Effect of Silver Diamine Fluoride and Potassium Iodide on Shear Bond Strength of Glass Ionomer Cements to Caries-Affected Dentine

Abstract

Objective: *To investigate the effect of silver diamine fluoride (SDF) and potassium iodide (KI) treatment on dentine discolouration and the shear bond strength (SBS) of glass ionomer cements (GICs) to artificial caries-affected dentine.*

Methods: *Dentine slices from human molars were demineralised to mimic caries-affected dentine. They were randomly allocated for treatment (n=20 per treatment) with SDF+KI, SDF (positive control) or water (negative control). All slices were immersed in the artificial saliva for 24 hours after treatments. The colour of the treated surfaces was assessed using the CIELAB system. Lightness values were measured. Total colour change (ΔE) was calculated using water as the reference group and was visible to naked eyes if ΔE>3.7. All dentine slices were bonded with GICs. The SBS was assessed using a universal testing machine. Colour parameters and the SBS were analysed using a one-way ANOVA test.*

Results: *The slices treated with SDF+KI had a higher lightness value than the reference group water, whereas those treated with SDF presented a lower lightness value compared to those treated with water. The treatment with SDF+KI did not introduce any adverse colour effect to demineralised dentine (ΔE=14.4), whereas the application of SDF alone caused significant staining (ΔE=24.6). The SBS (mean±SD) after treatment with SDF+KI, SDF and water were 3.0±1.4 MPa, 2.3±0.9 MPa and 2.6±1.1 MPa, respectively (p=0.217).*

***Conclusion**: The immediate application of KI solution after SDF treatment does not negatively affect adhesion of GICs to artificial caries-affected dentine. Moreover, KI treatment can reduce discolouration of demineralised dentine caused by SDF.*

1. Introduction

Dental caries, defined as a localised, pathological process with multiple factors, softening dental hard tissues and proceeding to the formation of cavities, continues to be a prevalent disease all over the world [1]. In recent years, the development of restorative materials and advancement in our conception of the caries process have created the capability to practice in consideration of a minimally invasive dentistry philosophy [2]. It requires performing management with as little tissue loss as possible and without causing any destruction to the adjacent healthy tooth tissues [3]. Carious dentine lesions were characteristically defined as comprising two different layers: an outer layer of bacterially infected dentine (caries-infected dentine) and an inner layer of caries-affected dentine [4]. The outer layer has been regarded as being highly demineralised and exhibiting irreversible denatured collagen fibrils with an obvious disappearance of cross-linkages, whereas the inner layer is not affected with bacteria and is partially demineralised and physiologically remineralisable [5]. Thus, caries-affected dentine should be preserved in clinical treatment based on the philosophy of minimally invasive dentistry. Consequently, caries-affected dentine other than normal dentine has commonly been the bonding substrate in clinical settings.

Silver diamine fluoride [$Ag(NH_3)_2F$] (SDF) is a topical fluoride solution which has been used to halt dental caries in a concentration of 38% (44,800 ppm fluoride) throughout the world since the early 1970s [6]. SDF currently has approval from the Food and Drug

Administration in the United States as a Class-II medical device for the management of tooth hypersensitivity. It has also been used as an anticariogenic agent to reduce the growth of cariogenic biofilms [7]. In addition, SDF positively influences dentine remineralisation [8], inhibits dentine demineralisation and prevents dentine collagen from degradation [9]. Moreover, it can promote the transformation of hydroxyapatite into fluorapatite with reduced solubility [10], which increases resistance of dental hard tissues to acidic challenge. The resistance of proteins to collagenase attacks also increases due to the reaction between SDF and dentine organic matrix [9, 11]. A randomised, controlled trial concluded that 38% SDF had a better result than the interim restorative treatment using glass ionomer cements (GICs) to arrest cavitated caries in primary teeth [12]. A systematic review confirmed that 38% SDF was effective in arresting dentine caries in deciduous teeth among children [13]. Additionally, caries removal is not needed before application of SDF, which can simplify treatment procedures. Nevertheless, SDF has a major adverse effect that stains the caries lesion black because of the reaction of SDF products with tooth tissues [6]. SDF has not been widely accepted by patients with aesthetic concerns due to its inherent disadvantage. Two alternatives have been suggested to minimise this side effect. One is to use a saturated potassium iodide (KI) solution, which can react with residual silver ions, to eliminate the staining effect [14]. However, the colour-eliminating effect is still not well understood when SDF and KI are applied to caries-affected dentine. The other alternative is to apply GICs or composites over SDF to mask the stained carious lesion and as a direct restoration after application of SDF [15].

Recently published studies [16, 17] reported that SDF arrested caries lesions with no further progression after 2–3 years when it was applied to cavitated caries with no excavation. It is noteworthy that cavities were left open without filling in these studies after application of

SDF. Restorations are generally needed for cavitated lesions to allow for an easily cleanable surface that may reduce the potential for secondary caries initiation. Although laboratory studies [14, 18] report that application of SDF is compatible with restorations with GICs, to the authors' knowledge there is insufficient evidence concerning the adhesion properties of GIC restorations when they are bonded to caries-affected dentine surfaces previously treated with SDF and KI. Therefore, the aim of this in vitro study was to investigate the effect of SDF and KI treatment on tooth discolouration and the shear bond strength (SBS) of GICs to artificial caries-affected dentine. Two null hypotheses were tested: SDF+KI treatment does not introduce staining effect on artificial caries-affected dentine, and SDF+KI treatment does not affect adhesion of GICs to artificial caries-affected dentine.

2. Materials and Methods

2.1 Sample preparation

Sixty non-carious human third molars were collected with the patients' written consent according to regulations at the University of Hong Kong. The research protocol was reviewed and approved by the local Institutional Review Board of the University of Hong Kong/Hospital Authority Hong Kong West Cluster (IRB UW 18-404). This research was conducted in full accordance with the World Medical Association Declaration of Helsinki. The teeth were stored in a 0.1% thymol solution at 4˚C prior to section. The study design is shown in Figure 1. Sixty dentine slices with 2-mm thickness were prepared from 60 sound third molars using a low-speed saw with a diamond blade (ISOMET 1000, Buehler, Lake Bluff, IL, USA). All dentine slices were embedded using a dental cold-cured acrylic (ProBase Cold, Ivoclar Vivadent AG, Liechtenstein). The surfaces of the dentine slices were polished with micro-fine 2000-grit sanding paper under running water. All samples were immersed in a demineralising solution (pH 4.4, 50 mM acetate, 2.2 mM KH_2PO_4, 2.2 mM

$CaCl_2$) for 7 days at 25 ˚C [9]. They were then allocated to three groups (n=20 per group). For group SDF+KI, the demineralised surfaces were treated with SDF+KI (Riva Star, SDI, Bayswater, Australia). A 38% SDF solution from silver capsules was topically applied to the demineralised surfaces, with immediately applying a saturated KI solution from green capsules to treatment site until creamy white turned clear. Treated surfaces were adequately washed with distilled water [19]. For group SDF, the positive control group, the demineralised surfaces were treated with a 38% SDF solution (Saforide, Toyo Seiyaku Kasei Co. Ltd., Osaka, Japan). For group water, the negative control group, the demineralised surfaces received application of water. After 30 min, all samples were immersed in the artificial saliva at 25 ˚C for 24 hours.

2.2 Colour assessment

Colour assessments (n=20 per group) of dentine samples were performed after 1-day immersion in the artificial saliva. The colour of the treated dentine surface was assessed using a colorimeter (NR10QC, General Colorimeter, 3nh, Shenzhen, China). The CIE system (Commission International del'Eclairage) was used to three-dimensionally elucidate the colour by recording the $L^* a^* b^*$ colour coordinates. The L^* axis represented lightness ranging from black (0) to white (100), the a^* axis represented red ($+a^*$) to green ($-a^*$) and the b^* axis described yellow ($+b^*$) to blue ($-b^*$). The measurements of L^*, a^* and b^* were triplicate. The mathematical equation $\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$ were employed to calculate the colour difference between the experimental groups and the negative control group [20]. The discolouration of each tooth was clinically perceptible to naked eyes if ΔE was more than 3.7 units (perceptibility threshold) [21].

2.3 SBS test and failure mode analysis

After colour assessment, all dentine samples (n=20 per group) were bonded with GICs (Ketac-Molar, 3M/ESPE Dental Products, St. Paul, MN, USA). A Teflon mould with 4-mm height and 3-mm diameter was placed on the demineralised dentine surfaces [22]. GICs in capsules were mixed using a rotational/centrifugal capsule mixing unit (RotoMix, 3M ESPE, St Paul, MN, USA) for 10 s, and then the mixture was applied in the Teflon mould to form a cylindrical button. All GICs were chemically cured. After bonding, samples were stored in 100% humidity at 37 ˚C for 24 hours after removal from the mould to allow complete setting of GICs. The SBS test was performed with a universal testing machine which had a flat-edge loading head (ElectroPuls 3000, Instron, Norwood, MA, USA). A shear force was applied perpendicularly to the GIC cylindrical button at a distance of 1 mm from the dentine surface to the loading head. The loading head moved at a fixed rate of 1 mm/min. The load necessary to debond GICs was recorded in newtons. The bond strength was expressed in Mega Pascals (MPa) by dividing the load at failure by the bonded surface area in square mm.

The debonded samples were examined under an optical microscope at 20× magnification. The failure modes were categorized as three types: Type 1; adhesive failure between dentine and GICs with exposing dentine surfaces, Type 2; cohesive failure in GICs or in dentine, Type 3; partially adhesive failure and partially cohesive failure (mixed failure) [23]. Some of the fractured samples were sputter-coated with gold and viewed by a scanning electron microscope (SEM, Hitachi S-4800 FEG Scanning Electron Microscope, Hitachi Ltd., Tokyo, Japan) to obtain images with higher magnification.

2.4 Statistical analysis

The data were analysed with IBM SPSS Version 25.0 software (IBM Corporation, Armonk, NY, USA). All data were checked for normality using the Shapiro-Wilk test (p>0.05). Colour parameters and the SBS were analysed using a one-way ANOVA with post hoc test. The distribution of failure modes for the three groups were analysed by a chi-square and Fisher's exact test. A p value lower than 5% was considered as statistically significant for all tests.

3 Results

3.1 Colour assessment

The colour characteristics for each dentine sample were examined to investigate whether the treatments introduced any discolouration effect compared to the colour of the negative control dentine (Table 1). The total colour change (ΔE) and values of L^* a^* b^* and of the three groups are shown in Table 1. Data confirmed that group SDF+KI had significantly higher L^* values than that of the negative control group, whereas group SDF presented a significantly lower lightness compared to the negative control group. The colour differences (ΔE) between the treated dentine and the non-treated ones were calculated based on the colour difference formula using the mean values of L^*, a^* and b^*. Both of the ΔE values for group SDF+KI and group SDF were more than 3.7 units. The treatment with SDF+KI did not introduce any adverse colour effect to dentine surfaces, whereas the application of SDF alone caused significant staining.

3.2 SBS test and failure mode analysis

The mean bond strengths of GICs to dentine are shown in Figure 2 (n=19 per group). Each group had one sample fractured pretest. The SBS (mean ± SD) for groups SDF+KI, SDF and water were 3.0±1.4 MPa, 2.3±0.9 MPa and 2.6±1.1 MPa, respectively (p=0.217). The bond strengths of the three groups were not significantly different from each other.

The failure modes of all samples are shown in Table 2. Cohesive failure was only observed within GICs but not in the demineralised dentine layer. No significant difference was found in the distribution of fracture modes for group SDF+KI, group SDF and group water (p=0.487). The general trend showed that cohesive failure within GICs was less frequent in the three groups compared to the other two failure types (p<0.05). Representative SEM images of GIC-dentine interfaces are displayed in Figure 3.

4 Discussion

This study sought first to investigate if pretreating caries-affected dentine with SDF+KI adversely affected adhesion of GICs to dentine. The results from this study indicated no significant difference in SBS between the negative control group and the experimental groups (pretreatment with SDF or SDF+KI). In addition, SDF+KI treatment had no adverse colour effect on the surface of caries-affected dentine. The two null hypotheses were validated based on the findings of the current study. The clinical implications of these findings are that if KI is applied after the application of SDF to arrest or prevent dentine caries in a tooth, discolouration caused by SDF can be significantly reduced and bond strength to the caries-affected dentine of that tooth will not be affected. The current work was conducted in a controlled laboratory condition in which dentine was artificially demineralised to mimic caries-affected dentine. It is worth noting that natural caries-affected dentine, compared with demineralised dentine lesions, has more complex microstructure. There may also be permeability differences between natural caries-affected dentine and artificial caries-affected dentine because the presences of mineral crystals in natural caries-affected dentine are considered to be effective in reducing fluid movement within dentinal tubules [24]. The two different substrates may therefore offer different conditions that will most likely lead to different adhesive properties. Furthermore, controlled laboratory conditions are different from the real oral environment. It is unclear whether the mechanical properties of GICs would be affected in a long-term exposure in the oral environment. Thus, caution should be taken when these findings are extrapolated to the clinical situation.

The traditional approach of managing cavitated carious lesions is drilling and filling, which refers to mechanically removing the soft and bacteria-infected dentine before filling the cavity with a proper restorative material. It is still reasonable to conduct the excavation because infected dentine is highly demineralised and physiologically not remineralisable based on the current evidence [2]. Nevertheless, there is evidence showing that removal of soft carious lesions may not be necessary. A clinical trial reported that no significant differences were found in the number of arrested tooth surfaces for children who had caries excavation prior to application of SDF compared with those that did not have caries removal [16]. Additionally, it has been demonstrated that bacterial count and activity were diminished over time if infected dentine in a cavitated caries lesion was restored with a well-sealed resin restoration [2]. From a biological view, the need of excavation prior to restoration or fluoride application is facing an intriguing challenge according to these findings. However, the need of caries excavation seems still to be controversial because it has been reported that the fracture strength of composite resin fillings may be compromised by the underlying soft, infected dentine [25]. On the contrary, caries-affected dentin structurally reserves enough collagen fibres to be remineralised and is relatively low in bacterial count. Dentine caries, which is either affected or arrested, tends to present lower bacterial activity compared to infected dentin. Thus, caries removal is not needed prior to restorative filling for caries-affected dentine or for arrested lesions.

SDF has been concluded as a bactericidal chemical that can reduce the adherence and growth of cariogenic bacteria [26]. Moreover, it can be used to prevent the formation of secondary dentine caries around GIC restorations [27]. Thus, SDF can be a promising biological approach in the practice of minimally invasive dentistry against conventional restorative methods. The use of SDF, however, has been generally limited to primary teeth because of the discolouration effect associated with its application. According to the results of this study, SDF can be used as a liner so that the dentine base for restoration with remaining bacteria are nonviable. Because SDF can cause staining, an Australian group suggested using a saturated KI solution to mask the staining by reacting with silver ions [14]. Additionally, SDF+KI treatment has been investigated to be effective in increasing resistance to cariogenic challenge [27]. SDF products that are readily commercially available were selected in this study to make the current work more related to dentists. SDF at a concentration of 38% can be found from Saforide, Advantage Arrest and Riva Star. Saforide was chosen as the positive control in this study since it is the most commonly used SDF in previous clinical and laboratory studies. Riva Star is the only commercially available product of SDF+KI. Hence, it was selected as the experimental group.

It is important for patients to consider the aesthetic appearance of a restoration. In the present study, discolouration was evaluated quantitatively by instruments instead of naked eyes, which is more accurate with high repeatability [20]. Metallic silver was formed by the reaction of SDF and hydroxyapatite, and its production was accelerated when exposed to light and high temperature [26]. This may be why SDF stains teeth black. It has been suggested that silver iodide (a bright-yellow solid compound) can be formed when KI solution reacts with SDF and that the excess free silver ions that cause the black staining could be reduced by this reaction [14]. A higher lightness value and a perceptible total colour change (ΔE) were detected on the caries-affected dentine in the SDF+KI treatment group in this study. The possible explanations may be that the formation of silver iodide attached to demineralised dentine surfaces which were relatively loosened and rough compared to normal dentine, even though the dentine surfaces were washed immediately after creamy white precipitates appeared according to the manufacturer's instruction. Silver iodide, however, is believed to be highly photosensitive and may dissociate into metallic silver and iodine by exposure to light. Ultimately, discolouration might still occur on tooth surfaces. Thus, the long-term effect of this treatment to eliminate staining, as well as its possible interaction with different restorative materials, needs to be determined.

GICs are regarded as one of the best options of fluoride-releasing restorative materials, which have been considered to be superior to compomers and giomer from the aspects of continuous fluoride release and recharge [22]. Nevertheless, the fluoride-releasing and anti-microbial effect of GICs are usually limited and insufficient. Hence, pretreating dentine surfaces with SDF or SDF+KI before GIC restoration has been proposed by some researchers [14, 27] to enhance antimicrobial and remineralising ability of GICs. The result of this study demonstrated that pretreatment with SDF or SDF+KI did not adversely affect adhesion of restoration to dentine, which is consistent with the previous findings of other laboratory studies [2, 14]. Nevertheless, another study reported that there was an improvement in adhesion properties of fissure sealants applied after treating a tooth surface with SDF [28]. The differences in the outcomes may result from different techniques or different characteristics of tooth substrates. Cohesive failure within GICs was reported as the most common fracture mode in terms of adhesion between GICs and dentine in a previous study [29], whereas this type of failure was less frequent than the other two failure modes in all groups in the present study. This variance might be explained by the different experimental conditions.

5 Conclusion

With the limitations of this laboratory study, the following conclusions were drawn: The immediate application of KI solution after SDF treatment can reduce dentine discolouration caused by SDF. Furthermore, SDF+KI treatment does not negatively affect bonding of GICs to artificial caries-affected dentine.

Conflict of interest

The authors declare that they have no conflict of interest.

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Table 1 Comparison of the colour parameters between natural dentine (negative control) and dentine after treatments (n=20)

Group	L*	a*	b*	ΔE
Water	55.6 ± 4.3	4.6 ± 2.3	9.6 ± 6.0	N/A
SDF+KI	69.8 ± 7.1^{+}	2.1 ± 0.6^{+}	10.5 ± 4.8	14.4
SDF	32.1 ± 4.5^{+}	8.6 ± 1.5^{+}	3.6 ± 2.5*	24.6

L*, a* and b* refer to the colour coordinates. ΔE is the calculated colour difference between treated and control dentine. Data are means ± standard deviation. + indicates significant difference from control dentine within each colour coordinate (one-way ANOVA, $p < 0.05$).

Table 2 The distribution of failure modes of the three groups (n=19)

Group	Failure modes (n)			Total (n)
	Cohesive	Adhesive	Mixed	
SDF+KI	1	8	10	19
SDF	1	12	6	19
Water	2	12	5	19

Figure 1 Flow chart of the study

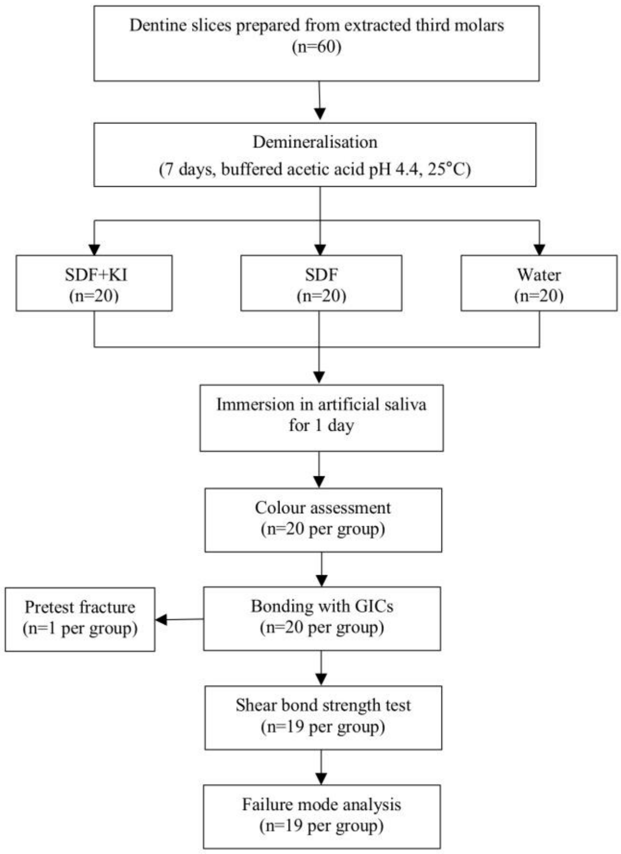
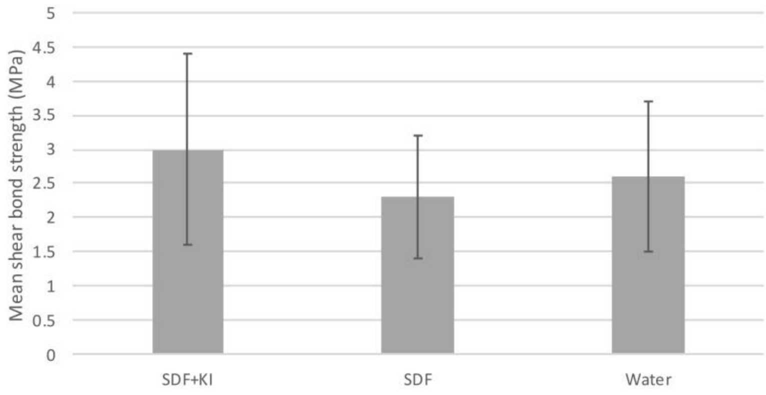
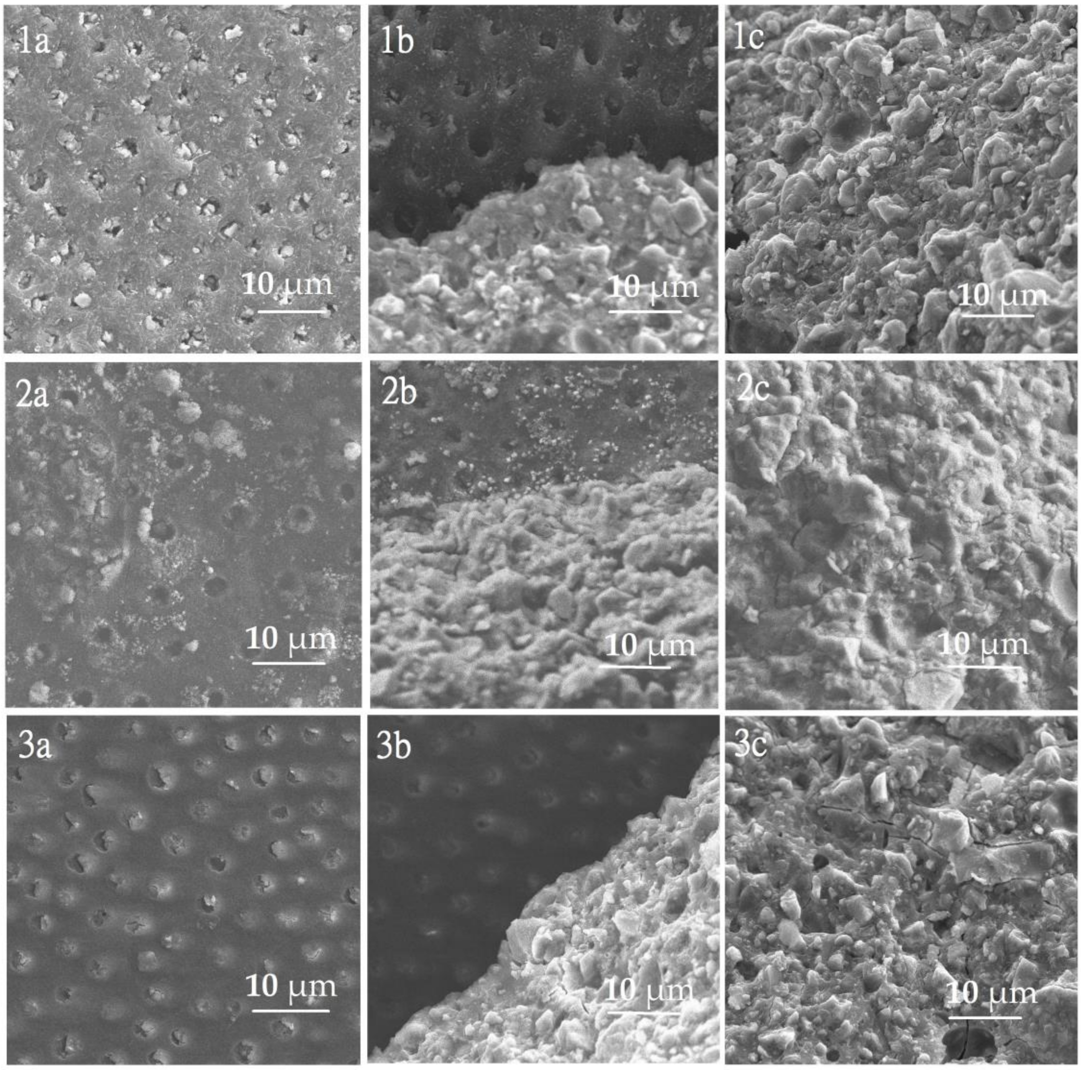


Figure 2 Mean shear bond strength between GIC and dentine of the three groups (n=19)



No difference in mean shear bond strength was found among the three groups (p=0.217).

Figure 3 Failure modes of dentine–GIC interface under SEM at 1000× magnification



Adhesive failure: SDF+KI (1a), SDF (2a), water (3a)

Mixed failure: SDF+KI (1b), SDF (2b), water (3b)

Cohesive failure: SDF+KI (1c), SDF (2c), water (3c)