ORIGINAL RESEARCH

The effect of dentin desensitizers on shear bond strengths and vickers surface hardness of self-adhesive resin cement

Dentin hassasiyet gidericilerin self-adesiv resin simanın yüzey sertliğine ve makaslama bağlantı dayanıklılığına etkisi

Assist. Prof. Murat Alkurt

Recep Tayyip Erdoğan University, Faculty of Dentistry, Department of Prosthodontics, Rize

Prof. Zeynep Yeşil Duymuş

Recep Tayyip Erdoğan University, Faculty of Dentistry, Department of Prosthodontics, Rize

Assist. Prof. Mustafa Gündoğdu

Atatürk University, Faculty of Dentistry, Department of Prosthodontics, Erzurum

Dr. Fikret Özgür Coşkun

Recep Tayyip Erdoğan University, Faculty of Dentistry, Department of Prosthodontics, Rize

Dr. Tugay Şişçi

Recep Tayyip Erdoğan University, Faculty of Dentistry, Department of Prosthodontics, Rize

Dr. Mustafa Yıldırım

Recep Tayyip Erdoğan University, Faculty of Dentistry, Department of Prosthodontics, Rize

Received: 22 October 2017

Accepted: 31 January 2018

doi: 10.5505/yeditepe.2018.96720

Corresponding author:

Assist. Prof. Murat Alkurt Recep Tayyip Erdoğan University, Faculty of Dentistry, Department of Prosthodontics 53100 Rize - Türkiye Phone:+90 545 878 81 63 E-mail: muratalkurt@hotmail.com

SUMMARY

Aim: The aim of study is to evaluate the effects of desensitizing agents on the shear bond strengths (SBS) and Vickers surface hardness (HV) of self-adhesive resin cement.

Materials and Methods: One-hundred twenty human molars were sectioned parallel to the occlusal plane, polished and randomly divided into 6 groups: Control (CO); Teethmate (TM); Bifluorid (BF); Gluma (GL); Smartprotect (SP); Bisblock (BB). Self-adhesive cement was applied into cylinder mold (3mm×3mm) (N=10). SBS was measured using a Universal testing machine at a 0.5mm/min. Statistical analysis was performed using one-way ANOVA and Tukey's test (α =0.05). The tooth surface was examined by scanning electron microscopy (SEM). To evaluate HV, self-adhesive cement was applied into cylinder mold (3mm×100µm) (n=10). HV values at the dentin side of cement were evaluated at four times. A load of 200 g was applied for 15 s, and vertical and horizontal indentation diagonals were measured under microscope. All measurements were converted to the Vickers surface hardness (HV) value by using Vickers hardness formula. Data were analyzed by one-way ANOVA and Tukey's tests (α =0.05).

Results: SP group showed the greatest MPa (4.05 ± 1.34 MPa) followed by BB (2.71 ± 1.39 MPa), CO (2.12 ± 0.76 MPa), GL (1.96 ± 1.23 MPa), TM (1.80 ± 0.66 MPa) and BF (1.30 ± 0.57 MPa). BF group showed the lowest HV (11.34 ± 2.24 HV) followed by SP (14.27 ± 3.65 HV), GL (15.58 ± 1.77 HV), BB (16.06 ± 5.73 HV), CO (18.04 ± 3.05 HV), and TM (19.22 ± 2.10 HV). BF desensitizer showed lowest the MPa and HV value. BF desensitizer shows the lowest SBS desensitizer, but this effect is not statically important (p>.05).

Conclusions: SP and BB desensitizers have a positive effect on the SBS of self-adhesive cement. For Vickers surface hardness, BF and SP desensitizers showed statically decrease in surface hardness of resin cement (p<.05).

Keywords: Shear bond strength, densesitizer, surface hardness, self-adhesive resin cement

ÖZET

Amaç: Bu çalışmanın amacı hassasiyet gidericilerin self-adesiv resin simanın yüzey sertliğine ve makaslama bağlanma dayanıklılığına etkisini değerlendirmektir.

Gereç ve Yöntem: 120 adet insan molar dişin oküzal yüzeyine paralel bir kesim işlemi yapıldı, yüzeyinin pürüzlülüğü giderildi ve rastgele 6 gruba [Control (CO); Teethmate (TM); Bifluorid (BF); Gluma (GL); Smartprotect (SP); Bisblock (BB)] ayrıldı. Silindir kalıp (3mm×3mm) içine self-adesiv resin siman uygulandı (n=10). Makaslama bağlanma dayanıklılığı Universal test cihazı yardımıyla (0,5mm/dk) ölçüldü. Makaslama bağlanma dayanıklılığı verilerinin istatistiksel analizi tek yönlü ANOVA ve Tukey's testi kullanılarak yapıldı (α=0,05). Diş yüzeyi taramalı elektron mikroskobu (SEM) yardımıyla değerlendirildi. Yüzey sertliğini ölçmek için self-adhesiv resin siman silindir





kalıp içine (3mm×100µm) uygulandı (n=10). Yüzey sertlik ölçümü resin simanın dentin yüzeyine temas eden yüzeyinden 4'er kez ölçümü yapılarak değerlendirildi. Yüzeye 200 gr kuvvet 15 sn boyunca uygulandı ve yüzeyde oluşan diagonal çentik şeklinin mikroskop altında vertikal ve horizontal köşegen uzunlukları ölçüldü. Elde edilen tüm ölçümler Vickers sertlik formül kullanılarak yüzey sertlik değerine çevrildi. Yüzey sertliği verileri tek yönlü ANOVA ve Tukey's testi yöntemiyle istatistiksel olarak değerlendirildi (α =0,05).

Bulgular: En yüksek makaslama bağlanma dayanıklılığı SP (4,05±1,34 MPa) gurubunda görüldü, bunu BB (2,71±1,39 MPa), CO (2,12±0,76 MPa), GL (1,96±1,23 MPa), TM (1,80±0,66 MPa) ve BF (1,30±0,57 MPa) grubu takip etti. BF grubu en düşük yüzey sertliği değeri (11,34±2,24 HV) gösterdi, bunu SP (14,27±3,65 HV), GL (15,58±1,77 HV), BB (16,06±5,73 HV), CO (18,04±3,05 HV), and TM (19,22±2,10 HV) grubu takip etti. En düşük yüzey sertliği ve makaslama bağlanma dayanıklılığı BF hassasiyet giderici uygulamasında görüldü. Fakat BF hassasiyet gidericinin makaslama bağlanma dayanıklılığı üzerine etkisi istatistiksel olarak anlamlı bulunmadı (p>,05).

Sonuçlar: SP ve BB hassasiyet gidericiler self-adesiv resin simanların makaslama bağlanma dayanıklılığına olumlu etkisi bulunmuştur. Yüzey sertliği açısından değerlendirildiğinde BF ve SP hassasiyet gidericiler resin simanın yüzey sertliğinde istatistiksel olarak azalmaya neden olduğu görülmüştür (p<,05).

Anahtar kelimeler: Makaslama bağlanma dayanımı, hasssiyet giderci, yüzey sertliği, self-adesiv resin siman.

INTRODUCTION

Normal dentin, enamel-dentine junction extending to the wall of the pulp, consists of millions of tubules through the intertubuler region rich in collagen and calcium. Inside the tubules is filled with collagen, liquid and odontoblast extension. There are also rarely non-myelinated pulpal nerves which are approximately 150µ in length towards the dentin-enamel junction.¹

Dentin hypersensitivity, which happens against chemical, thermal and mechanical stimuli; are short and sharp pains caused by open dentin surface without any pathology or dental defect.² The hydrodynamic theory is the most widely accepted theory explaining the transmission of stimuli, which occur by the movement of fluid within the tubules. According to this theory, stimuli cause the dentinal hypersensitive with the effect of stimulation of nerve terminations close to the odontoblastic layer by fluid within the tubules to flow inward or outward.³

Clinically, dentin hyper-sensitivity after crown or cavity preparation is one of the primary challenges.⁴ Nearly 1 to 2 million dentinal tubules exposed during prosthetic

preparation of posterior crown.⁵ Exposed dentinal tubules became sensitive to the external stimuli and so patients experienced discomfort situation.⁶ Also, dentin hyper-sensitivity was observed in exposed dentin surface as a result of the loss of protective enamel structure by chemical agent (erosion), mechanical (abrasion), parafunctional habits or gingival recession.7-9 Dentin hyper-sensitivity is a common prevalence (57% of the population) seen in every age group and both gender. In the United Kingdom, 25% of dental patients in total are diagnosed with dentin hyper-sensitivity, within 10% acute, by the dentists. Usually, dentin hyper-sensitivity is treated with application of desensitizer agents on exposed dentinal surfaces. These agents cover the surface as a thin layer through precipitating on the dentinal surface.¹⁰⁻¹² HEMA/glutaraldehyde, oxalate, fluoride based desensitizers are commonly used for the treatment of dentin hyper-sensitivity. HEMA/glutaraldehyde agents are dentin occlusive including benzalkonium chloride and fluoride. Glutaraldehyde agent causes protein coagulation in dentin tubules. Also, HEMA agent occludes dentin tubules and creates a deep tag for the penetration.¹³ Oxalate based desensitizer reacts with calcium and creates calcium oxalate crystals occluding dentin tubules. Fluoride based agent reacts with calcium and generates calcium fluoride form and decreases dentin permeability in dentin tubules.¹² Recently, triclosan based desensitizer which is affected by anti-inflammatory and calcium phosphate based desensitizer is being used for the treatment of dentin hyper-sensitivity.14,15

A pretreatment process is applied to the dentin surface before the application of the conventional resin cement. This pretreatment process consumes time and requires technical precision. For this purpose, the self-adhesive cement that does not require any pretreatment on the tooth surface has been developed.¹⁶ In this system, adhesive mechanism happens with infiltration of the acidic monomer to demineralize collagen network.¹⁷

Surface hardness is defined as resistance which the surface resists the indentation force that is applied to the material surface.¹⁸ Vickers (HV) and Knoop (HK) micro hardness test is a method used to measure surface hardness. In previous studies, it was indicated that physical properties of the resin material such as conversion rate and abrasion value were assessed by micro hardness test method.¹⁹

There are studies evaluating the terms of the bonding strength of traditional cement and self-adhesive cement. In the results of some studies bond strength values of resin cements did not show any significant differences, while in some studies bond strength values of self-adhesive cement were found to be lower.^{20,21} However, the studies on evaluating the shear bond strength and Vickers surface hardness of self-adhesive resin cement after application of desensitizer on the dentin surface are limited.^{21,22}



For this purpose, this study evaluated the shear bond strength and Vickers surface hardness values of the resin cement.

The hypotheses of this study are as the following: 1) desensitizing agents do not have an effect on the shear bond strengths of self-adhesive resin cement; 2) desensitizers do not affect the surface hardness of the contact side of resin cement.

MATERIALS AND METHODS

Preparation of dentinal surfaces

In this study, 120 fresh human third molars without caries and restorations were extracted and restored in a 0.5% chloramine solution at 5°C until use. The teeth were cleaned from residual periodontal tissues. Then, all teeth were embedded in the cylindrical mold using autopolymerizing acrylic resin (Imicryl, Konya, Turkey) in the way that the occlusal surfaces stay out of the resin surface. The enamel parts of the teeth were separated as parallel to the occlusal plane, under water cooling. The exposed dentinal surfaces were polished with dental sandpapers with 200, 400, 600 and 800 grits under water to standard smear layer in clinical condition. The teeth were divided into six different groups according to the pretreatment of desensitizer; 1) Group CO, Control; 2) Group TM, Teethmate (Kuraray); 3) Group BF, Bifluorid 10 (Voco); 4) Group GL, Gluma (Heraeus Kulzer); 5) Group SP, Smartprotect (Detax); 6) Group BB, Bisblock (Bisco).

Following the manufacturer's instructions, desensitizers were applied on the surfaces of the teeth specimen (Table 1).

	Table 1. M	lanufacturer's	instructions of	of desensitize
--	------------	----------------	-----------------	----------------

Desensitize Agent Use Instructions		Composition	Manufacturer	
Teethmate	Mix	powder and liquid (more than 15 sec)	Tetra calcium phosphate, di	Kuraray Noritake
(TM) to make slurry and then apply the s		nake slurry and then apply the slurry to	calcium phosphate	Dental, Tokyo, Japan
(1111)	den	tin surface using with cotton pellet	anhidrosis, water	
	(rub	bing more than 30 sec).Rinse the excess		
	slur	ry with water spray (5 sec), and clean		
	the	dentin surface by wet cotton (more than		
	10 s	ec).		
Bifluorid10	Mat	erial is taken onto Pele Tim and it is	Sodium and calcium	VOCO GmbH P.O.
(BF)	app	lied on the humid dentin. It is waited to	fluoride	Box 767 27457
()	be a	bsorbed for 10-20 seconds and then		Cuxhaven, Germany
	drie	d with air.		
Gluma	Ap	plied on air dried dentin (5 sec) and	%35 HEMA,	Heraeus Kulzer
(GL)	rubl	ping (30 sec) with a cotton pellet. The		GmbH& Co,
	den	dentin surface was dried until fluid film		Hanau, Germany
	disa	ppear and then rinse with water.		
Smartprotect	Clea	an and dry the dentine surfaces. Apply	Aqueous alcoholic solution	Detax, GmbH & Co.
(SP)	on c	fried dentin and actively via a brush for	with antibacterial content	KG,
(51)	10 s	econds. Allow to take effect for a	(%2-10 gluteraldehyde and	Ettlingen, Germany
	furt	her 20 sec. Do not rinse the tooth	%20-30 isopropyl alcohol)	
	surf	ace and slightly dried with air.	and %0.14 fluoride,	
			triclosan	
Pichloak	Etal	with 22% phosphoric acid for 15 costs	Ovalia agid < %5	Pisao Ina
DISUICK	dan	late calcium ions from dontin surface	Oxalic aciu < 765	Sahaumhura
(BB)	uep	then since with water. Conthe oil dry 2.2		T LISA
	enc	Apply BisBlock for 30 seconds		112,037
	sec.	wing calcium ovalate crustals to form in		
	the	tubulas Dinse with water		
	me	uoures. Kinse with water.		

The control group of dentin surfaces were not treated. Application of desensitizers were performed by a single operator to standardization. The teeth were stored in distilled water for 24 hours at room temperature. After 24 hours, dual cure self-adhesive cement (Panavia SA Automix, Kuraray Noritake Dental Inc.) was applied on the processed dentinal surface.

Shear bond strengths (SBS) test -Bonding procedure

A transparent cylinder plastic mold (3mm internal diameter and 3mm height) was used as spacers for resin cement and fixed perpendicularly to the center dentin surface. Before the application of resin cement, applied desensitizer dentin surfaces were rinsed with distilled water and air dried. In this study used dual cured self-adhesive cement which not requiring etchant and adhesive system on dentin surface before the cementation. Equal amounts of base and catalyst paste of dual cure self-adhesive resin cement (Panavia SA Automix) were automatically mixed and injected into the plastic mold. The dual-cured self-adhesive resin cement material was polymerized using a LED curing unit (Woodpecker, Guangxi, China) with an intensity of 850-1000 mW/cm². The first light cure was applied for 2 to 5 seconds, and then the excess cement was removed. Final light-cure was applied to the entire surface and margins for 10 seconds, and then we waited for chemical polymerization for a total of twelve minutes (23°C/ 73°F). Application of resin cements were done by a single operator to standardization.

All specimens were stored in distilled water for 24 hours at 37°C prior to testing. Shear bond strength was tested using a Universal Testing Machine (Model 2519-106; Instron Corp, USA) at a crosshead speed of 5mm/min. Maximum loads at bond failure were recorded in Newtons (N), and bond strengths were calculated in megapascals (MPa)

SEM analysis

To evaluate change in dentin surface after bonding test, resin-dentin interface was studied by selecting examples close to the average shear bonding strength value in each experimental group. The fracture of samples was evaluated under SEM (Zeiss EVO LS 10; Carl Zeiss) with 15kV at magnification 1000x. The surface image of fractured samples, coated with 80% gold and 20% palladium at the 3µm thickness, was taken.

Vickers surface hardness (HV) test -Preparation of zirconia samples

Zirconia samples (3M ESPE Dental, Seefeld, Germany) that are in the shape of a disc with 8 mm diameter and 1.0 mm height were prepared by CAD-CAM system and sintered to full density in a furnace according to the manufacturer's instructions. Using a stainless steel mold, a 1.0mm-thick Vita VM9 ceramic porcelain (Zahnfabrik, Bad



Säckingen, Germany) layer was applied to the surface of the zirconia framework according to the manufacturer's recommendations (Figure 1A-1B).



Figure 1A. Stainless steel mold



Figure 1B. Without porcelain-zirconia sample and bilayered zirconia sample

The self-adhesive resin cements were polymerized under the bilayered zirconia-porcelain samples by light irritation.

Bonding procedure

An extremely thin layer of Vaseline was applied to two surfaces of a polyester strip as a separating medium between zirconia samples and resin cement. A 100 μ m thick, black Teflon sheet (5x5 mm) with a hole (3 mm diameter and 100 μ m depth) at the center was used as a spacer for resin cement on dentin surface. A dual cure self-adhesive resin cement (Panavia SA Automix) was mixed and then, a small amount of resin cement was placed on each dentin surface within the hole. The polyester strip and zirconia sample were placed on the hole of Teflon sheet, respectively (Figure 2).



Figure 2. Schematic view of diagram for hardness surface test

Then, the dual-cured resin cement material was polymerized through zirconia samples using a LED curing unit (Woodpecker, Guangxi, China) with an intensity of 850-1000 mW/cm². The first light cure was applied for 2 to 5 seconds, and then the excess cement was removed. Final light-cure was applied to the entire surface and margins for 10 seconds, and then we waited for chemical polymerization for a total of twelve minutes (23°C/ 73°F). After chemical and light curing, the zirconia samples, polyester strip and teflon sheet were removed from the dentin surface, respectively. After the bonding procedure, the samples were stored in distilled water for 1 day.

-Vickers surface hardness measurement

The adherent surface of resin cements was carefully detached from dentin by plastic spatula. Then, Vickers surface hardness measurements were carried out from the dentin side of cement (side of resin cement bonded to dentin).²³ Vickers surface hardness was measured using a micro hardness tester (TMTeck HV, Beijing, China) and performed at 1mm interval four points on diameter of resin cement contact side. A load of 200g was applied for 15s, and vertical and horizontal indentation diagonals (mm) were measured (Figure 3).

Duramin 5 - Video Measurement System		
Measurements Image Zoom Options	Egtras	
Section of the sectio		
and the second second		
Contraction and and and and		
Constant Lot and		
Contract Territoria		
and a strange of the		
and the second s		
Barrier and the second s		
and the second s		
CONTRACTOR OF		
the second second		
Carl and the second second		
Harris and the second		
the second second second second		

Figure 3. A microscope image of Vickers surface hardness test

Afterwards, all measurements were converted to the Vickers surface hardness (HV) value by using the HV=1.8544 F/d2 formula. [F= indentation load (kgf), d= the arithmetic mean of two diagonals (mm)].

Statistical analyses

The data were analyzed by one-way ANOVA with the shear bond strength and surface hardness of resin cement as an independent factor. Post-hoc multiple comparisons were performed using the Tukey test. Statistical significance was preset at $\alpha = 0.05$.

RESULTS

Means and standard deviations of the shear bond strength (SBS) are presented in Figure 4.



Figure 4. Means of the shear bond strength (SBS)

The results of the one-way ANOVA and Tukey (Table 2) test showed that dentin desensitizers affected SBS of resin cement (p<.05). The group of SP desensitizer exhibited the highest (4.05 ± 1.34 MPa) mean SBS, while the group of BF desensitizer showed the lowest (1.30 ± 0.57 MPa) mean SBS. The experimental groups CO (2.12 ± 0.76 MPa), GL (1.96 ± 1.23 MPa), TM (1.80 ± 0.66 MPa), BF (1.30 ± 0.57 MPa) showed similar mean SBS value (p> .05). However, the experimental test group treated with SP (4.05 ± 1.34 MPa) showed a significantly higher mean SBS value than the BF, TM, and GL desensitizer groups and CO group (p< .05). No statically significant was observed between SP (4.05 ± 1.34 MPa) and BB groups (2.71 ± 1.39 MPa) (p> .05).

Table 2. Means (Standard Deviations in parentheses) of SBS

	Groups	Means (SD)
Tukey HSD	BF	1.30 ^a (0.571)
	ТМ	1.80ª (0.664)
	GL	1.96 ^a (1.239)
	со	2.12 ^a (0.761)
	BB	2.71 ^{ab} (1.393)
	SP	4.05 ^b (1.345)
Groups with same	superscripts are not significan	tly different $(p \ge 0.5)$

The microstructure of the dentin-resin interface was examined by SEM (X1000). Each group was tagged between Figure 5A-F. Enlarging images including were divided into subgroups. According to the SEM images of CO group, most of dentinal tubules opened and not sealed with resin cement (Figure 5A).



Figure 5A. SEM micrographs of dentin surfaces of CO group (1000x)

When SEM images of the BF desensitizing agent were investigated, it was seen that the diameter of the dentinal tubules became more open and wider when compared to other desensitizer groups. Thin and rare remnant resin cement was observed in a small area of the dentin surface (Figure 5B).



Figure 5B-SEM micrographs of dentin surfaces of BF group (1000x)

When SEM images of the TM desensitizing agent were investigated, residual resin cement, open and sealed dentin tubules was seen in Figure 5C.



Figure 5C. SEM micrographs of dentin surfaces of TM group (1000x)

SEM images of the GL desensitizing agent showed precipitation in the dentin tubules because of the protein coagulation. Also, it was seen that most of the dentin tubules were sealed and residual resin cement was in a small area on the dentin surface (Figure 5D).



Figure 5D. SEM micrographs of dentin surfaces of GL group (1000x)



The fracture surface of SP and BB group showed more resin composite remnants than the other desensitizer group (Figure 5E and 5F).



Figure 5E. SEM micrographs of dentin surfaces of SP group (1000x)



Figure 5F. SEM micrographs of dentin surfaces of BB group (1000x)

Means and standard deviations of the Vickers surface hardness (HV) obtained from self-adhesive dual-cured cement of the examined surface which contacts with the dentin desensitizer are presented in Table 3.

Groups	Means (SD)
BF	11.34ª (2.24)
SP	14.27 ^a (3.65)
GL	15.58 ^{ab} (1.77)
BB	16.06 ^{ab} (5.73)
СО	18.04 ^b (3.05)
TM	19.22 ^b (2.10)

Table 3. Means (Standard Deviations in parentheses) of HV

Groups with same superscripts are not significantly different (p>.05)

The results of the one-way ANOVA and Tukey test showed that dentin desensitizer significantly affected surface hard-

ness of contact side of resin cement (p< .05). The group of TM desensitizer exhibited the highest (19. 22±2.10 HV) mean surface hardness while the group of BF desensitizer showed the lowest (11.34±2.24 HV) mean surface hardness. The experimental groups TM (19.22±2.10 HV), CO (18.04±3.05 HV), BB (16.06±5.73 HV), GL (15.58±1.77 HV) showed similar surface hardness values (p> .05). However, the experimental test groups treated with BF (11.34±2.24 HV) and SP (14.27±3.65 HV) showed a significantly lower surface hardness value when compared other desensitizers group and CO group (p< .05).

DISCUSSION

In this study, the bonding strength of self-adhesive cement was examined after the application of desensitizer to dentin. It was seen that the group of SP desensitizer exhibited the highest mean SBS while the group of BF desensitizer showed the lowest mean SBS. However, the use of GL, TM, and BF desensitizers does not considerably affect the bonding strength of the self-adhesive cement. Therefore, the first null hypothesis was partially rejected.

In the present study; TM, BB, and GL desensitizers did not affect the surface hardness of the contact side of resin cement, whereas BF and SP desensitizers decreased the surface hardness of self-adhesive resin cement. Therefore, the second null hypothesis was partially rejected.

Rueggeberg and Craig²⁴ showed that the hardness is too sensitive and affected by any slight alteration in polymer structure in their study. A lower hardness value indicates the lower density of polymer structure.²⁵ There is a correlation between conversion and polymerization rate of a resin material and micro surface hardness of the material during the polymerization, and it gives information about monomer conversion.²⁶ Also, mixing conditions of adhesive resin cement polymerization and additional materials in an environment have an effect on polymerization reaction.²⁷ The present study evaluated the effects of the desensitizers on the contact side of resin cement's hardness during polymerization.

Previous studies showed that presence of antibacterial agent or desensitizer in environmental conditions affects physical property of resin cement.^{5,28-33} Oguz Ahmet³⁰ assessed the effect of the antibacterial agent (benzalkonium chloride) on the dual resin cement of surface hardness. The result of this study showed that the antibacterial agent leads to 20% and 25% decrease in surface hardness on light applied to side and the bottom side, respectively. In the current study, the results showed that the addition of TM, BB, and GL desensitizers does not cause any significant changes in surface hardness of the self-adhesive resin (p> .05). However, the addition of BF and SP desensitizers significantly decreases the surface hardness of self-adhesive resin cement compared to the control

group (p<.05). In BF desensitizer group, it also observed that there was a linear relation between decrease of surface hardness and reduce in SBS. The changes in the surface hardness of BF group may be related to the high amount of F ions. The presence of high amount of F ions on environmental condition can be negatively affect polymerization or conversion rate of resin cement.^{33,34} In SP desensitizer group, reverse relation was observed between decrease of surface hardness and increase in SBS value. This may be related to the presence of different chemical ions (glutaraldehyde, isopropyl alcohol, fluoride, triclosan) in SP desensitizer. While some of ions such as glutaraldehyde may have positive affect on SBS,^{29,35} some of ions like a fluoride may have negative affect on polymerization or conversation rate of resin cement.^{33,34}

Many vitro studies evaluated the effect of glutaraldehyde and HEMA containing desensitizer (Gluma) on the bonding strength of the traditional resin cement. Some studies concluded that glutaraldehyde and HEMA containing desensitizer has a negative effect, while some studies found that it does not have a significant effect on the bonding strength of the traditional resin cement.^{6,22,28,33} However, some of studies on the self-adhesive resin cement showed that the application of glutaraldehyde and HEMA containing desensitizer (Gluma) increased the bonding strength of resin cement.^{6,22,29,31,32} It was considered that increasing of bonding strength may be related to the hydrophilic property of HEMA and glutaraldehyde. Self-adhesive resin cement contains phosphate group or phosphoric ester monomer (methacrylate phosphoric ester). The HEMA provides an elimination of the water on the surface by forming condensation reaction with the phosphate group in the content of the dentine and also contributes to the diffusion of the monomer to the structure of the dentin.^{29,31} Also, the reaction of glutaraldehyde with phosphate in the structure of the dentin may cause an increase in the bonding strength.^{29,35} However, in this study HEMA- glutaraldehyde and HEMA containing desensitizer (GL) did not cause a change in the bonding strength. This result may be related to the fact that GL desensitizer causes the coagulation of dentin protein in the dentinal tubules and plugging of the tubules.²⁸

Recently, anti-inflammatory desensitizers containing triclosan and calcium phosphate containing desensitizers are being used for dentin hypersensitivity treatment.^{28,37} Triclosan containing desensitizer forms has a low surface energy and so it caused a decrease in wettability level of dentin surface.³⁸ Reduced wettability level may adversely affect the connection between dentin and resin. However, the glutaraldehyde including triclosan desensitizer is reacted with dentin phosphate and it may cause the bonding of the self-adhesive cement to improve.³⁵ Also, Dündar³⁹ examined the effect on the bonding strength of the two and three stage resin cement of triclosan containing desensitizer (Seal&Protect, Dentsply Co., UK) and concluded that it has an adverse effect on the bonding strength. In this study, triclosan containing desensitizer (SP) increased the bonding strength of the self-adhesive resin cement. This increase in the bonding strength may be related to the fact that triclosan containing desensitizer involves the glutaraldehyde.^{29,35,36}

The application of fluoride containing desensitizers on the dentin surface showed a decrease in the bonding strength of resin cement.^{33,34,40} Saraç³⁴ have stated that an increase in the fluoride amount of desensitizers may reduce connection between dentin and resin. They argued that an increase in the crystal structure precipitated on the dentin surface causes a decrease in the bond strength. These crystals have resistance to acids or chemically and physically affect, and it can block the penetration of the resin components of the dual cement. In this study, it was seen that fluoride containing desensitizers (BF) applied on the dentin surface reduces the bonding strength between dentin and self-adhesive resin, which is consistent with previous studies.^{33,34,40} However, this decrease was not statistically significant.

Potassium oxalate forms calcium oxalate crystals by reacting with the calcium containing oral liquid or the structure of the dentin. This crystal structure covers the dentin surface by blocking dentinal tubules. Consequently, adhesive resin cannot provide good contact with the dentine surface containing oxalate crystals.^{40,41} In this study, in accordance with the manufacturer's recommendations, the etching process was applied on the dentine surface for 15 seconds before the application of oxalate containing desensitizer (BB). Pashley⁴² stated that the crystals applied on the acid-etched dentin surface are too deep from the surface of the dentin tubules. However, Tay43 compared desensitizer with acid-etched and non-etching dentine surface and stated that the bonding strength of the acid etched surface is close to the non-etching surface. In addition, Huh²⁸ noted that the oxalate containing desensitizers does not affect the bonding strength of self-adhesive systems. In this study, it has been seen that the oxalate containing desensitizer (BB) increases the bonding strength of self-adhesive cement. However, this increase was not statistically significant. Use of phosphoric acid before desensitizer application and self-adhesive cementation may cause an increase in bond strength.42 Teethmate desensitizer is a calcium-phosphate desensitizer agent that contains TTCP (tetracalcium phosphate) and DCPA (dicalcium phosphate anhydrous) with water. The combinations of the two components (TTCP and DCPA) transform to hydroxyapatite and provided a thick layer penetrating into the dentinal tubules and occluding the dentin tubules, hence, decrease of clinical dentin sen-

sitivity is observed.³⁷ To this study, calcium-phosphate containing desensitizer (TM) showed a decrease in bonding strength of self-adhesive cement, but this decrease was not statistically important. A correlation was observed between the SEM images of the dentin-resin interface and the effect on the bonding strength of desensitizers. When SEM image of desensitizers having a decrease in bonding strength was examined, an increase was seen in the number of the open dentine tubules and a decrease was found in the amount of residual resin cement. The limitation of this study is that the effects of dentin desensitizers were evaluated based on only one type of resin cement. Further studies are recommended to evaluate according to the application (self-etch and etch and rinse) or type of polymerization (chemical, light and dual-cured).

CONCLUSIONS

The TM, BB, and GL groups show similar surface hardness values and do not significantly affect surface hardness. However, the BF and SP desensitizers statically affect the surface hardness of contact side of resin cement. BF desensitizer shows the lowest SBS desensitizer, but this effect is not statically significant. SP and BB desensitizers have a positive effect on the SBS of self-adhesive cement. However; the use of other desensitizing agents does not significantly change the SBS values.

Also, we concluded that BF desensitizer showed a linear relation, while SP desensitizer showed reverse relation between surface hardness and SBS value.

REFERENCES

1. Hafez AA, Cox CF, Mills JC. 78th General session of the International Association for Dental Research, in conjunction with the 29th annual meeting of the American Association for Dental Research and the 24th annual meeting of the Canadian Association for Dental Research Washington, DC, USA. April 5-8 [abstract] J Dent Res 2000; 79: 431.

2. Orchardson R, Collins WJ. Clinical features of hypersensitive teeth. Br Dent J 1987; 162: 253-256.

3. Braennstroem M, Astroem A. A Study on the mechanism of pain elicited from the dentin. J Dent Res 1964; 43: 619-625.

4. Felton DA, Bergenholtz G, Kanoy BE. Evaluation of the desensitizing effect of Gluma Dentin Bond on teeth prepared for complete-coverage restorations. Int J Prosthodont 1991; 4: 292-298.

5. Richardson D, Tao L, Pashley DH. Dentin permeability: effects of crown preparation. Int J Prosthodont 1991; 4: 219-225.

6. Yim NH, Rueggeberg FA, Caughman WF, Gardner FM, Pashley DH. Effect of dentin desensitizers and cementing agents on retention of full crowns using standardized

crown preparations. J Prosthet Dent 2000; 83: 459-465. 7. Perdigão J, Geraldeli S, Hodges JS. Total etch versus self-etch adhesive: effect on postoperative sensitivity. J Am Dent Assoc 2003; 134: 1621-1629.

8. Porto IC, Andrade AK, Montes MA. Diagnosis and treatment of dentinal hypersensitivity. J Oral Sci 2009; 51: 323-332.

9. Rosing CK, Fiorini T, Liberman DN, Cavagni J. Dentine hypersensitivity: analysis of self-care products. Braz Oral Res 2009; 23: 56-63.

10. Addy M, Smith SR. Dentin hypersensitivity: an overview on which to base tubule occlusion as a management concept. J Clin Dent 2010; 21: 25-30.

11. Gillam DG, Bulman JS, Eijkman MA, Newman HN. Dentists' perceptions of dentine hypersensitivity and knowledge of its treatment. J Oral Rehabil 2002; 29: 219-225.

12. Orchardson R, Gillam DG. Managing dentin hypersensitivity. J Am Dent Assoc 2006; 137: 990-998.

13. Dondi dall'Orologio G, Lone A, Finger WJ. Clinical evaluation of the role of glutardialdehyde in a one-bottle adhesive. Am J Dent 2002; 15: 330-334.

14. Chow LC. Next generation calcium phosphate-based biomaterials. Dent Mater J 2009; 28: 1-10.

15. Wara-aswapati N, et. al. The effect of a new toothpaste containing potassium nitrate and triclosan on gingival health, plaque formation and dentine hypersensitivity. J Clin Periodontol 2005; 32: 53-58.

16. Behr M, Rosentritt M, Regnet T, Lang R, Handel G. Marginal adaptation in dentin of a self-adhesive universal resin cement compared with well-tried systems. Dent Mater 2004; 20: 191-197.

17. Bayle MA, Gregoire G, Sharrock P. The role of acrylophosphonic acid monomers in the formation of hybrid layers based on self-etch adhesive. J Dent 2007; 35: 302-328.

18. Anusavice KJ. Mechanical Properties of Dental Materials. In: Anusavice KJ, Shen C, Rawls HR, editors. Phillips' Science of Dental Materials. 12nd ed. St. Louis; Mosby; 2013. p. 48-69.

19. Medina Tirado JI, Nagy WW, Dhuru VB, Ziebert AJ. The effect of thermocycling on the fracture toughness and hardness of core buildup materials. J Prosthet Dent 2001; 86: 474-480.

20. Abo-Hamar SE, et. al. Bond strength of a new universal self-adhesive resin luting cement to dentin and enamel. Clin Oral Investig 2005; 9: 161-167.

21. Yang B, Ludwig K, Adelung R, Kern M. Microtensile bond strength of three luting resins to human regional dentin. Dent Mater 2006; 22: 45-56.

22. Sailer I, Tettamanti S, Stawarczyk B, Fischer J, Hämmerle CH. In-vitro study of the influence of various dentin sealing and desensitizing methods on the shear bond strength of two universal resin cements. J Adhes Dent

2010; 12: 381-392.

23. Shiomuki D, Minami H, Tanaka T, Suzuki S. Influence of light irradiation on Vickers hardness of dual-cure cement polymerized under restorations. Dent Mater J 2003; 32: 449-455.

24. Rueggeberg FA, Craig RG. Correlation of parameters used to estimate monomer conversion in a light-cured composite J Dent Res 1988; 67: 932-937.

25. Calheiros FC, Daronch M, Rueggeberg FA, Braga RR. Degree of conversion and mechanical properties of a BisGMA:TEGDMA composite as a function of the applied radiant exposure. J Biomed Mater Res B Appl Biomater 2008; 84: 503-509.

26. Lee HH, Lee CJ, Asaoka K. Correlation in the mechanical properties of acrylic denture base resins. Dent Mater J 2012; 31: 157-164.

27. Van Meerbeek B, et. al. Dual cure luting composites-Part II: clinically related properties J Oral Rehabil 1994; 21: 57-66.

28. Huh JB, et. al. The effect of several dentin desensitizers on shear bond strength of adhesive resin luting cement using self-etching primer. J Dent 2008; 36: 1025-1032.

29. Munksgaard EC, Asmussen E. Bond strength between dentin and restorative resin mediated by mixtures of HEMA and glutaraldehyde. J Dent Res 1984; 63: 1087-1089.

30. Oguz Ahmet S, et. al. Addition of benzalkonium chloride to self-adhesive resin-cements: some clinically relevant properties. Acta Odontol Scand 2014; 72: 831-838.

31. Qiu C, Xu J, Zang Y. Spectroscopic investigation of the function of aqueous 2-hydroxyethylmethacrylate/glutaraldehyde solution as a dentin desensitizer. Eur J Oral Sci 2006; 114: 354-359.

32. Sailer I, Oendra AE, Stawarczyk B, Hämmerle CH. The effects of desensitizing resin, resin sealing, and provisional cements on the bond strength of dentin luted with self-adhesive and conventional resin cement. J Prosthet Dent 2012; 107: 252-260.

33. Soeno K, Taira Y, Matsumura H, Atsuta M. Effect of desensitizers on bond strength of adhesive luting agents to dentin. J Oral Rehabil 2001; 28: 1122-1128.

34. Saraç D, Külünk S, Saraç YS, 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.

35. Stawarczyk B, et. al. 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.

36. Guentsch A, et. al. Biomimetic mineralization: long-term observations in patients with dentin sensitivity. Dent Mater 2012; 28: 457-464.

37. Shetty S, Kohad R, Yeltiwar R. Hydroxyapatite as an in office agent for tooth hypersensitivity: a clinical and scanning electron microscopic study. J Periodontol 2010; 81: 1781-1789.

38. Arrais CA, Chan DC, Giannini M. Effects of desensitizing agents on dentinal tubule occlusion. J Appl Oral Sci 2004; 12: 144-148.

39. Dündar M, Cal E, Gökçe B, Türkün M, Ozcan M. Influence of fluoride- or triclosan-based desensitizing agents on adhesion of resin cements to dentin. Clin Oral Investig 2010; 14: 579-586.

40. Pashley EL, Tao L, Pashley DH. Effects of oxalate on dentin bonding. Am J Dent 1993; 16: 116-118.

41. De Andrade e Silva SM, Malacarne-Zanon J, Carvalho RM, Alves MC, De Goes MF, . Effect of oxalate desensitizer on the durability of resin bonded interfaces. Oper Dent 2010; 35: 610-617.

42. Pashley DH, Carvalho RM, Pereira JC, Villanueva R, Tay FR. The use of oxalate to reduce dentin permeability under adhesive restorations. Am J Dent 2001; 14: 89-94.

43. Tay FR, et. al. Integrating oxalate desensitizers with total-etch two-step adhesive. J Dent Res 2003; 82: 703-707.



90