

## Effect of silver diamine fluoride and potassium iodide on bonding to demineralized dentin

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**ABSTRACT: Purpose:** To evaluate the microtensile bond strength (MTBS) of resin composite to demineralized dentin treated with SDF or SDF-KI. **Methods:** Occlusal enamel of 30 caries-free extracted human molars was removed to expose flat dentin surfaces. Specimens were immersed in demineralizing solution (pH 4.5) for 7 days. A standard smear layer was created using 600-grit silicon carbide paper. Specimens were distributed into three groups (n=10). Control: Dentin rinsed with deionized water; SDF: Dentin treated with 38% SDF; and SDF-KI: Dentin treated with SDF and KI. Specimens were bonded with composite using Scotchbond Universal in etch-and-rinse mode. MTBS beams were prepared from each specimen after 24 hours and tested. Data was analyzed by one-way ANOVA with Tukey HSD post-hoc test. **Results:** MTBS test results ranged from 0 to 40 MPa. The highest values were obtained in the Control and lowest in SDF-KI, where pre-test failures were frequently observed. One-way ANOVA showed a significant difference among groups ( $P < 0.005$ ); post-hoc analysis suggested no statistical difference between Control and SDF, but both groups showed higher MTBS compared with SDF-KI. (*Am J Dent* 2019;32:143-146).

**CLINICAL SIGNIFICANCE:** Silver diamine fluoride can be applied precisely and regionally on demineralized dentin before bonding without compromising the bond strength of a universal adhesive used with phosphoric acid etching. Addition of potassium iodide to reduce discoloration will dramatically weaken the bond.

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### Introduction

Silver diamine fluoride (SDF) is a caries-arresting agent that has recently gained popularity in the United States.<sup>1</sup> In 2014, the United States Food and Drug Administration approved its use as a dentin desensitizer due to regulatory requirements, but it has also been shown to be useful in treating caries.<sup>2</sup> In addition to its well-known application as a caries arresting-agent in difficult to treat cases, root caries and pediatric cases, the use of SDF has potential value in contemporary restorations, for such cases as deep dentin caries approaching the pulp. Complete caries removal and conventional preparation of such tooth structure could cause permanently compromised pulpal health. Essentially, desensitization and caries-arresting tendencies are both invaluable when excavating decay in a vital tooth. In addition to anti-demineralization effects on the mineral phase,<sup>3</sup> a recent study showed that SDF may have significant effects on dentin collagen, reducing its degradation in an acidic environment.<sup>4</sup> SDF has also demonstrated a highly effective antibacterial action against cariogenic biofilm. Considering all these benefits, SDF may be a viable choice as a cavity cleansing agent.

Many contemporary restorations rely on adhesive bonding to the remaining tooth structure after caries excavation and tooth preparation; which may involve demineralized dentin. According to the minimal invasive concept for restoration of

On the other hand, dentistry is esthetically driven and potential drawbacks from the SDF must be considered. One of the main disadvantages of SDF is its discoloration effect. The silver ions in the composition of SDF turn to metallic silver in the presence of light, resulting in a dark discoloration that can spread deep into the tooth.<sup>5</sup> Therefore, restoration of tooth structure with contemporary materials over SDF-treated surfaces may result in a dark hue emitting underneath restorative materials. One strategy is SDF application in combination with a saturated solution of potassium iodide (KI),<sup>6</sup> which might reduce the dark staining of SDF on teeth. The SDF-KI produces a visible opaque precipitate over the tooth surface. Therefore, SDF-KI may be applied to a demineralized surface to both reduce tooth discoloration and achieve the beneficial effects of SDF.<sup>7</sup> The usage of SDF or the SDF-KI combination have also been reported to reduce occurrences of both recurrent decay and root caries.<sup>8</sup> However, insufficient research has been done to evaluate the effects of both SDF and SDF-KI on the bond strength of a common adhesive, the most basic property when evaluating the quality of bond.

SDF application significantly affected bond strength to sound dentin under certain conditions.<sup>9</sup> The adhesives least affected by SDF were etch-and-rinse adhesives<sup>9</sup> or glass-ionomer cement.<sup>10</sup> However, sound dentin may not be the primary bonding substrate in many clinical applications when restoring a deeply carious tooth. Sound dentin is composed of

cavities with dentin involvement, caries affected dentin (CAD) may remain after removal of the infected tissue. CAD is moderately demineralized and despite some organic matrix changes, dentin is considered remineralizable. CAD is the clinical substrate for bonding in many clinical situations; however, the effects of SDF on bonding to demineralized dentin is not known.

about 50 percent by volume inorganic mineral content (mostly apatite and calcium phosphates), and the rest organic matrix (mostly type-1 collagen) and water.<sup>11</sup> On the other hand, within a carious lesion, there are multiple layers of variously infected and damaged dentin. These layers are understood as a spectrum from the outermost caries-infected dentin (CID) filled with

bacteria and soft, heavily demineralized dentin to an inner layer of caries-affected dentin that has few bacteria and firm, less demineralized dentin.<sup>12,13</sup> CID can be easily removed and is very soft due to severe demineralization, and does not serve as a bonding substrate.<sup>14</sup> The collagen matrix is severely altered and its cross-linking destroyed; there are bacterial protein deposits, and the damage is considered irreversible.<sup>15</sup> In the inner layer of a carious lesion, caries-affected dentin (CAD) has moderate demineralization, collagen fibrils maintain their triple-helical shape, and there is reversible loss of some collagen cross-linking.<sup>11,16</sup> CAD is considered to be the main bonding substrate on the pulpal floor in contemporary and minimally invasive composite restorations, which if not removed may preserve the health of pulpal tissue.<sup>15</sup> However, bonding to CAD is weaker than bonding to sound dentin.<sup>17</sup>

With the caries-arresting properties of SDF and SDF-KI, no study has reported on the effect of SDF and SDF-KI on bond strength of adhesives to demineralized dentin or CAD. Whether bond strength is increased, unchanged, or decreased by their application may guide future clinical decisions. This study examined if treating demineralized dentin with SDF or SDF-KI prior to bond dental adhesive has any influence on the microtensile bond strength (MTBS) of the adhesive. The null hypothesis was that there was no difference in MTBS to demineralized dentin with or without pretreatment using SDF and SDF-KI.

### Materials and Methods

A total of 30 human extracted caries-free posterior teeth with no prior restorations were collected from oral surgery clinics in the greater Seattle area. The de-identified extracted teeth were cleaned from organic debris and stored in 0.1 wt% Thymol solution at 4°C for less than 6 months before the experiment. The use of human teeth in this study followed the ethical guidelines set by the University of Washington Human Subjects Division and the Declaration of Helsinki.

**Preparation of specimens** - Teeth were prepared by embedding the roots below the cemento-enamel junction (CEJ) in 10 mm × 10 mm blocks filled with fast-setting acrylic resin (CureGrace<sup>a</sup>) to provide a handle for manipulation. The occlusal enamel was then removed using a low-speed diamond saw (Isomet<sup>b</sup>) under running water to expose a flat dentin surface. The dentin surfaces were smoothed with 600-grit silicon carbide (SiC) paper<sup>c</sup> under wet conditions for 10 seconds.

Each specimen was then immersed in 15 mL of a demineralizing solution (containing 1.5 mM of CaCl<sub>2</sub>, 0.9 mM

Table. Microtensile bond strength (MPa) to demineralized dentin.

Group	Control	SDF	SDF-KI
MTBS Mean (SD)	23.5 (10.7)	19.8 (8.4)	7.9 (6.6)*
MTBS Range	12.5 - 40.4	8.1 - 33.8	2.4 - 18.5
MTBS with PTF = 0	21.2 (11.4)	18.5 (9.5)	1.5 (2.6)*
MTBS Range with PTF (PTF beam%)	7.6 - 40.4 (15.5%)	4.1 - 33.8 (10%)	0.0 - 5.9 (77.5%)

\* In each MTBS row, values marked by an asterisk are statistically significantly different from other (one-way ANOVA, P= 0.005).

The range presents range of averaged values per tooth

SD- Standard deviation; MTBS-Microtensile bonding strength; PTF-Pre-test failures

A universal bonding agent (Scotchbond Universal<sup>1</sup>) in total-etch mode with 37% phosphoric acid was used for the MTBS test, following the recommendation of Lutgen et al.<sup>18</sup> After application of the bonding agent to the treated dentin surfaces was completed, a 2 mm-high composite build-up (Clearfil Majesty, <sup>5</sup> Shade A2) was placed over the cured bonding agent.

**Microtensile bond strength test (MTBS)** - Specimens were stored in DI water for 24 hours at 37°C. Following their storage, several MTBS beams (approximately 0.7 mm × 0.7 mm in dimensions) were produced from each of the coronal surfaces using a low-speed diamond saw. Four beams were randomly selected which most closely approximated 0.7 mm × 0.7 mm parameters, and tested immediately. The beams were fixed on the MTBS jig using a cyanoacrylate glue (Model Repair II<sup>3</sup>) in a compact testing machine (Bisco Micro-tensile Tester<sup>1</sup>) to obtain load bearing force values at breakage for each beam. After testing, the dentin portion of each fractured beam was retrieved to measure its dimensions using a micrometer caliper.<sup>1</sup> In order to convert the force values (N) to bond strength (MPa), the force was divided over the surface area of bonding for each beam.

**Statistical analysis** - The force values of the four central beams from each tooth were averaged to obtain one strength value used for its data point. If the central MTBS beam fractured at the adhesion interface during preparation and before the test (pre-test failures, PTF), they were valued as 0 MPa. The resulting MTBS values were treated following two different approaches, with and without PTF values included.

**Scanning electron microscopy (SEM) imaging** - The adhesive interface in each group was observed under SEM. Additional teeth (n= 6) were sectioned using the diamond saw to create a 2 mm-thick coronal dentin slab. Three of the slabs were subjected

of  $\text{KH}_2\text{PO}_4$ , 50 mM of acetic acid, pH value adjusted to 4.5 using NaOH) at 37°C for 7 days to create artificial demineralized dentin, according to a protocol previously established.<sup>18</sup> Afterwards, the demineralized dentin surface was lightly polished using 600-grit silicon carbide paper to create a standard smear layer without removing the demineralized dentin layer.

**Experimental groups** - The demineralized dentin surfaces were distributed into three groups (n=10); Control: dentin rinsed with deionized water; SDF: A drop of 38% SDF (Advantage Arrest<sup>d</sup>) was applied using a microbrush and rinsed off after 1 minute; SDF-KI: dentin treated with SDF, then a saturated solution of KI,<sup>e</sup> made of 7,100 mg KI in 5 ml deionized water applied using a microbrush until white precipitate formed, and finally rinsed off after 1 minute.

After the bonding agent application, three of the discs were subjected to demineralization as described above. After surface treatment according to the experimental groups in the study, 2 mm-thick composite buildup was placed on dentin discs following the protocol used for the MTBS test. Specimens were stored in deionized water at 37°C for 24 hours. After that, the specimens were cross-sectioned through the bonded interface using the diamond saw and embedded in slow-setting epoxy resin (Total Boat<sup>h</sup>). The embedded cross-sections were polished sequentially using #400, #600, #800, #1,000, #1,500 and #2,000 SiC papers followed by diamond pastes with particle sizes of 6, 3, 1, 0.25  $\mu\text{m}$ , gold-sputter coated and observed at  $\times 1,500$  under 10kV SEM.<sup>3</sup>

## Results

MTBS test results are presented in the Table. The highest

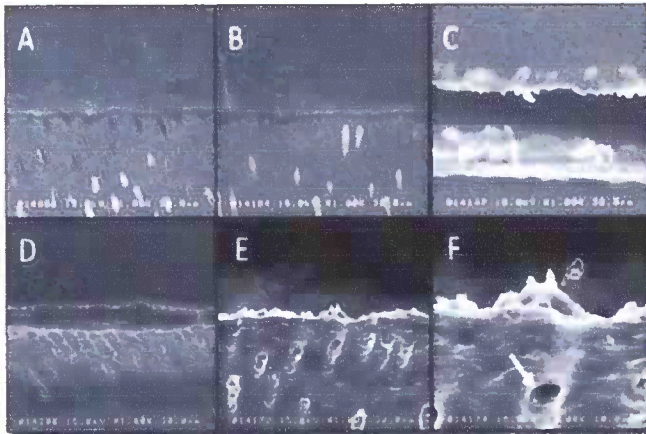


Figure. SEM micrographs of the adhesive interface. **A.** Sound dentin, dentin tubules are widened by etching and resin tags have formed. **B.** Sound dentin treated with SDF. Resin tag formation into tubules is not as prominent as in (A) but adhesive interface appears intact. **C.** Sound dentin treated with SDF-KI. Note that the composite has separated while some adhesive remnants remain on dentin. **D.** Demineralized dentin. A thick zone of demineralized dentin is distinctly observed under the adhesive. **E.** Demineralized dentin treated with SDF-KI, etchings effect is not observed in contrast to D. **F.** Higher magnification of E (Demineralized dentin with SDF-KI). SDF-KI precipitate on dentin surface (Hand pointer); Arrow: patent demineralized dentin tubule close to the interface has no resin tag.

values were obtained in the Control and the lowest in SDF-KI, where pre-test failures were frequently observed. One-way ANOVA showed a significant difference among groups ( $P < 0.005$ ); post-hoc analysis suggested that while there was no statistical difference between Control and SDF, both groups were significantly different from SDF-KI. When PTF beams were included, the trend was similar. The majority (77.5%) of beams in the SDF-KI group failed at the adhesive interface during preparation.

**SEM** - SEM images for both sound dentin and CAD bonded to composite were collected to compare interfaces. Sound dentin with no SDF or KI on its surface was bonded as a standard, showed typical presentation of resin tag formation on etched dentin and a thin bond layer ( $\pm 5 \mu\text{m}$ ) from a single application

challenging task. In the current study, demineralized dentin was achieved artificially through a previously established protocol.<sup>22</sup> Artificial demineralized dentin allowed better control of both the extension and depth of carious lesions across all 30 specimens. This protocol resulted in artificial demineralized dentin that was similar in intertubular nanoindentation hardness<sup>16</sup> and mineral density<sup>21</sup> with natural CAD up to the depth of around 150  $\mu\text{m}$ . Although natural CAD was softer than artificially demineralized dentin beyond this depth, the microtensile bond strength of various adhesives was similar between the artificial demineralized dentin and natural CAD.<sup>21</sup>

The more demineralized dentin is, the weaker the collagen fibrils become due to the gradual loss of collagen cross-linkers, resulting in lower cohesive strength of the dentin substrate.<sup>18</sup> Moreover, both natural and artificial demineralized dentin have been shown to have significantly lower hardness when compared with sound dentin.<sup>15</sup> Bond strength of adhesives is strongly affected by the mechanical properties of the substrate. All these differences explain the lower bond strength values to demineralized dentin that have been consistently reported in the literature.<sup>17,20</sup> However, the lower bond strength to demineralized dentin is thought to have less clinical importance if the margins of prepared teeth are on sound dentin and enamel, which is achievable in many restorations.<sup>19</sup>

A previous study<sup>22</sup> showed that SDF has weakening effects on bond strength of the universal adhesive involving sound dentin; SDF is a basic solution by design with a pH of around 10 according to the manufacturer. Therefore, it may significantly negate the acidity of an ultra mild self-etching adhesive such as the SBU with a pH of 2.7.<sup>23</sup> It was shown that when bonding to SDF-treated sound dentin, phosphoric acid etching prior to application of the adhesive improved the bonding performance when compared with the self-etch approach.<sup>3</sup>

In contrast to those reports on the negative effect of SDF in bonding to sound dentin, there was no significant difference between the control group and SDF to demineralized dentin in

of SBU (Fig., A). In Fig., B, sound dentin with SDF showed less resin tags in the dentin tubules comparatively. Sound dentin with SDF-KI showed a remarkable thickness of a precipitate, with separation between the adhesive layer and composite (Fig., C).

The control (Fig., D) presented a demineralized zone of dentin with multiple resin tags into the tubules. The SDF-KI group, which had the weakest bond, showed no adhesive layer on the dentin surface. Due to its fragility, the substrate bond failed prior to SEM imaging, revealing adhesive failures at both the composite and demineralized interfaces. (Fig., E and F).

### Discussion

Resin and dentin bond together through micro-mechanical adherence to exposed collagen fibrils and dentin tubules, as well as by chemical reaction of functional monomers with dentin substrate. CAD shows less mineral content and more water than sound dentin.<sup>16,19</sup> When bonding to natural CAD, mineral deposits in the demineralized dentin tubules may prevent penetration of resin tags.<sup>14</sup> The use of 32-37% phosphoric acid etching was suggested for natural CAD long ago; however, more recent literature has favored the self-etching approach.<sup>20</sup>

While the use of natural caries seems ideal for the study of bonding to CAD, standardizing natural caries would be a

this study. One explanation for this observation is that since the bonding to demineralized dentin is intrinsically limited by the substrate cohesive strength,<sup>24</sup> the potential adverse effects of SDF were not reflected. Different adhesives that show significant variations in bond strength to sound dentin as the substrate may show no significant difference in bond strength when demineralized dentin is the tested substrate.<sup>25</sup> However, it should be noted that SDF could have positive effects on preserving the dentin, since degradation of collagen at the interface is thought to affect the bonding interface over time.<sup>21</sup> SDF has shown a strong potential in the prevention of collagen matrix degradation, which is important in regards to the longevity of the deep demineralized dentin, since this substrate has little support for the organic phase from apatite. The silver nitrate of SDF binds to dentin forming yellow cubic crystals that are not rinsed away, thus altering the dentin substrate.<sup>11</sup>

Despite KI's ability to reduce the immediate staining effects of SDF, the null hypothesis was rejected because the bond strength of the SDF-KI group was significantly weaker than the other two groups. A large number of pre-test failures were observed in the SDF-KI group. This observation is uncommon with the contemporary adhesives tested using the MTBS test on a flat surface.<sup>26</sup> When SDF-KI was applied, it formed a white-yellow precipitate on the surface of demineralized dentin, which

was not removed by etching or rinsing. From SEM observation (Fig., E, F), it appears that the precipitate then served as the actual bonding substrate for these specimens. One possible reason why the precipitate over the substrate weakened the bond strength is that it prevented penetration of resin into the dentin, as suggested by empty tubules close to the surface (Fig., F). These results are consistent with a previous study<sup>10</sup> on bonding to SDF-KI treated sound dentin, which showed the formation of a precipitate and decreased resin tag formation. However, the bond strength values reported for adhesive resins and resin-modified glass ionomer to sound dentin were not as dramatically reduced as observed for demineralized dentin in the current study.

With increased popularity of SDF, the use of SDF-KI is expected to rise. SDF-KI has similar caries-arresting and prevention effects as SDF.<sup>27,28</sup> It should be mentioned that the long term benefit of KI application to reduce staining have been questioned. Some studies suggested that the staining effects of SDF and SDF-KI were not significantly different and darkening occurred in SDF-KI over the long term,<sup>25,29</sup> while another report showed acceptable results and minimal darkening after 6 months.<sup>30</sup>

Based on the current results, bonding of adhesive to demineralized dentin treated with SDF-KI would be severely compromised. Glass-ionomer-based restorations have been suggested on SDF and SDF-KI treated dentin; further study is required to investigate optimal approaches for adhesive restorations following SDF-KI treatment.

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In conclusion, application of SDF followed by phosphoric acid etching did not significantly alter bond strength of a universal adhesive to caries-affected dentin. However, SDF-KI had a dramatic weakening effect, compromising bonding to the demineralized dentin.

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