Shear bond strength and interfacial analysis of high-viscosity glass ionomer cement bonded to dentin with protocols including silver diammine fluoride

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Abstract

Purpose: High-viscosity glass ionomer cements (HV-GICs) are reinforced with ultrafine, highly reactive glass particles, as well as a higher-molecular-weight polyalkenoic acid component. Silver diammine fluoride (SDF) is an agent with promising activity against active caries. The present study aimed to evaluate the shear bond strength (SBS) and interfacial morphology of a new HV-GIC bonded to dentin after treatment with various adhesive protocols including SDF.

Methods: HV-GIC cylinders were bonded to dentin after various surface treatments (6 groups, n = 22): water; polyalkenoic acid; SDF; SDF + potassium iodide (KI); SDF + KI + polyalkenoic acid; SDF + KI + two weeks of storage in water + polyalkenoic acid. For each group, 20 samples were tested for SBS after 48 h, and 2 samples were cut and subjected to environmental scanning electron microscopy (E-SEM) and energy-dispersive X-ray (EDX) analysis.

Results: No significant differences in SBS were found between any of the protocols tested. However, E-SEM and EDX images showed different interfaces when SDF was applied.

Conclusion: SDF has no influence on the adhesion of HV-GIC to sound dentin and could potentially improve the cario-resistance of the dentin/HV-GIC interface.

Keywords: energy-dispersive X-ray analysis, high-viscosity glass ionomer cement, shear bond strength, silver diammine fluoride

Introduction

Since their introduction in 1972 by Wilson and Kent [1], glass ionomer cements (GICs) have been widely used for provisional dental restorations. GICs rely on an acid-base reaction between fluoroaluminosilicate glass and polyalkenoic acid [1]. These resin-free cements have specific properties directly derived from their composition, including self-adhesion to dental tissue [2], rechargeable fluoride release [3] and moisture tolerance [4]. Some studies have also demonstrated beneficial bacteriostatic and cariostatic effects [5,6] associated with GICs, although other studies have questioned the significance of this [7]. However, as GICs have poor aesthetic and mechanical properties and low resistance to wear, improvements in composition have been proposed [4].

Resin-modified glass ionomer cements (RMGICs) contain methacrylate monomers to provide an additional polymer network, thus strengthening the matrix after polymerization and improving the properties of the cement [8]. However, although RMGICs have improved aesthetic and mechanical properties, they have low resistance to wear [9] and a high water uptake due to the hydrophilic nature of hydroxethylmethacrylate monomers [10], resulting in hydrolytic degradation of the fillers and the resin matrix. The resin content of RMGICs also raises questions about their biocompatibility. It has been proposed that high-viscosity glass ionomer cements (HV-GICs) might represent an improvement over traditional GICs. New-generation HV-GICs contain ultrafine and highly reactive glass particles dispersed within the conventional GIC structure, as well as a higher-molecular-weight polyalkenoic acid that enhances the mechanical and wear properties [11]. New-generation HV-GICs have yielded promising clinical results as permanent fillings for class I and small class II restorations [12]. They can also be utilized as a base material for composite restorations after application of an adhesive system [13], they are resin-free with optimal biocompatibility and no polymerization shrinkage, and allow an optional cavity conditioning stage with polyalkenoic acid.

Another product that has gained popularity recently is silver diammine fluoride (SDF), which is used not only as a desensitizing agent [14], but also to prevent and arrest caries [15]. SDF is a colorless basic solution containing silver and fluoride, which forms a complex with ammonium and creates a salt in contact with the tooth structure [15]. Many recent studies have demonstrated its efficacy as a remineralizing and cariostatic agent [16-18]. However, the silver ions leave a permanent black stain on the decayed area of the tooth. To counter this effect, immediate application of potassium iodide (KI) after SDF, which leads to the precipitation of creamy white silver iodide crystals, has been suggested [19].

To date, few data are available on the effect of SDFs on the bonding strength of HV-GIC to dentine. However, a “symbiotic” relationship between the two materials has been suggested: HV-GICs could mask the stained carious lesion associated with SDF, while SDF could minimize the micro/nano leakage and secondary caries associated with HV-GICs. However, application of KI may decrease the bond strength.

Using shear bond strength tests, interfacial analysis and elemental analysis, the aim of the present study was to investigate the interface between a new HV-GIC (EQUIA Forte Fil, GC Corporation, Tokyo, Japan) and SDF-treated dentin, as well as the effects of various clinical protocols. The null hypotheses tested were that (i) various dentin surface treatments involving SDF would have an impact on the shear bond strength of HV-GIC, and (ii) there would be no differences in the interfacial characteristics of HV-GIC bonded to various dentin surfaces treated with SDF.

Materials and Methods

Sample preparation
One hundred and thirty-two freshly extracted human permanent molars were collected after extraction, cleaned of soft tissues, stored at 4°C in a solution of 1% chloramine, and used within 3 months. All teeth were obtained from the dental departments of AP-HP, France. All experiments were conducted in accordance with the declaration of Helsinki, and all patients provided informed oral consent according to the ethical guidelines stipulated by French law and with a dedicated authorization for the dental school of Paris University (n°DC-2009-927, Cellule Bioéthique DGRi/A5, 2010).
The occlusal surface of the crowns was then abraded on water-cooled sandpaper (80 grit) using a polishing machine (Planopoli 3, Struers, Copenhagen, Denmark). The occlusal surface of the crowns was then abraded on water-cooled sandpaper (800 grit) to expose a flat dentin surface (> 7 mm²) corresponding to the surface roughness obtained with a fine diamond bur. The residual crowns were embedded in self-cured acrylic resin (Plexcil, Escil, Chas-sieu, France) in plastic cylinders (diameter 25 mm, depth 15 mm) with the flat dentin surface exposed. The flat surfaces were inspected under ×40 magnification to ensure that the enamel had been completely removed and the dentin cleared of debris. These teeth were randomly assigned to six groups (n = 22).

For each group, 20 teeth were used for shear bond strength testing, and two for interfacial observation by environmental scanning electron microscopy (E-SEM) followed by energy-dispersive X-ray (EDX) microanalysis.

Materials and bonding procedures

Various adhesive protocols were utilized when applying the HV-GIC (EQUIA Forte Fil, GC Corporation) cylinders onto the dentin surface. Materials, abbreviations, manufacturers, batch numbers and composition are presented in Table 1.

The following dentin surface treatment were investigated:
- Group 1 (control): the dentin surface was cleaned with water spray for 10 s, then gently air-dried.
- Group 2 (polyalkenoic acid): the dentin surface was treated with polyalkenoic acid (Cavity Conditioner, GC Corporation) for 10 s, then rinsed with water spray for 10 s and gently air-dried.
- Group 3 (SDF): the dentin surface was treated with SDF (Riva Star, SDI, Bayswatch, Australia) using a microbrush with a scrubbing motion for 10 s, then gently air-dried.
- Group 4 (SDF + KI): the dentin surface was treated with SDF using a microbrush with a scrubbing motion for 10 s, then a generous amount of KI solution (Riva Star, SDI) was applied until the initial creamy-white precipitation turned clear. Thereafter, the dentin surface was gently air-dried.
- Group 5 (SDF + KI + polyalkenoic acid): the dentin surface was treated with SDF using a microbrush with a scrubbing motion for 10 s, then a generous amount of KI solution was applied until the initial creamy-white precipitation turned clear. Thereafter, the dentin surface was cleaned with polyalkenoic acid to remove KI precipitates for 10 s, rinsed with water spray for 10 s and gently air-dried.
- Group 6 (SDF + KI + two-week storage in water + polyalkenoic acid): the dentin surface was treated with SDF using a microbrush with a scrubbing motion for 10 s, then a generous amount of KI solution was applied until the initial creamy-white precipitation turned clear. Thereafter, the samples were stored in water for 2 weeks at 37°C. Finally, the dentin surface was cleaned with polyalkenoic acid for 10 s to remove KI precipitates, rinsed with water spray for 10 s and gently air-dried.

On each sample, a cylindrical silicone mold (EXA'lence, GC Corporation) was placed in order to build a 3-mm-high cylinder with a flat 2.3-mm² base. HV-GIC was inserted into the mold in one increment. After 10 min at room temperature, the silicone mold was removed, and the material excess, if present, was gently removed from around the base of the HV-GIC cylinder with a scalpel. All the samples were stored in tap water at 37°C for 48 h to allow proper setting of the HV-GIC.

Shear bond strength testing and failure mode determination

Shear bond strength (SBS) was determined in a universal testing machine (AGS-X, Shimadzu Corporation, Kyoto, Japan). The shear force was applied at the HV-GIC cylinder/dentin interface with a chisel-shaped blade, parallel to the dentin surface. A cross-head speed of 0.5 mm/min was chosen.

The debonded specimens were observed under a binocular microscope (BZH10, Olympus, Hamburg, Germany) at ×30 magnification and the failure modes were classified into the following five types:
- Type CF-D: failure is considered to be cohesive in dentin if more than 75% of the fracture involves the dentin.
- Type AF: failure is considered to be adhesive if more than 75% of the dentinal surface is intact and free of HV-GIC.
- Type MF: failure is considered to be mixed if the intact dentinal surface free of GIC is between 25% and 75%.
- Type CF-HV-GIC: failure is considered to be cohesive in HV-GIC if more than 75% of the fracture involves the HV-GIC.

Environmental scanning electron microscopy and energy-dispersive X-ray spectroscopy

The samples were sectioned perpendicularly to the bonded interface using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) with water cooling, as near as possible to the center of the cylinder. The sections obtained were polished with abrasive discs of decreasing grit size (400, 800, 1200, 2400 and 4000 SiC), followed by diamond particles of 3 and 1 μm. The samples were cleaned by ultrasonication after each polishing step and then examined using an environmental scanning electron microscope (E-SEM) (FEI Kanta 200, FEI, Hillsboro, OR, USA) with an energy-dispersive X-ray spectroscopy (EDX) analysis (EDAX Metek New XL30, FEI). E-SEM/EDX analysis made it possible to observe the interface between HV-GIC and dentin and to detect precipitates of silver in dentinal tubules. To this end, backscattered electron scanning assay and EDX analysis of dentinal tubules near the interface were performed to determine the elemental spectrum of the targeted areas. For groups showing no heavy precipitates, three different areas were studied (one close to the interface and two in dentinal tubules). For groups showing heavy precipitates, three areas were studied (one close to the GIC/dentin interface and two below the surface).

Statistical analysis

Normal distribution was checked by the Shapiro-Wilk test, and the equality of variances was assessed using the Levene-Wilk test, before the tests were performed. SBS data were expressed as mean values and standard deviations. One-way ANOVA followed by Tukey’s post hoc test was used to investigate the difference in SBS between the different groups. Failures were analyzed by chi-squared test. In all tests, the significance level was set at P < 0.05. Statistical calculations were performed using XLSTAT software (XLSTAT, Addinsoft, Paris, France).

Results

SBS and failure analysis

The Shapiro-Wilk test confirmed a normal distribution of SBS values among all the groups (P > 0.05). Levene’s test showed equality of variance (P > 0.05).

Data on shear bond strength for all experimental groups are summarized in Table 2. One-way ANOVA following the Tukey’s post hoc test revealed no significant differences in SBS values among any of the adhesive protocols tested.

All of the fracture profiles obtained in the different groups were adhe-
The chi-squared test indicated no significant differences among the various groups.

**E-SEM examination and EDX analysis**

Figure 1 shows the E-SEM images with back-scattered electron detection for each group at $\times 2,000$ magnification. Figure 2 shows the results of EDX analysis performed on dentinal tubules close to the interface.

For groups 1 and 2, where no SDF was applied, the HV-GIC/dentin interface appears continuous and the dentinal tubules non-occluded. EDX analysis of the dentinal tubules revealed the presence of oxygen, calcium, phosphorus, aluminum, fluoride, silicon and sodium compounds and confirmed the absence of silver precipitates.

For groups 3, 5 and 6, the HV-GIC/dentin interface appeared continuous and heavy precipitates were observed in dentinal tubules at various depths. EDX analysis of the dentinal tubules revealed the presence of silver compounds (and chlorine for group 3) in addition to the same elements listed above for groups 1 and 2. Analysis of the samples revealed that the silver compounds penetrated only into the dentinal tubules and were not blocked at the HV-GIC/dentin interface.

For group 4, the HV-GIC/dentin interface appeared continuous; heavy precipitates were observed in the dentinal tubules, but also at the interface between the HV-GIC and the dentin. EDX analysis revealed the presence of iodide and the same elements as those listed above for groups 5 and 6.

**Discussion**

An ideal restorative material would not only be biocompatible, mechanically resistant and aesthetic, but also demonstrate spontaneous adhesion to dental tissues and similar thermal expansion to tooth structure, thus ensuring a good marginal seal. HV-GICs demonstrate several of these criteria, and their superior mechanical properties compared to conventional GICs have yielded results better than those obtained using resin composites for some class I and class II cavities [20]. On the other hand, several studies have demonstrated microleakage at restorative-enamel and restorative-dentine margins when HV-GICs are used [21-23].

Similarly to HV-GIC, SDF is being increasingly utilized in young and elderly patients to remineralize tooth structures, arrest caries development and inhibit biofilm formation [24-27]. Some studies have also indicated

| Groups tested | Dentin surface treatment | SBS (±SD) |
|---------------|--------------------------|-----------|
| 1             | water                    | 6.9 (±3.3)$^a$ |
| 2             | CC                       | 7.3 (±2.5)$^a$ |
| 3             | SDF                      | 7.2 (±3.2)$^a$ |
| 4             | SDF + KI                 | 6.5 (±2.7)$^a$ |
| 5             | SDF + KI + CC            | 7.0 (±2.8)$^a$ |
| 6             | SDF + KI + 2 weeks + CC  | 6.8 (±2.6)$^a$ |

Values with the same superscript letter are not significantly different at $P < 0.05$
that SDF could prevent secondary caries associated with GICs and resin composite placement [28], due to the formation of silver fluorohydroxyapatite during remineralization with SDF. The resulting silver particles incorporated into the tooth structure exert a prolonged antimicrobial and anticariogenic effect [26]. However, one of the major drawbacks associated with SDF placement is black staining resulting from the formation of a silver precipitate by-product after placement. It has been suggested that immediate application of KI after SDF application can minimize this staining by forming a colorless precipitate by-product with the silver ions [29].

The present study is the first to have investigated the impact of different SDF pretreatments on the interface between dentin and HV-GIC using SBS tests, SEM, and EDX analysis. No significant differences were demonstrated in the SBS of EQUIA to dentin using all of the protocols tested, and thus the first null hypothesis was rejected.

The non-significant differences between group 1 (water + EQUIA), group 2 (CC + EQUIA) and group 3 (SDF + EQUIA) were in line with the EQUIA Forte manufacturer’s instructions for optional cavity conditioning, and also with other studies of dentin pretreatment for HV-GICs [30-32]. Cavity conditioning facilitates micro-interlocking between EQUIA Forte and dentin, thus conferring resistance to short-term debonding stress [31]. Accordingly, omission of surface pretreatment before bonding of HV-GIC could be a good option for temporary restorations, minimizing the risk of pulpal interference. However, to improve long-term bonding performance, which is crucial for long-lasting restorations, surface preconditioning would be recommended.

Some manufacturers claim that SDF pretreatment can improve the bond strength of GICs to dentin, although this has not been demonstrated in previous studies [30]. Despite this, pretreatment use of SDF could result in improved long-term survival due to the antibacterial and autoprotopalytic properties of the materials, as described earlier [26]. Previous studies have shown that KI precipitates have decreased SBS values with conventional GIC and adhesives [30], but not with RM-GIC [33]. In the present study, although the SBS values in group 4 (SDF + KI + EQUIA) were the lowest, no significant differences between groups were demonstrated. However, this might be attributable to not only the high standard deviation observed when HV-GIC were bonded to dentin, but also stronger dentin adhesion to HV-GIC than to GIC.

In Groups 5 and 6, where SDF and KI application was followed by immediate and delayed polyalkenoic acid treatment, respectively, no significant differences in SBS were noted. Clinical use of polyalkenoic acid after SDF application might be attributable to the remineralization capability of SDF. In non-clinical settings, pre co-operative patients or in cases of time constraint, delayed application of the definitive restoration after SDF application might be required. In group 6, which recreated this type of scenario, the associated delay led to no significant reduction or improvement.

In this study, SBS testing was utilized instead of tests of other similar bonding parameters such as micro-tensile bond strength. The reason for this choice was that due to the relatively low bond strength of HV-GICs in comparison to resin composites, other bonding tests would have been difficult to undertake [34]. The authors found SBS testing to be effective for overcoming this limitation.

No significant differences in failure mode were found among the various groups. All failures were adhesive failures, in accordance with previous studies showing essentially adhesive failure and some mixed failures [30,35,36]. The present results also indicated that the adhesive strength of EQUIA Forte to dentin was weaker than its cohesive strength. Since HV-GICs are materials containing water, they are difficult to observe by SEM without inducing artificial microcracks in both the material and at the bonding interface, due to dehydration and metalization. That is why wet and uncoated samples were examined by E-SEM, which does not require dehydration or metalization. This allowed better analysis of the interface, along with concomitant EDX analysis.

E-SEM/EDX analysis revealed no differences in groups 1 and 2. Both groups demonstrated a continuous adhesive interface, suggesting that the improved chemistry of EQUIA was efficient for infiltrating the smear layer and ensuring good coaptation with dentin. EDX analysis of dentinal tubules revealed that most of the elements, including oxygen, calcium, phosphorus, aluminum, fluoride, silicon and sodium, originated from components of the HV-GIC and tooth structure. The non-captioned peak to the right of the calcium peak (visible in all groups) was a secondary peak of calcium.

In groups 3 to 6, EDX analysis confirmed that SDF application resulted in the formation of stable silver precipitates in the dentinal tubules. As discussed previously, because GICs demonstrate greater inhibition of cariogenic bacteria in comparison to resin composites, HV-GICs are often used as transitional or definitive restorations for patients with high caries risk [6]. To improve the functional time of such restorations, improvements at the dentin/HV-GIC interface are essential. The presence of stable silver precipitates in dentin tubules could introduce antimicrobial and anticariogenic properties, and thus may be very useful for improving the resistance of restorations to secondary caries. Additionally, obliteration of dentinal tubules might also decrease postoperative sensitivity [37].

The presence of chlorine after SDF application, as demonstrated in groups 3 to 6, has also been demonstrated in a previous study [38]; possible explanations for this include the presence of chlorine in the product used, or contamination of the dentinal tubules by chloramine in which the tooth had been stored.

The present study did not examine the penetration depth of silver compounds into dentinal tubules. Previous studies of various dentin have demonstrated a high standard deviation of the penetration depth [38,39]. This can be explained by variations in patient physiological parameters such as age and the type of dentine, resulting in a range of tubule diameters or orientations, depending on the area of the tooth examined. Moreover, the lack of intra-pulpal pressure in an extracted tooth might lead to overestimation of the SDF penetration depth. In the present SEM/XRD analysis, groups 3 and 6, which showed the greatest depth of penetration of silver precipitates, also demonstrated the greatest tubule diameters, whereas conversely groups 4 and 5, which showed the least depth of penetration, also demonstrated the smallest dentinal tubule diameters.

In group 4, silver was found at the interface between HV-GIC and dentin. Although there were no significant differences among the groups with regards to SBS, this finding corroborates a previous study of conventional GIC [30], which found that SDF followed by KI application led to formation of precipitates on tooth surfaces that were difficult to remove and potentially reduced the period of HV-GIC adhesion to dentin. These precipitates might also explain the presence of iodide in group 4, as revealed by EDX analysis. Knight et al. [30] proposed that rinsing tooth surfaces for 30 s might remove these precipitates. However, in the present groups 5 and 6, where polyalkenoic acid was applied for 10 s either immediately or 2 weeks after SDF and KI application, no precipitates were demonstrated by SEM. This would suggest that application of polyalkenoic acid might be effective for removal of KI precipitates while also ensuring the preservation of silver in dentinal tubules. Thus, the second null hypothesis was rejected.

To conclude, no significant differences in SBS were demonstrated for EQUIA Forte Fil bound to sound dentin using all the tested protocols involving SDF. SEM analysis demonstrated intimate contact between EQUIA Forte Fil and dentin, thus supporting the good bond strength values obtained. SEM/EDX analysis demonstrated the presence of silver deposits in dentinal tubules for all groups with SDF application. When KI was applied after SDF and not cleaned with polyalkenoic acid, silver deposits were also found at the interface between HV-GIC and dentin.

Within the limitations of this in vitro study, it appears that SDF would be a suitable material for improving the resistance of HV-GIC restorations to secondary caries and for decreasing postoperative pain after application without significantly effecting bond strength. If KI is applied, additional surface pretreatment with polyalkenoic acid would be recommended to reduce surface silver contamination.

**Conflict of interest**

None.

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