

EFFECTS OF DESENSITIZERS ON RESIN CEMENT BONDING

DENTİN HASSASİYET GİDERİCİ AJANLARIN REZİN SİMANIN BAĞLANMASINA ETKİSİ

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ABSTRACT

Aim: The purpose of this in vitro study was to determine how pretreatment with desensitizers affects micro-tensile bond strength between dentin and self-etch resin cement (SEC).

Material and Methods: The occlusal surfaces of extracted teeth were abraded with a diamond bur to the dentin layer and divided into 5 equal groups (n = 20); G1- control (no desensitizer, only Clearfil aesthetic resin cement (CEC); G2-Gluma and CEC; G3-Bi-fluorid 12 and CEC; G4-FGM desensitizer and CEC; G5- D/Sense Crystal and CEC. Resin cement layers were added to a depth of 8 mm in the control group, and desensitizer was applied to the surfaces before resin cement application in the other experimental groups. A specimen from each group was examined with scanning electron microscopy. The specimens were sectioned to be 1 mm² perpendicular to the adhesive layer, and the microtensile test was applied. According to the results of the post hoc Tamhane test, there was a significant difference. (p<0.05).

Results: Only the Gluma desensitizer had a significantly lower bond strength value when compared with the other desensitizers and the control group.

Conclusions: While dentin with oxalate, fluoride, and strontium chloride-potassium nitrate contamination did not affect the bonding strength of SEC, dentin contamination with glutaraldehyde/HEMA had a negative effect. **Keywords.** Desensitizer; self-etch resin cement; microtensile bond strength.

ÖΖ

Amaç: Bu in vitro çalışmanın amacı, dentine hassasiyet giderici ajan uygulamasının, self-etch rezin simanın (SEC) mikrogerilim bağlanma dayanımını nasıl etkilediğini araştırmaktır.

Gereç ve Yöntem: Çekilmiş dişlerin oklüzal yüzleri elmas frez ile dentin tabakasına kadar aşındırıldı ve 5 eşit gruba ayrıldı (n = 20); G1- kontrol (hassasiyet giderici ajan kullanılmadı, sadece Clearfil estetik rezin siman (CEC); G2-Gluma ve CEC; G3-Bi-fluorid 12 ve CEC; G4-FGM ve CEC; G5-D/Sense Crystal ve CEC. Kontrol grubunda, dentin üzerine 8 mm yüksekliğinde rezin siman tabakası eklendi ve diğer deney gruplarına rezin simandan önce dentin hassasiyet giderici ajanlar uygulandı. Her gruptan bir örnek taramalı elektron mikroskobu ile incelendi. Örnekler dentin-siman bağlantısına dik olarak 1 mm² olacak şekilde kesildi ve mikrogerilim bağlanma dayanımı ölçüldü. Post-hoc Tamhane testinin sonuçlarına göre anlamlı bir fark vardı (p <0.05).

Bulgular: Dentin hassasiyet giderici ajanlar arasında sadece Gluma, diğer gruplar ve kontrol grubu ile karşılaştırıldığında anlamlı şekilde daha düşük bir bağlanma dayanımına sahipti.

Sonuç: Dentin üzerine oksalat, florür ve stronsiyum klorür-potasyum nitrat uygulaması, SEC ile arasındaki bağlanma dayanımını etkilemese de, glutaraldehit/HEMA uygulaması olumsuz etkilemiştir.

Anahtar kelimeler: Hassasiyet giderici ajan; Self-etch rezin siman; Mikrogerilim bağlanma testi

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INTRODUCTION

Dentin sensitivity after tooth preparation is a concern in clinical practice¹ and can be controlled by reducing the permeability of the dentin tubules.² Products available for such treatment include those incorporating fluoride,²⁻⁴ hydroxyethyl methacrylate (HEMA)/glutaraldehyde, 5-8 oxalate, 9-12 potassium nitrate, and calcium phosphate,¹³ of which HEMA/ glutaraldehyde is the most widely used.⁶ Glutaraldehyde works by coagulating proteins in the dentin tubules, and HEMA by forming resinous tags that occlude the dentinal tubules.14 Desensitizing agents (DAs) and their effects on the bond between dentin and resin cement have been studied, with some studies reporting that the administration of DAs prior to the cementation of restorations reduces crown retention^{10,15,16}; however, another study reported no adverse effects¹⁴ and even increased retention.

The application of calcium oxalate to dentin¹⁷ blocks the dentinal tubules,18 leading to immediate nerve fiber depolarization by potassium ions and then tubular occlusion from the gradual formation of calcium oxalate crystals.¹⁹ The action of acidic potassium oxalate solutions depends on the chemical interaction of the calcium ions and the dentin fluid caused by the calcium oxalate crystals occluding and/or obstructing the tubules.²⁰ The same applies to fluoride, which produces calcium fluoride crystals in the tubules and reduces the conductivity of dentin.¹⁸ D/Sense Crystal is a cavity liner and 1-step, dualaction gel desensitizer material that reacts with dentin and precipitates calcium oxalate microcrystals and potassium nitrate; these crystals go deep into the tubules, sealing the dentinal surface with an uninterrupted, acid-resistant complex.

Desensibilize (FGM Produtos Odontológicos) is a neutral viscous solution that combines 2 mechanisms for desensitization: occlusion of the dentinal tubules by strontium chloride and a neural action by potassium nitrate. The authors are unaware of studies that have investigated whether products of this type reduce the permeability of dentin and therefore affect the bonding of resin cement.

The general purpose of applying agents to block the dentin tubules is to reduce the permeability of the dentin.^{21,22} However, the effects of desensitizer treatment on bonding are not fully known. An oxalatecontaining DA has been reported to weaken resin infiltration due to precipitation onto the dentin layer as calcium oxalate crystals.^{20,23} However, it has been reported^{24,11} that potassium oxalate crystals that form a more stable and more homogeneous hybrid layer do not reduce the bonding between resin and dentin.

Whether the application of desensitizers containing different types of agents on the dentin surface affects the bonding between dentin and SEC is unclear. The null hypothesis of the study was that no variation in bonding would be found among the adhesive systems, regardless of the desensitizing agent applied. When using self-etch resin cement for cementation, it is preferable to use oxalate, fluoride, and strontium chloride-potassium nitrate when it is necessary to use desensitizing agents.

MATERIAL AND METHODS

The products and the methods used in this study are presented in Table 1. In the control group, the dentin surface was untreated. The Scientific Ethics Committee of Atatürk University approved the study protocol with the 8/2018 protocol, and a total of 30 extracted third molar teeth were collected. The teeth were ground with a diamond bur (KG Sorensen) under water so that the dentin surfaces were exposed. The occlusal surfaces of the teeth were ground with abrasive paper under water for 60 seconds, and the dentin surface was examined with an optical microscope (Olympus SZX7) to ensure that no enamel remained. The specimens were divided into 5 groups and stored in distilled water. The DA was applied according to the manufacturer's instructions (Table 1). Resin cement was applied (Clearfil aesthetic cement, Kuraray) on the dentin surfaces with a layering technique (total of 8 mm of resin cement), and the bonded resin cement was polymerized. The specimens were cut into microbars (10×1×1 mm) with a precision sectioning saw (Isomet 1000, Buehler, Evanston IL, USA), and 20 intact microbars were included in each group for microtensile bond strength (Mtbs) testing. Each specimen was centered using cyanoacrylate adhesive (Akfix 705, Turkey) that the resin-dentin interface was between the jaws of the unit. A crosshead speed of 0.5 mm/min was applied (T 610-10K, Bisco dental), and the load was recorded at specimen fracture in N/mm². The homogeneity of the variances was evaluated by the Levene test, and the difference between the groups was determined using one-way ANOVA (a=.05). The post hoc Tamhane statistical analysis was used to determine the different

groups when the p value was found to be significant, since the variances were not homogenous.

In this in vitro study, the Mtbs between the dentin surfaces on which various dentin desensitizing agents were applied and the self-etch, dual-cure resin cement were investigated.

Desensitizer	Contents	Application protocol	
Gluma	HEMA (25-50%),	After application on	Heraeus
Desensitizer	glutaraldehyde (5-	dried dentin, wait 30-	Germany
	10%), distilled water	60 seconds and gently	,
		dry with air.	
D/Sense	Potassium binoxalate	Apply on tooth	Centrix Inc.
Crystal	2,5%, Nitric acid 2,5%	surface, allow	
-		permeation for 20 s,	
		dry for 30 s, wait for	
		2 m (typically less	
		than 3 microns thick).	
	Sodium fluoride 60	After shaking, thinly	Voco,
Bi-fluoride 12	mg/g (6.0 % w/w),	paint tooth surfaces	Germany
Suspension	Calcium fluoride 60	to be treated. Special	
	mg/g (6.0 % w/w)	foam pellet ("Pele	
		Tim") and suspension	
		is adsorbed within 10-	
		20 seconds and then	
		dried with air.	
ECM	100/ strantium	Apply to tooth wait	Procil
FGM		Apply to tooth, wait	Brasii
Desensibilize	chionue 5% polassium	tooth with bruch	
	nitrate		
		matorial by oithor	
		material by either	
Clearfil	Major componente	Apply primar to	(Kurara)
Ecthotic	within the primer	Apply primer to	
Cement		for 30 seconds and	lanan)
cement	NMSA water	then cently dry with	Japanj
	accelerator The main	air after the excess is	
	components of resin	removed with cotton	
	cement are Bis-GMA	nellet. Then resin	
	TEGDMA hydronhobic	cement is applied	
	aromatic	and each laver is light	
	dimethacrylate	polymerized	
	monomers, silanated	porymenzedi	
	barium glass		
	elephants.		

Table 1. Composition of desensitizers and resin cement

Bis-GMA: bisphenol-A-diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; TEGDMA: Triethyleneglycol dimethacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA, Nmethacryloyl-5-aminosalicylic acid.

ESEM Analysis

Following the application of the DA and the attachment of resin cement, one specimen was selected from each group and cut vertically into 2 parts and abraded with 600 and 1200 grit abrasive papers then the resin-dentin interfaces were analyzed (Fig. 1).



A. Control group; no desensitizer contamination, only resin cement.



B. FGM group; Contamination of dentin with strontium chloride-potassium nitrate before resin cement.



C. Bi-fluoride group; Contamination of dentin with fluoride before resin cement.



D. Gluma group; Contamination of dentin with Glutaraldehyde/HEMA before resin cement.



E. D-sense group; Contamination of dentin with calcium oxalate and potassium nitrate before resin cement. Figure 1.



RESULTS

The mean values of the microtensile bond strength (MPa) obtained with the post hoc Tamhane test are shown in Table 2. The statistical analysis revealed significant differences between the mean values of Gluma and the other desensitizing treatment groups and the control group regarding bond strength. The bond strength of the Gluma group was significantly lower than that of the other experimental and control groups (p < 0.05) The results for the Gluma, D-sense, Bi-fluoride, and Desensibilize experimental groups were similar to those of the control group (p > F.05). Many of the specimens in which a desensitizer agent was applied had similar results to those of the specimens without any applied agent.

Table 2. Results of post hoc Tamhane test

Multiple Comparisons								
Dependent Variable: parameter								
Tamhane								
(I) grup	(J) grup	Mean	Std. Error	Sig.	95% Confidence			
		Difference			Interval			
		(I-J)			Lower	Upper		
					Bound	Bound		
control	gluma	2.63000 [*]	.83150	.040	.0784	5.1816		
	bifluoroid	18000	1.02944	1.000	-3.2419	2.8819		
	fgm	-1.88000	1.09605	.629	-5.1370	1.3770		
	dsense	24000	1.04108	1.000	-3.3355	2.8555		
Gluma	control	-2.63000 [*]	.83150	.040	-5.1816	0784		
	bifluoroid	-2.81000 [*]	.74910	.009	-5.0960	5240		
	fgm	-4.51000 [*]	.83829	.000	-7.0835	-1.9365		
	dsense	-2.87000 [*]	.76502	.009	-5.2073	5327		
bifluoroid	control	.18000	1.02944	1.000	-2.8819	3.2419		
	gluma	2.81000*	.74910	.009	.5240	5.0960		
	fgm	-1.70000	1.03494	.684	-4.7787	1.3787		
	dsense	06000	.97653	1.000	-2.9620	2.8420		
fgm	control	1.88000	1.09605	.629	-1.3770	5.1370		
	gluma	4.51000*	.83829	.000	1.9365	7.0835		
	bifluoroid	1.70000	1.03494	.684	-1.3787	4.7787		
	dsense	1.64000	1.04652	.738	-1.4720	4.7520		
dsense	control	.24000	1.04108	1.000	-2.8555	3.3355		
	gluma	2.87000*	.76502	.009	.5327	5.2073		
	bifluoroid	.06000	.97653	1.000	-2.8420	2.9620		
	fgm	-1.64000	1.04652	.738	-4.7520	1.4720		

An SEM examination of the control group revealed that the tubules were generally open and that only some tubules were obstructed (Figure 1a). FGM (Figure 1b), Bi-fluoride (Figure 1c), and, D-sense (Figure 1e) were treated, and occlusion was clearly observed in some dentin tubules. Resin tags were detected in Gluma (Figure 1d), but this finding was not as consistent in the other groups. Furthermore, as the result of the smooth surface, no evidence of resin penetration was present. A similar microstructure to that shown in Figure 1e was demonstrated in Figure 1b. Because of the thickness of the bifluoride layer, it was not possible to identify some resin tags.

DISCUSSION

The application of desensitizers had no effect on the Mtbs, except for Gluma, which had a reduced mean Mtbs SEC to dentin than the other DAs and the control group. Therefore, the null hypothesis was partly supported.

In a study investigating the effects of DAs on dentin permeability,¹² the agents were classified as those covering the tubules and those leading to the precipitation of proteins and crystals. The agents leading to the precipitation of crystals within the tubules reduced dentin permeability more than the others. While the fluoride-containing agents, used to treat extreme dentin hypersensitivity, precipitated sodium and calcium fluoride crystals³(E) within the dentin tubules, the agents containing resin provided long-lasting sensitivity relief through resin tag formation and the creation of a thin layer of polymerized resin surface covering the dentin tubules.⁴ The HEMA and glutaraldehyde contained in Gluma blocked the dentin tubules by coagulating the dentin fluid proteins.⁷ A limitation of in vitro studies is that the amount of dentin fluid protein in the tested extracted teeth is unknown.

The shear bond strength improved depending on glutaraldehyde/HEMA containing DA applied before the self-adhesive cement compared with the SEC luting process.⁷ Furthermore, in another study,¹⁴ shear bond strength increased after aging; this study also reported that Gluma influenced the bonding of selfadhesive resin cement (SAC) positively even when mastication was simulated.⁵ These results were not consistent with those of the current study where SEC was used instead of SAC. In the present study, pretreatment of the dentin surface with Gluma negatively influenced the Mtbs of the SEC to dentin. The bonding of the resin cement is substantially associated with the HEMA and glutaraldehyde concentration.⁶ Moreover, Qin et al.⁸ reported that the glutaraldehyde in Gluma was not able to cross-link mineralized dentin. Gluma has been reported to inhibit the bond strength of SEC as a result of the coagulation of proteins rather than the blocking of tubules.^{10,15} However, HEMA is hydrophilic and unstable²⁵ and can affect the bonding strength after aging or water storage.¹⁴ In a study examining the

effect of Gluma on dentin bonding after aging, better bonding was obtained with SAC compared with SEC. Therefore, the use of Gluma reduced the bond strength of self-etch resin cement.¹⁴ Additionally, Aranha et al²⁵ reported that Gluma had no effect on bond strength values.

The bonding of conventional resin cement was reduced after Gluma application.¹⁵ Huh et al¹⁰ reported that after Gluma application, the use of selfetching primer negatively affected resin cement bonding. In the current study, the bond strength of the Gluma-treated specimens was reduced compared with that of the controls. This reduction may be related to the use of different adhesives, resin cement types, and primers, methods of surface roughening with acid, and bonding strength tests. Gluma contains HEMA, which may rehydrate the collagen matrix after air-drying. The lower Mtbs²⁶ result for this desensitizer agent when compared with that of other agents is difficult to explain. Sealing of the dentinal tubules by the precipitates and also the funnel-shaped tubules as demonstrated by Arrais et al² may be factors.

Calcium oxalate crystals form as a result of the reaction of the ionized calcium present in the dentinal tubules with potassium oxalate. According to Acar et al,¹ the oxalate desensitizer reduced the Mtbs of SAC to dentin. These crystals cover the dentinal surface, including the tubule orifice; thus, the adhesive resins cannot bond adequately to the oxalate-applied dentin.^{17,20} Pashley et al,²¹ reported that treating etched dentin with potassium oxalate gel created crystals within the tubules, leading to reduced crystal formation on the surface. They also reported that the development of crystals within the tubules did not prevent the development of a hybrid layer. Like the current study, Tay et al,²⁴, reported that Mtbs values were similar to those of the nontreated dentin when oxalates were used. According to Shafiei et al,¹¹ oxalate had no effect on bonding in the etch and rinse resin cement system. Studies using all kinds of DA using recently introduced SECs are lacking.¹

The application of a desensitizer with oxalate has been reported not to affect the bonding between resin cement (Panavia F2.0, SEC) and dentin.¹⁰ A low pH value oxalate application replaces the smear layer with calcium oxide crystals.^{9,10} According to the SEM images, open tubules were detected with oxalate particles that had entered the tubules, enabling the development of resin tags.¹⁰ Kolker et al,¹² reported that some of the tubules appeared to be closed. D-

sense crystal precipitate was found adjacent to the tubular walls, and the tubules apparently extended toward the dentin surface. Although the tubules were obstructed as a result of the development of insoluble crystals on the peritubular dentin, the reason some tubules were open may be related to the development of a hybrid layer. ¹¹

This study was not able to fully replicate the clinical performance of desensitizing agents with resin cement. However, without direct evidence regarding the way that desensitizers react with resin cement, clinicians should avoid their use before cementing indirect restorations. Other limitations of the study include the limited number of DAs tested. Additionally, DAs were applied according to the manufacturer's recommendations, and it may be that thickness can also affect bond strength. Comparing different types of adhesive resin cements in future studies is recommended. Additionally, the evaluation of different situations such as fatigue or aging treatments may affect the outcomes.

CONCLUSIONS

Considering the limitations of this study, using D-Sense, Desensibilize, and Bi-fluoride on dentin can be recommended if used together with a self-etch resin cement when required. Additionally, pretreating dentin with D-Sense, Desensibilize, and Bi-fluoride had no negative effect. However, Gluma may negatively affect bonding with SEC.

NOT: Çalışmada herhangi bir yazar, kurum ya da kuruluş ile çıkar çatışması içerisinde bulunmamaktadır. Makale daha önce hiçbir yerde yayınlanmamış ve yayınlanmak üzere işlem görmemektedir

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