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Effect of dentin pretreatment on shear bond strength of three resin-based luting cements

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Purpose
The aims of this study were; to compare the shear bond strength values of resin-based luting cements using etch-and-rinse, self-etching or self-adhesive techniques and to evaluate the effects of pretreatment with 0.2%, and 2% chlorhexidine (CHX) solutions on the bonding stability to dentin.

Materials and methods
Ninety specimens were divided into 9 groups of equal sample size (0.2% CHX, 2% CHX and no CHX application groups). Variolink N (multi-step etch-and-rinse technique), Panavia F2.0 (self-etching technique), or RelyX U200 (self-adhesive technique) resin-based luting cement was applied. All specimens were subjected to shear bond strength test (SBS) after bonding procedure. The mode of failure was analyzed by using a stereomicroscope.

Results
There were no significant differences among study groups for the dentin treatment factor. However, for luting cement factor, significant differences were found and Variolink N showed the highest SBS values.

Conclusion
CHX application has no immediate effect on the SBS values of any resin cements. Despite the development of simplified cementation techniques, etch-and-rinse technique is still the most reliable technique because of its high bond strength to dentin.

Keywords: *RelyX U200; Panavia-F 2.0; Variolink; chlorhexidine; shear strength*

Introduction

Chlorhexidine digluconate (CHX) is one of the most commonly used antimicrobial agent in dentistry and is commercially available as mouthwash, irrigation solution, gel, spray, and aerosol formulations (1). Previous studies (2-6) have shown that CHX demonstrates anti-microbial activity, substantivity, biocompatibility and it also inhibits proteolytic enzymes referred to as metalloproteinases (MMPs) and cathepsins (CTs). These enzymes are responsible for the degradation of bonding interface and can compromise the longevity of the luting cement (7-9). Degradation retarding effects of CHX on the bonding interface have been previously examined by different authors. Kul *et al.* (5) reported the efficacy of different irrigation solutions on the bond strength of a fiber post attached with a self-adhesive resin cement and authors found no difference among the CHX, phosphoric acid, and distilled water groups. In addition, CHX activity on the bonding process of root dentin on different luting agents has been widely examined. De Araújo *et al.* (7) investigated the influence of 2% CHX solution on the bond strength of glass fiber posts to root dentin

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Table 1. *Commercial brands, compositions and manufacturers of luting cements and chlorhexidine digluconate (CHX) solutions used in this study*

Material	Composition	Manufacturer
Panavia F 2.0	ED Primer II: liquid A: 10- methacryloloxydecyl dihydrogenphosphate, 2-hydroxyethyl methacrylate, N,N-diethanol-p-toluidine, N-methacryloyl 5-aminosalicylic acid, water; liquid B: N,N-diethanol-p-toluidine; sodium benzen sulphinate, N-methacryloyl 5-amino-salicylic acid, water. Panavia F: paste A: silanated barium glass, colloidal silica, bisphenol A polyethoxy dimethacrylate, 10- methacryloloxydecyl dihydrogenphosphate, hydrophilic dimethacrylate, hydrophobic dimethacrylate, benzoil peroxide, dl- camphoroquinone; paste B: silanated barium glass, silanated titanium oxide, sodium fluoride colloidal silica, bisphenol A polyethoxy dimethacrylate, hydrophilic dimethacrylate, hydrophobic dimethacrylate, N,N-diethanol-p-toluidine, sodium	Kuraray, Osaka, Japan
Variolink N	Monomer matrix: bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate; inorganic fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, spheroid mixed oxide, initiators, stabilizers, pigments. Syntac primer: triethyleneglycol methacrylate, polyethyleneglycol dimethacrylate, maleic acid, ketone; syntac adhesive: polyethyleneglycol dimethacrylate, glutaraldehyde. Heliobond: bis-GMA, triethyleneglycol dimethatcrylate, stabilizers, initiators.	Ivoclar Vivadent, Schaan, Liechtenstein
RelyX U200	Base paste: glass powder treated with silane, 2-propenoic acid, 2-methyl 1,1′-(1-[hydroxymetil]-1,2-ethanodlyl) ester dimethacrylate, triethylene glycol dimethacrylate (TEGDMA), silica treated silane, glass fiber, sodium persulfate, per-3,5,5-trimethyl hexanoate t-butyl. Catalyst paste: glass powder treated with silane, substitute dimethacrylate, silica-treated silane, sodium p-toluenesulfonate, 1-benzyl-5-phenyl-acid barium, calcium, 1,12-dodecane dimethacrylate, calcium hydroxide, titanium dioxide.	3M ESPE, Seefeld, Germany
Klorhex (0.2% CHX)	0.2% Chlorhexidine digluconate	Drogsan Pharmaceuticals, Ankara, Turkey
Cavity Cleanser (2% CHX)	2% Chlorhexidine digluconate	Bisco Inc., Schaumburg, IL, USA

using two luting cements. Authors reported that CHX did not improve the bond strength of any luting cement.

Post and core restorations are mainly luted to the root dentin with adhesive resin-based luting agents to provide a reliable bonding to the tooth structure. Furthermore, resin-based luting cements have popularized all-ceramic systems because of their bond strength and for increasing the fracture resistance properties of ceramics (10). Various studies investigated CHX activity on bond strength of resin-based luting cements to root dentin. However, the influence of CHX on the bond strength between self-adhesive luting cements and dentin has not been clearly examined.

The aims of this study were twofold. First, to investigate the influence of different concentrations of CHX on the bond strength of luting cements to prepared teeth using three resin-based luting cements, two different dual-cured composite resin cements and a self-adhesive dual-cured resin cement, and second, to compare the shear bond strength of these luting cements to dentin specimens. The null hypothesis tested in this study were; there are no differences in bond strength according to luting cement and the use of different concentrations of CHX has no effect on the luting cements' shear bond strength.

Materials and methods

This *in vitro* study involved the analysis of two main factors: type of resin-based luting cement (three types); and dentin pre-treatment using different CHX concentrations (0.2% and 2%) (Table 1).

Specimen preparation

Ninety mandibular third molar teeth (N=90) were collected, cleaned and stored in 0.5% Chloramine-T (9.0 g sodium chloride and 5.0 g chloramine–trihydrate dissolved in 1000 mL distilled water) solution to prevent dehydration at 4°C for a maximum of 1 month until use. Inclusion criteria were the absence of caries and/or restorations. After cleaning and drying, teeth were embedded in autopolymerizing acrylic resin (Simplex Rapid, KemDent, Wiltshire, UK). Specimens were wet-ground flat with silicon carbide abrasive papers ending with 600 grit (11) to obtain flat dentin surface at 1 to 2 mm distance from the pulp, which was measured by using a digital caliper. For each type of resin-based cement and concentration of CHX, the specimens were randomly divided into nine groups of ten specimens each (n=10) (0.2% CHX, 2% CHX and no CHX groups for each of the three resin-based luting cements) (Table 2).

Ninety disk shaped specimens, 4 mm in diameter and 2 mm in height, were produced with IPS e.max Press lithium disilicate glass ceramic system (IPSe.maxPress, Ivoclar Vivadent, Schaan, Liechtenstein). IPS e.max Press ingots (MO1 shade, IPSe.maxPress, Ivoclar Vivadent, Schaan, Liechtenstein) were heat pressed with the lost wax/heat pressed technique according to the manufacturer's instructions. Ceramic disks were then allowed to bench cool at room temperature and divested by 50-μm Al_2O_3 at 0.2 MPa pressure from a distance of 10 mm. Ceramic disks were ultrasonically cleaned (Invex-Liquid, IvoclarVivadent, Schaan, Liechtenstein) for 10 minutes to remove the reaction layer and polished with 600 grit silicon carbide paper under water-cooling to adjust final thickness

***Table 2.** Group descriptions stratified by dentin pre-treatment solutions and luting cements*

Luting cement	Dentin pre-treatment with CHX	Group description
Panavia F 2.0	0.2% CHX	GR1
	2% CHX	GR2
	-	GR3
Variolink N	0.2% CHX	GR4
	2% CHX	GR5
	-	GR6
RelyX U200	0.2% CHX	GR7
	2% CHX	GR8
	-	GR9

CHX: chlorhexidine digluconate

***Figure 1.** All specimens prepared and embedded in autopolymerizing acrylic resin.*

***Figure 2.** All specimens were stabilized and shear bond strength tests were performed in an universal testinh machine.*

and to standardize surface roughness. All ceramic disk surfaces were acid-etched for 20 seconds with hydrofluoric acid (HF) in less than 5% concentration (IPS Ceramic Kit Etching Gel, Ivoclar Vivadent, Schaan, Lichtenstein), rinsed thoroughly under running water for 60 seconds and dried for pre-cementation surface treatment (Figure 1).

Bonding procedure

Three resin-based luting cements (Panavia F2.0/ Kuraray, Osaka, Japan; Variolink N/ Ivoclar Vivadent, Schaan, Liechtenstein; RelyX U200/ 3M ESPE, Seefeld, Germany) were used in this study. All ceramic disks received pre-treatment in the bonding areas, according to the luting cement, study group, and manufacturer's instructions (Table 3). After pre-treatment, bonding areas were isolated by adhesive tape with a 4 mm in diameter circular hole to prevent excess flash adhering to the specimens. Finger pressure was used for cementing all specimens with an approximate thickness of 5 µm. Dentin-cement-ceramic specimens were left in the air for polymerization of the cement according to the manufacturer's instructions. Before shear bond strength test (SBS), all specimens were stored in distilled water for 24 hours at 37°C.

Shear bond strength (SBS) test

All specimens were mounted to a universal testing machine (Autograph AG-IS Series, Shimadzu, Japan) and SBS tests were performed at a crosshead speed of 1mm per minute until fracture occurred. Bond strength was recorded in Newtons (N) and converted into Megapascals (MPa). Average shear bond strength (MPa) was calculated by dividing the load (N) at which failure occurred by the bonding area (mm^2) (Figure 2).

Fracture types

Bond failure sites and fracture analysis on all specimens were performed visually with a stereomicroscope (OlympusSZ61, Olympus Optical Co., Tokyo, Japan) at 40x magnification. Fractures were classified into one of the three categories as: adhesive failure (if the complete fracture was seen at the luting-dentin interface) or cohesive failure (if the cohesive fracture was seen in the luting cement) or mixed failure (if the adhesive fracture was seen at the resin-based luting cement-dentin interface combined with cohesive fracture in the luting material).

Ethics committee approval and informed consent were not considered to be necessary.

Statistical analysis

Statistical Package for Social Sciences (SPSS) software version 15.0 (SPSS Inc.; Chicago, IL, USA) was used for statistical analysis. The Kolmogorov–Smirnov test was used to determine whether the distribution characteristics of the data meet the requirements of normality assumptions. Levene's test was employed to check the homogeneity of variances. As the data is normally distributed and the variances are homogenous, two-way analysis of variance (ANOVA) and post-hoc Tukey's Honestly Significant Difference (HSD) tests were used for multiple and pairwise comparisons, respectively. Confidence interval was set to 95% and p values less than 0.05 were considered as statistically significant.

Table 3. *Details of surface treatments and luting protocols used in this study*

Luting cement	Dentin pre-treatment	Ceramic surface pre-treatment	Protocol
Panavia F 2.0	0.2% CHX was applied with microbrush for 1 min and dried with absorbent paper; ED primer II: drop each of liquid A and liquid B mixed, 30 s, dried with gentle air flow.(GR1) 2% CHX solution was applied by lightly scrubbing with a microbrush for 5 s and surface was rinsed with distilled water for 2 s; ED primer II was applied according to previous description.(GR2)	Acid-etched for 20 s with <5% HF acid (IPS Ceramic Kit Etching Gel, Ivoclar Vivadent, Schaan, Lichtenstein), rinsed with water for 60 s and dried. CCP (Kuraray, Osaka, Japan) was applied and left to dry for 5 min.ED primer II was applied according to previous description.(GR3)	Mixed paste A + B (1:1) for 20 s. Oxyguard II (Kuraray, Osaka, Japan) was applied and light cured for 20 s. Oxyguard II was removed after 3 min.
Variolink N	Total etch for 15 s, rinsed with water, gently air-dried; 0.2% CHX was applied according to previous description; Syntac primer for 15 s, Syntac adhesive for 10 s; Heliobond blown to a thin layer, light cured for 20 s.(GR4) Total etch for 15 s, rinsed with water, gently air-dried; 2% CHX solution was applied according to previous description; Syntac primer for 15 s, Syntac adhesive for 10 s; Heliobond blown to a thin layer, light cured for 20 s.(GR5) Total etch for 15 s, rinsed with water, gently air-dried; Syntac primer for 15 s, Syntac adhesive for 10 s; Heliobond blown to a thin layer, light cured for 20 s.(GR6)	Acid-etched for 20 s with <5% HF acid (IPS Ceramic Kit Etching Gel, Ivoclar Vivadent, Schaan, Lichtenstein), rinsed with water for 60 s and dried. Monobond S (Ivoclar Vivadent, Schaan, Liechtenstein) was applied for 60 s and dried with air.	Transparent base paste and high viscosity transparent catalyst paste was mixed (1:1). Liquid strip (Ivoclar Vivadent, Schaan, Liechtenstein) was applied and light cured for 40 s.
RelyX U200	0.2% CHX was applied according to previous description.(GR7) 2% CHX solution was applied according to previous description. (GR8) No dentin pre-treatment.(GR9)	Acid-etched for 20 s with <5% HF acid (IPS Ceramic Kit Etching Gel, Ivoclar Vivadent, Schaan, Lichtenstein), rinsed with water for 60 s and dried. RelyX Ceramic Primer (3M/Espe, St. Paul, MN, USA) was applied for 5 s and dried with air.	Base and catalyst paste was applied through an automix syringe and light cured for 40 s.

CHX: chlorhexidine digluconate

Results

Shear bond strength to dentin

The results of the two-way ANOVA of SBS data are presented in Table 4. Results of two-way ANOVA indicated that the type of luting cement affected the bond strength values ($p<0.05$). Tukey's HSD test (Table 5) showed that there were significant differences between Variolink N and the other luting cement groups. Variolink N exhibited significantly higher SBS value than Panavia F2.0 ($p=0.021$) and RelyX U200 ($p=0.031$). There was no significant difference between the SBS values of Panavia F2.0 and RelyX U200. As presented in Table 5, dentin pre-treatment with any of the two CHX solutions demonstrated no significant difference in the SBS values.

Fracture types

Figure 3 presents the distribution of the various fracture types in percentage. Majority of the fractures occurred during the SBS tests were adhesive type failures (56.67%). Adhesive failures were associated with the lowest SBS values while cohesive failures were associated with greater SBS values. 40% cohesive and 70% adhesive failure rates were found in GR6 GR2 groups, respectively.

Table 4. Two-way analysis of variance test results for luting cement and dentin pre-treatment with chlorhexidine digluconate (CHX)

Source	Sum of Squares	df	Mean squares	F	p
Luting cement	116.573	2	58.286	3.304	0.042
Dentin pre-treatment with CHX	13.805	2	6.903	0.391	0.677
Luting cement*dentin pre-treatment with CHX	22.883	4	5.721	0.324	0.861

Statistically significant p values are written in bold

Table 5. Pairwise comparisons of the study groups

Factor	Luting system or pre-treatment	p
Luting cement	Panavia F2.0 / Variolink N	0.021
	Panavia F2.0 / RelyX U200	0.873
	Variolink N / RelyX U200	0.031
Dentin pre-treatment with CHX	0.2% CHX / 2% CHX	0.966
	0.2% CHX / No dentin pre-treatment with CHX	0.82
	2% CHX / No dentin pre-treatment with CHX	0.672

CHX: chlorhexidine digluconate
Statistically significant p values are written in bold

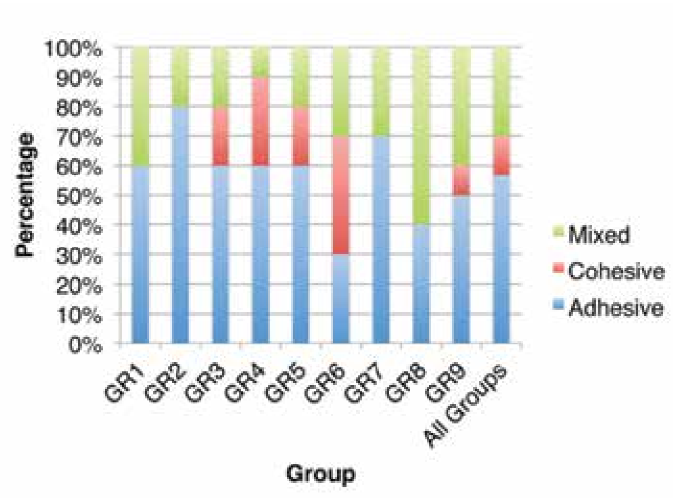

***Figure 3.** Percentage distribution of the failure types in study groups.*

Discussion

This study investigated the shear bond strength of three resin-based luting cements to dentin with and without prior CHX application. Because there were statistically significant differences in bond strength according to luting cement in dentin, the first null hypothesis was rejected. In the present experimental settings, three resin-based luting cements with different dentin pre-treatment protocols in terms of the number of steps of adhesive application were used according to the manufacturer's instructions. Variolink N luting cement was used with multi-step etch-and-rinse technique including etching and rinsing, application of primer and bonding agent. Panavia F2.0 luting cement was used with self-etching technique in which application of self-etching primer and application of adhesive paste applied step by step. RelyX U200 luting cement system uses a self-adhesive application technique and no dentin pre-treatment is required according to the manufacturer's claim.

When compared to that of RelyX U200 Variolink N revealed significantly higher bond strength to dentin (p=0.031). This can be explained by the removal of the smear layer and dissolving of the mineral during the rinsing step of etch-and-rinse technique. According to the manufacturer, RelyX U200 luting cement consists of methacrylate monomers modified with phosphoric acid that can mineralize the dentin, and cement infiltrates the hybrid layer with resin tags, thus, no prior removal of the modified smear layer is needed. In contrast, it was reported that adhesive resin composites which contain phosphoric acid, have very low pH and these systems appear not to have a chemical affinity for bonding to the dentin (12, 13). In addition, this poor micromechanical infiltration for attachment to the dentin can be explained by inadequate demineralization on dentin layer and weak formation of the hybrid layer (14).

Variolink N also revealed significantly higher bond strength to dentin, compared to that of Panavia F2.0 (p=0.021), which is in accordance with the results of previous studies (15-18). This may be explained by, firstly, the high filler content and viscosity of the Panavia F2.0 luting cement, which may decrease infiltration depth of the adhesive into the primed dentin (18, 19). Secondly, water diffusion may occur from the dentin across the ED Primer during the slow polymerization in the dual cured mode and water droplets along the primer-cement interface may affect adhesive permeability (20), and finally, the residual acids of ED primer may impede the chemical curing of the luting cement (21).The results of the present study showed that RelyX U200 specimens' bond strength to dentin was not statistically different from those of Panavia F2.0. These results are in agreement with previous literature. Using the microtensile bond strength (µTBS) test of simplified resin-based luting cements, Bacchi *et al.* (22) observed no statistically significant difference and the self-etching primer along with a conventional dual-curing cement (ED Primer+Panavia F2.0) led to µTBS similar to that of the self-adhesive resin cement (RelyX U200).

The results of the present study showed that dentin pre-treatment with CHX did not significantly affect the bond strength to dentin in any group; therefore, the second null hypothesis was accepted. CHX is a non-specific MMP inhibitor and it has shown success in inhibiting both MMPs and cysteine cathepsin; thus preserving the integrity of the hybrid layer (23). To improve bond strength, CHX can be applied to dentin as a primer after phosphoric acid-etching for rehydration (24, 25), be incorporated in the acid etchant (26, 27), or with CHX-containing dental adhesives (28, 29). However, the effectiveness of using CHX as a primer after phosphoric acid application on bond strength in previous studies is controversial. The aforementioned studies reported that CHX used as a therapeutic primer on acid-etched dentin does not interfere with immediate bond durability and significantly higher bond strengths were observed after only 6 to 12 months (24, 25). A recent study controversially reported that the application of the 2% CHX as dentin pretreatment decreased the number of adhesive failures compared with untreated controls after 9 months of aging, however, this effect was not significant and was seen on the etch-and-rinse adhesive but not on the self-etching adhesive (30). In contrast, Ricci *et al.* (31) found that 2% CHX application significantly increased the µTBS values of adhesives to the acid-etched dentin and positive immediate efficacy on bond durability.

The bond strength test results of the present study confirmed that different concentrations of CHX used as a therapeutic primer in etch-and-rinse adhesive, primer self-etching adhesive and self-adhesive groups have no positive or negative immediate effect on bond strength, which is mostly in accordance with the literature. On the other hand, authors show, for the first time, that RelyX U200, a self-adhesive luting cement that consists of methacrylate monomers modified with phosphoric acid, produced similar improvement in SBS values when applied to CHX-treated dentin. Although no negative or positive effects of 0.2% or 2% CHX solutions were observed, further studies are needed to ascertain the time-dependent efficacy of CHX on dentin bond strength.

Conclusion

Within the limitations of this *in vitro* study, the etch-and-rinse technique shows highest bond strength to dentin and CHX is not effective on the bond strength of luting cements to dentin. The benefits of CHX application prior to bonding may still be observed after long-term clinical studies, encouraging further clinical investigations in the evaluation of bond strength over longer periods of time. Despite the development of simplified cementation techniques, etch-and-rinse technique seems to be the most reliable one because of high bond strength to dentin.

Ethics Committee Approval: Ethics committee approval was considered not to be necessary.

Informed Consent: There were no participated patients in this study and written informed consent was not obtained.

Peer-review: Externally peer-reviewed.

Author Contributions: NBB and GE designed the study and generated the data. NBB, GE and BGR gathered and analyzed the data. NBB, GE, BGR and TÇ wrote the majority of the original draft. All authors approved the final version of the paper.

Conflict of Interest: The authors have no conflicts of interest to declare.

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Türkçe öz: *Dentin yüzeyine uygulanan ön işlemlerin üç farklı rezin bazlı yapıştırma simanının makaslama bağlanma dayanım kuvvetlerinin üzerindeki etkileri. Amaç: Bu çalışmanın amaçları; "etch-and-rinse", "self-etch" ya da "self-adeziv" teknikleri kullanılarak, rezin bazlı yapıştırma simanlarının dentine olan makaslama bağlanma dayanım kuvvetlerinin karşılaştırılması ve dentin yüzeyine ön işlem olarak uygulanan %0.2 ve %2'lik klorheksidin solüsyonlarının simantasyon esnasındaki bağlanma stabilitesine olan etkisinin değerlendirilmesidir. Gereç ve Yöntem: 90 adet numune eşit örnek büyüklüğüne sahip 9 gruba ayrılmıştır. (%0.2 klorheksidin grupları, %2 klorheksidin grupları ve ön işlem görmeyen gruplar). Variolink N (çok aşamalı "etch-and-rinse" tekniği), Panavia F2.0 ("self-etch" tekniği), ya da RelyX U200 ("self-adeziv" tekniği) rezin bazlı yapıştırma simanları uygulanmıştır. Tüm örneklere yapıştırma aşamalarından sonra makaslama bağlanma dayanım testi (SBS) uygulanmıştır. Kırılma tipleri stereomikroskop kullanılarak analiz edilmiştir. Bulgular: Dentin yüzeyine işlemine göre değerlendirildiğinde, grupların kırılma dirençleri arasında anlamlı bir fark bulunamamıştır. Kullanılan simana göre değerlendirildiğinde, gruplar arası istatistiksel olarak anlamlı farklılıklar bulunmuştur ve Variolink N en yüksek kırılma değerlerini vermiştir. Sonuç: Dentin yüzeyine uygulanan klorheksidin ajanının kırılma değerlerine erken dönem etkisi bulunmamaktadır. Bununla birlikte "etch-and-rinse" tekniği, güncel teknolojiler ile geliştirilmiş olan basitleştirilmiş simantasyon tekniklerine göre, dentine yüksek bağlanma kapasitesi sebebiyle en güvenilir yöntemdir. Anahtar kelimeler: RelyX U200; Panavia-F 2.0; Variolink; klorheksidin; makaslama kuvveti*

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