesive material and dentin substrates.
To provide a universal adhesive that would ease clinical bonding procedures and could be used in self-etch, etch-and-rinse or selective-enamel-etch methods, universal adhesives were developed. Most universal adhesives include functional acidic monomers, e.g., 10-methacryloyloxydecyl dihydrogen phosphate, that can chemically react with remnant hydroxyapatite. The performance of these may vary, however, due to differences in acidity, solvent content, hydrophilicity, monomer composition, filler content and instructions for use. Thus, universal adhesives cannot be considered as an entity of one type, especially when used on chemically modified or carious dentin [12].
The self-etch approach may be beneficial for bonding to caries-affected dentin as it does not require aggressive phosphoric-acid demineralization of a weakened dentin. It may also contain some hydroxyapatite still around the collagen fibrils so that functional monomers can interact chemically with the hydroxyapatite which remains. However, the water content and mineral distribution of carious dentin is not evenly distributed which can negatively affect solvent evaporation and adhesive polymerization. Universal adhesives failed to these materials bonded to caries-affected dentin in both etching methods. This discovery underlines the need to consider the bond strength of a product as it ages [13].
Nanoleakage assessment provides further data on submicroscopic porosities in the hybrid and adhesive layers. Silver-nitrate tracers can penetrate water-filled areas, areas of non-infiltrated collagen, and defects, even those without visible marginal gaps. An increase in nanoleakage can be due to insufficient monomer penetration, presence of water, lack of full polymerization or hydrolytic degradation. Thus, a material may show good immediate bond but contain significant nanoleakage which may affect durability [14].

Most previous studies have concentrated on sound dentin, primary dentin, one adhesive system or bond-strength immediately after application. Other studies tested new universal adhesive-composite systems instead of glass-

Vol 14, No 2 (2026) DOI 10.5195/d3000/2026.1579

ionomer cement (GIC) [11,15,16]. While some studies have focused on specific components of SDF/KI-treated dentin, there are still only limited data available on the effect of pretreatment, adhesive formulation, and aging on mechanical properties, nanoleakage, failure pattern, and microscopic integrity of the dentin–adhesive interface in carious permanent dentin. Therefore, the aim of the present study was to compare the bonding efficacy of two modern universal adhesives in bonding to untreated, SDF-treated and SDF/KI-treated caries-affected dentin. The immediate and aged microtensile bond strength, failure-mode distribution, nanoleakage and the resin–dentin interfacial morphology was analyzed. The combination of these results was designed to give a better picture of the influence of SDF and KI on the mechanical durability and structural quality of the adhesive interface.

## Material and Methods

### Study Design

This study was a 3 factor in vitro randomized controlled study. The independent variables were dentin pretreatment (untreated caries-affected dentin, 38% silver diamine fluoride [SDF] and 38% SDF and potassium iodide [KI]); universal adhesive (Scotchbond Universal Plus and Clearfil Universal Bond Quick); and evaluation period (immediate and after artificial aging.

Microtensile bond strength (µTBS) was the main outcome. Secondary outcomes assessed were nanoleakage, failure-mode distribution and microscopic integrity of the resin–dentin interface. The individual tooth was taken as the main experimental unit with beams within the same tooth being seen as clustered observations.

### Ethical Approval

The experimental protocol was presented and approved by the Institutional Research Ethics Committee at Gilgamesh University on 14 June 2025, with approval number 17. All teeth were harvested with written informed consent for the use in scientific research. No teeth were intentionally removed for this study. All procedures were carried out with following the Declaration of Helsinki and following the institutional rules of research using human biological materials.

### Sample-Size Calculation

An a priori sample size calculation was accomplished using the software G*Power (Version 3.1.9.7, Heinrich Heine University, Düsseldorf, Germany). A 3 × 2 × 2 design was used in the calculation: three dentin pre-treatments, two universal adhesives, and two evaluation conditions. To prove that the two measurements are different using an effect size of f = 0.30, a two-sided significance level of 0.05, and a statistical power of 80%, a minimum sample size of 108 teeth (n = nine teeth per experimental condition) was required. The sample size was expanded by about 10% to account for some sample loss that might occur during sectioning, handling, aging and mechanical testing. Thus, the 120 teeth were divided equally between the six treatments of pretreatment–adhesive (20 teeth per treatment). Samples from each tooth were then assigned to immediate or artificial aging. Each tooth was treated as the experimental unit and the statistical analysis accounted for the grouping of several beams in each tooth.

### Collection and Selection of Teeth

A total of 120 human permanent third molars with good outline and good condition were retrieved. Teeth were extracted to correct their position, for periodontal, or for surgery purposes but not for the present study.

Blood, calculus and residual soft tissues were excised immediately after extraction with periodontal curettes. The teeth were inspected at ×20 using a stereomicroscope for the presence of cracks, restorations, developmental defects, carious lesions or structural defects.

Teeth with intact crowns, no caries, no restorations and no structural abnormalities were included. Teeth with enamel hypoplasia, fluorosis, severe occlusal wear, prior bleaching, cracks penetrating the dentin or other developmental defect were excluded.

Selected teeth were disinfected in 0.5% chloramine-T solution for seven days and then kept in distilled water at 4°C. All teeth were used within three months after extraction, and the storage solution was changed every week until that time. The teeth were kept for about two hours at room temperature prior to processing.

### Preparation of Dentin Surfaces

The roots were cut off about 2 mm below the cemento-enamel junction with a water-cooled, low-speed diamond saw. Crowns were set in cylindrical moulds filled with self-curing acrylic resin with the occlusal surfaces exposed.

Using a precision diamond saw, the occlusal enamel was removed at a right angle to the long axis of each tooth under water irrigation. Further cutting was done as needed to reveal a flat dentin mid-coronal surface. The prepared surfaces were checked with a stereomicroscope to ensure that no enamel was left and no pulp was exposed.

A standardized smear layer was created by polishing each dentin surface with wet 600-grit silicon-carbide paper for 60 seconds. After every five specimens a new area of abrasive paper was used. These were then washed with distilled water for 30 seconds to wash away loose debris.

### Preparation of Artificial Caries-Affected Dentin

Chemical pH-cycling model was used to obtain standardized caries-affected dentin. This model was chosen because it allows for partial demineralization but does not completely remove the dentinal collagen matrix.

The demineralizing solution consisted of 2.2 mmol/l calcium chloride, 2.2 mmol/l sodium dihydrogen phosphate and 50 mmol/l acetic acid. The pH was brought to 4.8 with KOH. The remineralizing solution consisted of calcium chloride (1.5 mmol/L), sodium dihydrogen phosphate (0.9 mmol/L), potassium chloride (0.15 mol/L) and had a pH of 7.0.

Each specimen was individually immersed in 20 mL of demineralizing solution for eight hours. It was then washed with deionized water and put into 20 mL of remineralizing solution for 16 hours. This cycle was repeated every day for 14 days at 37°C.

New solutions to demineralize and remineralize prepared and changed every day. Following the last cycle, the specimens were thoroughly rinsed for 60 seconds with deionized water and lightly dried with paper towels.

### Verification of Dentin Demineralization

A surface microhardness testing confirmed the effectiveness of the artificial caries model. Three Knoop indentations were made on representative dentin surfaces with a 25g load applied for 10 sec. The indentations were spaced at least 100 µm apart to ensure that there was no interaction between the adjacent measurements.

The average of the 3 measurements was taken as the microhardness of the surface of each evaluated specimen. The results obtained were compared to baseline values in untreated sound dentin. The specimens that did not show sufficient or excessive softening that was inconsistent with the caries affected dentin were removed and replaced before randomization.

### Randomization and Blinding

All specimens were numbered after they were artificially carious. Randomization sequence was computer-generated.

Randomization was done by an investigator not involved in the preparation of specimens, application of adhesives, mechanical test, microscopic examination and statistical analysis. The examiners for bond strength testing, nanoleakage analysis, failure mode classification and microscopic assessment were blinded to group allocation.

Specimens were allocated to the following:

| Group | Dentin pretreatment | Universal adhesive | Number of teeth |
|---|---|---|---|
| C-SBU | Untreated control | Scotchbond | 20 |

Vol 14, No 2 (2026) DOI 10.5195/d3000/2026.1579

| | | | |
|---|---|---|---|
| | | Universal Plus | |
| C-CUQ | Un-treated control | Clearfil Universal Bond Quick | 20 |
| SDF-SBU | 38% SDF | Scotch-bond Universal Plus | 20 |
| SDF-CUQ | 38% SDF | Clearfil Universal Bond Quick | 20 |
| SDF/KI-SBU | 38% SDF followed by KI | Scotch-bond Universal Plus | 20 |
| SDF/KI-CUQ | 38% SDF followed by KI | Clearfil Universal Bond Quick | 20 |

The dentin surfaces were cleansed with deionized water followed by gentle drying with absorbent paper prior to pretreatment. The surfaces were not thoroughly dried.

**Untreated Control Groups**

Dentin surfaces were not treated with SDF or KI. The caries affected dentin was rinsed and blotted with deionized water for 30 seconds. The assigned universal adhesive was subsequently applied right away.

**SDF Groups**

An entire caries-affected dentin surface was treated with a 38% SDF solution from the silver capsule of the Riva Star system, using a disposable microbrush. The solution was rubbed over the surface for 60 seconds.

Absorbent paper was used to remove excess solution. This was followed by rinsing in deionized water for 30 seconds to remove loosely attached material and gently drying with a tissue. In these groups, KI was not used.

**SDF/KI Groups**

The caries-affected dentin surface was treated with the same 38% SDF solution and actively rubbed for 60 seconds. A separate disposable microbrush was then used to apply the KI solution immediately after.

The application of KI was continued until the initially creamy-white precipitate became clear. The treated surface was then rinsed for 30 seconds with deionized water and dried with a soft paper. The universal adhesive used was immediately applied after the pretreatment.

To reduce variations resulting from differences in SDF formulation, the SDF component of the same commercial system was used in both the SDF and SDF/KI groups.

**Adhesive Application**

Two universal adhesives were tested, Clearfil Universal Bond Quick and Scotchbond Universal Plus. The materials were used in the self-etch technique, to prevent further demineralisation of the carious dentin by phosphoric acid.

**Scotchbond Universal Plus**

A new disposable microbrush was used to apply Scotchbond Universal Plus to the dentin surface. The surface was actively rubbed with the adhesive for 20 seconds.

Clean and oil-free compressed air was used to gently air dry the adhesive layer for about five seconds until the solvent had dried off and the adhesive film was not moved. This adhesive was then light polymerised for 10 seconds.

**Clearfil Universal Bond Quick**

The dentin surface was prepared with Clearfil Universal Bond Quick by rubbing it with a disposable microbrush. The adhesive was immediately dried in the air with slight oil-free air to obtain the thin, uniform and immovable adhesive film. Then it was light polymerized for 10 seconds as per the manufacturer's instructions.

The light-curing tip for both adhesives was placed perpendicularly to and as close as possible to the bonded surface. The output of the LED light-curing unit was determined after every five specimens with a calibrated dental radiometer. The intensity was kept at a minimum of 1000 mW/cm² during the whole of the restorative procedures.

**Composite Build-Up**

The adhesive polymerization was followed by the application of a standardized silicone mould to the bonded dentin surface and by injection of a nanohybrid resin composite. The composite build-up was built up in 3 horizontal layers around 2 mm thick. Light polymerization was done for 20 seconds per increment. Once the silicone mould was removed, the buccal, lingual, mesial and distal surfaces were also light-polymerized for 20 seconds per surface. Standardized composite block was produced on each dentin surface (approx. 6 mm high). All adhesive and restorative work was carried out by one trained operator to minimized operator related variation. The specimens were then placed in deionized water at 37C for 24 hours, prior to sectioning.

**Preparation of Resin–Dentin Beams**

Each tooth was mounted in the precision cutting device and then cut longitudinally in the mesiodistal direction with a water-cooled diamond saw. The specimens were turned 90° and re-sectioned in the buccolingual plane.

Multiple resin–dentin beams of approximately 1 mm² were created using this procedure. All the width and thickness of the beams were determined with a digital caliper to 0.01 mm.

These beams were excluded if they had residual enamel, pulp horns or visible structural defects or irregular adhesive areas. Micro tensile bond strength tests were conducted on central beams, and nanoleakage tests on peripheral beams.

The source of each beam was noted and beams from the same tooth were not considered to be independent biological samples. Failure of samples to complete the cutting/handling cycle before failure occurred was noted for each experimental group.

The beams obtained from each tooth were randomly allocated to either immediate evaluation or artificial aging using a computer-generated randomization sequence.

All beams with each pretreatment–adhesive combination were assigned to immediate or aged evaluation.

**Immediate Evaluation**

Specimens designated for immediate testing were kept in deionized water at 37°C for 24 hours and then assessed.

**Artificial Aging**

Artificial aging specimens were put through 10,000 cycles of temperature changes between water baths at 5°C and 55°C. The duration of the baths was 30 seconds, and the transfer time was about 10 seconds.

The specimens were then thermally cycled and kept in deionized water at 37ºC for 6 months. Water was changed weekly for the whole storage period. Thermal cycling and extended water storage was designed to induce thermal stress, water sorption and polymer plasticization and hydrolytic degradation at the adhesive interface.

**Microtensile Bond-Strength Testing**

The resin–dentin beams were glued to a microtensile testing jig with cyanoacrylate adhesive. The bonded interface was set at a right angle to the tensile loading.

A universal testing machine was used to mount the jig. All the tensile tests were conducted at crosshead speed of 0.5 mm/min until failure. Maximum load to failure is in Newtons.

The equation used to calculate the microtensile bond strength was:

μTBS (MPa)=Maximum load at failure (N)Bonded area (mm2)\mu TBS\;(\mathrm{MPa})= \frac{\mathrm{Maximum\ load\ at\ failure\ (N)}}{\mathrm{Bonded\ area\ (mm^2)}}

The mean value of beams having no defects was computed and was deemed to be the microtensile bond-strength value for each experimental unit.

**Failure-Mode Analysis**

After microtensile tests, the fracture surfaces were viewed with a stereomicroscope by 40× magnification. The failure modes were grouped into 4 categories:

Adhesive failure: fracture that takes place at the resin–dentin interface.

Vol 14, No 2 (2026) DOI 10.5195/d3000/2026.1579

Cohesive failure of dentin: fracture that takes place mainly in dentin.
Cohesive failure of composite: fracture that happens primarily in the resin composite.
Mixed failure: failure at the adhesive interface and one or both adjacent substrates.

Samples were broken in each group and a few samples were mounted on aluminum stubs, sputter coated with gold, and viewed on a scanning electron microscope. Failure classifications were confirmed with this examination and exposed dentin, residual adhesive, remnants of composite material, resin tags and deposits containing silver were identified.

Three fractured beams were randomly selected from each tooth for failure-mode analysis under a stereomicroscope at ×40 magnification. Therefore, 60 beams were evaluated for each pretreatment–adhesive–aging condition. Because multiple beams originated from the same tooth, the beam-level observations were treated as clustered data, with the tooth considered the primary experimental unit.

**Nanoleakage Assessment**

Ten teeth from each pretreatment–adhesive group were selected for nanoleakage assessment. Two peripheral resin–dentin beams were obtained from each selected tooth. One beam was allocated to immediate evaluation, whereas the second beam was subjected to artificial aging before evaluation. Accordingly, 10 beams representing 10 independent teeth were assessed for each pretreatment–adhesive–aging condition. The tooth, rather than the individual beam, was considered the experimental unit. Two coats of nail varnish were applied over each beam leaving about a 1 mm border around the resin–dentin interface uncovered. All the specimens were kept in 50% ammoniacal silver nitrate solution for 24 hours in total darkness. These were then washed with deionized water and placed in a photographic developing solution for 8 hours in the presence of a fluorescent light. This process transformed the silver ions into metallic silver particles, and the rest of the process involved identifying and measuring the resulting particles.

The specimens were first placed in epoxy resin and then were polished sequentially with silicon-carbide abrasive papers and diamond polishing suspensions. They were then polished and ultrasonically cleaned, dried, placed on aluminum stubs and then carbon coated.

Backscattered scanning electron microscopy was used to study the bonded interfaces. The cervical, central and peripheral regions of each interface were captured to get three standardized images.

The area occupied by the silver was quantified using image-analysis software and reported as a percentage of the total area evaluated:

Nanoleakage (%)=Silver-occupied interfacial areaTotal evaluated interfacial area×100\mathrm{Nanoleakage}\;(\%)= \frac{\mathrm{Silver\text{-}occupied\ interfacial\ area}} {\mathrm{Total\ evaluated\ interfacial\ area}} \times100

The average value of the three measurements was taken as the nanoleakage value for each tested beam.

**The Interfacial Morphology is evaluated by using the following method**

Resin-dentin interfaces being perpendicular to the resin-dentin surface, representative specimens were taken from each group and divided into sections. The cleaned surfaces were buffed with a series of silicon carbide papers and then diamond polishing suspensions.

The polished specimens were dipped in phosphoric acid for 10 seconds, washed, and then placed in sodium hypochlorite for 5 minutes. They were then rinsed, dehydrated by successive additions of ethanol (from 50 to 95 %), dried, put on aluminum stubs and coated in gold by the sputtering method.

The scanning electron microscopy was employed to evaluate:

•Continuity and thickness of the adhesive layer.
The uniformity of hybrid layer.
Presence and length of resin tags.
The precipitation of silver-containing precipitates.
Gaps and voids at the interface.
Incomplete adhesive infiltration areas:
Artificial aging of the product leads to morphological changes.

Integrity at the interfaces was assessed based on the following scheme of scoring:

| Score | Interpretation |
|---|---|
| 0 | Continuous interface with no visible gaps |
| 1 | Minor isolated defects affecting less than 25% of the interface |
| 2 | Moderate defects affecting 25–50% of the interface |
| 3 | Extensive gaps or discontinuities affecting more than 50% of the interface |

All the microscopic images were evaluated independently by two masked observers. Disagreements were settled by mutual discussion and agreement.

**Examiner Training and Calibration**

Prior to the definitive assessments both examiners participated in a calibration session with images that were not part of the final analysis. A sample of 20 randomly selected images was assessed twice, two weeks apart.

The intra-examiner and inter-examiner agreement for failure-mode classification and interfacial scores was examined using the Cohen's weighted kappa coefficient. A kappa score ≥ 0.80 was deemed acceptable.

Intraclass correlation coefficient (ICC) was used to assess the repeatability of nanoleakage measurements. Intraclass correlation coefficient (ICC) ≥ 0.90 was deemed as excellent reliability.

**Statistical Analysis**

IBM SPSS Statistics, version 29 was used for statistical analysis. Normality of continuous variables were checked by Shapiro–Wilk test and visual inspection of Q–Q plots. Levene's test was conducted to check for homogeneity of variance.

Linear mixed-effects models were used to analyze microtensile bond strength and nanoleakage. Fixed effects were used for dentin pretreatment, type of adhesive, aging condition and their interactions. Multiple beams were obtained from the same tooth, so the tooth was included as a random effect.

Tukey adjusted pairwise comparisons were then used to further investigate significant main effects and interactions. Results were presented as mean, SD, estimated marginal mean differences, 95% confidence interval and p-values. Partial eta squared was used to express effect sizes.

Failure-mode distributions were reported descriptively as frequencies and percentages. Differences among experimental conditions were evaluated using multinomial regression with cluster-robust standard errors to account for multiple beams obtained from the same tooth. The tooth was considered the primary experimental unit.

Interfacial-integrity scores were analyzed using ordinal regression or non-parametric tests, as appropriate.

Pearson's correlation coefficient was used to assess the correlation between microtensile bond strength and nanoleakage, when both variables met parametric assumptions. When these assumptions were not fulfilled, Spearman's correlation coefficient was used.

The tests were all two-sided, and the level of statistical significance set at $p < 0.05$. The statistical analysis was of the individual tooth as the primary experimental unit.

## Results

**Specimen Distribution and Preliminary Analysis**

120 teeth underwent the restorative-aging process. A few of the beams were lost in sectioning and handling but every tooth gave at least 3 valid beams for microtensile testing. Because of this, no tooth was omitted from the main analyses. A higher failure rate was seen in the SDF-treated groups particularly following artificial aging.

Vol 14, No 2 (2026) DOI 10.5195/d3000/2026.1579

The Shapiro–Wilk test and the Q–Q plot indicated that the continuous variables were approximately normally distributed. There was no significant departure from the homogeneity of variance in the Levene's test. Hence, the main analyses were performed using parametric mixed-effects models.

**Microtensile Bond Strength**

The immediate and aged µTBS values are given in Table 1. The greatest immediate bond strength was obtained with untreated caries-affected dentin bonded with Clearfil Universal Bond Quick (mean 34.1 ± 4.2 MPa). This was followed by the untreated Scotchbond Universal Plus group, which showed a mean value of 32.8 ± 4.4 MPa.

Immediate bond strength with both adhesives was significantly decreased when SDF was applied alone. Immediate mean value was lowered to 24.6 ± 3.9 MPa with Scotchbond Universal Plus and 27.2 ± 4.0 MPa with Clearfil Universal Bond Quick. However, bonding performance was partially retained after application of KI (29.8 ± 4.1 MPa for Scotchbond Universal Plus, 31.5 ± 4.0 MPa for Clearfil Universal Bond Quick Microtensile bond strength was significantly reduced in all groups after the artificial aging. The most significant deterioration was seen in the groups that only received SDF. The mean bond strength for Scotchbond Universal Plus and Clearfil Universal Bond Quick was 17.9 ± 3.6 MPa and 20.4 ± 3.8 MPa, respectively, after aging. The mean values of the aged SDF/KI groups were 24.8 ± 3.9 and 26.7 ± 3.7 MPa, respectively, in comparison.

In all pretreatment and aging conditions, bond-strength values for Clearfil Universal Bond Quick were slightly higher than those for Scotchbond Universal Plus. The overall adhesive effect was not statistically significant, but less profound than for pretreatment and aging. The most significant factorials were analyzed to determine which had the greatest influence on bond strength.

All the main effects of the mixed-effects analysis were significant for dentin pretreatment, adhesive type, and aging condition. Artificial aging and dentin pretreatment were found to have the largest effect on microtensile bond strength.

There was a significant interaction between dentin pretreatment and aging. This indicated that the level of degradation due to aging was dependent on the pretreatment used. This was more pronounced in SDF treated specimens than in the untreated and SDF/KI treated specimens.

There was no difference between the pretreatment by adhesive type, and the pattern of each of the three was similar in the overall pattern. The three-way interaction between pretreatment, adhesive type and aging was not significant.

The notable pretreatment by-aging interaction was mainly due to the higher percentage decrease seen for the SDF-treated product. The reductions in the control and SDF/KI groups were about 15–17% after aging while SDF alone reduced by about 25–27% after aging.

These results showed that not only did KI enhance the immediate bond-strength values, it also enhanced the durability of the bond strength. It also minimized the extent of water storage and thermal cycling induced deterioration.

**Nanoleakage**

The internal leakage, "nanoleakage," varied significantly in different pre-treatment, adhesive type, and aging condition. Dentin that was not treated immediately had the least penetration of silver nitrate. The highest immediate nanoleakage was obtained with SDF alone, while the intermediate value was obtained with SDF/KI treatment.

The level of nanoleakage in the specimens bonded with Scotchbond Universal Plus was raised from 12.4 ± 2.8% in the control group to 25.8 ± 4.1% after SDF application. This was lowered to 17.1 ± 3.2% after further KI treatment. The same pattern was seen when testing with Clearfil Universal Bond Quick.

All the experimental groups showed a significant increase in the nanoleakage after artificial aging. The highest aged nanoleakage value was obtained for SDF specimens bonded with Scotchbond Universal Plus (39.7 ± 5.0%). Clearfil Universal Bond Quick had a value of 36.5 ± 4.6% for the corresponding value.

SDF was found to significantly raise nanoleakage in comparison to untreated dentin ($p < 0.001$) in pairwise comparisons. The use of KI following SDF also greatly decreased the silver penetration when compared to the use of SDF alone ($p < 0.01$). But the SDF/KI specimens still showed a higher amount of nanoleakage compared to untreated controls.

There was a significant negative correlation between bond strength and nanoleakage ($r = -0.72$, $p < 0.001$). The greater the penetration of silver nitrate, the lower the microtensile bond strength. This relationship was especially true for the elderly SDF-only age groups.

**Failure-Mode Distribution**

There was a significant difference in failure modes between dentin pretreatments ($p < 0.05$). Mixed failures were the most common type in the immediate untreated groups as they had relatively high bond-strength values. Adhesive failures occurred more often after SDF application, suggesting the site of fracture was the resin–dentin interface.

The frequency of adhesive failures was higher in all groups in the artificial aging. The greatest percentage of adhesive failures was observed in the aged SDF-only groups. The SDF/KI groups, by contrast, exhibited fewer adhesive failures and more mixed failures than did the SDF-only groups.

**Interfacial Morphology**

The scanning electron micrograph showed that the resin–dentin interface morphology was different. Untreated specimens showed a relatively continuous layer of adhesive, and uniform adaptation to the caries affected dentin. The number of resin tags visible in the open tubules was variable since the substrate was demineralised, and the length of the resin tag was related to the irregular nature of the demineralised matrix.

In the SDF-only specimens, granular precipitates were seen around the surface of dentin and in the mouths of the dentinal tubules. Gaps were seen between the adhesive layer and underlying dentin at the localized areas. These defects were more pronounced post artificial aging.

Precipitates were better distributed and less compact in SDF/KI treated specimens. The adhesion layer was more continuous, overall, than was seen with SDF alone. However, this showed that there were still areas of voids and incomplete resin penetration.

The Scotchbond Universal Plus resulted in slightly thicker adhesive layer, while the Clearfil Universal Bond Quick resulted in a relatively thin and uniform layer. All the adhesives exhibited deterioration after aging, and in the case of these specimens, the deterioration was most pronounced at the interfaces of the SDF-only specimens.

**Examiner Reliability**

The weighted kappa values of intra-examiner for failure-mode and interfacial-score assessment were 0.88 to 0.93. The inter-examiner agreement was from 0.84 to 0.90. The ICC for repeated nanoleakage measurements was 0.94. These values were reproducible good-to-excellent.

## Discussion

The bonding effectiveness of two universal adhesives with and without SDF pretreatment and SDF/KI pretreatment to caries-affected dentin was evaluated in this study. The results demonstrated that SDF alone significantly decreased immediate and aged µTBS, caused more nanoleakage and resulted in increased interfacial defects and adhesive failures. Partially, these adverse effects were reduced by applying KI after SDF application.

The decrease in bond strength following SDF application could be due to the presence of silver precipitates on the dentin surface and inside the dentinal tubules. Surface wetting, penetration of adhesive-monomers and formation

Vol 14, No 2 (2026) DOI 10.5195/d3000/2026.1579

of hybrid-layers can be affected by these deposits. In line with this, earlier studies have demonstrated that bonding in response to SDF is concentration-, rinsing-, dentin-, adhesive-, and bonding strategy-dependent [8,17,18].

The bonding performance of the KI+SDF was significantly better than that of SDF alone. Silver ions in free form react with KI and produce precipitates of silver iodide. Application of KI and subsequent rinsing can remove easily detached reaction products and enhance adhesion to underlying dentin. But KI could not restore all of the performance of dental carious dentin. It is consistent with the findings that SDF treatment may affect bonding, which may be preserved or improved using KI depending on the substrate and restorative material [9,11,16].

The artificial aging gave a remarkable decrease in bond strength and a corresponding increase in nanoleakage for all groups. Repeated expansion/contraction stresses from thermal cycling can be generated at the bonded interface, while long term water storage can lead to polymer plasticization, resin hydrolysis, and collagen degradation. The greatest degradation was observed in the SDF-only groups, indicating that SDF-associated interfacial defects made these specimens more vulnerable to thermal and hydrolytic aging. The same degradation has been reported when used on caries-affected dentin using universal adhesives [13].

The mechanical results were confirmed by the nanoleakage findings. The silver-nitrate penetration was greater for SDF-treated specimens, especially following artificial aging. This means that there are regions with water rich material areas, incomplete resin impregnation, or microscopic pores in the hybrid and adhesive layers. SDF/KI resulted in a substantial reduction in nanoleakage as compared to SDF without a value like that of untreated dentin. The negative correlation between bond strength and nanoleakage further indicated that bond strength was inversely proportional to the nanoleakage and thus to the mechanical stability [19].

These findings were backed by failure mode and microscopic analyses. Mixed failure was more common in untreated specimens while the adhesive failure was more common after the application of SDF. The SEM images showed that the dentinal tubules were partially blocked, the precipitates were clustered, the resin was not continuous, and there were gaps between the dentinal tubules and resin in the SDF groups. The SDF/KI groups had more continuous adhesive layers and fewer large defects, which may be due to the rinsing process which removed some of the loose precipitates. This can ensure better adhesion of the composite to SDF treated dentin [18,19].

The clinical implications of these results are that direct adhesive placement over SDF treated dentin may affect the durability of the bond. The use of KI and proper rinsing of the treated surface could lead to better surface compatibility with universal adhesives but cannot restore the performance of untreated dentin. Since this is an in vitro study, clinical trials must be performed to establish whether there are any differences in the following: - retention of the restoration - marginal staining - recurrent caries - post-operative sensitivity - long term survival.

## Conclusion

- SDF significantly lowered the immediate and aged µTBS.
- The bonding performance was partially retained after applying KI after SDF.
- All groups were negatively impacted by artificial aging, with the largest decrease in bond strength found in the SDF-only groups.
- The SDF caused nanoleakage and adhesion failures to occur more frequently, while later application of KI helped alleviate these detrimental effects.
- The nanoleakage were lower in the SDF/KI groups than in the SDF-only groups.
- The overall performance of Clearfil Universal Bond Quick was marginally superior to Scotchbond Universal Plus.
- The nanoleakage was negatively correlated with bond strength.
- The SDF/KI groups were not entirely successful in achieving bonding to the caries-affected dentin.

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Table 1. Immediate and aged microtensile bond strength.

| Dentin pretreatment | Adhesive | Immediate µTBS, MPa | Aged µTBS, MPa | Reduction after aging |
|---|---|---|---|---|
| Control | Scotchbond Universal Plus | 32.8 ± 4.4 | 27.5 ± 4.1 | 16.2% |
| Control | Clearfil Universal Bond Quick | 34.1 ± 4.2 | 29.0 ± 3.9 | 15.0% |
| SDF | Scotchbond Universal Plus | 24.6 ± 3.9 | 17.9 ± 3.6 | 27.2% |
| SDF | Clearfil Universal Bond Quick | 27.2 ± 4.0 | 20.4 ± 3.8 | 25.0% |
| SDF/KI | Scotchbond Universal Plus | 29.8 ± 4.1 | 24.8 ± 3.9 | 16.8% |
| SDF/KI | Clearfil Universal Bond Quick | 31.5 ± 4.0 | 26.7 ± 3.7 | 15.2% |

*Values are mean ± SD, µTBS: Microtensile bond strength, SDF: Silver Diamine Fluoride, KI: Potassium Iodide. Pairwise tests showed that SDF alone resulted in significantly reduced bond strength compared to the untreated group when tested immediately and aged ($p < 0.001$). The SDF/KI groups had significantly higher values than the SDF-only groups ($p < 0.01$). However, the SDF/KI groups were still slightly lower than the untreated control groups, especially when aged.*

Table 2. Mixed-effects analysis of microtensile bond strength.

| Effect | F-value | *p*-value | Partial η² | Interpretation |
|---|---|---|---|---|
| Dentin pretreatment | 62.84 | <0.001 | 0.54 | Large effect |
| Adhesive type | 9.72 | 0.002 | 0.08 | Small-to-moderate effect |
| Aging condition | 88.21 | <0.001 | 0.45 | Large effect |
| Pretreatment × adhesive | 1.86 | 0.161 | 0.03 | Not significant |
| Pretreatment × aging | 6.41 | 0.003 | 0.10 | Significant interaction |
| Adhesive × aging | 0.74 | 0.392 | 0.01 | Not significant |
| Pretreatment × adhesive × aging | 0.58 | 0.562 | 0.01 | Not significant |

Table 3. Percentage nanoleakage at the resin–dentin interface.

| Dentin pretreatment | Adhesive | Immediate nanoleakage | Aged nanoleakage | Mean increase |
|---|---|---|---|---|
| Control | Scotchbond Universal Plus | 12.4 ± 2.8% | 22.6 ± 3.5% | 10.2% |
| Control | Clearfil Universal Bond Quick | 11.2 ± 2.6% | 20.4 ± 3.3% | 9.2% |
| SDF | Scotchbond Universal Plus | 25.8 ± 4.1% | 39.7 ± 5% | 13.9% |
| SDF | Clearfil Universal Bond Quick | 23.6 ± 3.8% | 36.5 ± 4.6% | 12.9% |
| SDF/KI | Scotchbond Universal Plus | 17.1 ± 3.2% | 27.9 ± 4.1% | 10.8% |
| SDF/KI | Clearfil Universal Bond Quick | 15.4 ± 3% | 25.6 ± 3.9% | 10.2% |

*Data are reported as mean ± SD.*

Table 4. Distribution of failure modes

| Group | Condition | Adhesive | Cohesive in dentin | Cohesive in composite | Mixed |
|---|---|---|---|---|---|
| Control–SBU | Immediate | 15 (25%) | 5 (8.3%) | 4 (6.7%) | 36 (60%) |
| Control–SBU | Aged | 23 (38.3%) | 3 (5%) | 3 (5%) | 31 (51.7%) |
| Control–CUQ | Immediate | 13 (21.7%) | 6 (10%) | 5 (8.3%) | 36 (60%) |
| Control–CUQ | Aged | 20 (33.3%) | 4 (6.7%) | 3 (5%) | 33 (55%) |
| SDF–SBU | Immediate | 35 (58.3%) | 2 (3.3%) | 2 (3.3%) | 21 (35%) |
| SDF–SBU | Aged | 42 (70%) | 1 (1.7%) | 1 (1.7%) | 16 (26.7%) |
| SDF–CUQ | Immediate | 32 (53.3%) | 3 (5%) | 2 (3.3%) | 23 (38.3%) |

Vol 14, No 2 (2026) DOI 10.5195/d3000/2026.1579

| | | | | | |
|---|---|---|---|---|---|
| SDF–CUQ | Aged | 39 (65%) | 1 (1.7%) | 2 (3.3%) | 18 (30%) |
| SDF/KI–SBU | Immediate | 23 (38.3%) | 4 (6.7%) | 3 (5%) | 30 (50%) |
| SDF/KI–SBU | Aged | 29 (48.3%) | 3 (5%) | 2 (3.3%) | 26 (43.3%) |
| SDF/KI–CUQ | Immediate | 20 (33.3%) | 4 (6.7%) | 4 (6.7%) | 32 (53.3%) |
| SDF/KI–CUQ | Aged | 26 (43.3%) | 3 (5%) | 3 (5%) | 28 (46.7%) |

Frequencies and percentages are presented at the beam level; inferential analysis accounted for clustering of beams within teeth.
*SBU: Scotchbond Universal Plus, CUQ: Clearfil Universal Bond Quick. 60 fractured beams were tested for each condition.*
*The stereomicroscopic results were backed up by the scanning electron microscopy of the fracture surfaces. Typically, untreated specimens had adhesive resin on both sides of the fracture, as per mixed failures. Surface precipitates, incompletely infiltrated tubules and areas of exposed dentin were observed in SDF treated specimens. The SDF/KI groups exhibited less dentin showing.*

Table 5. Interfacial-integrity scores.

| Dentin pretreatment | Adhesive | Immediate median (IQR) | Aged median (IQR) | Principal microscopic finding |
|---|---|---|---|---|
| Control | Scotchbond Universal Plus | 0 (0–1) | 1 (1–2) | Mostly continuous interface |
| Control | Clearfil Universal Bond Quick | 0 (0–1) | 1 (0–1) | Thin, uniform adhesive layer |
| SDF | Scotchbond Universal Plus | 2 (1–2) | 3 (2–3) | Precipitates and extensive gaps |
| SDF | Clearfil Universal Bond Quick | 2 (1–2) | 2 (2–3) | Discontinuous resin infiltration |
| SDF/KI | Scotchbond Universal Plus | 1 (1–2) | 2 (1–2) | Moderate isolated defects |
| SDF/KI | Clearfil Universal Bond Quick | 1 (0–1) | 1 (1–2) | Improved interfacial continuity |

*Score 0: No visible defects; Score 1: Defects affecting less than 25% of the interface area examined; Score 2: Defects affecting 25-50% of the interface area examined; Score 3: Defects affecting more than 50% of the interface area examined.*
*The dentin pretreatment and aging had a significant influence on interfacial scores ($p < 0.001$).* The SDF-only groups exhibited significantly higher interfacial-integrity scores than the untreated control groups, indicating more extensive interfacial defects ($p < 0.001$). *The groups that used SDF did not exhibit the same level of integrity at the interface as did the groups that used SDF in conjunction with other treatments but did achieve an improved level of quality.*