The Effect of Silane Content on Microleakage of the **Adhesive Systems**

Adeziv Sistemlerin Silan İçeriklerinin Mikrosızıntı Üzerine Etkisi

ABSTRACT

Aim: The aim of this study is to compare to microleakage levels of two silane based adhesive systems and one silane free adhesive system.

Material and Method: Thirty standardized freshly extracted sound premolar teeth were used in study. Teeth were randomly and divided into three groups of 10 teeth each, according to the adhesive systems (n=20). Class V cavities were prepared (mesio-distal 4 mm, and occluso-gingival 3 mm, and 2 mm depth). After the adhesive systems were applied (Clearfil Universal Bond- silane based, Single Bond Universal-silane based and Adper Single Bond 2-silane free) composite resins (Filtek Z 250) were built up to the cavities. The specimens were aged with 5,000 thermocycles and immersed in 0.5% basic fuchsine solution during 24 hours. Then the samples were sectioned longitudinally in bucco-lingual and mesiodistal directions. The slices were observed under a stereomicroscope (X40 magnification). The scores were statistically analyzed using the Kruskal-Wallis and the Mann Whitney U tests.

Results: Significant differences were found in microleakage values among the adhesive systems (p<0.05). The lowest microleakage value was recorded in the Single Bond universal (p<0.05). There were no significant differences in microleakage values among the Clearfil Universal Bond Adper Single Bond 2 (p>0.05). Statistical analysis revealed significant differences between the occlusal and gingival in all restorations (p < 0.05).

Conclusion: For all groups, microleakage values were higher at gingival margins than at occlusal margins. None of the materials tested in this study completely eliminated microleakage at both the enamel and gingival margin.

Key words: Microleakage, Silan content, Adhesive systems.

ÖΖ

Amaç: Bu çalışmanın amacı yeni geliştirilen silan içerikli iki farklı bonding ajan ile silan içermeyen bir bonding ajan kullanılarak yapılan restorasyonların mikrosızıntı yönünden incelenmesidir.

Gereç ve Yöntem: Çalışmada, 30 adet çürüksüz yeni çekilmiş premolar diş kullanıldı. Dişler rastgele 3 gruba ayrıldıktan sonra tüm dişlerin bukkal ve lingual yüzeylerine sınıf V kaviteler açıldı (4 mm uzunluk, 3 mm genişlik ve 2 mm derinlik) Black V kaviteler açıldı (n=20). Bonding ajanlar uygulandıktan sonra tüm kaviteler (silan içerikli Clearfil Universal Bond ve Single Bond Universal, silan içermeyen Adper Single Bond 2) Filtek Z250 (3M, ESPE) ile restore edildi. Termal siklus (5000 kez 50C-550C) ile yaşlandırılan örnekler 24 saat bazik fuksin (%0.5) içerisinde bekletildi. Daha sonra dişler isomet cihazı ile bucco-lingual yönde ikiye ayrıldı. Kesitler stereomikroskopta 40X büyütmede fotoğraflanarak sızıntı skorları belirlendi. Elde edilen veriler Kruskall-Wallis H ve Mann-Whitney U testleriyle istatistiksel olarak değerlendirildi.

Bulgular: En az sızıntı Single Bond Universal grubunda tespit edilirken (p<0,05), diğer iki bonding ajan arasında istatistiksel açıdan bir fark tespit edilemedi (p>0,05). Okluzal ile gingival bölgeler arasında istatistiksel açıdan fark tespit edildi (p<0,05).

Sonuç: Gingival bölgede daha fazla sızıntı tespit edilirken hiç bir materyal mine ve gingival kenarlarda sızıntıyı tamamen engelleyememiştir.

Anahtar sözcükler: Mikrosızıntı, Silan, Adeziv sistemler

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Received / Geliş tarihi : 31.08.2015 Accepted / Kabul tarihi: 10.10.2015 DOI: 10.21306/jids.2015.1.03

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INTRODUCTION

In recent years, composite resins give excellent results in restorative dentistry because of high quality aesthetic expectation. Nevertheless, despite the continuous evolution of these resins, still several problems like a polymerization shrinkage and marginal microleakage (1). However, composite resins, compomer, glass ionomer are used cervical erosion and abrasions in the treatment of caries because of more aesthetic than amalgam (2, 3). Microleakage means between cavity and restoration various ions, microorganism and occurred with the passage liquids and causing postoperative sensitivity, recurrent caries, coloration, and inflammation and pulpal pathology (4-6). Microleakage reasons are thermal expansion difference of cavity and restorative materials and enamel and dentin, shrinkage during polymerization, elastic deformation, surface erosion and carelessness of physicians (7, 8).

Self-etch adhesive systems consist of aqueous mixtures of acidic functional monomers without the need for separate acid etching and subsequent rinsing methods. Acid monomers partially dissolve hydroxyapatite structure; therefore, primers penetrate into the collagen network (9, 10). Self-etching dental adhesives have been developed to simplify bonding procedure and to make their application less time-consuming. In two-step systems, the primer and adhesives are combined into one solution unlike the one-step systems the etchant, primer, and adhesives are combined into one solution (11).

In etch-and-rinse systems, the bonding mechanism is micromechanical and is based on the formation of a hybrid layer. In addition to micromechanical adhesion, diffusion and infiltration of resin within etched collagen fibrils are also effective in bonding to dentin (12). In studies recently, cervical cavities in the dentin layer using all-in-one system is less microleakage than the self-etch systems (13, 14). However some researchers found no difference between self-etch bonding systems and all-inone system in Class V cavity (15, 16).

Artificial aging that imitates environmental influences is important in composite repair. Thermocycling and water storage are the oft-used methods to simulate aging and to stress interfacial bonds (17). In in vitro studies, different periods of water storage and thermocycling are used in the aging process of dental materials (18, 19).

The aim of this in vitro study was to assess the marginal microleakage of different adhesive systems in Class V cavities of premolar teeth. The null hypothesis to be

investigated in this study was that there are no differences among microleakage values of the adhesives.

MATERIALS and METHODS

The study was performed at Department of Restorative Dentistry, Gaziantep University, Faculty of Dentistry, Gaziantep, Turkey. Thirty sound human premolar teeth used (extracted for some kind of reasons) for the current study. The teeth were then stored in distilled water until use. Then, the teeth were cleaned with slurry of pumice and water, rinsed thoroughly with tap water, and then examined macroscopically with magnification for defects in the enamel and cement. Teeth were randomly and divided into three groups of 10 teeth each, according to the adhesive systems. Class V cavities were prepared on the buccal and lingual surfaces with the occlusal margins in enamel and the gingival margins located 1.5 mm apical to the cemento-enamel junction (n=20). Cavity dimensions were standardized, (4.0 mm in width, 3.0 mm in height, and 2 mm in depth) using a marked bur.

The groups of this study are following as:

Group 1: one-step self-etch adhesive system-silane based (Clearfil Universal Bond, Kuraray)

Group 2: one-step self-etch adhesive system-silane based (Single Bond Universal, 3M ESPE)

Group 3: two-step total-etch adhesive system-silane free (Adper Single Bond 2, 3M ESPE)

Adhesives were applied according to manufacturers' instructions. Table I shows the adhesive systems used in the present study including manufacturers' instructions, batch numbers, compositions. Following the application of the adhesives, composite resins (Filtek Z 250, 3M ESPE Dental Products, St. Paul, USA) were built up incrementally. Each layer was polymerized for 20 s with a LED Lamp (Valo, Cordless, Ultradent, Germany). After 24 h, the restorations were finished with fine-grit diamond bur, polished with a composite polishing disc (Optidisc, Kerr, Switzerland).

Then, the teeth were aged with thermocycles at 5-55°C for 5000 cycles with a dwell time of 30 seconds. Apical margins of teeth were covered by flowable composite (Competence Flow Willmman & Pein Gmbh, Germany). The exposed crown and root structure was covered with two coats of nail varnish, extending 1 mm beyond the margins of the restoration. Specimens were then immersed in a 0.5% basic fuchsine dye buffered at pH=7 at 37°C for 24 hours. After this procedure, teeth were washed, and dried. In the vertical plane, each

Materials	Composition	Manufacturer	Lot no
Clearfil Universal Bond	Adhesive: 10-MDP, Bis-GMA, 2-HEMA, Hydrophilic aliphatic dimethacrylate, Colloidal silica, Silane coupling agent, dl-Camphorquinone, Ethanol, Water	Kuraray Europe GmbH BU Medical Products Philipp-Reis-Strasse 4 65795 Hattersheim am Main	3D0006
Single Bond Universal	Etchant: $35\% H_3PO_4$ with silica Adhesive: ethanol, water Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, initiator, silane	3M ESPE Dental product, St. Paul, MN, USA	499405
Adper Single Bond 2	Etchant: 35% phosphoric acid Adhesive: ethyl alcohol, Bis-GMA, silica nanoparticles treated, HEMA, glycerol 1,3 dimethacrylate, acrylic acid copolymer and itaconic acid, diurethane dimethacrylate, water	3M ESPE Dental product, St. Paul, MN, USA	N614336
Filtek Z250	Bis-GMA, UDMA, Bis-EMA, Silica, zirconia filler, average cluster particle size 0.01 μm to 3.5 μm	3M ESPE Dental product, St. Paul, MN, USA	N613265

Table I: Details of materials used in the study.

Abraviation; MDP = Methacryloyloxydecyl dihydrogen phosphate, HEMA= Hydroxyethyl methacrylate, Bis-GMA = Bisphenol A-Glycidyl Methacrylate, Bis-EMA = Bisphenol-A ethoxylated dimethacrylate, TEGDMA = Triethylene glycol dimethacrylate, UEDMA = Urethane Dimethacrylate.

tooth was sectioned bucco-lingually across the center of the restorations using with a slow-speed diamond saw (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) machine. The cut sections were examined under a stereomicroscope at X40 magnification and the teeth were scored using the linear scoring criteria.

Dye penetrations at the occlusal and gingival margins were assessed by one examiner to determine the extent of microleakage according to a five-point scale as follows (20);

0: No dye penetration,

1: Dye penetration within 1/3 of the cavity wall,

2: Dye penetration within 2/3 of the cavity wall,

3: Dye penetration within the last 1/3 of the cavity wall without reaching the axial wall,

4: Dye penetration spreading along the axial wall.

Statistical analysis

To understand the significance of differences between the groups, the data were analyzed with the Kruskal-Wallis non-parametric test. Pairwise comparisons were made using the Mann-Whitney U test. The preset level of significance was 0.05.

RESULTS

According to the statistical analysis results, there were significant differences in microleakage scores among adhesive systems (p=0.002), (Table II). There were significant differences in microleakage values among Single Bond Universal and Clearfil Universal Bond adhesives. Also, there were significant differences in microleakage values among Single Bond Universal and Adper Single Bond 2 adhesives (p<0.05). But, there were no significant differences in microleakage values among the Clearfil Universal Bond and Adper Single Bond 2

Table II: Comparison of median of microleakage scores between three groups.

Materials		Mean rank	Sig.
Clearfil Universal Bond	40	62.08	
Single Bond Universal		46.65	.002
Adper Single Bond 2	40	72.78	

(p>0.05), (Table III). Occlusal and gingival microleakage scores of groups were shown in Table IV.

Less microleakage was observed at the occlusal margins than at the gingival margins for all restorations p=0.001 (p<0.05), (Table V). For Single Bond universal, the lowest microleakage value was recorded in both enamel and gingival margins. The highest microleakage value was recorded in Adper Single Bond 2 adhesives for enamel and gingival margins (p<0.05) (Table VI).

DISCUSSION

This in vitro study compared the marginal microleakage of three resin-based adhesives. The results of this study did not support the hypothesis that there are no differences among microleakage values of the adhesives. In fact, there were significant differences in microleakage scores among adhesive systems.

Composite restorative materials represent one of the many successes of modern biomaterials research, as they replace biological tissue both in appearance and function (21). The major drawbacks of these material include polymerization shrinkage limited toughness, microleakage and the presence of unreacted monomers. composite polymerization always involves some degree of shrinkage depending on the organic matrix (22). The quality of bonding is affected by numerous factors such as variations in resin penetration into the demineralized surface and subsequent polymerization, along with the stresses that develop at the adhesive-dentine interface during curing and function. All these variables might

Table III: The comparison of the adhesive systems according to microleakage scores.				
Groups		Mean ranks	Sig.	
Group1-Group2	20	45.54-35.46	.044	
Group1-Group3	20	43.96-37.04	.171	
Group2-Group3	20	31.69-49.31	.000	

Table III: The comparison of the adhesive systems according to microleakage scores.

Table IV: Occlusal and gingiv	al microleakage	scores of groups.
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Groups	Occlusal scores 0 1 2 3 4	Gingival scores 0 1 2 3 4
Clearfil Universal	59321	52157
Single Bond Universal	5 12 1 2 -	10 3 2 2 3
Adper Single Bond 2	58241	1 - 1 11 7

Table V: The comparison of microleakage scores between microleakage region.

Microleakage region		Mean rank	Sig.	
Occlusal	60	49.99	.001	
Gingival	60	71.01		

Table VI: Statistical values for microleakage of occlusal and gingival margin.

Croups		Occlusal		Gingival	
Groups	n	Mean ranks	Sig.	Mean ranks	Sig.
Group1-Group2	20	21.60-19.40	.516	24.08-16.93	.044
Group1-Group3	20	19.95-21.05	.754	18.45-22.55	.240
Group2-Group3	20	18.93-22.08	.357	14.28-26.73	.000

be influenced by the operator and are likely to cause variations in results (23, 24). Free radical polymerization of methacrylate-based composites, monomer molecules come closer to each other during the polymerization process, which results in polymerization shrinkage (25). Polymerization contraction stress produces powerful forces that can result separate the restoration from the tooth resulting in marginal microleakage (25).

Current single-step self-etching adhesives produce simultaneous conditioning and priming effects on dental substrates (26). These systems do not remove the smear layer, instead, modify it (26) dependent on pH, composition, and concentration of polymerizable acids (27). The acidic monomers of self-etching adhesives promote conditioning of the smear layer and underlying enamel/dentin substrates, resulting in a 'typical hybrid layer,' which is divided into an upper portion with a thick hybridized smear layer (resin infiltration into the demineralized organic material layer) and a lower portion with a thin and homogeneous true hybrid layer in the demineralized dental substrate (28). The demineralization of dentin and enamel in mild self-etch adhesives occurs using acidic primers, so that hydroxyapatite crystals exposed to acidic monomers remain around collagen fibrils in dentin tubules. It is suggested that crystals have a chemical reaction with functional monomers and can prevent marginal microleakage (12, 29).

Some studies (30, 31) investigated the bond strengths of different composite resins to pretreated teeth. It was showed that acid etching produces a hybrid layer and the characteristic funnel-shaped resin tags, regardless of the type of surface preparation (bur/laser) (32). According to the results of the current study, the highest microleakage value was recorded in Adper Single Bond 2 adhesive for enamel and gingival margins. Some researchers have shown differences in the sealing ability of restoration margins between self-etch and total etch adhesives (14, 33) by Mitsui and others (33) and Pradelle and others (14); both researchers stated that etch-and-rinse and self-etch systems are no difference in dentin margin microleakage. Additionally, one of the factors involved in microleakage is bond strength. Different studies have shown that these two types of adhesive systems have nearly similar bond strengths (12, 34). Some researchers said that 3-monthstorage had no effect on microleakage (35) but some other studies concluded that storage time increases the microleakage in some adhesive systems (36, 37).

Silane within Single Bond Universal Adhesive enables the adhesive to chemically bond to glass ceramic surfaces

without using a separate ceramic primer. At the same time, the Single Bond Universal Adhesive contains the Vitrebond Copolymer, HEMA, and water in a balanced manner (38). In the current study, it was found that the lowest microleakage value was recorded in Single Bond Universal Adhesive, which contains silane.

The thermocycling aging method, which is a combination of hydrolytic and thermal degradation, simulates temperature-related breakdown through repeated sudden temperature changes (39). As reported in the literature, 10,000 thermocycling corresponds to approximately one year of in vivo functioning (40). As artificial aging plays a crucial role in composite repair, the composite specimens in this study were subjected to a 5,000 thermocycling procedure before and after the repair.

CONCLUSIONS

Within the limitations of this study, we can draw the following conclusions:

- Microleakage values were higher at gingival margins than at occlusal margins. None of the materials tested in this study completely eliminated microleakage at both the enamel and gingival margin.
- The microleakage values of silane based adhesive systems were lower than silane free adhesive system. The silane-based adhesive systems may be recommended. Further in vitro and in vivo studies should be continued for clinical practice.

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