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Shear Bond Strength of Glass Ionomer Cement to Silver Diamine Fluoride Treated Caries

Elizabeth Ng

Abstract

Purpose: *The purpose of this study is to measure microshear bond strength* ***(µSBS)*** *of glass ionomer cement* ***(GIC)*** *to carious dentin with and without silver diamine fluoride* ***(SDF)*** *treatment.*

Methods: *Permanent molars were sectioned and demineralized to create artificial carious lesions. Variables tested included the demineralization of the dentin, application of SDF, use of conditioner, and time between SDF and restoration. µSBS was measured after 24 hours using an UltraTester machine.*

Results: *The strongest bond strength was found when GIC was placed on conditioned and demineralized dentin treated with SDF one week prior. There was no statistical difference to µSBS with and without SDF. Statistically significant increases in bond strength were found when the dentin was demineralized, when conditioner was applied before SDF, and when one week elapsed between SDF application and GIC placement. The lowest bond strength was found with immediate GIC application after SDF.*

Conclusions: *Results suggest that optimal retention is obtained by conditioning with polyacrylic acid and allowing SDF treatment to set for one week prior to GIC placement.*

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Introduction

Dental caries is the most common disease affecting childhood. [1] Recent traditions in the comprehensive treatment of caries in pediatric dentistry have focused on removing infected tooth structure and filling with restorative materials such as amalgam, composite, or glass ionomer cement **(GIC)**. However, arresting caries progression with topical silver solutions also has a long history.[2] As recently encouraged by the American Dental Association, arresting carious lesions is an appropriate treatment option.[3] This may be the most realistic option in certain clinical scenarios such as holding care, special needs dentistry, and the pre-cooperative child. The best result may be achieved by combining these two options: applying a caries arresting medicament and following with a restorative material, as in the so-called silver-modified atraumatic restorative treatment **(SMART)**.[4]

Aqueous silver diamine fluoride 38% **(SDF)** has been utilized for decades in various countries including China, Japan, Germany, Nepal, Brazil, Argentina, New Zealand, and Australia, to arrest caries.[5, 6] In 2014, SDF was cleared by the US FDA as a class II medical device for the treatment of dentinal hypersensitivity. Silver diamine fluoride contains two ammine groups and the chemical name should be silver diammine fluoride to illustrate the formation of an ion complex with silver in solution.[7, 8] Recent literature uses "diamine", which was used for this paper.

The mechanism of action for SDF mediated caries arrest is not fully understood, and is multifactorial in nature. SDF targets both organic and inorganic components in the carious lesion. Locally, the insoluble layer formed by precipitated oxidized silver (silver phosphate, silver oxide, and silver chloride) increases remineralization, obturates dentinal tubules, and inhibits enzymes that break down the organic dentin matrix such as matrix metalloproteinases and cathepsins.[9] These phenomena increase the tooth's resistance to acid dissolution and enzymatic digestion, while plugging of dentinal tubules decreases sensitivity.[10] In regards to antibacterial effects, silver ions and possibly metallic silver inhibit bacterial enzymes such as collagenase, cell processes such as DNA replication, cell membranes, cell wall function, and biofilm formation.[10-14] Furthermore, dying bacteria release silver into the environment, thus "re-activating" the SDF to repeatedly act on live bacteria (so called the "zombie effect").[15] *In vitro*, it has been shown that SDF has antibacterial action against *S. mutans*, *S. sobrinus, L. acidophilus, Lactobacillus rhamnosus, Actinomycesnaeslundii*, and *E. faecalis*.[10, 16-18]

Numerous studies demonstrate the clinical effectiveness of SDF in arresting caries. In randomized controlled trials, Chu CH, 2002 and many others have demonstrated caries arrest.[19] Caries prevention is shown by Llodra JC, 2005 and Tan HP, 2010 in primary and permanent teeth.[20, 21] Many more studies have confirmed these findings. Meta-analysis showed that SDF was more effective than placebo in carious lesion arrest in primary teeth.[22] Additionally, research has shown a 400-fold margin of safety, normal pulpal response, and few minor adverse events such as staining of gingiva and clothes.[23, 24]

In cavitated lesions, SDF can be used in conjunction with GIC to combine the benefits of caries arrest and a restoration.[4] The term "SMART restoration", or Silver-Modified Atraumatic Restorative Treatment has been used to describe this treatment. Modern caries management emphasizes selective caries removal. Yet few studies have examined the effects of SDF on bond strength to carious lesions. The purpose of this study is to measure µSBS of a GIC to dentin with artificial carious lesions with and without the application of SDF. In addition, the effect of conditioner use and time lapse between SDF application and restoration placement will be examined.

Materials and Methods

Non-carious extracted human permanent molars were used for this study. Teeth were extracted for clinical reasons only and collected without documentation or personal identifiers and thus this study is exempt from need for human subject board review.

In all groups, extracted molars were gamma irradiated for 24 h for sterilization.[25] Then, they were sectioned along the occlusal plane to expose dentin just gingival to the dentino-enamel junction and polished to a 400-grit with silicone carbide sandpaper. Samples were stored in de-ionized water. Sectioned molars were coated with nail varnish (Revlon #270) to expose a 3 mm x 3 mm window of dentin. Groups 2 - 6 specimens were exposed to a demineralizing solution of acetic acid to create artificial carious lesions (66 h on a rocker in 0.05 M acetate buffer containing 2.2 mM calcium and phosphate at pH 5.0), as described elsewhere.[26] Previous studies have shown that this treatment creates an artificial lesion of about 140 µm depth and yields a reproducible flat demineralized zone consistent with standards required for measuring shear bond strength. All samples were then mounted in dental microstone (2.5 cm diameter x 3 cm height cylinder). At this point, all samples were stored in 100% humidity.

Six groups of samples were prepared in a microshear bond strength mold (UltraTester Bonding Clamp, Bonding Mold Inserts, Ultradent Products, Inc., South Jordan, Utah, USA) with the following materials where indicated below following manufacturers' instructions. Conditioner: GC Cavity Conditioner (GC America Inc., Alsip, Illinois, USA) (20% polyacrylic acid and 3% aluminum chloride hexahydrate) was applied with a microbrush for 30s and rinsed for 10s. SDF: Advantage Arrest Silver Diamine Fluoride 38% (Elevate Oral Care LLC, West Palm Beach, Florida, USA) was applied with a microbrush and excess removed with a cotton roll. GIC: GC Fuji IX GP capsules (GC America Inc., Alsip, Illinois, USA) (100% high viscosity glass ionomer cement) was triturated for 10s and applied with a plastic instrument. Fuji IX was chosen for its indication for use in posterior restorations. Zinc phosphate: zinc phosphate cement (Prime Dental Manufacturing, Inc., Chicago, Illinois, USA) was incrementally mixed on a refrigerator chilled glass slab and applied within two minutes. Zinc phosphate cement was included as one of the testing groups as it is a gold standard in dental cements.

Restoration Preparation Groups:

1. **Sound dentin**, conditioner, GIC

2. **Demineralized dentin**, conditioner, GIC

3. **Demineralized dentin**, conditioner, **SDF**, GIC

4. **Demineralized dentin**, SDF, GIC, **(no conditioner)**

5. **Demineralized dentin**, conditioner, SDF, GIC placed **immediately** after SDF

6. **Demineralized dentin**, SDF, zinc phosphate cement

In groups 1 - 4 and 6, the cement was placed one week after SDF placement. In group 5, GIC was placed immediately after SDF. In all groups, samples were incubated at 100% humidity at 37˚C for 24h to mature the cement prior to bond strength testing. The microshear **(µSBS)** bond strength in MPa was measured using the UltraTester Bond Strength Testing Machine (Ultradent Products, Inc., South Jordan, Utah, USA).

A sample size of 10 was chosen per group. Statistical analysis was performed using ANOVA and heteroscedastic paired T-tests with Microsoft Excel. The statistician was blinded to the groups.

Results

Results are summarized in Figure 1 and Table 1. Group 1 (n=10) had a mean µSBS of 6.5 MPa (standard deviation (SD)=2.0). Group 2 (n=10) had a mean bond strength of 10.4 (SD=4.5), showing that demineralization increases bond strength by 0.60X (p=0.02). Group 3 (n=10) had a mean of 13.2 (SD=3.4), which is 0.27X higher but not significantly different (p=0.13) than that for group 2, for which the only preparation difference was SDF treatment.

Group 4 (n=11) had a mean bond strength of 7.4 (SD=3.8). This was significantly different from group 3 (p=0.002): the use of conditioner created a 0.78X higher bond strength.

Group 5 (n=12) had a mean bond strength of 5.0 (SD=3.4). This was statistically significantly different from group 3: the immediate placement of GIC after SDF treatment resulted in 62% lower bond strength than GIC placed after one week after SDF (p<0.001). This group had the lowest bond strength of any GIC preparation.

In group 6 (n=6), all samples debonded in the bonding jig the cement fell off. Therefore no bond strength measurements were performed.

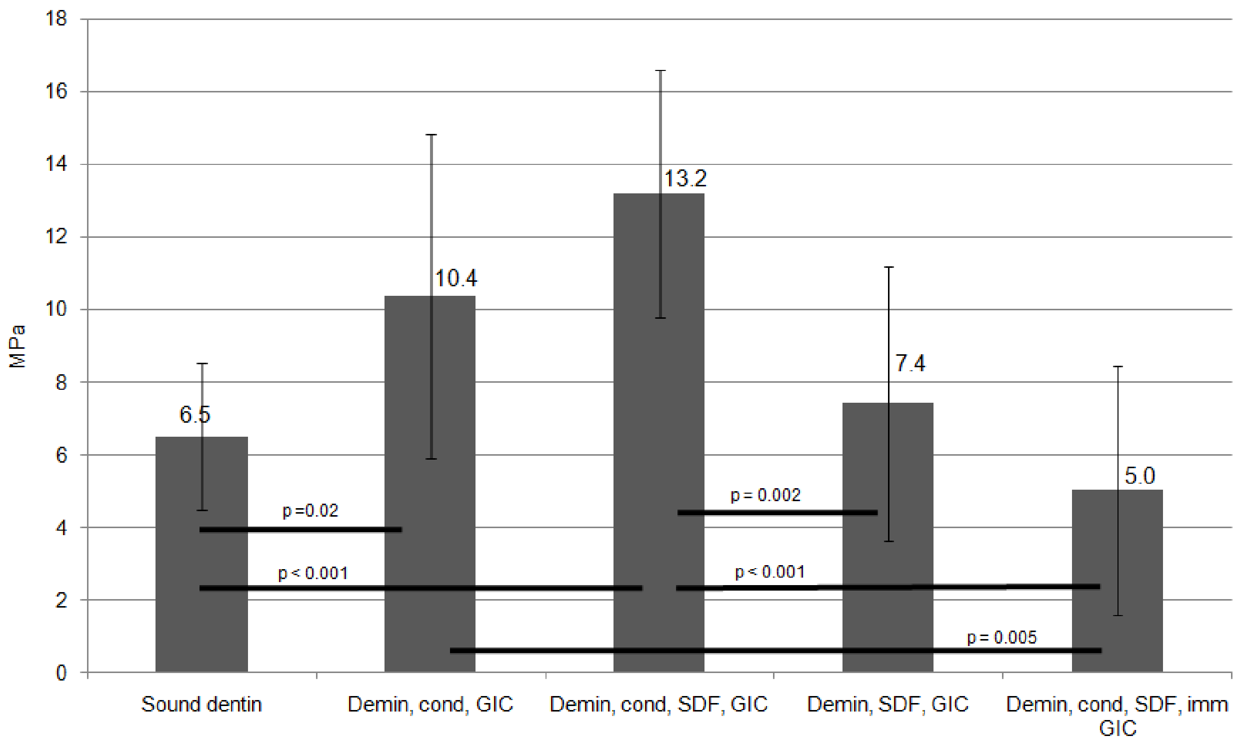

Figure 1. Microshear bond strength of glass ionomer cement to dentin samples. Demin: demineralized dentin. Cond: conditioner. GIC: GIC placed one week after SDF. Imm GIC: GIC placed immediately after SDF.

Group	n	Mean (MPa)	Standard Deviation (MPa)
1: Sound dentin	10	6.5	2.0
2: Demin, cond, GIC	10	10.4	4.5
3: Demin, cond, SDF, GIC	10	13.2	3.4
4. Demin, SDF, GIC	11	7.4	3.8
5. Demin, cond, SDF, imm GIC	12	5.0	3.4
6. Demin, SDF, zinc phosphate	6		

Table 1. Microshear bond strength to dentin samples. Demin: demineralized dentin. Cond: conditioner. GIC: GIC placed one week after SDF. Imm GIC: GIC placed immediately after SDF.

Discussion

Puwanawiroj and colleagues tested microtensile bond strength of GIC and SDF treated carious primary dentin.[27] Although they conditioned after, not before SDF treatment, their results were comparable to data obtained here, showing a non-statistically significant trend of higher bond strength in the SDF treatment group. They also evaluated the failure mode with microscopy and found that the majority of the samples displayed mixed cohesion and adhesion failure. Kucukyilmaz and colleagues found that SDF and ammonium hexafluorosilicate (SiF) adversely affected microtensile bond strength of resin composite to dentin and artificial carious lesions in dentin. In the same study, Er:YAG laser irradiation increased the bond strength in the SiF group but not in the SDF group.[28] Selvaraj and colleagues showed that treatment with SDF/KI trended towards increasing the µSBS of resin composite in both etch-and-rinse and self-etch systems, but this finding was not statistically significant. They also used a transmisson electron microscope to show that SDF/KI reduced nanoleakage in all testing groups.[29] In 2012, Quock RL showed that with sound dentin, SDF did not affect microtensile bond strength of resin composite with either etch-and-rinse or self-etch systems.[30] An evaluation by Yamaga showed that SDF uniformly increased the bond strength of four GIC mixes with 0 to 10% tannin fluoride to bovine dentin.[31]

Therefore, it is evident that while some studies exist in this area, there is a lack of information for clinicians. With only one previous study, there was previously insufficient evidence studying the effect of SDF on bond strength with carious dentin to optimize the clinical application of silver-modified atraumatic restorative treatment.

Group 1 (Mean 6.5 MPa, SD=2.0) demonstrated a mean µSBS well within reported literature values (1.9 to 9.96 MPa) of various GICs applied to sound dentin.[32] This validates the materials and methods used in this study. GIC has two-fold bonding, utilizing both micromechanical interlocking and chemical bonding mechanisms. Weak polyacrylic acid in the cement and also in the cavity conditioner start a demineralization process that is less potent than the etchant used for composite restorations. Once surface debris is removed, micromechanical interlocking and infiltrations by the cement occur. Chemically, ionic bonding occurs between the polyacrylic acid carboxylate groups and the calcium ions in both enamel and dentin.[33]

When comparing groups 2 and 3, the addition of SDF had no statistically significant effect on the bond strength. Yet the average value for µSBS did improve from 10.4 to 13.2 MPa when SDF was applied, thus supporting a restorative procedure of SDF treated caries with a GIC in a clinical setting. In group 3, GIC was bonded one week after SDF was applied. In this week, SDF penetrated into the dentinal tubules and also formed a hardened layer on the surface comprised of silver oxide conjugates. Studies show silver and fluoride ions penetrate 200-500 microns into dentin.[34, 35] In the oral environment, the chemical reaction products of SDF $[Ag(NH_3)]_2F$ and hydroxyapatite $[Ca_{10}(PO_4)_6(OH)_2]$ are hypothesized to be calcium fluoride CaF_2, silver phosphate Ag_3PO_4, and ammonium hydroxide NH_4OH.[5, 36] It is possible allowing one week for the SDF to penetrate and form a hardened surface layer resulted in a GIC bond to this layer at a similar strength to demineralized dentin. Therefore, although our study found no significant differences in bond strength with the application of SDF, a clinical difference may exist.

There was a statistically significant difference between groups 3 and 5. In group 3, the GIC was bonded one week after SDF application. In contrast, GIC was bonded immediately after SDF application in group 5. Group 5 had a mean of 5.0 MPa, compared to group 3 with a mean of 13.2 MPa. GIC placement one week after SDF application resulted in no statistically significant changes to bond strength to demineralized dentin. However, when GIC was placed immediately following SDF placement, there was a statistically significant decrease in bond strength. It is possible that when SDF is not given sufficient time to fully solidify, a layer of unreacted remnant SDF lays on the dentin surface and reduces the biomechanical bonding to GIC. The layer of aqueous SDF, with silver, ammonia, and fluoride ions, may have affected both mechanical interlocking and chemical bonding of the GIC bond.

Group 4 was statistically significant from group 3 ($p=0.002$), with a higher mean bond strength (13.2 MPa) with the use of conditioner than without (7.4 MPa). Clinically, the addition of conditioner, which is diluted polyacrylic acid (20% by weight), etches the dentin surface and removes the smear layer. This clean interface between dentin hydroxyapatite and GIC allows tags to form which contribute to mechanical bonding. In this study, no significant smear layer was formed after demineralization and the sample was not prepped mechanically. It is possible that residual polyacrylic acid remained after the conditioner was rinsed off, and was incorporated into the GIC, contributing to ionic bonding and creating a stronger chemical bond. This may have contributed to an increase in µSBS when conditioning before SDF application.

Most importantly, our results show that the 24 hour bond strength of GIC to conditioned demineralized dentin is significantly decreased (62%) by SDF when the GIC is placed immediately after SDF. When SDF is allowed to set one week prior to GIC placement, a non-statistically significant increase in bond strength is observed (27%). This may be due to slow reaction and penetration kinetics of SDF, which are still ongoing when GIC is placed immediately. Our results show that the use of conditioner prior to GIC placement increases 24 hour dentin bond strength. Clinically, these results suggest to condition prior to GIC placement, and to separate SDF treatment and GIC placement by a week or more.

In addition to restoration of function and reduced food impaction in cavitated lesions, GIC placement after SDF has shown to increase resistance to marginal caries. Mei, Zhao, and colleagues have demonstrated *ex vivo* this benefit, which further supports SDF-modified atraumatic restorative treatment to prevent restorative failure.[37, 38, 39]

Limitations and Future Studies

Limitations to this study include the inherit difference in shear bond strength between *in vitro* artificial carious lesions and clinical caries. Shear bond strength testing also has limited generalizability to ART restorations' clinical longevity. Ideally, longevity of SDF + ART restorations would be tested clinically. In fact, a clinical trial has recently begun comparing precisely the GIC conditions that we find here to be most optimal: Groups 2 and 3.[40]

Another limitation is the use of permanent molars as opposed to primary dentition, partially due to differences in dentinal tubule structure. A flat surface is necessary for reproducible measurements of shear bond strength. Given the limitations and subjectivity of using clinical carious lesions that have been ground down to a flat surface into affected dentin and a small dentin surface area available on primary dentition for creating artificial carious lesions, the decision was made to use permanent molars.

Future studies could examine the time component between SDF placement and GIC bonding. In this study, the time points studied were bonding immediately and one week later. Clinically, it may be advantageous to study bonding after five minutes, ten minutes, one day, etc. ART restorations may be used as holding care prior to definitive restoration, while the patient matures, while waiting for conscious sedation or general anesthesia, or even as a definitive restoration in select patients. Therefore, another time point that is of importance for practitioners is the bond strength of GIC after several months or years. In this study, GC Fuji IX was chosen as it is indicated for posterior restorations, which is one of the more common types of ART. However, future studies could examine the effect on bond strength of composite resins, resin modified GICs, luting cements, and zinc oxide based materials.

Conclusion

This study supports the application of chemical cured glass ionomer cements to caries lesions directly or after treatment with 38% silver diamine fluoride solution. In particular, in children, a direct application of SDF onto the carious cavity will substantially alleviate suffering from the restorative process as it can eliminate or delay surgical removal of the lesion. When SDF is followed with a GIC restoration, the combination is a durable treatment choice that synergizes caries arrest, fluoride release, and cleansibility. Clinicians may use this data to inform treatment plans which include SDF application, holding care, atraumatic restorative treatment, and SMART fillings for the pediatric and special needs populations.

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