

Original Article

The effects of various desensitizers on the bond strengths of different cements to dentin

Mengqin Gu, Yujie Tan, Wangyang Li, Jialin Huang, Zhenzhen Zhang, Ting Xiong, Ling Guo

Department of Prosthodontics, Hospital of Stomatology Southwest Medical University, Luzhou, Sichuan, China

Received March 2, 2018; Accepted January 10, 2019; Epub April 15, 2019; Published April 30, 2019

Abstract: The aim was to evaluate the shear bond strength (SBS) of dentin to resin composites with different desensitizers, cements, and thermal-cycling. This study consists of the SBS test (60 premolars) and the scanning electron microscopy (SEM/8 premolars) test. 60 premolars were sectioned yielding 120 specimens and divided into four groups ($n=30$ each): control, Duraphat, Bifluorid 12, and Prime & Bond NT. Each group was divided into three subgroups ($n=10$ each): RelyX™ U200, Glass ionomer cement (GIC), and Resin Modified Glass Ionomer Cement (RMGIC/RelyX™ Luting Cement). After 24 hours storage in a 37 °C water bath and thermal-cycling (5 °C/55 °C), the SBS was tested by a universal testing machine. We checked the failure modes under a magnifying glass and analyzed the data. Additionally, the effect of desensitizers on the dentin surface was observed with SEM. The application of Duraphat and Bifluorid 12 reduced the SBS value significantly ($P<0.05$), and the Prime & Bond NT combined with RelyX™ U200 improved the retentive strength ($P<0.05$), and with GIC, the SBS was reduced ($P<0.05$), and with RMGIC, there was no significant difference from the untreated group ($P>0.05$). The SBS values of all the groups was lower after thermal-cycling than in their corresponding 24 h groups ($P<0.05$). The three desensitizers have different effects on the three cements, and the Prime & Bond NT can enhance the SBS of RelyX™ U200.

Keywords: Desensitizer, cement, shear bond strength, thermal-cycling

Introduction

One of the most common complaints of patients undergoing full crown and bridge restoration on account of exposure of the dentin tubules on vital abutment teeth is dentinal hypersensitivity (DH), which is defined as a transient and intense pain, caused by any form of thermal, evaporative, tactile, osmotic or chemical stimulation after the dentin is exposed, pain that cannot be attributed to any other dental disease or defect [1].

There are some theories about DH, including neural theory, transduction theory and hydrodynamic theory [2]. The phenomenon with the best explanation is hydrodynamic theory, which states that outside stimuli, such as thermal, evaporative, tactile, osmotic or chemical stimuli, lead to the flow of liquids within the dentin tubules, and the fluid movement triggers nerve fibers in the pulp, provoking a stimulation that is interpreted as pain or sensitivity [3]. DH is more frequently encountered in patients with

vital teeth prepared for restoration because 1.2-1.5 mm of tooth structure is removed and approximately 1-2 million dentin tubules are exposed in the process of tooth preparation [4]. Additionally, the generation of heat by friction is an important hypersensitivity factor [5].

Several medicines and treatments have been recommended to relieve sensitivity, such as Duraphat, Bifluorid 12 and Prime & Bond NT, which basically work by occluding the dentin tubules in varying degrees and restraining the movement of liquid [6-9]. However, a question has been raised about that the application of desensitizers may affect bond strengths on account that they seal the dentin tubules or induce a chemical interaction with the dentin organic matrix [10]. Many scholars have investigated the influences of desensitizers on the bond strength of cements and have drawn different conclusions. Some studies stated that the tensile bond strength of GIC is decreased significantly after treatment with desensitizing

Shear bond strength of various treatments to dentin

Table 1. Names, lot numbers, and manufacturers of the tested materials

Name	Lot Number	Manufacturer
Duraphat	184339	Colgate, New York, NY, USA
Bifluorid 12	1616623/1611624	Voco GmbH, Cuxhaven, Germany
Prime & Bond NT	1612000416	Dentsply, Konstanz, Germany
RelyX™ U200	625134	3M ESPE, St Paul, MN, USA
GIC	1539532/1534398	Voco GmbH, Cuxhaven, Germany
RMGIC	N836563/N837397	3M ESPE, St Paul, MN, USA
Fitek™ Z250	N655907	3M ESPE, St Paul, MN, USA

materials, but the retentive strength of resin cements is largely increased [11]. Sarac et al. found that all desensitizers decreased the bond strength of resin cements to dentin, except for hydroxyethyl methacrylate (HEMA) containing a desensitizing agent [12].

This project selected three common desensitizing materials (Duraphat, Bifluorid 12 and Prime & Bond NT) and bonding systems (RelyX™ U200, GIC and RMGIC). Duraphat is a varnish with 2.3% F and has been established that it has effective desensitization. Some scholars have suggested that the application of Duraphat could form a spherical calcium fluoride (CaF₂) on the dentin surface [6, 13]. Bifluorid 12 contains 5.6% F, and sodium fluoride particles penetrate into tubules to prevent hypersensitivity and CaF₂ or CaF₂-like deposits on the dentin surface which decrease the dentin permeability [7, 13]. A previous study indicated that the application of Bifluorid 12 prior to cementation prevented the forming of a stable hybrid layer resulting in a bond strength reduction [14]. Prime & Bond NT possesses a favorable penetrability and adhesiveness quality, and it exposes dentin tubules by acid-etching, then resin penetrates into tubules, a polymerizing resin tag to block tubules mechanically [8].

Numerous studies have investigated the strengths of GIC and RelyX™ U200 after coating with fluoride containing desensitizers [8, 15]. Most of studies stated that desensitizing paste can be safely used on dentin to alleviate sensitivity after cementation without compromising the retention of crowns luted with RelyX™ U200, but the bond strength of GIC would decrease by a large margin [15]. Additionally, the adhesion persistence of cements is critical for the long-range success of dentin [16]. However,

a comparison of the bond strength and durability of RelyX™ U200, GIC, and RMGIC with the desensitizers covered in this study has rarely been reported. Moreover, few studies have compared the effects of the desensitization and adhesion strengths of Duraphat and Bifluorid 12. Therefore, the aim of this study is to compare desensitizing materials (Duraphat, Bi-

fluorid 12 and Prime & Bond NT) on the bond strength and adhesion persistence of cements (RelyX™ U200, GIC and RelyX™ Luting cement), so as to help clinicians select the most effective therapy for vital teeth preparation. The null hypotheses were as follows: (1) the bond strength would not be affected by desensitizers; (2) thermal-cycling would not affect the bond strength.

Materials and methods

Three desensitizing materials, three bonding agents, and resin prostheses materials were evaluated in this study. The brands, lot numbers, and manufacturers of the tested materials are listed in **Table 1**, and their handling procedures are listed in **Table 2**.

Specimens storage

We collected 68 intact and plesiomorphic premolars extracted for orthodontic reasons at the Hospital of Stomatology, Southwest Medical University and cleaned them mechanically, then we stored in distilled water at 4°C until the experiment began (we changed the distilled water once a week, and the total storage time was less than 2 months). The study consisted of an SBS test (60 premolars) and an SEM test (8 premolars).

SBS test

The flow chart of the shear test is shown in **Figure 1**. The teeth were located parallel to their long axis on a custom-designed cup filled with die stone materials and placed on a milling machine. The buccal or lingual surfaces of the teeth structure were removed a uniform depth of 1.2-1.5 mm until superficial dentin was exposed, then we cut these samples trans-

Shear bond strength of various treatments to dentin

Table 2. Handling procedures of the tested materials

Material	Handling procedures
Duraphat	Apply pea-sized Duraphat to prepared surface with a brush and massage it gently, then remove it with water and air dry.
Bifluorid 12	Apply it to the tooth surface, and allow it to be absorbed for 10-20 s and air dry.
Prime & Bond NT	Etch enamel and dentin, with 37% phosphoric acid, rinse with water, and blot dry, apply Prime & Bond NT to the tooth surface and light-cure for 20 s.
RelyX™ U200	The base paste and catalyst paste mix within 20 s and apply it to the tooth surface. Finally, cure it for 20 s.
GIC	Mix the powder with liquid in accordance with ratio of 1:1, and apply to the tooth surface.
RMGIC	Dispense powder and liquid according to powder/liquid ratio of 1.6:1 by weight, mix them within 30 s and apply it to prosthesis.
Fitek™ Z250	Apply resin into plastic matrix to 2 mm height and light cure for 20 s.

Shear bond strength of various treatments to dentin

Figure 1. Distribution of specimens.

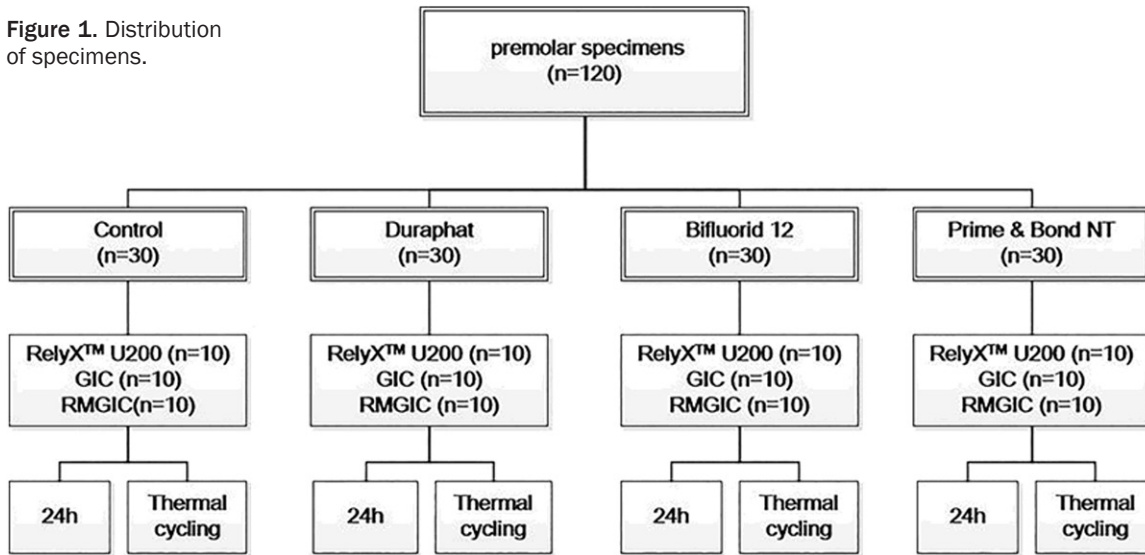


Table 3. SBS of the different groups (MPa, $\bar{x} \pm s$)

		RelyX U200	GIC	RMGIC
24 h	Control	7.19±0.91	2.84±0.78	5.81±0.91
	Duraphat	4.16±1.41	1.33±0.63	1.66±0.54
	Bifluorid 12	4.73±1.73	0.90±0.47	1.03±0.46
	Prime & Bond NT	12.67±0.72	1.16±0.92	5.66±0.64
24 h+tc	Control	4.97±1.02	1.85±0.99	4.31±0.80
	Duraphat	1.00±0.56	0.66±0.51	0.80±0.56
	Bifluorid 12	1.05±0.51	0.75±0.45	0.64±0.51
	Prime & Bond NT	7.30±0.67	0.53±0.44	3.31±0.64

versely at a 2 mm distance from the cemento-enamel junction (CEJ) toward the apex with cold water spraying and separated along the central groove, yielding 120 specimens with osteotome, and polished the bonding plane with a wheel rotational polishing machine using 320, 400, and 600-grit, respectively, silica carbide abrasive paper under a steady stream of water. All specimens were immersed in 14% EDTA (PH=7.4) for 1 min to pretreat the smear layer-covered dentin surfaces [17], rinsed with distilled water repeatedly, submerged into 75% ethyl alcohol for 5 min, then dried and made ready for use. Finally, these samples were divided into four groups at random each containing 30 samples. The dentin surfaces in the control group did not receive any treatment, and Duraphat (Colgate, New York, NY, USA), Bifluorid 12 (Voco GmbH, Cuxhaven, Germany) and Prime & Bond NT (Dentsply, Konstanz, Germany) were applied to the remaining three groups, respectively. After 30 min, all the gr-

roups were subdivided into three subgroups of 10 each in which RelyX™ U200 (3M ESPE, St Paul, MN, USA), Glass ionomer luting cement (Voco GmbH, Cuxhaven, Germany) or RelyX™ Luting Cement (3M ESPE, St Paul, MN, USA) was applied to the specimens. A transparent circular plastic matrix (r=2 mm, h=5 mm) was used to fabricate resin prostheses (Fitek™ Z250, 3M ESPE, St Paul, MN, USA). All specimens were pressed onto the resin prostheses with a force of 30 N

for 5 min. After 30 min standing, these samples were embedded in cylinders (r=1 cm, h=1 cm) full of die stone, and the bonding interface was slightly higher than plaster surface. Until the plaster completely hardened, all the samples were submerged in water at 37°C for 24 h. After this storage time, half of the samples were tested immediately, and the other half were treated with thermal-cycling 5000 times (5°C/55°C) in a thermocycling machine (TC-501F, Well, Suzhou, China) with a dwell time of 30 s and a transfer time of 10 s [18, 19]. The SBS values of all the specimens were measured at a crosshead speed of 0.5 mm/min in a universal testing machine (WDW-20, Jinan, China) until failure [19, 20]. The SBS was expressed with MPa (P=F/S, namely the force of dislodgment divided by the bonding area is equal to the SBS value). Additionally, the debonded surfaces of the teeth and prostheses were identified with a magnifier at 20 × magnification, and the failure modes were catego-

Shear bond strength of various treatments to dentin

Table 4. Three-way ANOVA test of factors influencing SBS

Source	Freedom of Motion	Mean Square	F	P
D	3	106.901	165.609	0.000*
C	2	172.941	267.915	0.000*
T	1	100.438	155.595	0.000*
D × C	6	32.504	50.354	0.000*
D × T	3	3.058	4.738	0.004*
C × T	2	24.756	38.352	0.000*
D × C × T	6	1.703	2.638	0.021*
Error	96	0.646	-	-
Total	120	-	-	-

D: desensitizers, C: cements, T: thermal-cycling.

* $P < 0.05$: statistically significant difference.

rized into four groups as follows: Type I: cement principally on the tooth surface (>75%); Type II: cement equally distributed on the tooth and prosthesis surfaces (25%-75%); Type III: cement principally on prosthesis surface (>75%); Type IV: tooth fracture [21].

SEM test

There were 8 premolars which were divided into four groups according to desensitizing materials. All the samples were removed occlusal structures with a uniform depth of 2 mm, and they were sectioned perpendicularly to the long axis of the teeth at a level of 2.0 mm distance from the CEJ toward the apex, then we smoothed the dentin surfaces with 320, 400, and 600-grit, respectively, silica carbide abrasive paper under a steady stream of water, and then we immersed them in 14% EDTA (PH=7.4) for 1 min, and rinsed them with distilled water repeatedly to ensure removal of any excess product from the surfaces. All desensitizing materials were applied on the dentin surfaces following the manufacturer's instructions, and then the control samples were gently rinsed with distilled water for 10 s. After the application of the desensitizers, all the specimens were split along the central groove with osteotome and were coated with a layer of palladium using an ion sputter coater (E-1010, HITACHI, Japan), then we observed the dentinal tubules' occlusions and the tubules' profiles under SEM (SU1510, HITACHI, Japan) at 15.0 KV. The SEM photomicrographs acquired at × 2,500 magnification were evaluated by one

examiner who was blinded to the experiment program.

Statistical analysis

The SBS values data were analyzed using a three-way ANOVA (desensitizer, cement and thermal-cycling), a two-way ANOVA and an LSD test to analyze the multiple comparisons with the help of SPSS 21.0. The level of statistical significance was set at 0.05.

Results

Shear bond test

The SBS mean values and standard deviations of all the groups are presented in **Table 3**. The RelyX™ U200+Prime & Bond NT treatment without thermal-cycling showed the highest SBS mean value (12.67 ± 0.72 Mpa), while the GIC+Prime & Bond NT treatment with thermal-cycling showed the lowest value (0.53 ± 0.44 Mpa). As seen in **Table 4**, a three-way analysis of variance (ANOVA) demonstrated that there were statistically significant differences for the factors “desensitizers” ($F=165.609$, $P < 0.05$), “cements” ($F=267.915$, $P < 0.05$), “exposure to thermal-cycling” ($F=155.595$, $P < 0.05$), the interaction between desensitizers and cements ($F=50.354$, $P < 0.05$), the interaction between desensitizers and thermal-cycling ($F=4.738$, $P=0.004$), and the interaction between cements and thermal-cycling ($F=38.352$, $P < 0.05$), as well as the interaction among three factors ($F=2.638$, $P=0.021$).

For the RelyX™ U200 groups, a two-way ANOVA and an LSD test showed that the application of the desensitizers (Duraphat, Bifluorid 12, Prime & Bond NT) prior to cementation with RelyX™ U200 significantly affected the SBS values ($P < 0.05$), but there were no significant difference between the Duraphat and Bifluorid 12 groups ($P=0.501$). That is to say, Prime & Bond NT increased the SBS value of RelyX™ U200, but Duraphat and Bifluorid 12 decreased it, and the reduction has no significant difference.

For GIC groups, pretreating the dentin surfaces with desensitizers (Duraphat, Bifluorid 12, Prime & Bond NT) significantly reduced the bond strength ($P < 0.05$), but no significant difference was found among the three groups ($P > 0.05$).

Shear bond strength of various treatments to dentin

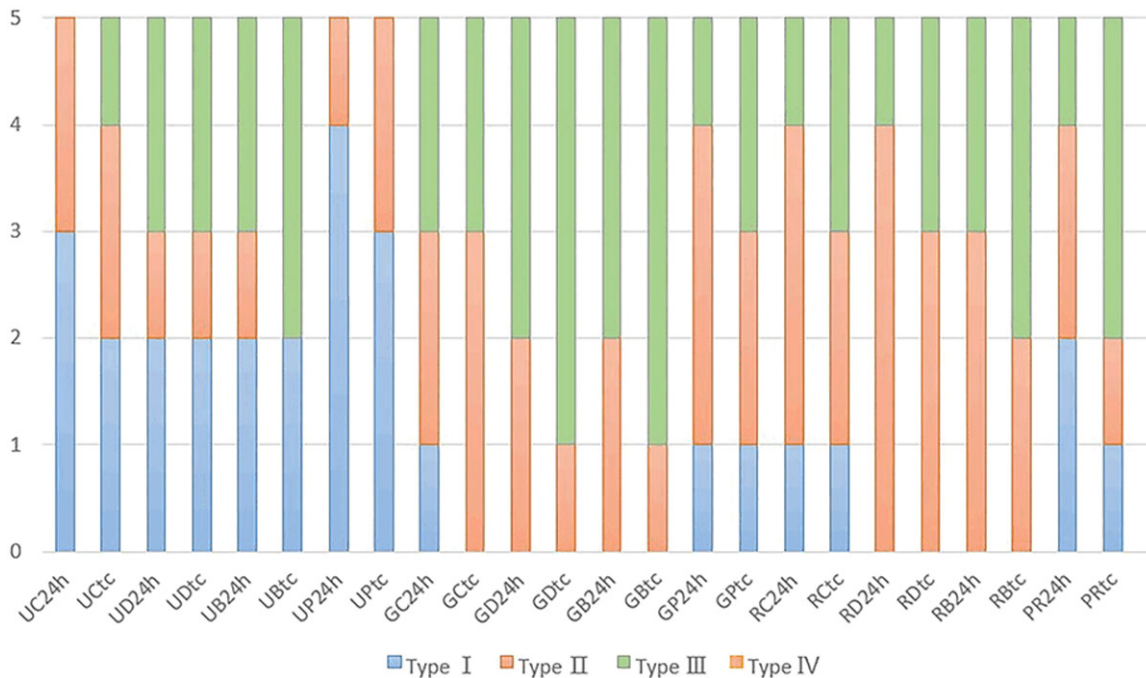


Figure 2. Distribution of failure modes (number of surfaces) for each cement group according to the applied desensitizers. U: RelyX™ U200, G: GIC, R: RelyX™ Luting cement, C: control group, D: Duraphat, B: Bifluorid 12, P: Prime & Bond NT, 24 h: store in water at 37 °C for 24 h, tc: store in water at 37 °C for 24 h and thermal-cycling 5000 times. Type I: cement principally on tooth surface (>75%); Type II: Cement equally distributed on tooth and prosthesis surfaces (25%-75%); Type III: cement principally on prosthesis surface (>75%); Type IV: tooth fracture.

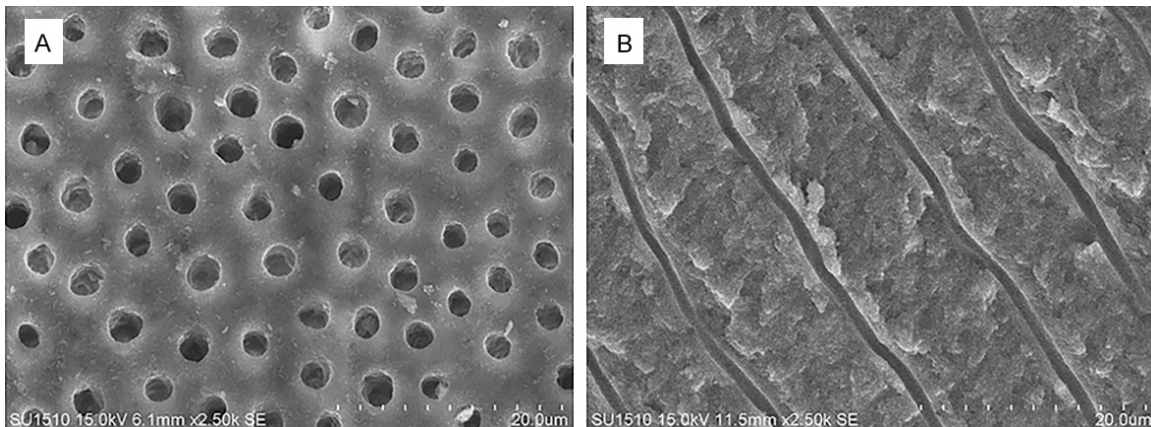


Figure 3. The SEM micrographs (2500 ×) of cross-sectional view (A) and lateral view (B) of dentin tubules with no treatment.

For the RMGIC groups, there were significant differences between each of the two groups ($P < 0.05$), except for the untreated group with the Prime & Bond NT group ($P = 0.056$), and the Duraphat group with the Bifluorid 12 group ($P = 0.186$). In other words, Prime & Bond NT would not influence the bond strength of RMGIC, and values for Duraphat and Bifluorid 12 were significantly lower than the control group, but the Duraphat group and the Bifluorid 12

group did not show any significant influence in the strength values.

According to the LSD test for cements, RelyX™ U200 showed a higher SBS value than other groups ($P < 0.05$), and the lowest was obtained with GIC ($P < 0.05$). In addition, thermal cycling decreased the SBS values for all the cement groups with various desensitizers ($P < 0.05$).

Shear bond strength of various treatments to dentin

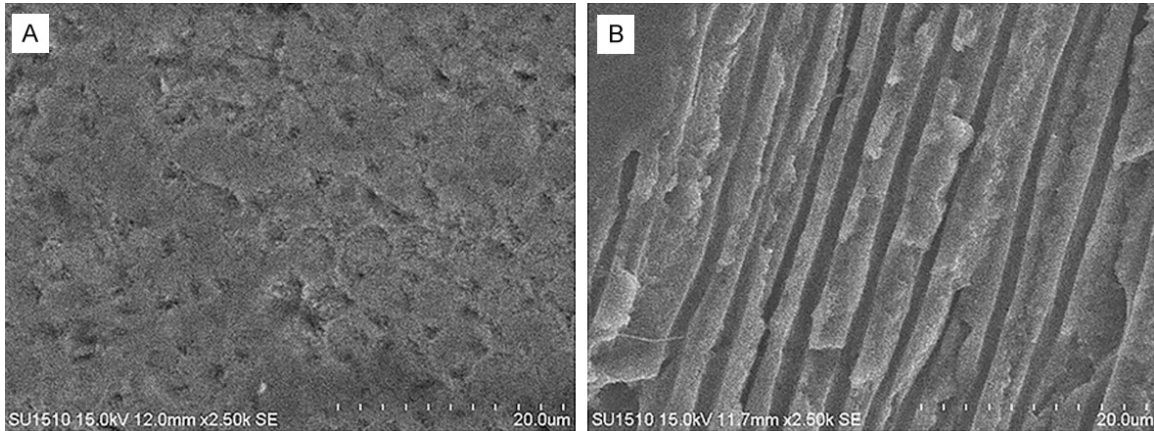


Figure 4. The SEM micrographs (2500 ×) of the cross-sectional view (A) and the lateral view (B) of dentin tubules treated with Duraphat.

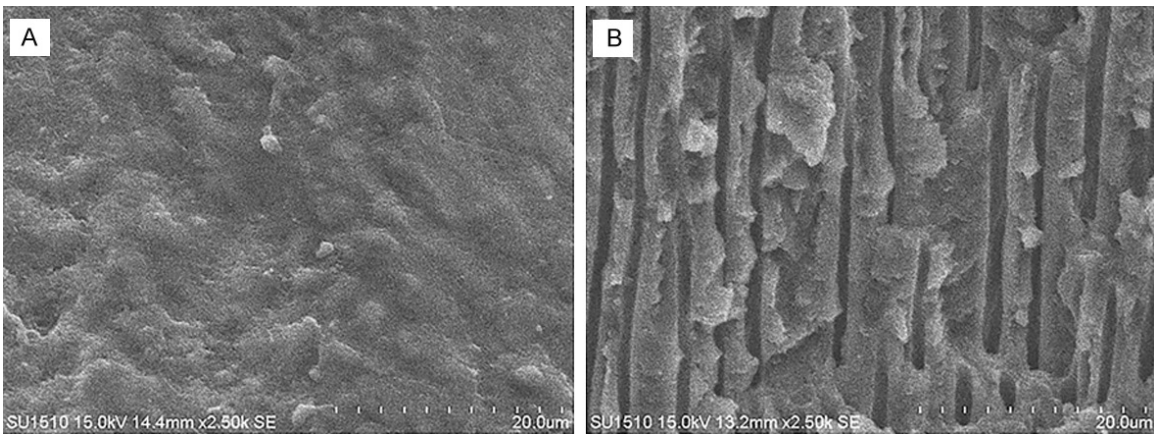


Figure 5. The SEM micrographs (2500 ×) of the cross-sectional view (A) and the lateral view (B) of dentin tubules treated with Bifluorid 12.

Figure 2 presents the failure modes after separation. For RelyX™ U200, the predominant debonding failures were Type I (50%); for GIC, but the failure modes were predominantly Type III (52.5%) and Type II (40%); the main dislodgment modes of RMGIC were Type II (50%) and Type III (37.5%).

SEM test

Photomicrographs of all groups are shown in **Figures 3-6**. Dentin tubule orifices of the control groups have no smear plugobliterating the tubules, and the tubules were straight and parallel (**Figure 3A, 3B**). The Duraphat groups revealed that fluoride precipitants were on the dentin surface, occluding the dentin tubules partially, and from a lateral view, most of tubules were blocked by sedimentation (**Figure 4A, 4B**). The SEM observation of the Bifluorid

12 groups showed remarkable depositions on the dentin surfaces and within the tubules, sealing the dentin tubules completely (**Figure 5A, 5B**). The Prime & Bond NT groups produced a uniform hybrid layer with less-exposed dentin tubules, and convex structures within the tubules could also be observed (**Figure 6A, 6B**).

Discussion

Both null hypotheses claiming that the bond strength would not be affected by desensitizers and thermal-cycling would not influence the bond strength were rejected. Because some groups with desensitizing materials had decreased the bond strength compared to the untreated group, the thermal-cycling was not beneficial for the bond strengths of all the groups.

Shear bond strength of various treatments to dentin

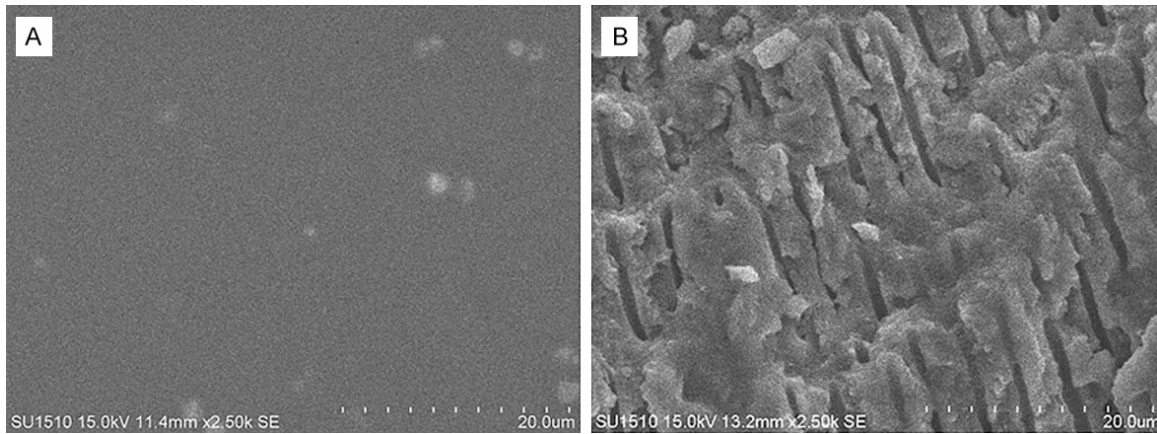


Figure 6. The SEM micrographs (2500 ×) of the cross-sectional view (A) and the lateral view (B) of dentin tubules treated with Prime & Bond NT.

In this study, the mean SBS value required to dislodge the crowns luted on untreated specimens with RelyX™ U200, GIC and RMGIC were 7.19 ± 0.91 Mpa, 2.84 ± 0.78 Mpa, 5.81 ± 0.91 Mpa, respectively.

For the Duraphat applied specimens, the bond strengths required to dislodge the crowns luted with RelyX U200, GIC and RMGIC were 4.16 ± 1.41 Mpa, 1.33 ± 0.63 Mpa, 1.66 ± 0.54 Mpa, respectively, which were found to be lower when compared with untreated specimens ($P < 0.05$). The finding is corroborated by previous observations demonstrating that the fluoride-containing agents lead to a lower retentive strength value [22-24]. Acar et al. stated that pretreatment of dentin surfaces with fluoride reduced the retentive strength of resin cements [23]. For RelyX™ U200, adhesion is probably achieved by micromechanical retention which occurs between demineralized dentin and the resin cement. Duraphat occluding the dentin tubules leads to a loss of micromechanical interlocking, thus decreasing the bond strength.

As shown in **Table 3**, when Bifluorid 12 was applied as a desensitizer on dentin surfaces, the bond strength of RelyX™ U200, GIC and RMGIC were 4.73 ± 1.73 Mpa, 0.90 ± 0.47 Mpa, 1.03 ± 0.46 Mpa, respectively. These findings implied that Bifluorid 12 caused a great degree loss of the bond strengths of the three cements compared with untreated specimens ($P < 0.05$). This conformed to the conclusions of the previous study, which reported that Bifluorid 12 significantly decreased the SBS values on account of the morphological changes

of the dentin surface and the dentin tubules [25]. Külünk et al. also demonstrated that the bond strengths of resin cements decreased when desensitizers containing sodium fluoride and calcium fluoride were applied to a dentin surface [26].

Duraphat is a varnish with 2.26% sodium fluoride (PH=4.5), and the principal components of Bifluorid 12 are 2.71% sodium fluoride and 2.92% calcium fluoride. Fluorination infiltrates into the dentin tubules and produces fluorapatite depositions, which seal the dentin tubules and relieve them from any outside stimulus that might reduce sensitivity [27]. In addition, the fluoride concentration compensates those dissolved apatite minerals and facilitates remineralization, and the CaF_2 or CaF_2 -like depositions on the dentin surface reduced the dentin permeability [13, 28, 29]. In general, chemical protection and mechanical barriers guarantee its desensitization [13, 28, 29]. After employing Duraphat or Bifluorid 12 to a dentin surface, the bond strength values of the three cements significantly decreased. There are some reasons for this: (1) as presented in the SEM micrographs, fluorapatite not only occludes the dentin tubules, but it also fills in the irregular dental surface; this way, it decreases the mechanical retention of cements; (2) they could form a spherical calcium fluoride on the dentin surface which could change tooth surface morphology, hindering the formation of resin tags and affecting bonding strength [6]; (3) although Duraphat possesses stickiness, it is easy to soften and shield when it encounters mechani-

Shear bond strength of various treatments to dentin

cal and chemical stimuli, and tiny cracks would further reduce the retentive strength; (4) the products of Bifluorid 12 on the dentin surface neutralize the acid from the etching agents, obstructing the sufficient infiltration of the resin monomer, preventing the formation of a stable hybrid layer, and leading to a decline in the bond strength [14]. The consequences of this study manifested that the bond strengths obtained from groups exposed to Bifluorid 12 were lower than those exposed to Duraphat (Table 3), although it has no statistical significance ($P>0.05$). This conclusion conformed to the findings of a previous study by Sarac et al. [12] who stated that the retentive strength value between the resin cements and dentin surface showed an adverse relationship with the fluoride content of desensitizers [12].

When on Prime & Bond NT applied specimens, the necessary bond strength to dislodge crowns cemented with RelyX™ U200 was 12.67 ± 0.72 Mpa, a significantly higher bond strength of RelyX™ U200 compared to the control group ($P<0.05$). This conclusion is contrary to a previous investigation which claimed that the application of Prime & Bond NT did not affect the retentive bond of resin cement (Panavia) [30]. The difference may be attributed to the resin cement types and their compositions in the mentioned studies. But above all, the bonding property of resin cements rests with the quality of the hybrid layer, which is formed during dental preparation [31, 32]. Otherwise, we also thermo-cycled these specimens, a step which was not performed in the earlier studies. RelyX™ U200 resin cement is a dual-curing self-adhesive cement, and it is incapable of demineralizing the dentin fully, which hinders the formation of the hybrid layer and resin tags [33]. However, Prime & Bond NT exposes dentin tubules and the collagen network of demineralized dentin by acid-etching, then the resin penetrates into the tubules, polymerizing the resin tags and the hybrid layer to block the tubules mechanically and reduce the movement of the fluid [8], which is verified in the SEM pictures. HEMA monomers in RelyX™ U200 have a chemical affinity to resin layers [34]. Therefore, the joint utilization of RelyX™ U200 and Prime & Bond NT have the desired effect of desensitization and adhesion.

After treating the samples with Prime & Bond NT, however, the bond strength required to dislodge crowns luted with GIC was 1.16 ± 0.92

Mpa, which was found to be significantly reduced compared to the untreated samples ($P<0.05$). The conclusion was consistent with earlier study stating that resin desensitizing agents reduced retention with GIC [35]. The adhesive mechanism of GIC to dentin surfaces consists of the mechanical retention force (48.3%) and chemical bonding (51.7%) [36]. Desensitizers weaken the mechanical interlocking by occluding the dentin tubules, and the coverage of the desensitizers prevents ionic interaction between the carboxyl group of polyacid chains and calcium on the tooth surface [37]. Therefore, for GIC, any type of desensitizing material (Duraphat, Bifluorid 12, Prime & Bond NT) would lead to a significantly decreased SBS value.

When we applied Prime & Bond NT prior to the cementation with RMGIC, the bond strength required to dislodge the crown was 5.66 ± 0.64 Mpa, and this finding implies that the retentive ability of RMGIC is unaffected by Prime & Bond NT ($P>0.05$), which was in agreement with the finding of earlier study by Swift et al., who reported that resin desensitizing agents had little or no effect on the retention of crowns cemented with RMGIC [38]. A possible reason could be that the adhesive mechanism of RMGIC is achieved through chemical bonding between negatively charged carboxylic acid groups of cement and the positively charged polyacid calcium ions of dentin, in combination with micromechanical bonding through infiltration into the collagen network [39].

Thermal-cycling was an artificial aging technique to verify the durability of cements simulating the oral conditions. The effect of thermal-cycling on the bond strength is affected by the number of cycles, temperature settings, dwell time and so on [40]. The number of cycles is supposed to be the crucial facet [41]. The ISO standard addressed that it was an appropriate test for aging cements to be subjected to 500 thermal cycles between 5°C and 55°C [41]. However, results of previous studies reported that 500 thermal cycles were not effective in reducing the SBS between cements and dentin surfaces but 5000 thermal cycles at least, since the performance improvement of cements [18, 42]. Many researchers stated that 10000 thermal cycles were equivalent to 1 year of in-vivo functioning [43]. In this study, thermal-cycling significantly decreased the me-

Shear bond strength of various treatments to dentin

an SBS values of all groups ($P < 0.05$), which conformed to the previous studies claiming that thermal-cycling had an adverse effect on bond strength [20, 32, 44]. The possible explanations could be as follows: (1) hot water may accelerate the movement of water on the bonding interface, the extraction of poorly polymerized resin oligomers, the hydrolysis of interface materials, as well as the aging and plastifying of resin, resulting in decreasing mechanical properties of the bonding systems [45-47]; (2) on account of the different dilatation coefficients, temperature variations produce internal stress and cracks at the tooth-biomaterial interface, leading to the degradation of the bond strength [36, 48, 49].

When the failure modes were inspected, the observations also reflected these characteristics. In this study, a lower type value is beneficial, because the bond strength between cement and the dentin is greater than prosthesis and there is less cement to be removed from the crown after debonding [50]. For RelyX™ U200, the predominant debonding failures were Type I (50%), and Type III was more pronounced in those groups in combination with Duraphat or Bifluorid 12; for GIC, the failure modes were predominantly Type III (52.5%) and Type II (40%); for RMGIC, the value mainly was Type II (50%) and Type III (37.5%); the application of Duraphat and Bifluorid 12 lead to higher type values, which implied that less cement remains on the tooth surface, corresponding to lower SBS values. The combination between Prime & Bond NT and various cements have different effects on failure mode, which also agreed with the changes in the SBS values. Additionally, after thermal-cycling, the increasing type values implied that the interaction between the dentin surfaces and the cements decreased.

Acknowledgements

This work was supported by the Fund of Luzhou Science and Technology Project under Grant no. 2010-S-16(1/3) and the Fund of the Sichuan Medical Association under Grant no. S16078.

Disclosure of conflict of interest

None.

Address correspondence to: Ling Guo, Department of Prosthodontics, Hospital of Stomatology Southwest Medical University, 2 Jiangyang South Road, Luzhou 646000, Sichuan, China. Tel: +86-83-0310-7522; E-mail: 372083745@qq.com

References

- [1] Abed AM, Mahdian M, Seifi M, Ziaei SA and Shamsaei M. Comparative assessment of the sealing ability of Nd: YAG laser versus a new desensitizing agent in human dentinal tubules: a pilot study. *Odontology* 2011; 99: 45-48.
- [2] Jacobsen PL and Bruce G. Clinical dentin hypersensitivity: understanding the causes and prescribing a treatment. *J Contemp Dent Pract* 2001; 2: 1-12.
- [3] Brannstrom M and Astrom A. The hydrodynamics of the dentine; its possible relationship to dentinal pain. *Int Dent J* 1972; 22: 219-227.
- [4] Richardson D, Tao L and Pashley DH. Dentin permeability: effects of crown preparation. *Int J Prosthodont* 1991; 4: 219-225.
- [5] Rosenstiel SF and Rashid RG. Postcementation hypersensitivity: scientific data versus dentists' perceptions. *J Prosthodont* 2003; 12: 73-81.
- [6] Chu CH and Lo EC. A review of sodium fluoride varnish. *Gen Dent* 2006; 54: 247-253.
- [7] Antoniazzi RP, Machado ME, Grellmann AP, Santos RC and Zanatta FB. Effectiveness of a desensitizing agent for topical and home use for dentin hypersensitivity: a randomized clinical trial. *Am J Dent* 2014; 27: 251-257.
- [8] Shafiei F and Memarpour M. Effect of repeated use on dentin bond strength of two adhesive systems: all-in-one and one-bottle. *Indian J Dent Res* 2009; 20: 180-184.
- [9] Ozen T, Orhan K, Avsever H, Tunca YM, Ulker AE and Akyol M. Dentin hypersensitivity: a randomized clinical comparison of three different agents in a short-term treatment period. *Oper Dent* 2009; 34: 392-398.
- [10] Al Qahtani MQ, Platt JA, Moore BK and Cochran MA. The effect on shear bond strength of rewetting dry dentin with two desensitizers. *Oper Dent* 2003; 28: 287-296.
- [11] Kumar S, Rupesh PL, Daokar SG, Yadao AK, Ghunawat DB and Sayed SS. Effect of desensitizing laser treatment on the bond strength of full metal crowns: an in vitro comparative study. *J Int Oral Health* 2015; 7: 36-41.
- [12] Sarac D, Kulunk S, Sarac YS and Karakas O. Effect of fluoride-containing desensitizing agents on the bond strength of resin-based cements to dentin, *J Appl Oral Sci* 2009; 17: 495-500.
- [13] Bergstrom EK, Birkhed D, Granlund C and Skold UM. Approximal caries increment in ado-

Shear bond strength of various treatments to dentin

- lescents in a low caries prevalence area in Sweden after a 3.5-year school-based fluoride varnish programme with Bifluorid 12 and Duraphat. *Community Dent Oral Epidemiol* 2014; 42: 404-411.
- [14] Tay FR, Pashley DH, Mak YF, Carvalho RM, Lai SC and Suh BI. Integrating oxalate desensitizers with total-etch two-step adhesive. *J Dent Res* 2003; 82: 703-707.
- [15] Pilo R, Harel N, Nissan J and Levartovsky S. The retentive strength of cemented zirconium oxide crowns after dentin pretreatment with desensitizing paste containing 8% arginine and calcium carbonate. *Int J Mol Sci* 2016; 17: 426.
- [16] AlRabiah M, Labban N, Levon JA, Brown DT, Chu T-M, Bottino MC and Platt JA. Bond strength and durability of universal adhesive agents with lithium disilicate ceramics: a shear bond strength study. *Oper Dent* 2017; 1-10.
- [17] Kambara K, Nakajima M, Hosaka K, Takahashi M, Thanatvarakorn O, Ichinose S, Foxton RM and Tagami J. Effect of smear layer treatment on dentin bond of self-adhesive cements. *Dent Mater J* 2012; 31: 980-987.
- [18] Magni E, Ferrari M, Papacchini F, Hickel R and Ilie N. Influence of ozone on the composite-to-composite bond. *Clin Oral Investig* 2011; 15: 249-256.
- [19] Nima G, Ferreira PVC, Paula AB, Consani S and Giannini M. Effect of metal primers on bond strength of a composite resin to nickel-chrome metal alloy. *Braz Dent J* 2017; 28: 210-215.
- [20] Ozel Bektas O, Eren D, Herguner Siso S and Akin GE. Effect of thermocycling on the bond strength of composite resin to bur and laser treated composite resin. *Lasers Med Sci* 2012; 27: 723-728.
- [21] Johnson GH, Lepe X and Bales DJ. Crown retention with use of a 5% glutaraldehyde sealer on prepared dentin. *J Prosthet Dent* 1998; 79: 671-676.
- [22] Shahabi M, Ahrari F, Mohamadipour H and Moosavi H. Microleakage and shear bond strength of orthodontic brackets bonded to hypomineralized enamel following different surface preparations. *J Clin Exp Dent* 2014; 6: e110-115.
- [23] Acar O, Tuncer D, Yuzugullu B and Celik C. The effect of dentin desensitizers and Nd: YAG laser pre-treatment on microtensile bond strength of self-adhesive resin cement to dentin. *J Adv Prosthodont* 2014; 6: 88-95.
- [24] Lewinstein I, Stoleru-Baron J, Block J, Kfir A, Matalon S and Ormianer Z. Antibacterial activity and tensile strength of provisional cements modified with fluoridecontaining varnish. *Quintessence Int* 2013; 44: 107-112.
- [25] Yilmaz NA, Ertas E and Orucoglu H. Evaluation of five different desensitizers: a comparative dentin permeability and SEM investigation in vitro. *Open Dent J* 2017; 11: 15-33.
- [26] Kulunk S, Sarac D, Kulunk T and Karakas O. The effects of different desensitizing agents on the shear bond strength of adhesive resin cement to dentin. *J Esthet Restor Dent* 2011; 23: 380-387.
- [27] Rosin-Grget K and Lincir I. Current concept on the anticaries fluoride mechanism of the action. *Coll Antropol* 2001; 25: 703-712.
- [28] Margolis HC, Moreno EC and Murphy BJ. Effect of low levels of fluoride in solution on enamel demineralization in vitro. *J Dent Res* 1986; 65: 23-29.
- [29] Lodha E, Hamba H, Nakashima S, Sadr A, Nikaido T and Tagami J. Effect of different desensitizers on inhibition of bovine dentin demineralization: micro-computed tomography assessment. *Eur J Oral Sci* 2014; 122: 404-410.
- [30] Chen L, Xu F, Guan Z, Zhang W and Wang X. Comparison of the occlusion effect of three kinds of desensitizers on dentinal tubules. *Zhong Nan Da Xue Xue Bao Yi Xue Ban* 2014; 39: 959-963.
- [31] Triolo PT, Kelsey WP 3rd and Barkmeier WW. Bond strength of an adhesive resin system with various dental substrates. *J Prosthet Dent* 1995; 74: 463-468.
- [32] Stawarczyk B, Hartmann R, Hartmann L, Roos M, Ozcan M, Sailer I and Hammerle CH. The effect of dentin desensitizer on shear bond strength of conventional and self-adhesive resin luting cements after aging. *Oper Dent* 2011; 36: 492-501.
- [33] Youm SH, Jung KH, Son SA, Kwon YH and Park JK. Effect of dentin pretreatment and curing mode on the microtensile bond strength of self-adhesive resin cements. *J Adv Prosthodont* 2015; 7: 317-322.
- [34] Jalandar SS, Pandharinath DS, Arun K and Smita V. Comparison of effect of desensitizing agents on the retention of crowns cemented with luting agents: an in vitro study. *J Adv Prosthodont* 2012; 4: 127-133.
- [35] Mausner IK, Goldstein GR and Georgescu M. Effect of two dentinal desensitizing agents on retention of complete cast coping using four cements. *J Prosthet Dent* 1996; 75: 129-134.
- [36] De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M and Van Meerbeek B. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res* 2005; 84: 118-132.
- [37] Tyas MJ and Burrow MF. Adhesive restorative materials: a review. *Aust Dent J* 2004; 49: 112-121; quiz 154.
- [38] Swift EJ Jr, Lloyd AH and Felton DA. The effect of resin desensitizing agents on crown retention. *J Am Dent Assoc* 1997; 128: 195-200.

Shear bond strength of various treatments to dentin

- [39] Yoshioka M, Yoshida Y, Inoue S, Lambrechts P, Vanherle G, Nomura Y, Okazaki M, Shintani H and Van Meerbeek B. Adhesion/decalcification mechanisms of acid interactions with human hard tissues. *J Biomed Mater Res* 2002; 59: 56-62.
- [40] Wadleigh RG, Redman RS, Graham ML, Krasnow SH, Anderson A and Cohen MH. Vitamin E in the treatment of chemotherapy-induced mucositis. *Am J Med* 1992; 92: 481-484.
- [41] Amaral FL, Colucci V, Palma-Dibb RG and Corona SA. Assessment of in vitro methods used to promote adhesive interface degradation: a critical review. *J Esthet Restor Dent* 2007; 19: 340-353; discussion 354.
- [42] Ozcan M, Barbosa SH, Melo RM, Galhano GA and Bottino MA. Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. *Dent Mater* 2007; 23: 1276-1282.
- [43] Xie C, Han Y, Zhao XY, Wang ZY and He HM. Microtensile bond strength of one- and two-step self-etching adhesives on sclerotic dentine: the effects of thermocycling. *Oper Dent* 2010; 35: 547-555.
- [44] Jurubeba JEP, Costa AR, Correr-Sobrinho L, Tubel CAM, Correr AB, Vedovello SA, Crepaldi MV and Vedovello MF. Influence of thermal cycles number on bond strength of metallic brackets to ceramic. *Braz Dent J* 2017; 28: 206-209.
- [45] Miyazaki M, Sato M and Onose H. Durability of enamel bond strength of simplified bonding systems. *Oper Dent* 2000; 25: 75-80.
- [46] Hass V, Dobrovolski M, Zander-Grande C, Martins GC, Gordillo LA, Rodrigues Accorinte Mde L, Gomes OM, Loguercio AD and Reis A. Correlation between degree of conversion, resin-dentin bond strength and nanoleakage of simplified etch-and-rinse adhesives. *Dent Mater* 2013; 29: 921-928.
- [47] Dickens SH and Cho BH. Interpretation of bond failure through conversion and residual solvent measurements and Weibull analyses of flexural and microtensile bond strengths of bonding agents. *Dent Mater* 2005; 21: 354-364.
- [48] Vasquez V, Ozcan M, Nishioka R, Souza R, Mesquita A and Pavanelli C. Mechanical and thermal cycling effects on the flexural strength of glass ceramics fused to titanium. *Dent Mater J* 2008; 27: 7-15.
- [49] Cadenaro M, Antonioli F, Codan B, Agee K, Tay FR, Dorigo Ede S, Pashley DH and Breschi L. Influence of different initiators on the degree of conversion of experimental adhesive blends in relation to their hydrophilicity and solvent content. *Dent Mater* 2010; 26: 288-294.
- [50] Horiuchi S, Kaneko K, Mori H, Kawakami E, Tsukahara T, Yamamoto K, Hamada K, Asaoka K and Tanaka E. Enamel bonding of self-etching and phosphoric acid-etching orthodontic adhesives in simulated clinical conditions: debonding force and enamel surface. *Dent Mater J* 2009; 28: 419-425.