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Original Article

Shear bond strength of different tooth color restorative materials after using silver diamine fluoride in primary tooth dentin: An *in vitro* study

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ABSTRACT

Background: The main disadvantage of silver diamine fluoride (SDF) is its persistent dark coloring. The aim of this study was to cover this discoloration on affected primary tooth dentin with different materials and subsequently measure their shear bond strength (SBS).

Materials and Methods: In this *in vitro* study total of 60 primary teeth were demineralized and randomly divided into five groups (n = 12). The tooth surfaces were treated with 38% SDF, and restorative cylinders were built on the dentin as follows: (1) phosphoric acid etching + GLUMA Universal Adhesive (GUA; etch-and-rinse mode) + composite resin (CR); (2) GUA (self-etch mode) + CR; (3) resin-modified glass ionomer (RMGI; Fuji II); (4) Surefil One (self-adhesive CR); and (5) TheraCem (self-adhesive resin cement) + CR. After restoration, the specimens were tested for SBS. Failure mode was determined by digital analysis and scanning electron microscopy. Data were analyzed by one-way analysis of variance and Tukey's honest significant difference *post hoc* test. P <0.05 indicated statistical significance.

Results: Group 1 had significantly higher mean SBS ($P < 0.05$) compared to Groups 2–5, while Group 5 had the least SBS ($P < 0.001$). Mean SBS differences between Groups 2 and 3 were not significant ($P = 0.328$). Group 4 had lower mean SBS than Groups 1 ($P < 0.001$) and 2 ($P = 0.17$). Most groups showed adhesive failure.

Conclusion: CR associated with the universal adhesive in etch-and-rinse mode had much higher SBS than the other groups; therefore, we recommend it to cover the dark discoloration generated by SDF.

Key Words: Composite resins, primary teeth, scanning electron microscope, topical fluoride

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INTRODUCTION

Dental caries is a dynamic phenomenon that undergoes periodic cycles of demineralization and remineralization. Numerous materials are used to promote enamel or dentin remineralization.[1] Silver diamine fluoride (SDF) is an effective preventive agent with antibacterial and remineralizing properties attributed to its silver and

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fluoride concentrations.[2,3] The exact mechanism of SDF is unknown; however, insoluble silver chloride deposits on the tooth surface form a protective layer. By limiting the loss of calcium and phosphate ions, SDF inhibits additional demineralization. Calcium fluoride is created when the highly alkaline SDF dissolves in saliva, and this increases the release of fluoride ions.[3] A disadvantage of SDF is the persistent dark coloring of the treated teeth, which is caused by the deposition of silver ions on the demineralized surface. This discoloration results in an undesirable appearance, which results in parental complaints and resistance to treatment.[4] Methods used to decrease this dark coloring include treating the tooth surface with iodide potassium or covering the discoloration with tooth-colored restorative materials such as composite resin (CR), glass ionomer (GI), or Biosilicate.[5-7]

CR is frequently used to restore anterior teeth and non-stress-bearing areas. The results of one study indicated that the color-masking effect of CR on primary teeth treated with SDF was permanent and unaffected by aging.[5] Different types of dental adhesives that promote CR adhesion to tooth structures include the "multipurpose" or "universal" adhesives, which are developed adhesives that can be used in conjunction with the etch-and-rinse or self-etch techniques. Although the clinical benefits associated with these adhesives in CR restoration of permanent and primary teeth are documented,[8,9] other studies indicated a reduction in shear bond strength (SBS) of the universal adhesive to enamel and dentin, especially with the self-etch technique.[10,11]

Novel self-adhesive CR and self-etch resin cement materials eliminate the need for acid etching or the application of bonding agents before use to decrease the procedure time, and this makes them a great option for pediatric dentistry. Some of these materials release fluoride and calcium, which might enhance remineralization.[7,12-15]

Pediatric dentists frequently use GI-based materials because of their biocompatibility and ability to release fluoride. These characteristics can enhance the cariostatic and remineralization effects. Other advantages of GI include ease of use and cost-effectiveness. However, the limitations include a susceptibility to wear and unsatisfactory esthetic outcomes compared to CR.[16] Comparisons of the ability of CR and GI to cover SDF black discoloration showed that the SBS of CR was better.[12,17]

The purpose of this *in vitro* study was to determine the SBS of various tooth color restorative materials and techniques on artificially affected dentin of anterior primary teeth treated with SDF, regarding the considerable incidence of dental caries in young children. The null hypothesis was that the SBS of various tooth color restorative materials was equivalent in the presence of SDF-treated dentin.

MATERIALS AND METHODS

Study design

The Human Ethics Review Committee of the Faculty of Dentistry, Shiraz University of Medical Sciences, approved this *in vitro* study. The approval code was: IR.SUMS.REC.1401.099. The parents provided written informed consent authorizing the use of their children's teeth after receiving a thorough explanation of the purposes of this research. A total of 72 intact primary canine teeth extracted during orthodontic treatment were collected over 3 months and kept in distilled water. The teeth were thoroughly cleansed of all supporting tissues, and any remaining debris was removed with a prophylaxis brush. The cleaned specimens were immersed in a disinfectant that contained 0.1% chloramine-T for 1 month. The roots were cut 2 mm below the cementoenamel junction. Then, each tooth was mounted with the buccal surface aligned parallel to the acrylic surface. The outer surfaces of the crown were abraded using a diamond bur to reveal the dentin. The dentin was smoothed with sandpaper (600–1000 size grit) with simultaneous use of water for cooling. Subsequently, an aluminum oxide compound with a particle size of 0.5–3 µm was applied to level the surface. Finally, the specimens were immersed in an ultrasonic bath for 5 min. The samples were then observed under a stereomicroscope (magnification: ×40) to confirm the complete removal of the enamel. Samples with cracks or defects were not assessed in this study. A total of 60 teeth passed the required selection criterion.

Artificial caries induction by pH cycling

A 3 mm × 3 mm paper label was attached to the dentin surface, and we applied two coats of nail varnish adjacent to the label. After the nail varnish dried, we removed the label, and this left a 3 mm × 3 mm window of dentin. Each sample was individually immersed in 10 mL of a pH 4.8 demineralization solution (50 mM acetic acid, 2.2 mM NaH_2PO_4, and 2.2 mM $CaCl_2$) for 8 h, followed by 16 h immersion in

10 mL of a pH 7 remineralization solution (0.15 mM KCl, 0.9 mM NaH_2PO, and 1.5 mM $CaCl_2$). The samples were then rinsed with deionized water and stored for 24 h. This procedure was repeated at room temperature without any disturbances for 14 days.[18]

Experimental groups

A single drop of 38% SDF (Dengen Dental, Inc., India) was placed on the prepared surfaces, which were then agitated for 2 min with a microbrush. A cotton swab was used to remove any unreacted or excess SDF. The samples were stored for 2 weeks in artificial saliva and then randomly divided into the following five treatment groups (n = 12 per group).

- Group 1: Etch (3M ESPE, St. Paul, MN, USA) + GLUMA Universal Adhesive (Kulzer GmbH, Hanau, Germany) (GUA; etch-and-rinse mode) + Charisma Smart CR (Kulzer GmbH, Hanau, Germany)
- Group 2: GUA (self-etch mode) + Charisma Smart CR
- Group 3: Conditioner (GC, Tokyo, Japan) + resin-modified GI (RMGI) (GC, Tokyo, Japan)
- Group 4: Surefil One self-adhesive CR (DENTSPLY Sirona GmbH, Konstanz, Germany)
- Group 5: TheraCem self-adhesive resin cement (BISCO, Inc., Schaumburg, IL, USA) + Charisma Smart CR.

Table 1 shows the composition of the materials and instructions for their proper use.

A rubber cylindrical mold with a 3 mm internal diameter and height of 3 mm was used to bond the CR or RMGI to the treated dentin. The cylinder was filled with RMGI or CR by using an incremental technique. A halogen light curing unit (Coltolux, Coltène/Whaledent AG, Altstätten, Switzerland) was used to polymerize each 2 mm layer for 20 s at a power density of 550 mW/cm^2. The specimens were then kept for 7 days at 37°C in a humid environment. One operator performed all of the procedures according to standard protocols and the manufacturer's instructions.

The SBS test was performed using a universal testing machine (ZwickRoell, Zwick, Ulm, Germany) with a crosshead speed of 1 mm/min to the point of failure with an applied force measured in megapascals (MPa). Subsequently, two researchers evaluated the fracture's bond failure under a digital microscope (Dino-Lite, Taipei, Taiwan) at a magnification of ×20. The observers were blinded to the study group assignment. They recorded the adhesive fracture at the composite–dentin interface, cohesive fracture in the substrate (dentin), or mixed fracture of both adhesive and cohesive fracturing for each study group.

Table 1: Materials and procedures used in the current study

Materials	Chemical composition	Procedure	Manufacturer
Caries arrest (SDF)	SDF solution (38%)	Apply and agitate for 2 min, the excess and unreacted SDF are blotted with a cotton pellet	Dengen Dental, India
Phosphoric acid gel	Phosphoric acid gel (37%)	Apply for 20 s, rinse for 15 s, air-dry for 2 s	3M ESPE, St. Paul, MN, USA
GLUMA Bond Universal	4-META acid, methacrylate monomer, acetone, 10-MDP, water	Etch and rinse strategy: Apply etchant for 20 s, rinse for 15 s, air-dry for 2 s, apply adhesive for 20 s, air-dry and light cure for 10 s Self-etch strategy: Apply the adhesive for 20 s, air-dry for 5 s, light cure for 10 s	Kulzer GmbH, Hanau, Germany
Charisma Smart	Bis-GMA, UDMA, TEGDMA, barium aluminum fluoride glass filler (0.02–2 µm), 5 vol% pyrogenic silicon dioxide filler (0.02–0.07 µm) (78% wt), methacrylic acid	Apply in thin layers (maximum 2 mm) and light cure for 20 s. Incremental technique was used to build the cylindric CR	Kulzer GmbH, Hanau, Germany
Cavity conditioner	Polyacrylic acid, aluminum chloride, distilled water	Apply for 10 s, rinse thoroughly, and blot-dry	GC, Tokyo, Japan
Fuji II LC capsule	Fluoroaluminosilicate glass, polyacrylic acid, HEMA, UDMA, camphorquinone, water	Mix the capsule for 10 s at 4000 rpm by an amalgamator. Cure for 20 s	GC, Tokyo, Japan
Surefil One	Aluminum-phosphor-strontium-sodium-fluorosilicate glass, water, highly dispersed silicon dioxide, acrylic acid, polycarboxylic acid, ytterbium fluoride, bifunctional acrylate, self-cure initiator, pigments, camphorquinone, and stabilizer	The surface is air-dry for 20 s. Activate the capsule by amalgamator at a speed of 4200–5000 rpm for 10 s, apply and light cure for 20 s	DENTSPLY Sirona GmbH, Konstanz, Germany
TheraCem	Base: Calcium base filler, glass filler, dimethacrylates, ytterbium fluoride, initiator, amorphous silica Catalyst: Glass filler, MDP, amorphous silica	Rinse and air-dry (3–5 s) the surface, mix base and catalyst on a mixing pad, apply a thin layer of cement, and light cure for 20–30 s	BISCO, Inc., Schaumburg, IL, USA

4-META: 4-methacryloxyethyl trimellitate anhydride; MDP: Methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: Bisphenol glycidyl methacrylate; UDMA: Urethane dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; HEMA: Hydroxyethyl methacrylate; SDF: Silver diamine fluoride; CR: Composite resin

Scanning electron microscope observation

We used a scanning electron microscope (SEM) to evaluate the fracture surfaces of two samples from each experimental group (n = 10). Each tooth surface was meticulously polished using silicon carbide papers with grit sizes of 400, 600, 1000, and 2000 and constant application of water for cooling. Subsequently, the surfaces were thoroughly cleaned and dehydrated with 96% ethanol, followed by gold sputter coating using a vacuum evaporator. The fractured surfaces in the samples were assessed by SEM (KYKY-EM3200, Shanghai, China) at a magnification of ×35.

Statistical analysis

IBM SPSS for Windows version 22.0 (Armonk, NY, USA, IBM Corp.) was used for statistical analyses. The Shapiro–Wilk test was used to assess for data normality. We used one-way analysis of variance (ANOVA) and Tukey's honest significant difference *post hoc* tests to compare mean SBS between the groups. P <0.05 was considered statistically significant. Intra-class correlation coefficient was also used to assess agreements between the two observers.

RESULTS

Table 2 shows the mean (±standard deviation) SBS for the five groups. One-way ANOVA indicated a significant difference between the mean SBS values of the groups ($P < 0.001$). The greatest mean SBS belonged to Group 1 (3.16 ± 0.29), which was significantly higher than the other groups (all $P < 0.05$). Next, Group 2 had the highest mean SBS (2.48 ± 0.73), followed by Group 3 (2.03 ± 0.55). There was no significant difference between the mean SBS values of Groups 2 and 3 ($P = 0.328$). The mean SBS value for Group 2 was higher than Groups 4 (1.72 ± 0.70, $P = 0.017$) and 5 (0.81 ± 0.79, $P < 0.001$); however, the mean SBS value for Group 3 was only higher than Group 5 ($P < 0.001$). Group 5 had a significantly lower mean SBS than the other groups (all $P < 0.001$).

Failure mode analysis revealed that adhesive fractures were most frequent in Groups 2–5. Mixed failure was mainly documented for Group 1. SEM findings confirmed the results of the failure modes [Figure 1a-c]. Table 3 shows the frequencies of different failure modes in the groups.

Table 2: Comparison of mean ± standard deviation shear bond strength (MPa) between the groups (*n*=12)

Group	Mean±SD	*P*
GUA (etch-and-rinse mode) + CR	3.16^{A}±0.29	<0.001*
GUA (self-etch mode) + CR	2.48^{B}±0.73	
Conditioner + RMGI	$2.03^{B,C}$±0.55	
Surefil One (self-adhesive CR)	1.72^{C}±0.70	
TheraCem (self-adhesive resin cement) + CR	0.81^{D}±0.49	

*One-way ANOVA *F*-test (*P*<0.05). Mean values with at least a common letter in superscript are not statistically significant (Tukey's HSD *post hoc* test). SD: Standard deviation; GUA: GLUMA Universal Adhesive; CR: Composite resin; RMGI: Resin-modified glass ionomer; HSD: Honestly significant difference, ANOVA: Analysis of variance

Table 3: Comparison of the number of different failure modes (*n*=12)

Group	Failure mode		
	Adhesive	Cohesive	Mixed
GUA (etch-and-rinse mode) + CR	3	1	8
GUA (self-etch mode) + CR	10	0	2
Conditioner + RMGI	7	0	5
Surefil One (self-adhesive CR)	7	0	5
TheraCem (self-adhesive resin cement) + CR	10	0	2

GUA: GLUMA Universal Adhesive; CR: Composite resin; RMGI: Resin-modified glass ionomer

DISCUSSION

This *in vitro* study evaluated the adhesion ability of tooth-colored restorative materials on the affected dentin of primary teeth treated with SDF. The findings guide pediatric dentists to choose an optimal approach for masking the black discoloration caused by SDF. This includes taking into consideration the use of etch-and-rinse or self-adhesive modes with a universal self-adhesive system or RMGI restoration, as well as the potential use of the novel self-adhesive CR. Our data showed that the SBS of the tooth color restorative materials differed in the presence of SDF-treated dentin, and this finding did not support the null hypothesis.

We used 38% SDF because of its enhanced effectiveness in preventing tooth demineralization compared to 30% and 12% SDF.[19] Alsagob *et al.* reported that CR or GI cement restorations applied 2 weeks after SDF treatment led to less discoloration compared to immediate restoration. Therefore, we applied the restorations 2 weeks after the SDF application to reduce the color change.[6]

SDF forms an insoluble coating of silver deposits on the tooth surface that obstructs the dentinal tubules, and this may prevent the adhesive from penetrating

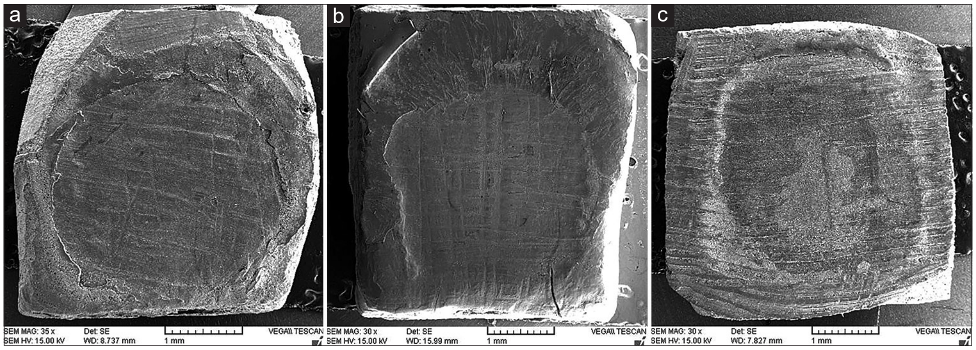

Figure 1: SEM images of failure mode of the surface fracture. (a) Adhesive fracture, (b) cohesive fracture, (c) mixed fracture (magnification: ×35). SEM: Scanning electron microscope.

the collagen matrix and dentinal tubules.[2,3,20] Many advised rinsing with water immediately following the application of SDF or superficial refreshing of SDF-treated dentin before adhesion to increase micro-SBS to the treated surface has been proposed.[21]

The hybrid layer created in the etch-and-rinse strategy is thicker, more continuous, and more consistent compared to the self-etch systems. The etching mode leads to the formation of deep dentinal furrows, which increases the surface area for adhesive bonding and generates a significant number of microtags that extend horizontally at right angles.[21,22]

According to our research, the GUA in the etch-and-rinse mode had a greater SBS of CR than the self-etch technique. This result supported earlier research findings.[21-23] However, one study reported higher adhesions to the primary teeth dentin after using UA in the self-etch mode.[8] We chose a universal adhesive because it is user-friendly and easily applied, both of which are important factors when selecting materials for children. We chose GUA for its pH of 1.6–1.8 and the inclusion of acetone, which allows adequate bonding, improves resin penetration into the dentin surface, and predicates the removal of silver deposits.[24]

GUA and most universal adhesives contain methacryloyloxydecyl dihydrogen phosphate (MDP). MDP is a hydrophilic monomer that possesses moderate etching characteristics with the capability to chemically bond to hydroxyapatite crystals and create a nanolayer. This nanolayer subsequently enhances the interface's mechanical strength.[11] In addition, the accumulation of stable MDP-calcium salts throughout the nanolayer increases the bond strength.[25,26] This functional monomer improves tooth wettability and penetration while preventing the hydrophobic monomer/water phase separation.[11] However, the existence of functional acidic monomers (4-MET acid) is not similar to MDP. The calcium salts that result from the acidic monomers with functional groups are susceptible to dissolution, and they are not hydrolytically stable.[27,28]

GI has a high fluoride release and exhibits anticariogenic properties. It is frequently used in pediatric dentistry. This material is composed of a polyhydrous copolymer, which is a polyalkenoic acid copolymer. The mechanism of adhesion of GI is predicated on the superficial hybridization of the linked carboxyl groups of the polyacrylic acid interaction and calcium in hydroxyapatite. Micromechanical retention is provided by the penetration of organic composition into treated dentin.[16,29,30] In the current study, the lower SBS of the RMGI and self-etch mode of GUA might be attributed to the inadequate acidity of these materials to treat dentin with SDF. A systematic meta-analysis reported that the application of SDF to dentin did not result in a reduction in the strength of the adhesion of GI to either sound or affected dentin when compared to an untreated surface. However, this meta-analysis included research that used a conditioning agent or a rinsing procedure immediately following the application of SDF.[31] In the present study, we used a conditioner before GI restoration.

The advantages of self-adhesive composites such as Surefil One make them good candidates for use as a restorative material in children. These materials are applicable even when there is uncertainty about the ability to control moisture contamination because they do not need etching and they use an adhesive.[32,33] In contrast to GI, Surefil One is a non-hybrid self-adhesive CR that offers an esthetic outcome. Its modified polyacid, acrylic acid, and functional monomer/polymer components cause two distinct modes of interaction with dental surfaces: a micromechanical interaction through etching and an ionic bonding with calcium ions in hydroxyapatite.[32]

Recent research compared the SBS of CR with a total-etch adhesive, RMGI, and Surefil One on permanent teeth. The study demonstrated that CR had better SBS than the other groups, followed by the self-adhesive CR and GI.[12] These findings approximated our results; however, we did not observe any significant difference between the SBS of GI and Surefil One. The differences between these studies may be related to the various protocols and the types of teeth assessed.

Self-adhesive resin cements eliminate the necessity for pretreatment and lead to decreased technical sensitivity, reduced risk of contamination, and reduced postoperative sensitivity. We used TheraCem because of its good adherence to both restorative material and dentin, as well as its ability to release calcium and fluoride.[14,15,34] We presumed that these qualities contribute to improved remineralization in the affected dentin and CR bonding. However, based on our research, the self-adhesive CR, or resin cement, had the lowest SBS. This could be attributed to the principal bonding mechanism of self-adhesive materials, which occurs through the chemical reaction between the acidic monomer of the materials and the calcium contained in the tooth structure. Insufficient pretreatment of the surface by monomers and the existence of silver deposits seem to impede chemical bonding between these functional monomers and calcium in hydroxyapatite.[35] Additionally, SDF reduces the availability of calcium ions for chemical interactions by increasing calcium fluoride. A study that supported our findings reported that the application of SDF through an intracanal led to a decrease in the push-out bond strength of fiber posts cemented with self-adhesive cement.[36]

We observed that adhesive failure was the most common finding, particularly with self-adhesive materials. This is likely due to a weaker bond between the materials and the treated surfaces, which supported the findings reported by Alrahlah *et al*.[20]

The present study assessed the SBS of restorative materials in primary affected dentin under controlled laboratory circumstances. This technique has been used to assess the resistance of materials to bite and contraction forces. Our methods attempted to mimic the clinic setting. However, *in vitro* experiments fail to accurately represent a clinical situation, and this is a limitation of the present study. In addition, the sample size may influence the outcome. Therefore, we recommend that researchers conduct studies of identical comparisons with infected, affected, and sound dentinal surfaces to determine the impact of carious dentin on the dentin bond strengths of self-adhesives after SDF application. Clinical studies that enroll large numbers of patients should be conducted to verify our findings.

CONCLUSION

Our first approach to cover the discoloration by SDF in the affected dentin of the primary teeth involved the use of CR and the etch-and-rinse technique with GUA. The SBS results indicated that the subsequent choice was the self-etch mode of the adhesive or RMGI restoration. The self-adhesive Surefil One CR was not significantly different from the RMGI. The SBS of the surface treated by SDF decreased when a self-etch resin cement (TheraCem) was applied before composite restoration.

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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 non-financial in this article.

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