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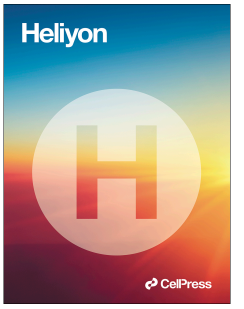

Impact of Silver Diamine Fluoride on Resin Composite Bond Strength: An In Vitro Study with Various Adhesive Systems.

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Title page:

Impact of Silver Diamine Fluoride on Resin Composite Bond Strength: An In Vitro Study with Various Adhesive Systems.

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Abstract:

Aim: This in vitro study investigates the effects of Silver Diamine Fluoride (SDF) application on the shear bond strength (SBS) of resin composite to sound and demineralized human teeth using different adhesive systems.

Methods: Eighty sound human third molars were sectioned, mounted in acrylic blocks, and prepared to expose a 2 mm thick dentin layer. The teeth were randomly divided into eight groups, each representing a combination of sound or demineralized dentin, SDF application, and adhesive system used. Demineralization was achieved using a pH cycling process. SDF was applied to the designated groups, followed by the use of either an etch-and-rinse or self-etch adhesive system. Resin composite was then applied, and the SBS was measured using a universal testing machine. Data were analyzed using three-way ANOVA and post-hoc tests to identify significant differences.

Results: The application of SDF generally led to a significant decrease in bond strength ($p<0.05$). In sound dentin, the SBS was higher compared to demineralized dentin ($p<0.05$). OptiBond FL showed higher bond strength than Clearfil SE Bond across all groups, although the difference was not statistically significant ($p>0.05$). The bond strength in the demineralized group using Clearfil SE Bond showed no significant difference between SDF application and non-application ($p>0.05$).

Conclusion: SDF application can significantly reduce the bond strength of resin composite to dentin, with the effect being dependent on the type of adhesive system and the state of the dentin. Further in vivo studies are needed to validate these findings.

Keywords: Silver Diamine Fluoride, Dental Bonding, Shear Strength, Composite Resins

Introduction:

Preserving tooth structure and preventing dental caries are fundamental objectives in dentistry [1]. Silver diamine fluoride (SDF), a topical fluoride agent, is widely used to arrest active dental caries and prevent new lesions in both primary and permanent teeth [2]. The US Food and Drug Administration (FDA) recognizes SDF as a Class 2 medical substance for managing dental hypersensitivity.

SDF reacts with hydroxyapatite in enamel and dentin to form calcium fluoride (CaF_2) and fluoroapatite ($Ca_{10}(PO_4)_6F_2$), which are less soluble in acidic environments. These reactions enhance the resistance of dental tissues to demineralization. Additionally, the silver ions in SDF inhibit bacterial metabolism, denature proteins, and interact with thiol groups in enzymes like collagenase to prevent the breakdown of dentin collagen. These combined effects make SDF effective in arresting dental caries and reinforcing dental structures [3]. Research suggests that caries removal prior to SDF application may not always be necessary, simplifying treatment and reducing invasiveness [4]. However, SDF can cause aesthetic side effects, which can be managed by placing a restoration over the SDF to hide its black color and restore the lost tooth structure [5].

In cases where composite restorations are used, achieving a strong and durable bond to the tooth surface is critical for restoration longevity and functionality. For composite restorations, achieving a durable bond involves a process called micromechanical hybridization, where adhesive resins infiltrate demineralized dentin tubules and interlock with exposed collagen fibers, thus forming a stable hybrid layer [6]. The durability of this bond depends on the degree of resin penetration into demineralized collagen and the stability of the hybrid layer, which is influenced by the matrix composition at the dentin-resin interface [7]. Insufficient resin penetration can activate matrix metalloproteinases (MMPs), leading to dentin matrix degradation [8]. Strategies such as pre-bonding dentin preparation using agents like chlorhexidine, modified polyglutamic acid, or SDF have been proposed to inhibit MMP activity and enhance bond stability [9]. SDF prevents collagen breakdown by inhibiting MMP-2, MMP-8, MMP-9, and cysteine cathepsin [3].

However, the effect of SDF on the bond strength of restorative materials remains debated. While some studies report a reduction in bond strength following SDF application, [10, 11] others find no significant impact [12, 13]. A systematic review by Jiang et al. [14] stated that no solid conclusion can be drawn on the effect of SDF application on the bond strength of dentine to adhesives due to the high degree of variation of the included studies. Moreover, differences in adhesive systems—such as the more technique-sensitive etch-and-rinse approach versus simplified self-etch adhesives—may influence how SDF interacts with the dentin substrate, potentially altering the hybrid layer's integrity. This variability creates uncertainty for clinicians, as a reduction in bond strength could compromise the durability of restorations.

The conflicting evidence regarding SDF's effect on bond strength underscores the need for further investigation to guide its clinical use. While the cariostatic properties of SDF are well-documented, its effects on the adhesion of restorative materials are less certain and may be influenced by various factors, including the type of adhesive system used and the choice of bonding protocol. This study examines how SDF affects the shear bond strength (SBS) of resin composite to sound and demineralized dentin using various adhesive systems. The null hypothesis for this study is that the application of SDF has no significant effect on the SBS of resin composite to sound and demineralized dentin, regardless of the adhesive system used in an in vitro setting.

Methods:

This in vitro study aimed to evaluate the effect of applying SDF on the SBS of resin composite to sound and demineralized human teeth using different adhesive systems. The research protocol was approved by the Ethics Committee of Isfahan University of Medical Sciences. Compositions, specifications, and manufacturers of the materials used in this study are displayed in Table 1.

Sample Size Justification:

The sample size was determined based on a power analysis to ensure that the study would have sufficient statistical power to detect a meaningful difference in SBS. The analysis was performed with the following parameters: a significance level (α) of 5% (0.05), a test power (1-β) of 80% (0.80), a standard deviation (S) of 0.06, and a clinically significant difference (d) of 0.85. These parameters were selected based on previous similar studies [15, 16] and the expected variability in the measurements. The sample size calculation is shown in Equation 1. This calculation indicated that a sample size of 10 specimens per group would be adequate to detect a clinically meaningful difference with the desired power and significance level.

$$n = \frac{(Z_{\frac{\alpha}{2}} + Z_{1-\beta})^2 \times 2S^2}{(\delta_1 - \delta_2)^2}$$

Equation 1 Sample size calculation formula

Experimental Procedure:

Eighty sound extracted human third molars were collected and immediately immersed in a 0.02% thymol solution in deionized water for preservation. Each tooth was sectioned below the cementoenamel junction using a high-speed handpiece bur with water spray. The coronal portion of each tooth was mounted on an acrylic block with cyanoacrylate adhesive to facilitate handling during testing. The occlusal surface of the teeth was aligned parallel to the transverse plane using a retaining jig designed on a slow-speed saw. Slow saws were then used to expose and cut a 2 mm thick layer of dentin. The thickness of the dental cuts was measured using a digital caliper. After dentin preparation, the bonding surface of each sample was rinsed with water and dried for five seconds with oil-free air spray to create a uniform smear layer. The bonding surface was then polished with 600 grit silicon carbide paper in an octagonal pattern, rinsed with water, and dried for five seconds with oil-free air spray to create a uniform smear layer. To ensure the absence of enamel on the dentin surfaces, all samples were examined under a stereo microscope (LeicaMS5, Wetzlar, Germany) at 20x magnification.

The teeth were randomly divided into eight groups with 10 samples per group, using a computer-generated randomization table. Each group represented a unique combination of sound or demineralized dentin, application of SDF, and the adhesive system used. Figure 1 provides a visual representation of the different study groups.

Demineralized dentin was prepared by subjecting the teeth to an eight-cycle pH cycling process, alternating between demineralizing and remineralizing solutions. The demineralizing solution contained 2.2 mol CaCl2, 2.2 mol NaH2PO4, and 50 mol acetic acid (pH = 4.8). The remineralizing solution contained 1.5 mmol CaCl2, 150 mmol KCl, and 0.9 mmol NaHPO4 (pH = 7). Each sample was immersed in the demineralizing solution for eight hours per cycle, followed by 16 hours in the remineralizing solution. [16, 17]

In half of the sound and demineralized samples, two drops of 38% SDF (Advantage arrest, Elevate Oral Care, USA) were placed on the tooth surface for 60 seconds, rinsed for 15 seconds, and dried for 5 seconds using oil-free air spray, following the manufacturer's instructions.

Two different adhesive systems were used in this study: an etch and rinse adhesive system (OptiBond FL, Kerr, CA, USA) and a self-etching adhesive (Clearfil Liner Bond F, Kuraray Medical Inc., Okayama, Japan). The adhesive systems were applied according to the manufacturer's instructions. A standardized clear mold (2 mm height and 1 mm diameter) was used to insert a 2 mm thick layer of hybrid resin composite material (Clearfil AP-X, Kuraray Medical Inc., Okayama, Japan) onto the prepared tooth surfaces. The composite was light cured for 40 seconds at an intensity of 1,200mW/cm2 using a Valo light cure unit (Ultradent, South Jordan, Utah).

The SBS of the resin composite to the tooth surface was measured using a universal testing machine (Zwick ROELL Z2.5 MA 18-1-3/7, Germany). Each specimen was subjected to a shearing force at a crosshead speed of 1 mm/min until bond failure occurred. The maximum force required for bond failure was recorded as the SBS value. The pattern of failure (adhesive or cohesive) in each specimen was determined using a stereomicroscope (LeicaMS5, Wetzlar, Germany) at 40x magnification.

Statistical Analysis:

All data were analyzed using SPSS software Version 23 (IBM Co., Armonk, NY, USA). The normality of the data was assessed using the Shapiro-Wilk test, and the homogeneity of variances across the groups was evaluated using Levene's test. Descriptive statistics including means and standard deviations were calculated. A three-way analysis of variance (ANOVA) was performed to examine the differences among the groups, with a significance level set at $p<0.05$. The factors included in the ANOVA were **dentin condition (sound vs. demineralized), SDF application, and adhesive system**, with the interaction terms also being analyzed. The results of the ANOVA indicated significant differences among the tested groups ($P<0.001$). To identify which specific groups differed significantly, the Tukey's HSD post hoc test was conducted. [18]

Results:

The mean SBS, standard deviation, and significance among study groups is presented in Table 2 and Table 3. To better comprehend the differences in SBS among these groups, a related graph was developed (Figure 2).

The highest mean SBS was observed in the Sound OptiBond FL group without SDF application at 26.63 ± 3.22 MPa, whereas the lowest mean SBS belonged to the Demineralized Clearfil SE Bond group with SDF application at 8.67 ± 1.51 MPa.

The SBS of Demineralized dentin bonded with OptiBond FL without SDF application was 15.10 ± 1.79 MPa, which was significantly higher than the SBS of Demineralized dentin bonded with OptiBond FL with SDF application, which had an SBS of 9.81 ± 1.74 MPa ($P<0.001$).

The SBS of Demineralized dentin bonded with Clearfil SE Bond without SDF application was 12.02 ± 1.46 MPa, which was not significantly different from the SBS of Demineralized dentin bonded with Clearfil SE Bond with SDF application, which had an SBS of 8.67 ± 1.51 MPa ($P>0.05$).

The SBS of Sound dentin bonded with OptiBond FL without SDF application was 26.63 ± 3.22 MPa. This value was significantly higher than the SBS of Sound dentin bonded with OptiBond FL with SDF application, which was 17.78 ± 2.60 MPa ($P<0.001$).

Similarly, for Clearfil SE Bond, the SBS of Sound dentin without SDF application was 23.53 ± 3.42 MPa, significantly higher than the SBS of Sound dentin with SDF application, which had an SBS of 16.29 ± 3.03 MPa ($P<0.001$).

In general, the bond strength in sound conditions was significantly higher than in demineralized conditions ($P<0.001$). The application of SDF significantly reduced bond strength ($P<0.001$), with the exception of the demineralized group using Clearfil SE Bond, which showed no significant difference between SDF application and non-application ($P>0.05$). Furthermore, the use of OptiBond FL resulted in higher bond strength than Clearfil SE Bond in all groups, but this difference was not statistically significant ($P>0.05$).

A three-way ANOVA evaluated the main effects of dentin condition (sound vs. demineralized), SDF application, and adhesive system. Dentin condition (mineralization) had a significant effect on SBS ($P < 0.001$), with the sound dentin group showing a higher mean SBS (21.046 MPa) compared to the demineralized dentin group (11.532 MPa).SDF application also had a significant effect ($P < 0.001$), where non-SDF-treated groups showed a higher mean SBS (19.366 MPa) than the SDF-treated groups (13.212 MPa). Adhesive system significantly affected SBS as well ($P < 0.001$), with OptiBond FL showing a higher mean SBS (17.461 MPa) compared to Clearfil SE Bond (15.117 MPa).

Discussion:

The primary objective of this in vitro study was to evaluate the impact of SDF application on the SBS of resin composite to sound and demineralized human teeth using two different adhesive systems, OptiBond FL (an etch and rinse adhesive) and Clearfil SE Bond (a self-etch adhesive).

Our study found that the use of SDF generally led to a significant reduction in bond strength. This aligns with previous literature, such as the study by Markham et al. [15], which also observed a decrease in the bond strength of three universal adhesive systems in self-etch mode to dentin after SDF application. John et al. [19] found that the average SBS of demineralized primary teeth using a self-etch adhesive with composites, dropped from 34.04 ± 8.2 Mpa to 18.81 ± 8.44 Mpa after applying SDF. This decrease in SBS was statistically significant. Abdullah et al. [20] also reported a significant decrease in SBS when using composite on SDF-treated demineralized dentine, compared to sound dentine without SDF treatment in primary teeth. The hybrid layer, a resin-infiltrated layer of demineralized dentin, is a key component in the adhesion of resin materials to dentin. However, the application of SDF may alter the composition and structure of this layer, potentially affecting the bond strength. It is therefore reasonable to anticipate a decrease in bond stability when dentin surfaces are contaminated with SDF application. These findings suggest that clinicians should carefully select adhesive systems when using SDF, as its application can significantly impact the bond strength, particularly in demineralized dentin.

However, when Clearfil SE Bond was used on the demineralized group, the bond strength did not show a significant difference whether SDF was applied or not. This implies that the impact of SDF on bond strength could be dependent on the type of adhesive system used and the state of the dentin. This is in line with the findings of Danaeifar et al. [21], where the pretreatment of dentin with SDF did not influence the SBS of the composite restorative material tested on primary sound teeth when a universal adhesive (Scotch Bond) was used. This suggest that the choice of adhesive can mitigate some of the negative effects of SDF. However, these finding should be interpreted with caution. While sdf did not significantly decreased bond strength in demineralized dentin with clearfil se bond, the bond strength was still less in compared with that on optibond fl.

OptiBond FL demonstrated a higher bond strength compared to Clearfil SE Bond across all groups, though this difference was not statistically significant. pH of SDF is alkaline, around 10-10.5 [22]. The demineralization of dentin surfaces by acid is crucial for hybrid layer formation [7]. Consequently, even after rinsing with water, the surface may remain alkaline, which could hinder the etching effect of an adhesive on enamel and dentin. This could explain the observed reduction in bond stability of self-etch adhesives compared to etch-and-rinse adhesives. These findings suggest that further studies should investigate the potential benefits of a stronger etching step for teeth treated with SDF.

In this study, we utilized the SBS test, which measures the resistance of a material to forces that can cause the internal structure of the material to slide against itself. However, there are various other testing methods such as Tensile Bond Strength (TBS), micro-Shear Bond Strength (mSBS), and micro-Tensile Bond Strength (mTBS). The SBS test applies force parallel to the adhesive interface, while the TBS test applies force perpendicular to the interface. The mSBS and mTBS tests are similar to their counterparts but are performed on a smaller scale. These tests can yield different results due to variations in the axis of force applied and the size of the bonding surface. Despite these differences, studies that used TBS [23], mTBS [24-26], and mSBS [11] tests have all reported a significant decrease in bond strength after SDF application, which is consistent with our findings.

This study's limitations include its in vitro nature, which may not fully replicate the clinical environment, and the lack of long-term durability testing of the bond strength. Bond strength tests are commonly used to evaluate restorative materials to predict their performance. Although the relationship between in vitro studies and clinical performance is difficult to establish, a material's adhesive ability is an indicator of restoration longevity; superior laboratory performance is probably indicative of better clinical performance [27]. Nevertheless, the results of in vitro studies should ideally be confirmed by long-term laboratory studies and randomized clinical trials, evaluating not only the effect of SDF pretreatment on bonding but also considering interface integrity, secondary caries, and staining. Given the valuable applications of SDF in dentistry, future research should include long-term in vivo studies to validate these findings and explore the effects of different concentrations and application protocols of SDF on bond strength.

Based on these findings, we reject the null hypothesis, as the application of SDF was found to significantly impact the SBS, with variations depending on the dentin condition and adhesive system.

In conclusion, this in vitro study examined the influence of SDF application on the SBS of resin composite to both sound and demineralized human teeth, utilizing two adhesive systems. Based on the results, we reject the null hypothesis, as the application of SDF was found to significantly reduce the bond strength, a finding that is consistent with prior research. The impact of SDF on bond strength seemed to be affected by the type of adhesive system employed and the state of the dentin. However, to fully understand the implications of SDF in a clinical context, further in vivo studies are recommended.

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Data availability statement:

Data associated with this study have not been deposited into a publicly available repository but data will be made available on request.

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Legends:

Figure 1. Visual representation of the different study groups.

Figure 2. Bond strenghth by mineralization, SDF application and bonding agent

Table 1. Compositions, specifications and manufacturers of the materials used in the study.

Table 2. The mean SBS, standard deviation, and significance among study groups. Groups with different significance letter are statistically significant.

Table 3. P-values for Comparative Analysis of different study groups.

Figure and Tables:

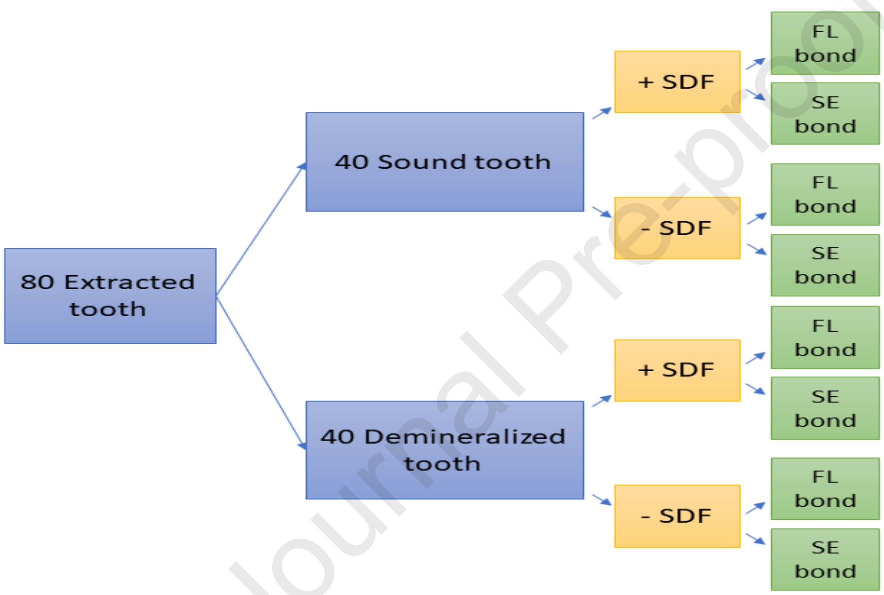
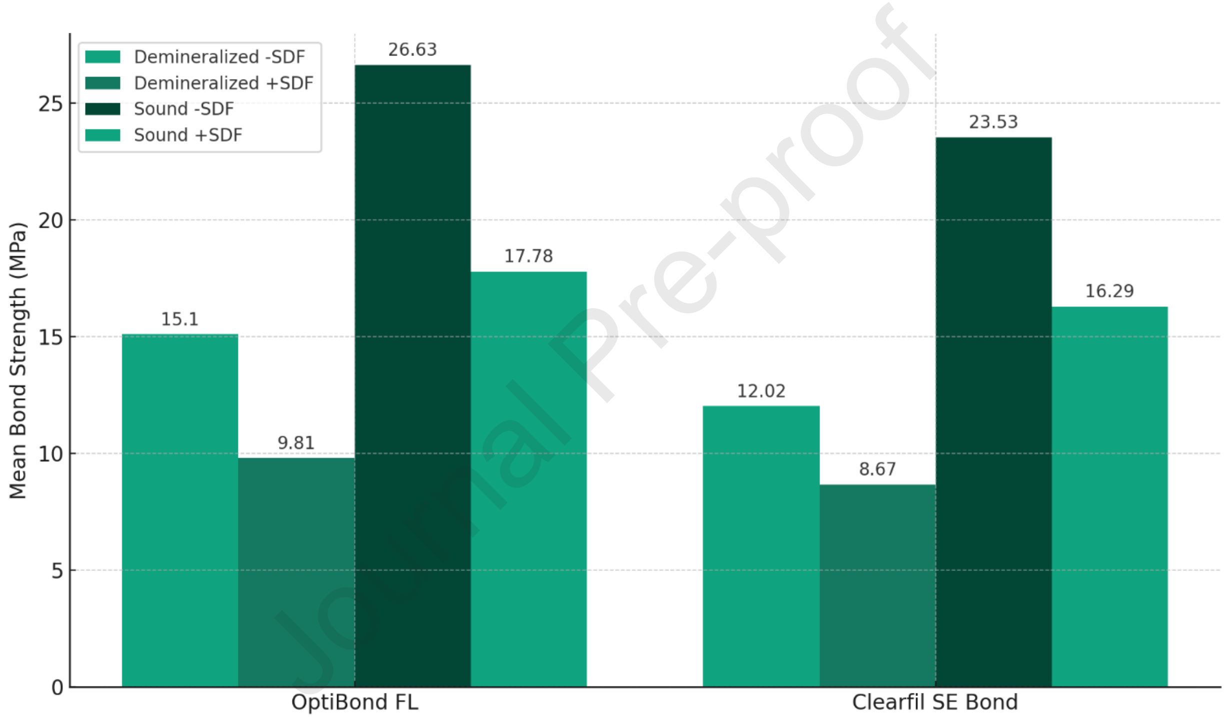


Figure 1



Demineralized -SDF
Demineralized +SDF
Sound -SDF
Sound +SDF
Mean Bond Strength (MPa)
0
5
10
15
20
25
15.1
9.81
26.63
17.78
12.02
8.67
23.53
16.29
OptiBond FL
Clearfil SE Bond

Figure 2

Materials	Type	Composition	Application
Advantage Arrest (Elevate Oral Care)	Silver Diammine Fluoride	38% Silver Diammine Fluoride pH: 10	SDF applied for 10 s, was allowed to absorb for 1 min. After 1 min, excess SDF was removed with a microbrush and specimens were rinsed with water for 15 s then air-dried for 5 s prior to bonding.
Scotchbond Etchant (3M)	Phosphoric Acid	37% phosphoric acid pH: 0.5	Apply and rub for 15 s; Rinse off for 15 s
OptiBond FL (Kerr)	Etch & Rinse adhesive	**Primer**: HEMA, GPDM, PAMM, ethanol, water, photoinitiator **Adhesive**: TEG-DMA, UDMA, GPDM, HEMA, Bis-GMA, filler, photoinitiator	Place applicator tip into the yellow Unidose capsule (OptiBond FL Prime). Apply material to the prepared dentin surfaces with a light scrubbing motion for 15 seconds. Gently air dry for approximately 5 seconds. At this point the dentin surface should have a slightly shiny appearance. Using the same applicator brush, place tip into the black Unidose capsule (OptiBond FL Adhesive). Apply material to the prepared dentin surfaces with a light scrubbing motion for 15 seconds. Blow to margin or to thin if necessary using a light application of air.
Clearfil SE Bond (Kuraray)	Two-step Self-etch Adhesive	**Primer**: 10-MDP, HEMA, hydrophilic dimethacrylate, dicamphorquinone , N,N-diethanol-p-toluidine, water. **Bond**: 10-MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, di-camphorquinone, N,N-diethanol-p-toluidine, silanated colloidal silica	Apply primer for 20 s, then gently air dry; Apply bonding agent, gently air blow; Light cure for 10 s
Clearfil AP-X (Kuraray)	Hybrid composite	Bis-GMA, silica fillers, silica-titania fillers(53% filler by volume, 0.04 to 0.6 µm particle size), CQ	Dispense in layers up to 2 mm in thickness; Light cure for 40 s

Table 1

Mineralization	Bonding	SDF	Mean ± Std Deviation (MPa)	Significance
Demineralized dentin	OptiBond FL	-SDF	15.10 ± 1.79	c
		+SDF	9.81 ± 1.74	d
	Clearfil SE Bond	-SDF	12.02 ± 1.46	cd
		+SDF	8.67 ± 1.51	d
Sound dentin	OptiBond FL	-SDF	26.63 ± 3.22	a
		+SDF	17.78 ± 2.60	b
	Clearfil SE Bond	-SDF	23.53 ± 3.42	a
		+SDF	16.29 ± 3.03	b

Table 2

Study groups		P-value
Demineralized Optibond FL (SDF-)	Demineralized Optibond FL (SDF+)	<0.001
	Sound Optibond FL (SDF-)	<0.001
	Sound Optibond FL (SDF+)	0.249
	Demineralized Clearfil SE (SDF-)	0.111
	Demineralized Clearfil SE (SDF+)	<0.001
	Sound Clearfil SE (SDF-)	<0.001
	Sound Clearfil SE (SDF+)	0.961
Demineralized Optibond FL (SDF+)	Sound Optibond FL (SDF-)	<0.001
	Sound Optibond FL (SDF+)	<0.001
	Demineralized Clearfil SE (SDF-)	0.490
	Demineralized Clearfil SE (SDF+)	0.968
	Sound Clearfil SE (SDF-)	<0.001
	Sound Clearfil SE (SDF+)	<0.001
Sound Optibond FL (SDF-)	Sound Optibond FL (SDF+)	<0.001
	Demineralized Clearfil SE (SDF-)	<0.001
	Demineralized Clearfil SE (SDF+)	<0.001
	Sound Clearfil SE (SDF-)	0.111
	Sound Clearfil SE (SDF+)	<0.001
Sound Optibond FL (SDF+)	Demineralized Clearfil SE (SDF-)	<0.001
	Demineralized Clearfil SE (SDF+)	<0.001
	Sound Clearfil SE (SDF-)	<0.001
	Sound Clearfil SE (SDF+)	0.877
Demineralized Clearfil SE (SDF-)	Demineralized Clearfil SE (SDF+)	0.063
	Sound Clearfil SE (SDF-)	<0.001
	Sound Clearfil SE (SDF+)	0.006
Demineralized Clearfil SE (SDF+)	Sound Clearfil SE (SDF-)	<0.001
	Sound Clearfil SE (SDF+)	<0.001

Sound Clearfil SE (SDF-)	Sound Clearfil SE (SDF+)	<0.001

Table 3

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: