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ORIGINAL ARTICLE

Influence of fluoride- or triclosan-based desensitizing agents on adhesion of resin cements to dentin

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Abstract Effect of desensitizers on the bond strength of resin cements to dentin was evaluated. Intact premolars (N=90) were embedded in polymethyl methacrylate; dentin surfaces were exposed, and they were randomly divided into two main groups of cements (Duolink (D), Variolink II (V); n=45 per group) and then into three desensitizer subgroups (n=15 per subgroup). Teeth in controls (C) were treated according to cements' adhesion protocols; the other two groups received either fluoride- [Aqua-Prep F (F)] or triclosan-based [Seal&Protect (T)] desensitizers. Ceramic disks (Empress 2) were adhered; specimens were thermocycled (×5,000 cycles, 5–55±1°C, dwell time 30 s) and subjected to shear bond strength test (MPa±SD) in a universal testing machine (crosshead speed 1 mm/min). Failure types were classified using scanning electron microscope. For V, application of both desensitizers (29.6±7.8 and 22.8±2.8 for F and T, respectively) did not present significantly different results than that of the VC (21.2±2.3; $p>0.05$, one-way ANOVA). In D, F (20.6±2.4) showed significantly higher results ($p<0.05$) than those in T (16.1±3.9) and DC group (15.2±2.3). V showed significantly higher results than D ($p<0.05$, Bonferroni). F and T did not negatively affect the bond strength results with D and V. Adhesive failures were more frequent with both T (84%) and F (66%) in D; cohesive failures in the cement (88%) were more commonly observed with F in V. Both F and T desensitizers can be safely used prior to final cementation but F in combination with V seems to be more reliable, considering both the bond strength and the failure types.

Keywords Adhesives · Ceramics · Desensitizers · Resin cement · Shear bond strength

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Introduction

In order to create sufficient space for the indirect fixed dental prosthesis, in clinical practice, most of the time enamel has to be removed with rotating instruments leading to exposure of the dentinal tubules. Dentin exposure may cause bacterial diffusion and trigger pulpal inflammatory response with subsequent formation of reparative dentin [1–3]. In several studies, a significant correlation between microbial micro-leakage and pulpal inflammation has been demonstrated [3–7].

The rate of bacterial invasion, which is approximately 1.6 μm/day, has been found to increase over time [8]. In a relatively short period of time (up to 4 days), bacteria can infiltrate the tubules. The odontoblastic processes, collagen fibers, kinetics of tubular fluid, and immunological function do not seem to be sufficient to inhibit this process. It is also inevitable that the exposed dentin subjected to mechanical, thermal, tactile, or osmotic stimuli results in hypersensitivity [1, 9].

It is generally recognized that dentin hypersensitivity is inhibited by the precipitation of water-insoluble substances at the orifices or inside the dentinal tubules. One minimal invasive approach for the treatment of hypersensitivity involves sealing of dentinal tubules or application of sedative agents and promotion of dentin remineralization [10]. Usually, potassium nitrate, oxalate, fluoride, and triclosan-based dentin desensitizers are recommended immediately after tooth preparation [11]. The use of such barriers to seal the dentinal tubules prior to cementation has been advocated in order to reduce the effect of external stimuli on hypersensitivity. Some ingredients present in dentin desensitizers may induce chemical interaction with organic substances of the dentin that may consequently affect the sealing and bonding characteristics of the adhesive cement system [10, 12–15]. Recent desensitizing products mainly involve fluoride and triclosan. In fact, the function of fluoride introduced to the desensitizers is to obturate the dentinal tubules like a funnel with incorporation of mainly 2-hydroxyethyl methacrylate (HEMA) hydrophilic monomer, which increases infiltration ability of the primer. On the other hand, triclosan acts as an anti-inflammatory agent [8, 16]. It can be anticipated that the presence of fluoride in the desensitizer may yield to obturation of dentinal tubules, and triclosan may create a low surface free energy [8] and thereby impair the adhesion of resin-based cements due to decreased wettability of the latter. However, it should also be taken into consideration that resin cements are not directly in contact with the desensitizer-treated dentin surfaces in that their adhesive systems are first applied which is followed by the resin cement itself. In that respect, among adhesive cements, those with their corresponding adhesive resins based on three-step etch-and-rinse systems may be expected to deliver improved adhesion to dentin as opposed to two-step etch-and-rinse systems. Nevertheless, limited studies are available concerning the influence of desensitizing agents containing fluoride and triclosan on the bond strength of the resin cements [5, 9, 13].

The objectives of this study therefore were to investigate the effect of fluoride- and triclosan-based desensitizers on the adhesion of a glassy matrix ceramic luted to dentin with two different resin cement systems and to evaluate the failure types after debonding.

Materials and methods

Preparation of the ceramic specimens

The brand names, manufacturers, chemical compositions, and batch numbers of the materials used in this study are listed in Table 1.

Lithia-based all-ceramic specimens (IPS Empress 2, Ivoclar Vivadent, Schaan, Liechtenstein; $N=90$; diameter 4 mm, height 3 mm) were prepared according to the lost-wax technique recommended by the manufacturer. Cementation surfaces were air-particle-abraded with 50 µm Al_2O_3 for the duration of 10 s from a distance of approximately 10 mm and ultrasonically cleaned (Sonorex, Bandeline, USA) for 15 min in deionized water. Then, the specimens for Variolink II (V) group were etched with 5% hydrofluoric acid (Ceramic Etching Gel, Ivoclar Vivadent) for 30 s, washed and rinsed thoroughly, air-dried, and silanized (Monobond-S, Ivoclar Vivadent). Ceramic specimens for Duolink (D) group were etched with 4% hydrofluoric acid (Ceramic Etching Gel, Bisco) for 30 s, washed and rinsed thoroughly, air-dried, and silanized (Porcelain Primer, Bisco). Reaction time of the silane coupling agent was 60 s for both resin groups. Then, the adhesives specific for each resin group (Heliobond, Ivoclar Vivadent and One-Step Plus, Bisco) were applied in a thin layer, gently air-blown, and photo-polymerized with a halogen photo-polymerization unit (Optilux 501, Kerr, USA) for 40 s from a constant distance of 2 mm from the surface. Light intensity was assured to be higher than 400 mW/cm^2, verified by a radiometer after every six specimens (Demetron LC, Kerr).

Preparation of tooth specimens

Sound maxillary human premolar teeth of similar sizes (minimum 4 mm mesiodistally and minimum 6.5 mm buccolingually at their largest section; $N=90$, $n=30$ per cement group), extracted for orthodontic purposes, were selected from a pool of recently extracted teeth that were stored in distilled water with 0.1% thymol solution at 4°C for a duration of 6 months [17]. The root surfaces were cleaned from debris using periodontal scalers under water. The clinical crowns were removed up to 2 mm below the buccal cementoenamel junction using a diamond bur (Brasseler, Savannah, GA, USA) under copious water spray. The teeth were then mounted in plastic rings using autopolymerizing polymethyl methacrylate (Palapress Vario, Hereaus Kulzer, Wehrheim, Germany). Dentin surfaces were exposed by cutting occlusal enamel and dentin perpendicular to the tooth axis using a slow-speed

Table 1 The brand names, manufacturers, chemical compositions, and batch numbers of the materials used for the experiments

Brand names and manufacturers	Chemical compositions	Batch numbers
Variolink II (V) (Ivoclar Vivadent, Schaan, Liechtenstein)	Cement base: Bis-GMA, UEDMA, TEGDMA, filler	E 51946
	Cement low viscosity catalyst: Bis-GMA, UEDMA, TEGDMA, filler	E 52170
Etching gel (total etch)	37% phosphoric acid	B 2835 B 28357
Primer (Syntac)	Tetraethylene glycol dimethacrylate, maleic acid, dimethyl ketone, water	E 34592
Adhesive (Syntac)	Polyethylene glycol dimethacrylate, glutaraldehyde, maleic acid, water	E 30794
Bond (Heliobond)	Bis-GMA, triethylene glycol dimethacrylate	E 51105
Ceramic etching gel	5% hydrofluoric acid	E 52923
Silane (Monobond-S)	3-Methacryloxy propyl-trimethoxysilane, water, ethanol	E 26882
Oxygen-inhibiting gel	Glycerine, silica	D 50843
Duolink (D) (Bisco Inc. Schaumburg, IL, USA)	Cement (base/catalyst): bis-GMA, TEGDMA, glass filler, urethane dimethacrylate	0400003526
Etching gel (Uni-Etch)	32% phosphoric acid	0400003457
Adhesive (One-Step Plus)	Monomer, BPDM, acetone	0400001415
Ceramic etching gel	4% hydrofluoric acid gel	0400009887
Silane (porcelain primer)	3-Methacryloxy propyl-trimethoxysilane, ethanol, acetone	0400003325
Aqua-Prep F (Bisco, Inc. Schaumburg, IL, USA) (F)	2.5 mg/m^3 fluoride, HEMA	0300011889
Seal&Protect (Dentsply Co., UK) (T)	Methacrylate resins, PENTA, nanofillers, triclosan	0503000759

Bis-GMA bis-phenol-A-glycidyl methacrylate, *UEDMA* urethane dimethacrylate, *TEGMA* triethylene glycol methacrylate, *HEMA* 2-hydroxyethyl methacrylate, *BPDM* biphenyl dimethacrylate, *PENTA* dipentaerythritol penta-acrylate monophosphate

saw with a diamond-coated disk (Isomet, Buehler, Lake Bluff, IL, USA) under water cooling. Then, the dentin surfaces were wet-polished with 600 grit silicon carbide paper which simulates the diamond-disk-prepared tooth surface with a standard smear layer in clinical situations [8]. The teeth were then stored in distilled water at 4°C.

The exposed dentin surfaces were further inspected with an optical microscope (MP 320, Carl Zeiss, Jena, Germany; ×50) to ensure that no enamel was left. The specimens were then randomly divided into two main groups according to the resin cements to be used, namely D (Bisco) and V (Ivoclar Vivadent). Specimens under each cement group were further randomly divided into three subgroups ($n=15$ per subgroup) to receive the desensitizing agents. While the teeth in control groups (C) were only treated according to each cement's adhesion protocol, the other two groups received either fluoride- [Aqua-Prep F (F)] or triclosan-based [Seal&Protect (T)] desensitizers.

Cementation procedure

The embedded and sectioned teeth were ultrasonically cleaned in distilled water for 60 s and gently dried with an air-spray. A thin adhesive tape with a 4-mm diameter opening in its center was securely stuck to the middle of the exposed dentin surface in order to limit the bonding surface area. The tooth surfaces were etched with phosphoric acids specific for each resin cement accordingly, washed, and dried gently. Desensitizing agents were applied according to each manufacturer's instructions.

Aqua-Prep F It was applied as a thin layer on the dentin surfaces with a brush and left in contact for 20 s. At this stage, care was taken to avoid pooling, and the excess was removed with 5 s light air-drying until a shiny appearance of the surface was obtained.

Seal&Protect One coat was applied with a brush on the dentin surfaces. A gentle stream of compressed air was used to volatilize the acetone solvent, and it was photo-polymerized for 20 s.

Control The specimens that were not treated with any of the desensitizers but with the corresponding adhesives of the cements acted as controls for each resin cement.

The conditioned surfaces of the ceramic disks were adhered onto the conditioned dentin surfaces with one of the two resin cements under a constant load of 300 g using an alignment device. The alignment device was custom-made by the modification of a parallelometer on which a knife-edge diamond tip was attached vertically in order to secure the vertical positioning of the specimen. For D cement groups, a photo-polymerized dentin adhesive (One-Step Plus) was applied for 15 s and polymerized for 10 s followed by the application of the dual-polymerized D cement. For V cement groups, primer (Syntac Primer, Ivoclar Vivadent) and adhesive (Syntac Adhesive, Ivoclar Vivadent) were applied for 15 and 10 s, respectively, and gently air-dried. Then, the bonding agent (Heliobond, Ivoclar Vivadent) was applied followed by the application of the dual-polymerized resin cement according to the manufacturer's instructions. It was light-polymerized circumferentially, from mesial, distal, buccal, and lingual sides for 40 s each. The dentin surfaces in the control groups were treated identically with the experimental groups, and the ceramic disks were adhered in the same manner as described above per cement. The free margins of the ceramic disks were coated with an oxygen-inhibiting gel (Oxyguard, Kuraray, Osaka, Japan). After 5 min, the cement was light-polymerized once again at four aspects for 40 s to ensure the transmission of light in case oxygen-inhibiting gel was unevenly distributed and created air bubbles. Finally, the specimens were rinsed with water, stored in distilled water in the dark at 37°C for 24 h, and then subjected to thermocycling for 5,000 cycles between 5°C and 55°C in deionized water (Willytech, Gräfelfing, Germany). The dwelling time at each temperature was 30 s, and the transfer time from one bath to the other was 2 s.

Shear bond strength test

Specimens were mounted in the jig of the universal testing machine (Autograph Model AG-50 kNG, Shimadzu, Japan), and the shear force was applied using a shearing blade with a 45° inclination to the ceramic/tooth interface until failure occurred. The load was applied to the adhesive interface, as close as possible to the surface of the substrate at a crosshead speed of 0.5 mm/min, and the stress–strain curve was analyzed with the software program.

Failure analysis

Cold field emission scanning electron microscope (SEM; JSM 5200, Kyoto, Japan) images were taken at 25 kV at a magnification of ×750. The debonded dentin surfaces were first sputter-coated with a 3-nm-thick layer of gold (80%)/palladium (20%) prior to examination. The failure types were defined as "adhesive" between the dentin and the resin cement and "cohesive" within the cement only. The term "mixed" failure was used to describe the combination of these two failure types.

Statistical analysis

Statistical analysis was performed using the SPSS 14.0 software for Windows (SPSS Inc., Chicago, IL, USA). The means of each group were analyzed by one-way analysis of variance (ANOVA) with the bond strength values as the dependent variable. Due to the significant difference between groups ($p=0.002$), Bonferroni post hoc test was performed. The failure types were analyzed using Kruskal–Wallis. p values less than 0.05 were considered to be statistically significant in all tests.

Table 2 The mean (±standard deviations) bond strength values (MPa) for the desensitizers in combination with two adhesive cement systems

Resin cement	Adhesive	Desensitizer	Mean (SD)
Duolink	One-Step Plus	Seal&Protect (T)	16.1 (3.9)a
Duolink	One-Step Plus	Aqua-Prep F (F)	20.6 (2.4)b
Duolink	One-Step Plus	Control (C)	15.2 (2.3)a
Variolink II	Syntac	Seal&Protect (T)	22.8 (2.8)b
Variolink II	Syntac	Aqua-Prep F (F)	29.6 (7.8)c
Variolink II	Syntac	Control (C)	21.2 (2.3)b

The same letters indicate no significant differences (two-way ANOVA, Bonferroni, $p<0.05$)

Results

The mean shear bond strength (SBS) values (MPa) of the groups are shown in Table 2.

When control groups are compared, V cement gave significantly higher results (21.2±2.3) than that of D cement (15.2±2.3; $p<0.05$). In the D cement system, application of F desensitizer (20.6±2.4) showed significantly higher results ($p<0.05$) than those of T desensitizer (16.1±3.9) and even the DC group (15.2±2.3). Overall, V cement system with its adhesive resin showed significantly higher results than that of D cement ($p<0.05$, Bonferroni). F and T desensitizers did not negatively affect the bond strength results with both D and V cements. Fluoride-containing desensitizing agent applied on dentin surfaces resulted in higher SBS values within each resin cement group.

While the incidence of adhesive failures was more frequent with both T (84%) and F (66%) desensitizers in the D cement group, cohesive failures in the cement (88%) were more commonly observed with F desensitizer in the V group (Table 3, Fig. 1a–c). V cement in particular showed more cohesive failures in the cement, and D cement showed more adhesive failures between the dentin and the resin cement.

Discussion

Shear test measurements have been reported as the most prevalent in the literature because of the simplicity and

Table 3 Number of specimens and percentage (%) of failure types

Resin cement	Adhesive	Desensitizer	Adhesive (%)	Cohesive (%)	Mixed (%)
Duolink	One-Step Plus	Seal&Protect (T)	84	0	16
Duolink	One-Step Plus	Aqua-Prep F (F)	66	34	0
Duolink	One-Step Plus	Control (C)	60	40	0
Variolink II	Syntac	Seal&Protect (T)	18	48	34
Variolink II	Syntac	Aqua-Prep F (F)	0	88	12
Variolink II	Syntac	Control (C)	36	42	22

Adhesive failure between dentin and resin cement, *Cohesive* cohesive failure of the cement, *Mixed* combination of adhesive and cohesive

rapid data retrieval by this method [1, 18, 19]. However, the stress distribution in such tests can be complex [20]. The main problem in SBS tests is the standardization of the test method. Therefore, the method employed in this study was based on the ISO 11405 norm [18]. To improve the usefulness of this in vitro test, an effort must be made to standardize SBS test methods. Some important aspects should be considered, such as storage conditions, type of substrate human or bovine tooth, tooth age, dentinal depth specimen preparation, rate of load application, presence of thermal cycling, film thickness, and cross-sectional surface area [21, 22]. In order to mimic the clinical situation as much as possible, ceramics were bonded to the dentin surfaces. The SBS of IPS Empress 2 on chlorhexidine-gluconate- (Consepsis) and benzalkonium-chloride-based (Tubulicid Red) disinfectant applied on dentin surfaces have been previously compared where V and ResiLute luting cements were used [23]. It has been reported that the chlorhexidine-gluconate-based disinfectant did not adversely affect the SBS to dentin after thermocycling (×500). The bond strengths of IPS Empress 2 to dentin with V were 20.5±5 (control), 24.2±3 (Consepsis), and 26.9±5 (Tubulicid Red) and with ResiLute 15.0±4 (control), 17.1±2 (Consepsis), and 20.9±4 (Tubulicid Red) [23]. In another study [24], progressive decrease in SBS after NaOCl application was observed. The results of our study indicated that the SBS of fluoride-based disinfectant groups were higher than triclosan-based desensitizing groups. Also, thermocycling did not effect the changes of SBS among groups when compared with the SBS values of the above-mentioned previous studies.

Earlier studies showed that antibacterial agents such as chlorhexidine gluconate and benzalkonium chloride that also contain fluoride had no adverse effect on bond strength of resin to dentin [24, 25]. The results of the present study also revealed that the fluoride-containing desensitizing agent had no adverse effect on the bond strength of the luting systems tested. Similar to the use of water or water-based adhesives, HEMA-containing hydrophilic monomers help to rehydrate the collapsed collagen matrix caused by air-drying [12, 26, 27]. This facilitates subsequent resin infiltration into the interfibrillar spaces of demineralized dentin [19]. Addition of HEMA to water lowers the vapor pressure of water and prevents water from evaporating prematurely during rehydration of the collapsed dentin matrix [28]. Therefore, the high SBS values obtained with the fluoride-containing desensitizing agent in the present study might be attributed to HEMA-induced rehydration mechanism allowing time for the penetration of the primer into dentin. SEM images also supported these high SBS values, namely dentinal tubules were adequately sealed with the resin. These results were in accordance with the results of a study by Pashley et al. [29]. The advised use of triclosan-based desensitizer is to relieve exposed hypersensitive root surfaces, and its recommended application requires light activation after two coats. In our study, this desensitizer was applied with one coat according to the manufacturer's instructions on prepared dentin surfaces. However, the resultant lower SBS values with triclosan desensitizer followed by single-step adhesive system combination, also supported by SEM images, demonstrated poor resin tag formation. This might be attributed to the single-step adhesive use.

It has been shown that fluoride ions penetrating into the dentin enhance mineralization of the dentin [30]. Fluoride treatment to sound dentin was demonstrated to decrease the bond strength of composite to the dentin [31–33]. However, it was reported that fluoride application to demineralized dentin might increase resin–dentin bond strengths by improving the mechanical properties of the dentin [34–44]. In the present study, the two resin cements and desensitizing agents applied on dentin surfaces resulted in higher SBS values with fluoride treatment irrespective of the resin cement used.

The application sequence of the disinfectant is also an important factor to be considered. While some clinicians prefer to apply disinfectants after tooth preparation, prior to the bonding procedure [13, 21, 37], others prefer to apply disinfectants after etching [24, 38, 39, 41, 45]. In this study, disinfectants were applied after etching procedures. The manufacturers recommend the use of F and T after etching. The use of dentin disinfectants would be more preferable after etching the dentin, as removal of the smear layer leads to the elimination of most microorganisms. Then, the use of disinfectants would be more beneficial for those microorganisms and their toxins that remain viable in the dentinal tubules [19, 23].

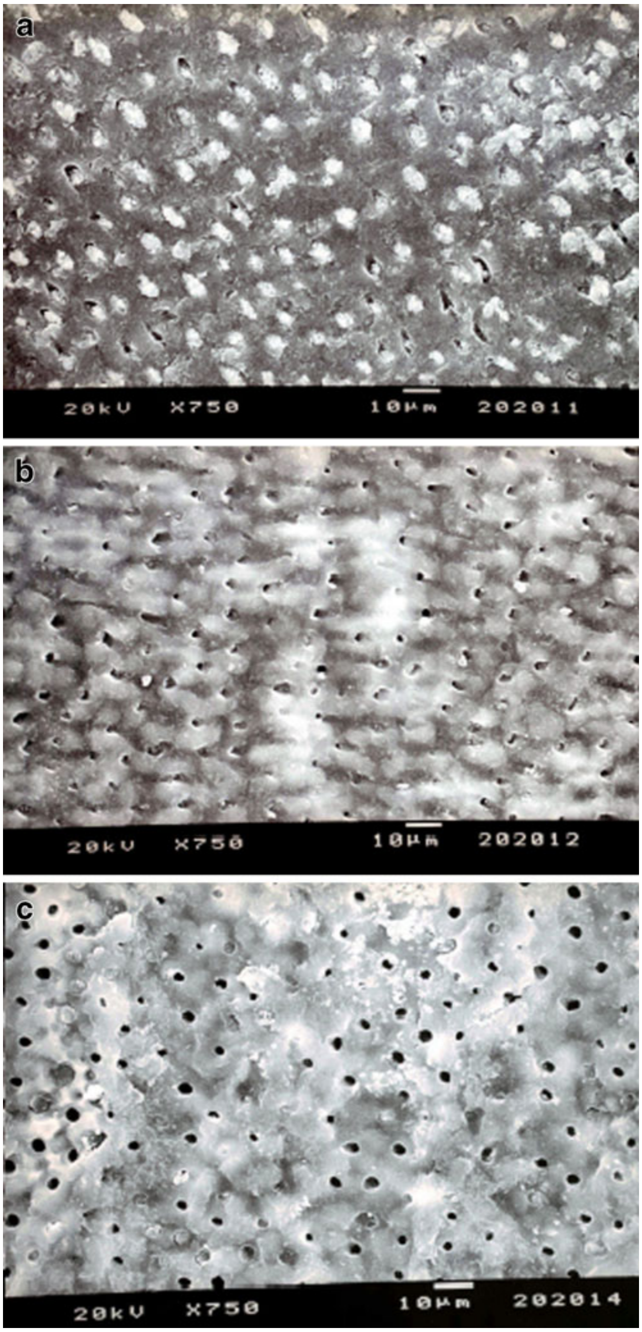

Fig. 1 **a**–**c** Representative SEM images (×750) of the typical failure types: **a** cohesive failure within the resin tags in V–F or T group. Note that the majority of the dentinal tubules were sealed with the resin tags; **b** adhesive failure in the D–F group. Note the partially sealed dentinal tubules with limited resin penetration; **c** adhesive failure in the D–T group. The opened dentinal tubules were not sealed with resin resulting in poor or no resin tag formation

This study did not primarily intend to compare the SBS of the two resin cements. However, it was interesting to note that the adhesive performances of these resin cements were irrespective of the use of a desensitizing agent. This may be due to the differences in chemical compositions of the resin cements. The overall higher SBS results for the V group might be related to the use of a three-step adhesive. One of the adhesives used in our study, Syntac, is a three-step (etching, priming, and bonding) dentin bonding agent. The other adhesive system, One-Step Plus, is a single-step bonding system. Single-step systems combine the primer and adhesives in one solution to be applied after enamel and dentin are etched simultaneously [29, 46]. In this system, the dentin surface should remain in a moist state to prevent collapse of the unsupported collagen and promote primer–resin infiltration [43].

Syntac adhesive system, on the other hand, is water-based, while One-Step Plus is acetone-based. Reis and others [43] have demonstrated that the moisture degree of dentin was effective on the bond strength of luting systems. By monitoring the amount of water used to rewet air-dried dentin surface, they have showed that total-etch adhesive systems achieve optimal bond strengths at different moisture degrees that usually depend on the solvent present in each system. Their data, confirmed in a quantitative manner, indicated that water-based systems require a rather drier dentin surface while acetone-based systems require a rather wetter dentin surface for improved bond strengths. The amount of dentin surface moisture was not assessed in the present study. However, the higher SBS values obtained in favor of water-based adhesive system might have resulted from this phenomenon. These findings support the findings of a previous study where adhesive system was more important than the disinfectant used [23]. Clinical studies are warranted to support this finding.

The failure types were observed mainly as adhesive in acetone-based adhesive system while they were mainly cohesive in water-based adhesive system. This finding confirms the above statements regarding the ease of penetration of resin into rehydrated dentinal matrix. The SBS values were higher for the water-based adhesive system in the control group, regardless of the desensitizing agent used, which might mean that the bond strength was not affected by the desensitizing agent alone, and the water-based systems performed better in terms of SBS. In addition, based on the increased SBS values with D resin cement following fluoride-containing desensitizer use, this desensitizer might be recommended for acetone-based adhesives in clinical practice where both improved adhesion and desensitizing effects are expected.

Although this study was performed under in vitro conditions, the results give insight into probable clinical behavior and provide guidance for clinical trials. In clinical applications, desensitizers are applied either immediately after tooth preparation prior to temporization or before definitive cementation of the restoration. This study investigated the application before definitive cementation. However, the effect of temporary cement might interfere with the tubule obliteration which might also affect the bond strength of the cement to dentin.

This aspect and the detection of penetration depths of the desensitizing agent types as well as their long-term degradation behavior require further research.

Conclusions

From this study, the following could be concluded:

1. Desensitizing agents used in the present study did not adversely affect the bond strengths of the resin cements tested.
2. Both fluoride- and triclosan-based desensitizers can be safely used prior to final cementation but fluoride-containing F in combination with V seems to be more reliable considering both the bond strength and the failure types.

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