Original Article

Investigation of the effect of simultaneous use of silver diamine fluoride and potassium iodide on the shear bond strength of total etch and universal adhesive systems to dentin

Farnaz Farahat¹, Abdolrahim Davari², Haleh Karami³

¹Department of Operative Dentistry, School of Dentistry, Shahid Sadoughi University of Medical Sciences, ²Department of Operative Dentistry, Member of Social Determinant of Oral Health Research Center, School of Dentistry, Shahid Sadoughi University of Medical Sciences, ³Department of Operative Dentistry, School of Dentistry, Hamadan University of Medical Science, Hamadan, Iran

ABSTRACT

Background: Applying silver diamine fluoride (SDF) is recommended to arrest and prevent dental caries. However, it may jeopardize the bond of the restorative materials to the tooth. The aim of the present in vitro study was to evaluate the effect of the simultaneous use of the SDF and potassium iodide (KI) on shear bond strength to the sound dentin.

Materials and Methods: In this in vitro study, on the buccal and lingual surfaces of the 48 human third molar teeth, dentinal surfaces with a diameter of 6 mm were created by removing the enamel. Then, the specimens were divided into four groups based on applying or not applying the KI/SDF and adhesive type (etch and rinse [E and R] or universal adhesive). Afterward, cylindrical composite restoration was made on the surfaces. Half of the specimens were subjected to 1000 cycles of thermocycling. Subsequently, the shear bond strength was evaluated by Universal testing machine. Furthermore, the type of failure was determined by a stereomicroscope. Data were analyzed by t-test and Chi-square at a significance level of P < 0.05.

Results: There were statistically significant differences in shear bond strength between Groups 1 and 3 (P < 0.05), whereas the bond strength difference between Groups 2 and 4 was not statistically significant (P = 0.609). Failures were predominantly of the mixed type in almost all groups.

Conclusion: Based on the results of this study, the superiority of shear bond strength of universal adhesive to the sound dentin compared to E and R adhesive was confirmed. Thus, using this type of adhesive is recommended under the condition that KI/SDF anticaries material is applied.

Key Words: Bond strength, dental adhesives, dentin, silver diamine fluoride

INTRODUCTION

Many studies have been conducted to introduce a method or a material to limit progression or stop active caries without eliminating sound dental structures. Since centuries ago in Japan, silver-containing compounds such as silver nitrate and combination of silver and fluoride (without stabilizing amine group) have been introduced as antimicrobial materials.[1] Silver diamine fluoride (SDF) with the chemical formula of (Ag(NH₃)₂F) was approved

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by the US FDA in 2016 as a substance which stops the process of tooth decay.[1,2] The positive effects of SDF are attributed to its constituent components. Silver salt stimulates sclerosis and calcification of dentin, silver nitrate kills bacteria, and fluoride causes remineralization and prevents the progression of demineralization.[3] According to the previous studies among fluoride-containing materials, the most effective material to prevent the degradation of collagen and demineralization is SDF.[2] However, applying SDF before composite restoration is not common for two reasons: (1) darkening the color of tooth and restoration and (2) failure to provide a proper method before placing the restoration in such a way as not to compromise the bond strength.[1,4] The recommended solution for the first problem is using some salts, such as potassium iodide (KI), which reacts with free silver ion that is formed after the application of SDF. The result of this reaction is the production of white silver iodide, which prevents changing the color of the tooth surface.[5] For the second problem, although several methods have been proposed, there is still no agreement on the implication of a specific method. Furthermore, in most of the studies except the study conducted by Selvaraj et al., examining the bond strength in the presence of SDF, the effect of use of KI has not been investigated.[6]

The results of the studies conducted to explore the impact of applying SDF on adhesive’s bond strength are inconsistent. Two studies concluded that applying SDF does not negatively affect the bond strength.[2,6] Opposing to the results of the aforementioned studies, a study conducted by Lutgen et al. showed that applying SDF before applying self-etch and universal adhesives with and without selective etch can reduce the bond strength to the dentin compared to the control groups.[7] Furthermore, Koizumi et al. showed that KI/SDF can reduce the bond strength of the three types of adhesives.[8] The Korkmaz et al. study also showed that in a case of using KI after applying SDF, the bond strength reduction still exists.[9]

However, all studies about SDF examined the initial bond strength. The various methods offered to check the bond strength during a long period of time include application of thermal and mechanical cycling and fatigue test. Thermocycling stimulates temperature changes in clinical conditions. According to some studies showed that thermocycling is able to accelerate the decay process of interface contact between dentin and restoration.[10]

Hence, the purpose of this study was to investigate the effect of simultaneous use of SDF and KI on the shear bond strength of composite restoration to dentin using total etch and universal adhesives after thermocycling. The null hypotheses were: (1) the bond strength of SDF/KI and control groups are similar. (2) The bond strength of the universal and etch and rinse (E and R) adhesives to SDF/KI treated surfaces are similar. (3) The thermocycling process has no effect on the shear bond strength of SDF/KI groups.

**MATERIALS AND METHODS**

**Sample preparation**

In this *in vitro* study, based on Lutgen et al.,[7] in which $\delta_1$ and $\delta_2$ were accordingly 7.1 and 4.6, to detect and eight-unit significant difference ($\Delta \mu = 8$), 95% confidence interval and 90% test power ($\beta = 0.1$), the desired sample size was calculated using the following formula:

$$n = \frac{(Z_{1-\alpha/2} + Z_{1-\beta})^2 (\delta_1^2 + \delta_2^2)}{\mu_1 - \mu_2}^2$$

Therefore, 48 human extracted third molar teeth without any cracks, decay, restoration, and enamel defects were used. The soft tissue residues were removed by curette and crown surfaces were cleaned with pumice powder, and then, they were rinsed with water. Afterward, the teeth were stored in 1% Coloramine T solution for 72 h. Then, the teeth were rinsed and kept in distilled water at 37°C until the practical phase began.

For the first step, 220 grit sandpaper (Klingspor/Germany) was used to remove the buccal and lingual enamel an evenly expose the dentin layer. To do so, the sandpaper was pulled 10 times clockwise until the surface of the sound dentin became 6 mm diameter. Then, to provide a standard smear layer, the bonded dentin surface was polished with 600 grit silicon carbide paper disk (PS11A, Auto-paper, p600A (Kingspor/Germany)), in the presence of moisture for 1 min. Finally, the specimens were rinsed and the excess moisture was gently wiped off with the wet cotton ball without completely drying their surfaces.

Considering that two buccal and lingual surfaces were prepared for each tooth, a total of 96 surfaces were provided for testing. Then, the teeth were embedded in autopolymerized resin. The prepared specimens
were divided into four groups \((n = 24)\), based on their adhesive types and applying or not-applying KI/SDF [Table 1].

- **Group 1:** On the prepared dentin surface, two drops of 38% SDF (advantage arrest SDF/elevate oral care/USA) were applied with microbrush for 10 s. Immediately afterward, KI was added to the surface by means of a microbrush, and consequently, a white precipitate is produced. Adding KI continued until the white precipitate was no longer produced. Afterward, rinsing the dentin surface was done for 30 s and the excess moisture was air-dried thoroughly for 5 s to evaporate the extra water. For the next step, the single bond Adper bond 2 (SB) (3M ESPE-ESPE/USA) adhesive was applied to the surface according to the factory instructions. To do so, first, the dentin surface was etched by 35% phosphoric acid/Ultra‑Etch (Ultradent products, Inc.) for 15 s and rinsed for 10 s, and then, adhesive was applied in two layers and cured by the LED (Kerr/USA) device with the intensity of 900 mw/cm\(^2\) for 15 s
- **Group 2:** On dentin surface, 35% phosphoric acid and SB were applied, as described in Group 1
- **Group 3:** Similar to Group 1, SDF and KI were applied on the surface, and then, with a microbrush, a drop of single bond universal adhesive (SBU) (3M ESPE/USA) was applied to the dentin surface for 20 s, and a light flow of air was blown for 5 s to evaporate the solvent. Then, the bonding agent was cured for 10 s
- **Group 4:** SBU was applied to the dentin surface in the same way which was described for Group 3.

It should be noted that in each tooth, the same adhesive system was used on the buccal and lingual surfaces, expect that on buccal surface, KI/SDF was applied but on lingual surface, no conditioning was considered.

In all groups before adhesive curing, the silicon mold (4 mm diameter and 3 mm length) was placed on the dentin surfaces, and it was fixed during the adhesive curing. The Z250 composite A2 shade (3M ESPE/USA) was applied in two increments and each layer cured for 40 s.

In each group, half of the specimens \((n = 12)\), were kept inside the incubator for 24 h at 37°C. The other half of the specimens \((n = 12)\) underwent thermocycling process. The specimens were subjected to 1000 thermal cycles at 5 and 55 with the dwell time of 30 s in the thermocycling device (Vafaei/Iran). Then, the specimens were dried and their shear bond strength was measured.

### Table 1: Materials used in the study

| Material                           | Composition                                                                 | Manufacturer                                      | Batch number |
|------------------------------------|-----------------------------------------------------------------------------|---------------------------------------------------|--------------|
| Advantage arrest silver            | Silver fluoride, ammonia, FD and C Blue#1, Deionized water                 | Advantage arrest SDF/elevate oral care/USA        |              |
| Composite resin material: Z250     | Resin matrix: BIS-GMA, BISEMA, UDMA with small amounts of TEGDMA filler loading: 60 vol% silanized zirconia/silica particles | 3M ESPE dental products, St. Paul, USA            | 6020         |
| A2 (3M/USA)                        | Size range: 0.01–3.5 μ, (average size: 0.6 micron)                          | Ultradent products; South Jordan, UT, USA         | R116         |
| Ultra‑Etch                         | 35% phosphoric acid, glycol, cobalt aluminate blue spinel                   | 3M ESPE, St Paul, MN, USA                         | 100076       |
| Adper single bond 2                | Ethyl alcohol (25%–30%), silane treated silica (nanofiller) (10%–20%), BIS-GMA (10%–20%), HEMA (5%–10%), glycerol 1,3-dimethacrylate (5%–10%), copolymer of acrylic and itaconic acids (5–10), water (5), diurethane dimethacrylate (1–5) | 3M oral care; St Paul, MN, USA                    | 619545       |
| Single bond universal              | 2-hydroxyethyl methacrylate, bisphenol A diglycidyl ether dimethacrylate, decamethylene dimethacrylate, ethanol, silane treated silica, water, 2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and phosphorous oxide, copolymer of acrylic and itaconic acid, dimethylamino ethyl methacrylate, camphorquinone, dimethylaminobenzoate, 2,6-dinitro-butyrate-P-cresol | 3M ESPE dental products, St. Paul, USA            |              |

**TEGDMA:** Triethylene glycol dimethacrylate; **UDMA:** Urethane dimethacrylate; **BIS-GMA:** Bisphenol A-glycidyl methacrylate; **Bis-EMA:** Bisphenol A ethoxylated dimethacrylate; **HEMA:** Hydroxyethyl methacrylate; **SDF:** Silver diamine fluoride; **ESPE:** European Society for Paediatric Endocrinology; **FD:** FDA has regulatory review for color additives used in foods, drugs, cosmetics, and medical devices. **FD&C Blue No. 1**, also known as Brilliant Blue FCF ("for coloring food"), is a water-soluble artificial blue dye allowed by the FDA for use in foods, drugs and cosmetics. **FD&C Blue No. 1** is widely used in food products (candies, confections, beverages, etc.) and there have been no reports of toxicity associated with this general food use.
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Shear bond strength test
To measure the shear bond strength, Universal testing machine Zwick/Roell Z050 (Zwick GmbH and Co. KG, Ulm, Germany) was used. To apply the shear force around the composite cylinder, the blade was inserted in a way that it was in the interface between the tooth and the composite and in contact with the dentin. The force was increasingly at a speed of 1 mm/min, perpendicular to the longitudinal axis of the composite cylinder until the bond failure occurred. In a case that before loading a specimen was deboned, a number of zero would be considered for it. Bond strength was reported as an amount of nominal stress value (in Mpa), which is the amount of load applied at the moment of failure (in N) divided by the total bonded surface (in mm²) (bond strength = F/A).

Evaluation of failure pattern
A stereo microscope (Olympus/Japan) with ×40 magnification was used to examine the failure mode of the specimens. Debonded surfaces were classified according to the mode of failure into one of the following three groups: (1) cohesive (completely in dentin or in resin composite substrate), (2) adhesive (in dentin resin interface), and (3) mixed (the combination of adhesive and cohesive failure).

Statistical analysis
Data were uploaded into SPSS17 software (Chicago, USA). The mean and standard deviation for each group were calculated. Data were subjected to Kolmogorov–Smirnoff analysis to examine normal distribution, in which case since the data distribution was normal, t-test was run to evaluate the shear bond strength, Chi-square was run to analyze the mode of failure. All analyses were performed at the statistical significance level of α = 0.05.

RESULTS
Shear bond strengths in MPa (means and standard deviations) for the study groups are represented in Table 2. There were statistically significant differences in shear bond strength values between the study groups (P < 0.05). The results revealed that using SDF/KI significantly reduced the shear bond strength compared to the control group, except for nonthermocycled samples of SBU adhesive (P = 0.486) [Figure 1].

The t-test revealed that there was statistically significant difference in shear bond strength between Group 1 and 3 (P < 0.05), whereas the bond strength of Groups 2 and 4 was not statistically significant (P = 0.609). Furthermore, the effect of thermocycling process on the shear bond strength of all groups was insignificant.

Failure modes of the study groups are shown in Table 3. Failures were predominantly of the mixed type in all groups, except for Group 4 which in the majority of failure mode was adhesive. According to P > 0.05, the difference between the studied groups was not significant in terms of the type of failure [Table 3].

DISCUSSION
The results of the present study showed that the use of KI/SDF reduces the shear bond strength compared to the control group. Furthermore, universal adhesive had higher shear bond strength with the prepared surfaces using KI/SDF. Therefore, the first two null hypotheses of the study were rejected. On the other hand, thermocycling did not have a significant effect on the shear bond strength of the studied groups. Hence, the third null hypothesis of the study was confirmed.

In the present study, the shear bond strength test was done to evaluate the bond strength of the specimens. While some believe that microtensile test is the preferred method for bond strength testing, shear bond testing provides more reliable results in examining the effect of dental materials on bonding. It is also easier and less time-consuming to prepare a specimen for a shear test.\cite{7}
The present study showed that KI/SDF reduces the bond strength of both types of adhesives compared to the control group. SDF can interfere with the penetration of primer and bonding into the intertubular and peritubular dentin which leads to the less hybrid layer formation with the lower collagen matrix. In a study Koizumi et al. showed that the use of KI/SDF reduces the dentin bond strength which is in line with the result of the present study, although in their study, no KI/SDF rinsing was done. Furthermore, Lutgen et al. showed that the use of SDF could reduce the bond strength, but they said that the severity of this negative effect depends on the method of applying SDF, in a way that rinsing the SDF led to the creation of a better bond strength. However, a study by RL QUACK showed that the use of 12% and 38% SDF did not negatively affect the dentin bond strength. In the mentioned study, unlike the present study, the effect of applying KI after SDF was not investigated, but rinsing was done for 30 s.

The types of adhesive used in the aforementioned studies were different. Furthermore, a possible reason for the inconsistency among the results of the different studies is the lack of standard sampling method and the SDF application protocol. In some studies, the dentin surface was rinsed immediately after applying SDF. While in other studies, SDF was allowed to be air dried. However, it should be noted that in clinical conditions, neither of these two methods is used. The patient is only asked to avoid eating and drinking for up to half an hour after the SDF application. SDF has a pH of about 10. If after applying SDF rinsing is not done, the surface becomes highly alkaline which interferes with self-etch adhesive and phosphoric acid and consequently reduces the bond strength. In fact, the rinsing step of the anticaries material left on the surface is an essential step in achieving the optimum bond of composite restoration. Three studies have shown that if KI/SDF residues were rinsed away, they did not affect the bond strength. On the other hand, one study showed that the bond strength remained low despite running the rinsing process. In the present study, rinsing was done for 30 s before applying each type of adhesive to remove excess KI/SDF deposits.

With the use of electron microscope, Lutgen et al. showed that when the surface was not rinsed after applying SDF, a thick layer of SDF covered the dentin surface and inside the tubules to a depth of 20 micron. Rinsing the SDF, the extra amount of it which has not been absorbed by the tooth was removed from peri and intertubular dentin. Observing the

| Group | KI/SDF application | Adhesive | Thermo cycling | Number | Mean±SD (MPa) |
|-------|--------------------|----------|----------------|--------|---------------|
| 1     | Yes                | SB       | No             | 12     | 7±2.96±*     |
| 2     | No                 | SB       | No             | 12     | 10.24±4.00    |
| 3     | Yes                | SBU      | No             | 12     | 11.79±2.52    |
| 4     | No                 | SBU      | No             | 12     | 12.84±4.49    |

Identical letters indicate no significant difference (P>0.05). KI: Potassium iodide; SDF: Silver diamine fluoride; SB: Single bond Adper bond 2; SBU: Single bond universal adhesive; SD: Standard deviation

| Group | KI/SDF application | Adhesive | Thermo cycling | n | Failure mood (%) | Total |
|-------|--------------------|----------|----------------|---|------------------|-------|
| 1     | Yes                | SB       | No             | 12 | 75               | 100   |
| 2     | No                 | SB       | No             | 12 | 66.7             | 100   |
| 3     | Yes                | SBU      | No             | 12 | 66.7             | 100   |
| 4     | No                 | SBU      | No             | 12 | 66.4             | 100   |

KI: Potassium iodide; SDF: Silver diamine fluoride; SB: Single bond Adper bond 2; SBU: Single bond universal adhesive
surface treated by SDF with scanning electron micrograph (SEM) shows that due to the small size of the SDF, it is possible for it to penetrate into the dentin to a depth of 200 μ. Studies have also shown that silver and fluoride ions penetrated to a depth of 450 nm into demineralized dentin. Therefore, it seems that rinsing only removes superficial SDF, so even by rinsing its therapeutic effect still exists. However, this layer can be mechanically removed with the help of 600 grit silicon carbide disc. However, there is a concern that the treated surface with the SDF and formed fluoridated hydroxyapatite may be eliminated.[7] The present study showed that after water rinsing the KI/SDF, the strength of the primary bond of the universal adhesive as well as its bond strength after thermocycling were significantly higher than E and R adhesive. According to what was recommended in the studies conducted by the results of the studies conducted by Rosa et al.[15] and Wagner et al.,[16] in this study, universal adhesive was applied on the dentin surface in a self-etch way. Koizumi showed that the bond strength reduction after applying KI/SDF was more considerable in 6th generation 2-step self-etch adhesive systems and it is less remarkable in the 3-step E and R systems. They hypothesized that phosphoric acid was responsible for removing deposit produced by KI/SDF. Therefore, to some extent, it improves the bond strength. Nevertheless, there is still enough sediment left on the surface to negatively affect the adhesion. It should be mentioned that the types of E and R and self-etch adhesive used in their studies were different from the materials used in the present study. They also did not rinse the KI/SDF away.[8] However, the study of RL Quack showed that there was no significant difference between the bond strength of 2-step self-etch adhesive and 3-step E and R adhesive in the control group and the 38% SDF group.[2] On the other hand, Lutgen et al. study revealed that, after applying the SDF, 2-step self-etch and universal adhesives with a selective etch performed better than the application universal adhesive alone.[7]

The highest SDF density is in the most superficial dentin layer, and therefore, the negative effect of SDF on bonding is greater in this area. Among the aforementioned studies, for those that rinsing was not done, the bond strength of the groups that etched by the phosphoric acid was higher.[17] However, according to the results of the present study, it seems that after rinsing KI/SDF, this surface layer is removed and therefore, the effect of two types of adhesive on dentin is similar to that of control group in a way that the bond strength of universal adhesive is significantly higher than the 2-step E and R, which is exactly the same as those groups in which KI/SDF was not used. The reason for this superiority can be attributed to the existence of 10 MDP monomer in universal adhesive, which can establish a long-lasting and effective bond by creating low-soluble calcium salt on hydroxyapatite. On the other hand, interlocking improves the bond strength of the self-etch adhesive by forming a resin tag and a hybrid layer. The weaker bond of E and R adhesive is attributed to the lack of this monomer and less than desired infiltration of the resin into the demineralized collagen network and also the weak bond to it. In addition, the process of etching and rinsing with 35% phosphoric acid can remove calcium from the surface and collagen network and it negatively affects the bond strength.[18]

In previous studies on the use of SDF or KI/SDF, the impact of aging on the bond strength has not been evaluated, so the available information is related to the initial bond strength. The durability of the resin-dentin interface depends on the formation of a compact and hemogen hybrid layer. The acceleration of hybrid layer degradation is stimulated by the thermocycling process. In the present study, the effect of aging on prepared specimens with anticaries material was investigated by applying 1000 thermal cycles. The result of this study revealed that the thermal aging process in groups applying KI/SDF reduces the dentin bond strength of both type of adhesives. Although the reduction was not statistically significant, it was higher in E and R adhesive than in universal adhesive. One study showed that although bond strength was higher in SDF-treated groups, their SEM images showed that limited resin tags were formed in dentin tubules. This contradiction negatively affects the reliability of the bonding result. In fact, in larger cavities, the bond strength is good in some areas and weak in others. Therefore, in the long time, the bonding consistency is compromised[17] which could be the reason for the decrease of bond strength in the groups treated with SDF after thermocycling. However, the reason for the insignificant decrease of bond strength after thermocycling can be attributed to the insufficient number of thermal cycles. Korkmaz et al., showed that the result of thermocycling was related to the number of cycles, restorative materials, and substrate.[9] Hariri et al. also observed a significant
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Dentin nonthermocycled shear bond strength. Therefore, better performance in terms of thermocycled and E and R adhesive was confirmed since it had significantly decreases which can be considered in future studies.

In the present study, failure mode in most cases was mixed and in the second place was adhesive. Furthermore, two cases of cohesive failure were reported. Cohesive failure may happen for some reasons such as an error in the specimen’s orientation relative to the longitudinal axis of the testing devices, microcracks in the specimens created during their preparation, and the fragility of the examined material. Therefore, data related to adhesive and mixed failure are more valuable. Since the methods of conducting different studies, the materials used in them, their failure modes, and the results of the similar studies are very diverse, it is not feasible to make an accurate comparison. In addition, while interpreting the results of various studies, this point should be considered that different methods have been used to evaluate the type of failure, including the use of optical microscope with magnification of 5–40 times, electron microscope, or unaided eye. On the other hand, the accuracy of an electron microscope is greater than that of eye observation or an optical microscope.

This study was conducted on sound dentin. It has been shown that the adhesion to the carious dentin was less successful than to the sound dentin. Dentin also undergoes structural changes over time. Hence, further studies are needed on adhesion to the carious dentin and it is recommended that different methods of surface preparation and different adhesives should be studied in sclerotic dentin treated with SDF. In addition, further changes in the application of SDF are needed to obtain the best method of applying SDF with adhesive restorative materials, so that the benefits of each will be well preserved.

CONCLUSION

This study showed that applying SDF/KI reduces the shear bond strength to the sound dentin. Furthermore, according to the results of the present study, the superiority of universal adhesive over E and R adhesive was confirmed since it had better performance in terms of thermocycled and nonthermocycled shear bond strength. Therefore, in a case of applying KI/SDF, the use of universal adhesive is recommended.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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