

EFFECT OF SILVER DIAMINE FLUORIDE PRETREATMENT ON MICRO-SHEAR BOND STRENGTH OF GIOMER AND SELF-ADHESIVE RESIN COMPOSITE TO DEMINERALIZED DENTIN OF PRIMARY MOLARS: AN IN VITRO STUDY

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ABSTRACT

Objectives: This study aimed to evaluate the effect of silver diamine fluoride (SDF) pretreatment on the micro-shear bond strength (μ SBS) and failure mode of two bioactive restorative materials—giomer (Beautifil Flow Plus) and self-adhesive resin composite (Surefil One)—when bonded to demineralized dentin of primary teeth.

Materials and Methods: Thirty extracted primary molars were sectioned into mesial and distal halves and prepared to expose dentin. Artificial demineralization was performed on two-thirds of specimens. Specimens were assigned to six groups based on dentin condition (sound or demineralized), surface pretreatment (with or without 38% SDF), and restorative material used (Beautifil Flow Plus or Surefil One). μ SBS was measured using a universal testing machine. Failure modes were analyzed under a stereomicroscope. Statistical analysis was performed using the Kruskal–Wallis H test, with significance set at $p \leq 0.05$.

Results: SDF application caused a non-significant reduction in Beautifil Flow Plus bond strength ($p = 0.402$) but significantly increased the μ SBS of Surefil One ($p < 0.001$).

Conclusion: SDF pretreatment had no significant adverse effect on the bond strength of Beautifil Flow Plus to demineralized dentin but significantly enhanced the bond strength of Surefil One. These findings suggest that the effect of SDF on bond strength may vary depending on the type of restorative material used.

KEYWORDS Silver Diamine Fluoride, Micro-Shear Bond Strength, Giomer, Self-Adhesive Resin Composite, Demineralized Dentin.

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INTRODUCTION

Dental caries represents the most widespread chronic disease in the pediatric population. If not properly managed, caries can lead to pain, oral discomfort, and impaired nutritional intake, which may subsequently have a negative impact on the child's overall quality of life.⁽¹⁾

Silver diamine fluoride (SDF) is widely employed as an anticariogenic topical agent due to its ability to suppress cariogenic biofilm activity, promote dentine remineralization, and inhibit further demineralization.⁽²⁾ Nevertheless, the principal drawback of SDF is the dark staining of treated dentin; moreover, cavitated lesions remain unsealed and can continue to accumulate plaque, potentially leading to caries reactivation. To mask the discoloration and restore the form and function of the tooth, many clinicians subsequently place tooth-colored restorations over SDF-treated lesions.^(3,4)

The use of bioactive restorative materials with antibacterial and remineralizing properties has gained popularity in recent years, aligning with the principles of minimally invasive dentistry.⁽⁵⁾

Giomers are bioactive materials that combine the properties of glass ionomers and composite resins. Beautifil Flow Plus, a light-cured direct restorative material, is one example of this category.⁽⁵⁾ Giomers integrate the mechanical and esthetic characteristics of composite resins with the caries-protective benefits of fluoride release, achieved by incorporating pre-reacted glass (PRG) fillers into the resin matrix.⁽⁶⁾

Surefil One is a novel self-adhesive hybrid composite that incorporates the fluoride-releasing ability and self-adhesive behavior of glass ionomers alongside the performance characteristics of resin composites. The self-adhesive property of Surefil One is attributed to its unique structure, the Modified Polyacid System (MOPOS), which promotes network formation and enhances mechanical per-

formance. Since the material is applied in a single step without requiring separate etching and bonding procedures, it is particularly advantageous in clinical situations where time or patient cooperation is limited, such as in pediatric dentistry.^(4,7)

Several studies have been conducted to better understand the effect of SDF application on the bond strength of esthetic restorations to carious primary dentin. However, studies in this aspect regarding bioactive restorative materials as giomers and self-adhesive resin composites are lacking. Therefore, this in-vitro study aimed to evaluate the effect of SDF pretreatment on micro-shear bond strength (μ SBS) and failure mode between giomer (Beautifil Flow Plus) or self-adhesive resin composite (Surefil One) and demineralized dentin of primary teeth.

The null hypothesis was that there is no significant difference in the μ SBS and failure mode between Beautifil Flow Plus or Surefil One and demineralized primary dentin in the SDF pretreated teeth compared to the control groups where no surface pretreatment is applied.

MATERIALS AND METHODS

Ethical regulations

Ethical approval was obtained from the Research Ethics Committee of the Faculty of Dentistry, Minia University (Reference No. 830, 2023; Meeting No. 100) prior to conducting this study. Informed consent was obtained from the legal guardians of the patients, confirming their agreement to use their children's extracted teeth for research purposes.

Sample size calculation

Sample size was calculated using G*power statistical power analysis program (version 3.1.9.4) for sample size determination.⁽⁸⁾ Based on the findings of previous studies^(1,4), a sample size of 8 per group ($n = 8$) was sufficient to detect a large effect size ($d = 0.5$) with a statistical power ($1 - \beta$) of 0.80

(80%) and a significance level (α) of 0.05 (5%) for a two-sided hypothesis test. The sample size was increased to 10 per group to account for potential disruptions that might interfere with the completion of the process.

Teeth selection

A total of 30 extracted human primary mandibular second molars with intact crowns were collected from the outpatient clinic of the Pediatric and Community Dentistry Department at Minia University Dental Hospital (MUDH), based on specific inclusion and exclusion criteria. Primary molars near their physiological exfoliation time, over-retained molars, and molars extracted for orthodontic reasons or due to periodontal involvement were included in the study.^(9,10)

Inclusion criteria

Primary molars with sound and caries-free crowns.⁽⁷⁾

Exclusion criteria

Primary molars with caries, restorations, developmental anomalies fractured crowns or cracks were excluded.^(1,11)

Tooth specimen preparation

The selected teeth were cleaned with a periodontal scaler (Nordent, #N5-5S, Illinois, USA) under running water to remove blood and adherent soft tissues.⁽¹⁾ They were then immersed in 0.5% chloramine solution for one week for disinfection.⁽¹²⁾ The teeth were then preserved in sterile distilled water at 4°C for a maximum of one month. The distilled water was replaced every week, and the storage process was conducted following local infection control protocols.⁽¹³⁾

The occlusal surfaces of the teeth were flattened using a diamond disc bur (VladMiVa, Russia) mounted on a high speed handpiece (Dentsply Sirona T3 Racer, Germany) under water coolant, until

dentin became visible and the enamel was completely removed. The exposed dentin surfaces were then smoothened and polished with #600 grit silicon carbide (Sic) sanding paper (Microcut, Buehler, Lake Bluff, IL, USA) under running water.^(14,15) Subsequently, the teeth were sectioned bucco-lingually into mesial and distal halves using a diamond disc rotating at high speed under water coolant.

Creation of artificial carious lesions in dentin

Two-thirds of the specimens (40 tooth halves) were randomly selected and immersed in a demineralizing solution containing 2.2 mM CaCl_2 , 2.2 mM KH_2PO_4 , 50 mM acetic acid (pH 4.4) for 96 hours to create a flat zone of artificial carious lesion in dentin.⁽¹¹⁾

The tooth specimens were then embedded in plastic molds filled with cold-cure acrylic resin (Acrostone, Egypt), with the dentin surfaces exposed and flushed with the upper rim of the mold.⁽¹⁵⁾

Grouping of specimens

Each specimen was assigned a code number, and randomization was then performed using an online random number generator (www.randomizer.org) to allocate the specimens into groups.

The 20 sound dentin specimens were divided into two subgroups (Ia and IIa) according to the type of restorative material used: either Beautifil Flow Plus F00 (BFP) (Shofu Inc., Kyoto, Japan) or Surefil One (Su-O) (Dentsply Sirona, Konstanz, Germany) ($n = 10$).

The 40 demineralized dentin specimens were assigned to four subgroups (Ib, IIb, Ic, and IIc) based on the surface pretreatment of dentin (with or without 38% SDF) and the type of restorative material used (BFP or Su-O) ($n = 10$).

Group Ia: Sound dentin + BFP ($n = 10$).

Group Ib: Demineralized dentin + BFP ($n = 10$).

Group Ic: Demineralized dentin + 38% SDF + BFP (n = 10).

Group IIa: Sound dentin + Su-O (n = 10).

Group IIb: Demineralized dentin + Su-O (n = 10).

Group IIc: Demineralized dentin + 38% SDF + Su-O (n = 10).

SDF application and restorative procedures

The SDF solution and all restorative materials were applied to the dentin surfaces in accordance with the manufacturer's instructions.

For the SDF-treated groups, a single drop of 38% SDF solution (Toothmate, Egypt) was applied to the dentin surface using a microbrush applicator (Cotisen, China). The solution was actively agitated for one minute and then left on the dentin surface for two minutes to be absorbed into the tooth.

To create a standardizing bonding area, a cylindrical split Teflon mold with a metal ring, having an internal diameter of 1 mm and a height of 2 mm, was used and fixed in place above the dentin surface using a custom made shear bond strength fixation device.

In the BFP groups, a universal adhesive (All-Bond Universal, Bisco Inc., Schaumburg, IL, USA) was applied to the dentin surface in self-etch mode and then light-cured for 10 seconds using an LED light curing unit (Bludent power pen cordless, #200-003p, BG LIGHT LTD, Bulgaria) with light intensity of 1100 mW/ cm² and 410- 490 nm wavelength. The mold was subsequently filled with Beautiful Flow Plus F00 and light-cured for 10 seconds.

In the Su-O groups, the capsule was mixed using an amalgamator at 4200– 5000 rpm for 10 seconds. The material was then applied directly into the mold using a capsule applicator gun and light-cured for 20 seconds.

Once the restorative material had fully cured, the fixation device and the Teflon mold were gently

removed to obtain a microcylinder of restorative material bonded to the dentin surface.

Micro-shear bond strength (μSBS) and failure mode analysis

A universal testing machine (Instron industrial products, Norwood, USA) was used to measure μSBS by applying a shear load parallel to the bonded interface using a chisel-shaped blade at a crosshead speed of 0.5 mm/min until bond failure occurred (Figure 1). The maximum load at fracture was recorded, and μSBS was calculated using the following formula:

$$\mu\text{SBS (MPa)} = \frac{\text{Maximum failure load (N)}}{\text{Bonded interface surface area (mm}^2\text{)}}^{(16)}$$

Following μSBS testing, all debonded surfaces were examined under a stereomicroscope (Leica Microsystems L2, Germany) at 25x magnification. Failure modes were classified as:

- Adhesive failure: at the interface between the dentin and restorative material.
- Cohesive failure: occurring within the restorative material.
- Mixed failure: involving both adhesive and cohesive components.⁽¹⁶⁾

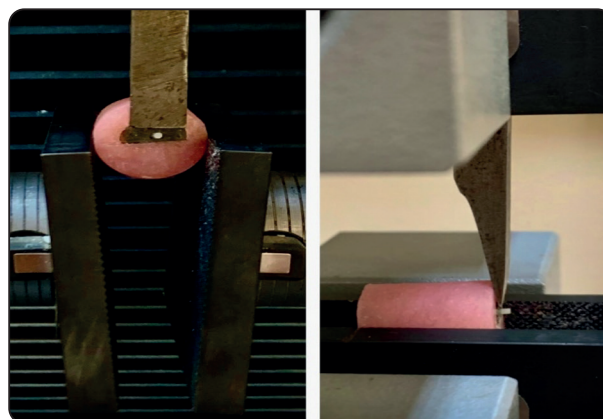


Fig. (1). Measuring μSBS using a universal testing machine.

Statistical analysis

All data were analyzed using SPSS software, version 27.0 (IBM Corp., Armonk, NY, USA). Continuous data were expressed as the median and interquartile range (median [IQR]) and analyzed using the Kruskal–Wallis H test, followed by post hoc pairwise comparisons with Bonferroni-adjusted Mann–Whitney U tests, since the data were not normally distributed according to the Kolmogorov–Smirnov and Shapiro–Wilk normality tests. Categorical data were expressed as frequencies and percentages (n, %), and Fisher’s exact test with Monte Carlo simulation was used to compare categorical variables between the study groups. A significance level of $p \leq 0.05$ was considered statistically significant for all tests.

RESULTS

I. Micro-Shear Bond Strength (μ SBS):

In general, the Beautifil Flow Plus (BFP) groups exhibited the highest micro-shear bond strength (μ SBS) values, whereas the Surefil One (Su-O) groups showed the lowest. The median μ SBS of

BFP was significantly higher in the sound dentin group compared to the demineralized dentin group ($p < 0.01$). Additionally, although the μ SBS of Su-O decreased in demineralized dentin compared to sound dentin, the difference was not statistically significant ($p = 0.191$). SDF application resulted in a non-significant decrease in the μ SBS of BFP compared to demineralized dentin not treated with SDF ($p = 0.402$). In contrast, the application of SDF significantly increased the μ SBS of Su-O compared to demineralized dentin not treated with SDF ($p < 0.001$) (Table 1).

II. Mode of failure:

A statistically significant overall difference was observed in the failure mode distribution among the study groups ($p = 0.011$). A statistically significant difference was found only between the sound dentin group restored with BFP and the demineralized dentin group restored with Su-O ($p = 0.03$).

Cohesive failures were observed exclusively in the BFP groups, with no cohesive failures recorded in any of the Su-O groups (Table 1).

TABLE (1) Median μ SBS in MPa with interquartile range [IQR] and failure mode distribution among the study groups.

Study group	μ SBS (MPa) Median [Q1–Q3]	Failure mode		
		Cohesive n (%)	Adhesive n (%)	Mixed n (%)
SD + BFP	46.97 [42.59–49.87] ^a	7 (70%)	1 (10%)	2 (20%)
DD + BFP	32.14 [28.46–34.7] ^b	3 (30%)	3 (30%)	4 (40%)
DD + SDF + BFB	26.66 [23.44–31.11] ^{bcef}	1 (10%)	4 (40%)	5 (50%)
SD + Su-O	12.92 [11.01–15.92] ^{cef}	0 (0%)	5 (50%)	5 (50%)
DD + Su-O	10.87 [10.03–11.72] ^e	0 (0%)	6 (60%)	4 (40%)
DD + SDF + Su-O	15.92 [13.33–18.32] ^{bf}	0 (0%)	4 (40%)	6 (60%)

Abbreviations: SD, sound dentin; DD, demineralized dentin. Median values with different superscript letters are statistically significant different ($p < 0.01$) based on post hoc analysis. Values sharing the same superscript letter are not significantly different ($p > 0.05$).

DISCUSSION

SDF is now commonly used for the prevention and arrest of carious lesions; however, its tendency to cause tooth discoloration remains a significant drawback.⁽¹⁵⁾ To minimize staining and restore the damaged tooth structure, esthetic restorative materials are commonly placed after the use of SDF.⁽¹⁷⁾

The aim of this in-vitro study was to evaluate the effect of SDF pretreatment on the μ SBS of two different esthetic restorative materials to demineralized dentin of primary teeth.

In this study, dentin was artificially demineralized to create a bonding substrate similar to natural carious lesions, thereby yielding results that are more applicable to clinical practice.⁽³⁾

Clinically, shear bond strength is particularly relevant because the predominant dislodging forces acting at the tooth–restoration interface are shear in nature.⁽¹⁸⁾ In the present study, the μ SBS test was used to reduce the uneven stress distribution often associated with conventional shear bond strength tests, thereby minimizing errors.⁽¹⁹⁾

The results of this study showed that the BFP groups exhibited higher bond strength values compared to the Su-O groups. This finding aligns with the results of **Abuljadayel et al. (2023)** and **Zahran et al. (2023)**, who found that the shear bond strength of giomer was higher than that of Su-O. They attributed the superior bonding efficiency of giomer to its high content of pre-reacted glass (PRG) fillers, and to the application of an adhesive agent prior to giomer placement, as recommended by the manufacturer, which promotes micromechanical retention and enhances adhesion to dentin. In addition, **Sharma et al. (2024)** attributed the high bonding performance of giomer to the modified surface layer created by surface pre-reacted glass (S-PRG) fillers, which improves substrate hydrophilicity and offers a more favorable bonding interface.

A possible explanation for the low bond strength observed with Su-O was provided by **Elraggal et al. (2024)**, who attributed the reduced bond strength of self-adhesive composites to their high viscosity and limited wettability, which impair their interaction with the tooth substrate and reduce micromechanical interlocking. Additionally, these materials generally contain lower concentrations of acidic monomers than self-etch or universal adhesive agents, further limiting their bonding capability, a finding supported by earlier studies by **Makishi et al. (2015)** and **Asiri et al. (2021)**.

The results also demonstrated that both BFP and Su-O exhibited higher bond strength when applied to sound dentin compared to demineralized dentin. This finding is in agreement with previous studies by **Keskin et al. (2021)**, **Khor et al. (2022)**, and **Sharma et al. (2024)**. This may be attributed to the decreased mineral content in demineralized dentin, which negatively influences bonding efficiency and subsequently affects the mechanical properties of the bonded restorative material.⁽²⁵⁾

Regarding the effect of SDF on μ SBS, our results revealed that its application reduced the bond strength of BFP to demineralized primary dentin, although this reduction was not statistically significant. In contrast, SDF application significantly enhanced the bond strength of Su-O. Therefore, the null hypothesis regarding the effect of SDF on μ SBS is accepted for BFP, while it is rejected for Su-O. This variation in SDF's impact on bond strength may be attributed to the distinct adhesion mechanisms employed by each material when bonding to dentin. These findings align with those of **Abuljadayel et al. (2023)**, who found that SDF impaired the bond strength of giomer (Beautifil II) to dentin while improving that of Surefil One.

Since giomer bonds to dentin primarily through micromechanical interlocking mediated by an adhesive agent, the occlusion of dentinal tubules by silver precipitates formed through the reaction of SDF with dentin, and the resulting impairment

of adhesive infiltration, may explain the reduced bond strength of BFP following SDF application.^(4,5,26) This finding is consistent with those of a systematic review by **Fröhlich et al. (2020)**, which reported that SDF application impairs the bonding effectiveness of adhesive systems to dentin.

Despite the similarity of results in several studies, a contrasting finding was reported by **Danaeifar et al. (2022)**, who found that SDF application did not affect the shear bond strength of giomer and composite materials to dentin. This discrepancy may be attributed to differences in experimental protocols, as their methodology included phosphoric acid etching followed by rinsing after SDF application. Such etching may have removed silver deposits and neutralized the alkaline surface layer formed by SDF, thereby enhancing resin infiltration and improving adhesive bond strength.

On the other hand, the bonding of Su-O to dentin is based on two mechanisms. The primary mechanism involves chemical interaction between calcium ions in the hydroxyapatite of the tooth substrate and the carboxylate groups of both MOPOS monomers and acrylic acid, which are present in Su-O. The secondary mechanism is micromechanical interlocking, achieved through partial etching and hybridization of the dentin surface.^(5,7)

A plausible explanation for the improved bond strength of Su-O following SDF application is that the silver deposits may enhance ionic interactions between the dentin and Su-O, as silver phosphate could chemically bond with the carboxylate groups in the material.^(5,27) Supporting this, **Abuljadayel et al. (2023)** observed an improved interfacial bond between Su-O and SDF-treated dentin under scanning electron microscopy compared to the control group, further validating the enhanced adhesion of Su-O achieved with SDF pretreatment.

Pertaining to the influence of SDF on the mode of failure, a decrease in cohesive failures and an increase in adhesive failures were observed in BFP specimens following SDF application. In contrast,

Su-O specimens demonstrated a reduction in adhesive failures and an increase in mixed failures. However, these differences in failure mode distribution were not statistically significant. Accordingly, the null hypothesis is accepted for both materials in terms of their failure modes. These findings are in agreement with those demonstrated by **Abuljadayel et al. (2023)**.

Limitations of this study include the challenge of replicating the complex oral environment, particularly factors such as temperature fluctuations, pH changes, and occlusal forces, all of which may influence bonding outcomes. Another limitation is that bond strength was assessed only immediately after SDF application. In clinical practice, a delay between SDF application and the placement of the restorative material may occur, which could affect bonding performance.

CONCLUSION

It was concluded that SDF application resulted in a non-significant reduction in the bonding efficiency of BFP to demineralized dentin, while significantly enhancing the bond strength of Su-O.

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