

Shear bond strength evaluation of bioactive restorative materials on pretreated carious dentin-influence on silver diamine fluoride, potassium iodide, and glutathione

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Abstract

Background: Silver diamine fluoride (SDF) has gained popularity for its caries-arresting properties, yet its tendency to cause esthetic concerns due to black-staining limits its widespread acceptance.

Aim: The aim of the study was to evaluate and compare the shear bond strength of Activa BioActive and Giomer restorative material with different protocols of SDF pretreatment on carious dentin.

Materials and Methods: Ninety-two extracted teeth were decoronated at the cemento-enamel junction, sectioned 1 mm into dentin, mounted in acrylic resin and randomly divided into four ($n = 8$) control and six ($n = 10$) experimental groups. Seventy-six samples underwent demineralization process for a period of 14 days for artificial carious dentin (ACD) formation. The samples categorized based on the dentin substrate (sound or ACD) were subjected to various SDF pretreatment protocols, i.e., only SDF, SDF with potassium iodide (KI), and SDF with glutathione (GSH). Further, bioactive restorative materials, i.e., Activa BioActive and Giomer (Beautiful II), were placed on it, and the samples were subjected to shear bond strength testing.

Statistical Analysis: Independent *t*-test was run to analyze the values obtained.

Results: Giomer exhibited better mean bond strength with SDF, SDF + KI, and SDF + GSH (6.56, 4.67, and 3.34 mega-pascals [MPa], respectively) compared to Activa BioActive (3.42, 3.27, and 2.96 MPa, respectively).

Conclusion: This study contributes to understanding the interplay between SDF application protocols, esthetic concerns, and the adhesive properties of bioactive restorative materials. Giomer exhibited enhanced bond strength after SDF application, unlike Activa BioActive. In addition, incorporation of KI or GSH adversely affected the bond strength of both the restorative materials, underscoring the critical need for cautious clinical application.

Clinical Relevance: This study highlights the importance of selecting appropriate dentin pretreatment agents to maximize the bond strength of bioactive restorative materials with carious dentin. SDF application significantly enhanced the bond strength of Giomer with carious dentin compared to Activa BioActive, thus, making it a good choice for restoring nonesthetic areas. In

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Date of submission : 28.07.2024

Review completed : 29.08.2024

Date of acceptance : 09.09.2024

Published : 11.11.2024

Access this article online

Quick Response Code:



Website:
<https://journals.lww.com/jcde>

DOI:
10.4103/JCDE.JCDE_523_24

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How to cite this article: Sharma N, Nikhil V, Mishra P, Ravinder R. Shear bond strength evaluation of bioactive restorative materials on pretreated carious dentin-influence on silver diamine fluoride, potassium iodide, and glutathione. *J Conserv Dent Endod* 2024;27:1146-51.

addition, the application of KI or GSH to mitigate discoloration of carious dentin negatively affected the bond strength of both, Activa BioActive and Giomer. Thus, the clinicians should weigh the benefits of SDF against potential bond strength reductions when using KI or GSH, especially for esthetic restorations.

Keywords: Activa BioActive; Beautifil II; bioactive restorative materials; carious dentin; dentin pretreatment; Giomer; glutathione; potassium iodide; silver diamine fluoride

INTRODUCTION

Most widely spread noncommunicable and preventable disease globally is dental caries, impacting 2.5 billion individuals.^[1] Its prevalence in the Indian population aged 3–75 years is 54.16%.^[2] Over the last century, management ideology of dental caries has undergone an evolution from a purely surgical “drill and fill” approach and G. V. Black’s cavity designs, emphasizing the “extension for prevention” paradigms to recently, the emergence of the minimal intervention dentistry, aiming at preserving the natural tooth structure and adopting a biological approach to treat caries. The ultraconservative treatment concepts involve the excavation of only the outer layer which is highly denatured and caries-infected dentin whereas, preservation of the inner layer consisting of intact, bacteria free, remineralizable, and caries-affected dentin during cavity preparation.^[3] Lately, various bioactive materials with antibacterial and/or mineralizing properties for the treatment of caries have emerged that are capable of hydroxyapatite crystals formation on tooth surfaces.

Silver diamine fluoride (SDF), due to its caries-arresting properties.^[4] Its application on teeth results in the formation of calcium fluoride, serving as a receptacle for fluoride ions, facilitating the formation of fluorapatite—a more acid-resistant compound than hydroxyapatite. In addition, SDF forms silver phosphate, which possesses antibacterial properties, is insoluble and precipitates on the tooth surface. This dual action helps prevent further dental caries and hardens existing lesions.^[5] Despite its benefits, SDF’s tendency to create a hard, blackened, and impermeable layer on tooth surfaces raises esthetic concerns. To mitigate this, potassium iodide (KI) can be applied after SDF treatment, forming a yellow, insoluble silver iodide precipitate that does not cause black staining on teeth.^[4] Another approach to reduce discoloration is the use of glutathione (GSH) biomolecules, which exhibit a strong affinity for metal surfaces such as silver, not only decreasing the rate of release of silver ions but also preventing their aggregation by formation of a protective coating over them.^[6] Several studies^[6–8] have explored the application of GSH with SDF to mitigate its potential toxic effects.

Various studies demonstrated the efficacy, safety, and caries-arresting properties of SDF, but fewer had evaluated the bond strength to caries dentin treated with SDF.

A review of the literature^[3,5,9] reveals a varied agreement on bond strength following SDF application. In addition, numerous studies^[8–11] have assessed the bonding of glass ionomer cements, resin-modified glass ionomer cements, and composite resins on SDF-treated dentin, while very few examined the bond strength of bioactive restorative materials in combination with SDF-treated dentin, thereby leading to a scarcity of literature on the same. To the best of our knowledge, only one study has evaluated the effect of GSH in combination with SDF on the bond strength of glass ionomer cement.^[8] However, no research has investigated the effect of GSH on the bond strength of bioactive restorative materials. Therefore, this study aimed to evaluate the bond strength of Activa Bioactive restorative and Giomer to KI or GSH in combination with SDF pretreated carious dentin. The null hypothesis tested was that the application of KI or GSH would not affect the bond strength of Activa Bioactive and Giomer when bonded to SDF-pretreated carious dentin.

MATERIALS AND METHODS

Freshly extracted human permanent maxillary and mandibular molars were collected and cleaned of any debris using ultrasonic scaler (BioSonic S1 L Piezo Ultrasonic Scaler System, Coltene, Switzerland). Teeth were then examined under magnification (3x Brilliance, Orikam, China) and illumination to select caries-free and intact nonrestored teeth. The selected teeth were stored in a 0.5% chloramine T (0.5% Ellis Horwood series in chemical science, Delhi) solution until further use.

Sample preparation

Sample size was estimated to be 10 for each group, following study of Kim *et al.*^[12] Ninety-two extracted teeth were decoronated at the level of cemento-enamel junction, and the samples were sectioned from occlusal surface approximately 1 mm into dentin from dentin-enamel junction, using a diamond-impregnated disc under continuous supply of water, to obtain dentin slices [Figure 1a]. Self-cure acrylic resin (Acryton-R, Uttar Pradesh, India) was mixed and poured into Teflon molds (25 mm × 20 mm). Dentin slices, with the dentin surface facing upward, were embedded in the resin [Figure 1b] and left to set for 24 h. Each sample was then polished with 600 grit sandpaper under running water to expose the desired dentin surface. Samples were then randomly and equally divided into groups ($n = 8$ for control groups and $n = 10$ for experimental groups)

according to dentin pretreatment agents and restorative materials used [Table 1].

Demineralization process

Seventy-six samples were subjected to pH cycling for artificial carious dentin (ACD) formation as per protocol followed by Follak *et al.*^[13] The pH cycling method involved two solutions: demineralizing solution (2.2 mM NaH₂PO₄, 2.2 mM CaCl₂, and 50 mM acetic acid adjusted at pH of 4.8 with 1M KOH) and remineralizing solution (0.9 mM NaH₂PO₄, 1.5 mM CaCl₂, and 0.15 M KCl adjusted at pH of 7.0). Each sample was cycled in 10 mL of both solutions, for 8 h in the demineralizing solution and 16 h in the remineralizing solution, over a period of 14 days at room temperature without agitation.

Dentin pretreatment agent application protocol

- Thirty-eight percent SDF solution (E-SDF, Kids-e-Dental, India) was applied to the dentin surface and agitated using an applicator tip for 1 min. It was left for 2 min and then, rinsed with a copious amount of distilled water for 30 s^[14]
- SDF with KI: SDF was applied to the dentin surface as described before. After its application, saturated 10% KI solution (Bangalore Fine Chemicals, Bengaluru, India) was applied. A creamy white precipitate formed,

which once turned clear was washed off with copious amounts of distilled water for 30 s^[8]

- SDF mixed with 20% GSH: 25 mL SDF solution was mixed with 5 mg GSH (Research-Lab Fine Chem Industries, Mumbai, India) by weight to prepare 20% GSH solution in SDF. Solution was vigorously stirred until a clear solution was obtained without any precipitates.^[15] It was applied on the sample in the same manner as the SDF solution described previously.

Samples were rinsed with water for 5 s and dried. Subsequently, 37% orthophosphoric acid etchant (Magic Acid Etchant Gel 37%, Coltene, Switzerland) was applied for 15 s^[16] and thoroughly rinsed for 20 s to ensure no residual color was visible, followed by drying for 5 s. The bonding agent (One Coat Bond SL, Coltene, Switzerland) was then applied with an applicator tip for 20 s, ensuring even coverage with continuous scrubbing, air dried, and light cured for 30 s according to the manufacturer’s protocol. Activa BioActive restorative (Pulpdent Corporation, Watertown, MA, USA) and Giomer (Beautiful II; Shofu Inc., Kyoto, Japan) were placed in 2 mm increments and shaped into cylindrical forms using a mold (4 mm × 4 mm) on the samples [Figure 1c], following manufacturer’s instructions. Each layer was light cured for 20 s using an LED light-curing unit (woodpecker Light cure LED Mini-S, China) with 1000 mW/cm² intensity.

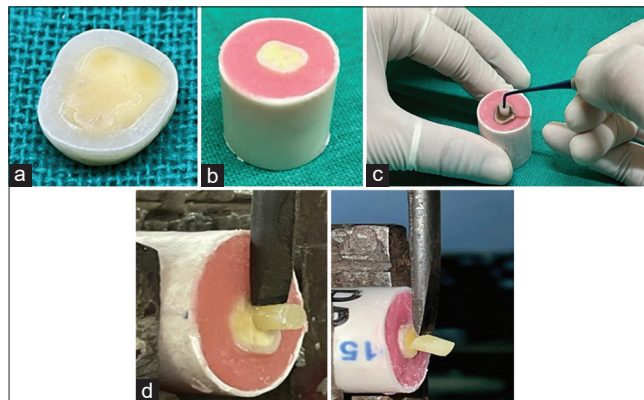


Figure 1: (a) Dentine slice, (b) dentin slice mounted on self-cure acrylic resin, (c) restorative material application, (d) shear bond strength testing

Samples stored for 24 h at 37°C in an incubator were subjected to 5000 thermal cycles (5°C/55°C, 20 s each cycle with 5 s interval) before shear bond strength testing. Shear bond strength testing was performed utilizing universal testing machine (Micronix, MI-16 × 2 ECO-TTM, Uttar Pradesh, India) at 1 mm/min crosshead speed with a blade parallel to the interface between restorative material and dentin [Figure 1d]. Load at failure was recorded in mega pascals (MPa), by dividing failure load by bonded surface area in square mm. Results were tabulated, and data were analyzed using SPSS 26.0 software (IBM, New York, USA). Mean and standard deviation were calculated for each group and an independent *t*-test assessed the shear bond strength. Level of significance was set at 0.05.

Table 1: Control and experimental groups according to dentin pretreatment agents and restorative materials

Group	Dentin type	Dentin pretreatment agent	Restorative material
Group 1 (BP) (n=8)	Sound dentin	-	Beautiful II
Group 2 (BN) (n=8)	ACD	-	Beautiful II
Group 3 (BS) (n=10)	ACD	SDF	Beautiful II
Group 4 (BSK) (n=10)	ACD	SDF with KI	Beautiful II
Group 5 (BSG) (n=10)	ACD	SDF with glutathione	Beautiful II
Group 6 (AP) (n=8)	Sound dentin	-	Activa BioActive restorative
Group 7 (AN) (n=8)	ACD	-	Activa BioActive restorative
Group 8 (AS) (n=10)	ACD	SDF	Activa BioActive restorative
Group 9 (ASK) (n=10)	ACD	SDF with KI	Activa BioActive restorative
Group 10 (ASG) (n=10)	ACD	SDF with glutathione	Activa BioActive restorative

ACD: Artificial carious dentin, SDF: Silver diamine fluoride

RESULTS

Table 2 demonstrates that Giomer had higher bond strength with sound dentin (6.73) compared to ACD (5.06). Similarly, Activa BioActive showed stronger bond with sound dentin (5.47) than ACD (4.12). Furthermore, Giomer’s shear bond strength significantly increased after SDF application ($P = 0.002$), while the AS group showed a nonsignificant decrease compared to the AN group ($P = 0.127$) [Figure 2]. BSK group had significantly lower shear bond strength than BS group ($P < 0.001$), and ASK group had an almost similar bond strength to AS group ($P = 0.647$). Table 2 also reveals that BSG group had significantly lower shear bond strength than BS group ($P < 0.001$), and ASG group showed no significant difference compared to AS group ($P = 0.230$).

DISCUSSION

The use of silver diamine treatments has significantly risen in recent years as an effective method for arresting carious lesions; however, their potential to cause discoloration remains a major concern. KI has been employed to address

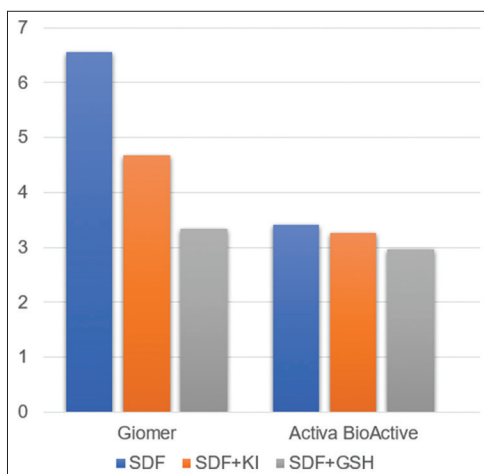


Figure 2: Shear bond strength (megapascals) of Giomer and Activa BioActive restorative materials to only Silver diamine fluoride (SDF), SDF with potassium iodide, and SDF + glutathione-treated carious dentin. SDF: Silver diamine fluoride, KI: Potassium iodide, GSH: Glutathione

tooth discoloration caused by SDF as it binds the free silver ions from the SDF by forming silver iodide crystals, preventing them from forming black precipitates on the teeth.^[17] GSH, which has been developed as an alternative to KI for combating tooth discoloration, has a strong affinity for metal surfaces. This enables GSH to form a protective layer around silver particles and reduce the release of silver ions, thereby playing a crucial role in minimizing the discoloration of a tooth treated with SDF.^[18,19]

The current study’s findings indicated that the use of KI or GSH with SDF protocols impacted the bond strength of both Activa BioActive and Giomer to demineralized dentin. Therefore, the null hypothesis was rejected.

In the present study, both Activa BioActive and Giomer bonded stronger with sound dentin compared to carious. Keskin *et al.* also observed that bond strength to caries-affected dentin was typically 20%–50% lower than to sound dentin.^[20] This may be because of morphological and chemical changes in mineralized tissues in caries-affected dentin.^[21] Due to denatured collagen fibrils, there is a lack of cross-linking and inadequate resin infiltration in interfiber collagen spaces, which could compromise the bonding.

On application of SDF, the bond strength of Giomer to carious dentin increased as compared to its negative control, approaching near-to-sound dentin, endorsing findings of Abuljadayel *et al.* This increase may be due to resin monomers that maintain mechanical strength and facilitate effective copolymerization with the adhesive, along with the presence of prepolymerized and surface prereacted glass-ionomer (S-PRG) fillers (constituting 83.3 wt%).^[22]

In this study, Giomer demonstrated significantly better bond strength to ACD compared to Activa BioActive. Gomers are resin-based restorative materials that release fluoride through S-PRG fillers, and during production, S-PRG fillers form a modified surface layer by an acid–base reaction in the presence of water.^[20] In our study, Gomers likely enhanced substrate hydrophilicity, providing a suitable bonding surface and facilitating effective and consistent

Table 2: Comparisons of shear bond strength of Giomer and Activa BioActive to sound dentin and artificial carious dentin in combination with only silver diamine fluoride, silver diamine fluoride with KI, and silver diamine fluoride with glutathione-treated carious dentin

Comparison	Group	Mean	SD	Group	Mean	SD	Difference	P
BN versus BP	BN	5.06	0.46	BP	6.73	1.18	–1.67	0.039*
AN versus AP	AN	4.12	0.36	AP	5.47	0.29	–1.35	0.001*
BN versus BS	BN	5.06	0.46	BS	6.56	0.71	–1.5	0.002*
AN versus AS	AN	4.12	0.36	AS	3.42	0.80	0.7	0.127
BSK versus BS	BSK	4.67	1.17	BS	6.56	0.71	–1.89	<0.001*
ASK versus AS	ASK	3.27	0.64	AS	3.42	0.80	–0.15	0.647
BSG versus BS	BSG	3.34	0.69	BS	6.56	0.71	–3.22	<0.001*
ASG versus AS	ASG	2.96	0.86	AS	3.42	0.80	–0.46	0.230

*Significant difference at $P \leq 0.0$. Independent t-test. SD: Standard deviation

reconstruction in demineralized dentin. This reconstructed mineralized surface, guided by a scaffold of collagen matrix, may have exhibited high surface energy and wettability for resin monomers. In addition, Giomer demonstrates low volumetric shrinkage (0.85%) and polymerization shrinkage stress (2.72 MPa); however, Kanchanasita *et al.* noted Activa BioActive's ability to absorb up to 7% of water by mass, attributed to its polyhydroxyethylmethacrylate content, which could potentially lower its bond strength.^[23]

Application of KI to SDF-treated carious dentin significantly reduced the bond strength of Giomer compared to dentin treated only with SDF. This finding aligns with Van Duker *et al.*, who found that despite rinsing to remove KI/SDF residues, bond strength remained low when SDF + KI was used. The application of SDF + KI resulted in a white-yellow substrate on demineralized dentin surfaces that resisted removal by etching or rinsing. Scanning electron microscopy observations indicated this precipitate acted as the bonding substrate, hindering resin penetration, and leaving tubules near the surface empty.^[24] Similar trends were noted for Activa BioActive, although the reduction was not statistically significant, consistent with studies by Kim *et al.*^[12] and Uctasli *et al.*^[25]

Applying GSH to SDF-treated carious dentin also reduced the bond strength of Giomer and Activa compared to dentin treated only with SDF. As previously mentioned, the use of GSH to reduce tooth staining after SDF application is a relatively new approach. Consequently, there is limited information available. Most studies have focused on evaluating its cytotoxicity and remineralizing capabilities, with only one study examining its bond strength – but not in the context of bioactive materials. Karuna *et al.* demonstrated that the SDF + GSH group showed lower remineralization microhardness values compared to the SDF and SDF + KI groups.^[7] Furthermore, Asghar *et al.* suggested that GSH concentrations above 15% could lead to saturation and crystallization.^[26] In the present study, the use of 20% GSH may have reduced the mechanical properties of caries-affected dentin, possibly hindering material adhesion due to the formation of precipitated crystals.

Limitations of this study include the inherent differences between *in vitro* and clinical settings. Artificially created demineralized carious lesions were used to standardize the samples for bond strength testing, unlike clinical carious lesions. Further research is needed to explore the impact of delayed bonding with various bioactive restorative materials over different time intervals following SDF pretreatment protocols. Future studies could also investigate the use of self-etch adhesives in conjunction with SDF pretreatment agents. In addition, both *in vivo* and *in vitro* studies are necessary to evaluate the

concentration-dependent effects of GSH combined with SDF on dentin discoloration, remineralization, and bond strength to bioactive restorative materials.

CONCLUSION

Within the constraints and limitations of this *in vitro* study, the conclusions drawn were that Activa BioActive and Giomer demonstrated superior bond strength to sound dentin compared to ACD. The application of SDF significantly increased the bond strength of the Giomer restorative material to demineralized dentin. Therefore, Giomer may be recommended in conjunction with SDF application in nonesthetic areas. Conversely, the bond strength of Activa BioActive decreased after SDF application, although the decrease was not statistically significant. In addition, the application of KI/GSH to reduce SDF-induced discoloration of carious dentin negatively affected the bond strength of the Giomer restorative material to ACD. Similar effects were observed for Activa BioActive, although the reduction was not significant. Given the limited research on the effect of GSH on bond strength, further studies are needed to better understand its impact in combination with SDF application on bioactive materials.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

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