Original Article

The effect of silane-containing universal adhesives on the immediate and delayed bond strength of repaired composite restorations

Razieh Hoseinifar[1], Niloofar Shadman[1], Farnaz Mirrashidi[2], Sajad Gholami[2]

[1]Department of Operative Dentistry, School of Dentistry, Kerman University of Medical Sciences, [2]Dentist, Private Practice, Kerman, Iran

ABSTRACT

Background: The repair of composite restorations is considered as a conservative treatment for avoiding the risk of pulp injury, the enlargement of cavity preparation, and excess removal of sound dental structure. The aim of this study was to evaluate the effect of silane-containing adhesives on immediate and delayed shear bond strength (SBS) of repaired composite restorations.

Materials and Methods: In this *in vitro* study, 132 discs of Z350 composite were fabricated and divided into fresh (10 min water storage) and aged (6-month water storage + 2000 thermal cycling). All composite surfaces were roughened and etched, and each group was divided equally into six subgroups: 1 (Single Bond 2), 2 (Single Bond Universal), 3 (Clearfil Universal Bond), 4 (silane + Single Bond 2), 5 (silane + Single Bond Universal), and 6 (silane + Clearfil Universal Bond). The specimens were restored with the same composite, thermocycled, and tested for SBS in a universal testing machine. Data were analyzed using one- and two-way ANOVA, *t*-test, and *post hoc* Tukey's tests. $P < 0.05$ was set as the level of significant.

Results: The highest and lowest SBS (in both fresh and aged groups) were related to Single Bond 2 with silane and Clearfil Universal Bond with silane, respectively. The delayed SBS of Single Bond 2 was significantly higher than universal adhesives (in both with and without silane application) ($P < 0.05$). Silane had no significant effect on the repair bond strength of Single Bond 2 and Single Bond Universal ($P > 0.05$), while silane application significantly decreased the delayed SBS of Clearfil Universal Bond.

Conclusion: The SBS of Single Bond 2 was significantly better than two other universal adhesives. The SBS of Single Bond Universal was not affected by silane application, while silane had a negative effect on delayed SBS of Clearfil Universal Bond.

Key Words: Adhesives, aging, composite resins, dental restoration repair

INTRODUCTION

Nowadays, composite resins are widely used in dentistry due to their aesthetics and adhesive properties, conservative tooth preparation, and repairability.[1]

Fracture and secondary caries are the main reasons for composite restorations failure.[2] The failed restorations are treated by complete replacement or repairing. According to the results of several clinical

studies, repair of the existing restoration is a more conservative treatment, which decreases the risk of pulp injury, preserves the sound tooth structure, increases the longevity of the restoration, and reduces the treatment time and cost.[3]

A successful repair of composite restoration requires an adequate bond between the old restoration and the new composite resin. Bonding to an old composite can be highly challenging due to composite water absorption over time and a decrease in the amount of available C=C bonds, which are required to react with the new composite. Therefore, the surface of an old composite that acts as a bonding substrate must be prepared by suitable treatment methods.[4]

The bond strength of repaired composite restorations is influenced by several factors such as adhesive's chemical composition, the method of aging, adhesive diffusion rate, age of the old restoration, oral conditions, type of composite, and surface roughness.[5,6]

While various methods including roughening of composite surface with diamond bur, etching with hydrofluoric acid, sandblasting, laser, and silane application have been suggested to improve the bond, no ideal technique has been found for the repair of composite restorations.[7]

According to some studies, the silane application on the surface of an old composite significantly increases the repair bond strength.[2,8-10] Silane can establish a chemical bond with filler particles of old composite, improve the adhesive wettability, and facilitate adhesive penetration into surface irregularities.[11] Silane makes the strongest adhesion with silica, glass, and quartz fillers.[9]

Recently, some manufacturers have produced silane-containing adhesives in their new products, which might eliminate the need to apply silane separately.[11] The present study aimed to evaluate the effect of silane-containing adhesives (Single Bond Universal and Clearfil Universal Bond) on the repair bond strength of fresh and 6-month aged composite restorations. A review of literatures revealed a lack of research on the evaluation of composite repair bond strength using Clearfil Universal Bond. On the other hand, studies conducted on Single Bond Universal have yielded conflicting results.[8,11-13]

The null hypothesis is that silane-containing adhesives have no effect on the repair bond strength of the composite (fresh and aged).

MATERIALS AND METHODS

In this *in vitro* experimental study, 137 composite discs were prepared using Z350 composite (Enamel A1, 3M ESPE, USA) with a diameter of 6 mm and a height of 3 mm using metal molds. The composite was placed in two increments with a thickness of 1.5 mm, and each layer was cured for 40 s using a light-emitting diode curing light device (Demetron, Kerr, USA) with a minimum intensity of 600 mW/cm². Five composite discs were considered as the control group to evaluate cohesive strength of composite. A plastic mold (with a diameter of 4 mm and a height of 3 mm) was immediately placed on the cured composite, and two composite increments were placed (1.5 mm each layer) and each layer was cured for the 40s. The remaining samples (132 discs) were randomly divided into two fresh and aged groups.

Fresh group
The composite discs were immersed in distilled water for 10 min, and then, the free surface of samples was roughened with a 320-grit silicon carbide sandpaper (Starcke, Germany) to create a surface roughness similar to one created by a diamond bur. The samples were washed, etched with 35% phosphoric acid for 15 s (Fine Etch 37, Spident, Korea), rinsed with air/water spray for 15 s, and then were randomly divided into the following two subgroups based on the use or lack of use of a separate silane. In the first subgroup, the silane (Ultradent, USA) was applied to the surface with a microbrush before the application of adhesive, followed by air drying after 60 s. However, only the adhesive was applied in the second subgroup.

In the next stage, the subgroups were randomly prepared by three different adhesives:
1. Clearfil Universal Bond (Kuraray, Japan)
2. Single Bond Universal (3M, ESPE, USA)
3. Single Bond 2 (3M, ESPE, USA).

All three adhesives were applied to the surface according to their manufacturer's instructions and then light cured for 20 s.

All samples were repaired using a plastic mold (with a diameter of 4 mm and a height of 3 mm) with Z350 composite. Two layers of new composite were placed, and each one was cured for 40 s. Afterward, the samples were stored in 37° distilled water for 24 h and then additionally thermocycled (Vafaei Industrial, Iran) for

2000 cycles (5°C–55°C) with a dwell time of 30 s and a transfer time of 10 s.

In the next stage, the shear bond strength (SBS) was evaluated by the universal testing machine (Testometric M350-10CT, England) at a crosshead speed of 1 mm/min using a parallel stainless-steel blade with a diameter of 1 mm in the interface area of new and old composites. The maximum force before fracture in each sample was recorded in Newton, and the bond strength was calculated in MPa by dividing the failure load into cross-sectional area of samples.

Aged group

The composite discs were stored in distilled water (replaced every 1 month) for 6 months to simulate the aging process and then were subjected to 2000 thermal cycles (between 5°C and 55°C). In the next stage, the free surface of composites was roughened with the 320-grit sandpaper, washed, etched with 35% phosphoric acid for 15 s, and rinsed with air/water spray for 15 s. Preparing the old composite surface (by silane and adhesives) and adding the new composite was done similar to the fresh group mentioned above.

The repaired samples were then placed in distilled water for 24 h, and the SBS was tested after 2000 thermal cycles similar to the fresh group.

In the next phase, all debonded surfaces in each group were evaluated for fracture patterns by two trained individuals under a stereomicroscope (Olympus, Dp12, Germany) with ×40 magnification. Fracture patterns were classified based on the following criteria:
1. Adhesive fracture: Remaining of <25% of the composite resin in the interface
2. Cohesive fracture: Remaining of about 75% of the composite resin in the interface (composite fracture)
3. Mixed: Remaining of 25%–75% of the composite resin in the interfacial.

Data analysis was performed in SPSS (version 18.0, SPSS Inc., Chicago, IL, USA) using two-way ANOVA, one-way ANOVA, *t*-test, and Tukey's test. In addition, *P* value was considered significant at the level of 0.05.

RESULTS

The mean and standard deviation of SBS values in various groups are shown in Table 1.

The highest and lowest bond strengths (in both fresh and age groups) were related to Single Bond 2 with silane and Clearfil Universal Bond with silane, respectively. The mean bond strength of the control group was 15.1 ± 4.2 MPa.

Regarding the bond strength in fresh groups, in silane-free subgroups, there was not seen a significant difference in bond strength of various adhesives. However, in the silane subgroup, only the bond strength of the Single Bond 2 was significantly higher than Clearfil Universal Bond. In addition, there was no significant difference among the control group and all adhesives (with or without silane) [Table 2].

The results of the aged groups showed that the SBS of Single Bond 2 was significantly higher than Single Bond Universal and Clearfil Universal Bond (in both with and without silane groups). However, the difference between Single Bond Universal and Clearfil Universal Bond was insignificant [Table 2].

A comparison of silane effect by *t*-test in the fresh groups revealed that the use of silane caused no statistically significant bond strength improvement.

Table 1: Mean shear bond strength±standard deviation of different groups (based on MPa)

Adhesive	Immediate	Delayed
Single bond 2		
Silane	19.1±3.2	19.7±4.2
Without silane	17.02±3.3	18.7±3.03
Single bond universal		
Silane	16.4±1.9	14.4±3.3
Without silane	15.4±6.05	13.2±4.2
Clearfil universal bond		
Silane	13.8±4.7	11.8±2.2
Without silane	15.06±4.4	14.9±3.01

Table 2: Two-by-two comparison of groups in terms of shear bond strength

Adhesive	*P*	
	Immediate	Delayed
Single bond 2+silane		
Single bond universal+silane	0.252	0.006
Clearfil universal bond+silane	0.01	0.001
Clearfil universal bond		
Single bond universal+silane	0.298	0.263
Single bond 2		
Single bond universal	0.88	0.006
Clearfil universal bond	0.81	0.04
Clearfil universal bond		
Single bond universal	0.99	0.53

In the aged groups, the application of silane had no significant effect on the SBS of Single Bond 2 and Single Bond Universal, while there was a significant decrease in the delayed SBS of Clearfil Universal Bond [Table 3].

There was no significant difference between the immediate and delayed SBS of all adhesives (with or without silane) [Table 4].

The fracture patterns of various groups are shown in Table 5. No statistically significant differences were observed among the different groups in terms of fracture pattern ($P > 0.05$).

DISCUSSION

One of the advantages of composite resins is their repairability. Repair of the composite restoration

Table 3: The effect of silane application on bond strength of adhesives

Adhesive	P	
	Immediate	Delayed
Single bond 2		
Silane	0.197	0.56
Without silane		
Single bond universal		
Silane	0.65	0.51
Without silane		
Clearfil universal bond		
Silane	0.59	0.022
Without silane		

Table 4: Comparison of fresh and age groups by *t*-test

Adhesive	P
Single bond 2+silane	
Fresh	0.71
Age	
Single bond universal+silane	
Fresh	0.25
Age	
Clearfil universal bond+silane	
Fresh	0.13
Age	
Single bond 2	
Fresh	0.26
Age	
Single bond universal	
Fresh	0.37
Age	
Clearfil universal bond	
Fresh	0.96
Age	

is preferred over the complete replacement of the restoration due to maintaining tooth structure and lower trauma to the pulp tissue.[3]

Removing the superficial layer of old composite and roughening the surface with diamond bur are necessary to achieve micromechanical retention.[8] In the present study, 320-grit silicon carbide sandpaper and phosphoric acid were applied to prepare the surface before the use of silane and adhesive. In general, bur and phosphoric acid are recommended for repairing nanofill composites.[3] Silicon carbide sandpaper is used in laboratory studies to create standard surface roughness. According to the results of profile meter, the 320-grit silicon carbide sandpaper creates surface roughness similar to the one created by medium grain diamond bur.[8]

To date, there is no consensus on the most effective composite aging technique. The most common aging method in the laboratory is water storage of samples for a long time.[14] Meanwhile, some studies have introduced thermocycling as the most effective method for degradation of composite resins.[15,16] In the current study, 2000 thermal cycles and 6-month water storage were used as the aging methods.

Effect of silane on repair bond strength
According to the results of this study, while the use of silane in Single Bond 2 and Single Bond Universal increased the SBS, this increase was not statistically significant. Various studies have evaluated the effect of silane application on repair bond strength of composite restorations, yielding contradictory results.[4,10,12,17] Silane is used to create a chemical bond between the glass filler particles of old composite and the fresh resin.[18] It is expected that after roughening of composite, fillers are exposed in 50% of the surface, and therefore, chemical bonds with fillers can be established by silane application, which improves bond strength. However, scanning electron microscopy (SEM) assessment showed that only 5.1% of the filler surfaces are exposed and nearly all small fillers are covered with the matrix, thereby making it impossible for silane to have a significant effect.[19]

Some studies have indicated that the effect of silane on the repair bond strength depends on the amount of filler in the surface, the nature and size of fillers, as well as the chemical formulation of commercial silane primers.[20,21]

In the present study, a prehydrolyzed one-bottle silane was used. According to a recent study, the

Table 5: Distribution (%) of failure modes of evaluated groups

Adhesive	Failure modes					
	Immediate			Delayed		
	Adhesive (%)	Cohesive (%)	Mixed (%)	Adhesive (%)	Cohesive (%)	Mixed (%)
Single bond 2						
Silane	27.3	18.2	54.5	36.3	0	63.6
Without silane	27.3	45.4	27.3	27.3	9.1	63.6
Single bond universal						
Silane	27.3	18.2	54.5	27.3	36.3	36.3
Without silane	36.3	36.3	27.3	18.2	45.4	36.3
Clearfil universal bond						
Silane	27.3	36.3	36.3	18.2	36.3	45.4
Without silane	27.3	27.3	45.4	27.3	18.2	54.5

two-bottle silane has better performance compared to single-bottle silane. The solution of one-bottle prehydrolyzed silane has a shorter shelf life, and its activity gradually decreases over time after opening the bottle, thereby preventing optimal adhesion.[22]

On the other hand, there was a significant decrease in the delayed SBS of Clearfil Universal Bond after the application of silane.

Dall'Oca *et al.*, in 2007, reported that the interface between the fresh and aged composites was the weakest zone in the repair of composite restoration. Therefore, the thickness of adhesive layer should be reduced as much as possible, and the thinner bonding layer will result in higher bond strength.[23] Chen *et al.* showed that elimination of the solvent was more difficult in Clearfil Universal Bond due to its high viscosity. This might be speculated that the use of a separate silane with Clearfil Universal Bond results in the formation of a thicker interfacial layer that is more vulnerable to failure.[24]

Effect of aging (6-month water storage + 2000 thermal cycles) on bond strength

According to the results of this study, there was no significant difference between the immediate and delayed bond strength of all adhesives (with or without silane application). Tantbirojn *et al.* evaluated the failure strength of repaired composites, indicating that adding a new layer to the cured and finished composite within the same session would significantly decrease the failure strength, compared to unrepaired monolithic samples.[25] In 2018, Altinci *et al.* assessed the microtensile bond strength of repaired composite with a universal adhesive (i Bond Universal) and reported a significantly higher bond strength in the fresh group (5 min after curing) compared to the aged group (8 h in boiling water + water

storage for 3 weeks), which is inconsistent with our findings.[4] This lack of consistency might be due to the difference in the type of bond strength test, type of used adhesives, and aging method. In addition, 2000 thermal cycles were used in the present study for aging. Meanwhile, some studies have introduced 5000 thermal cycles to be effective in aging and reducing repair bond strength.[2]

Effect of type of bonding on repair strength

According to the results of the present study, the delayed bond strength of Single Bond 2 was significantly higher than two studied universal adhesives. Previous studies have shown the high bond strength for Single Bond 2,[26,27] which was related to the superior chemical bonding ability of Single Bond 2 and its hydrolytic stability. Single bond 2 contains both hydrophobic and hydrophilic monomers and therefore is suitable for various surfaces with different wetting properties.[27,28] The superior bond strength of Single Bond 2 is due to the presence of 5-nm silica nanofillers (10% wt) that result in the formation of a resin film, which stabilizes the hybrid layer. This intermediate layer acts as an elastic zone and resists forces during polymerization shrinkage. In addition, the small size of nanofillers in Single Bond 2 improves its penetration and wettability.[26]

The lower bond strength of two studied universal adhesives, compared to Single Bond 2, could be related to factors such as high viscosity, the higher thickness of the adhesive layer, and greater hydrophilicity of these bondings, which are more explained below:

All universal adhesive systems contain water, which is necessary for the ionization of acidic functional monomers.[29] The residual water after air-drying results in the phase separation of monomers and

blister formation. According to Tsujimoto *et al.*, these blisters are weak point for Clearfil Universal Bond and adversely affected their bond durability.[30] In a study, the results of SEM were indicative of areas of discontinuity in Single Bond Universal after 24 h, which might be due to incomplete solvent evaporation.[31] As a result, one of the important steps in the application of universal adhesives is sufficient solvent evaporation by increasing solvent evaporation time.[32]

Luque-Martinez *et al.* reported that increasing the time of solvent evaporation from 5 to 25 s results in significantly higher bond strength of universal adhesives.[32] In the present study, solvent evaporation time was followed based on the manufacturer's instructions (5 s of gently air drying), and an increase of the solvent evaporation time might cause a better repair bond strength. In addition, the studied universal adhesives contain ethanol solvent. Water removal is easier in acetone base universal adhesives. Conversely, the solvent in adhesive with ethanol base has an increased ability to form hydrogen bonds, and consequently, the elimination of water from the adhesive is difficult.[24]

Some studies have shown that the thickness of the adhesive layer must decrease as much as possible in the repair of composite restorations, and adhesives with lower viscosity that create a thinner layer have higher bond strength.[8,31] Meanwhile, Single Bond Universal creates a thick adhesive layer due to the presence of filler and low solvent content (10–15 wt% ethanol).[33]

While the universal adhesives used in this study contained silane and were expected to improve bond strength, some studies have indicated that the presence of Bis-GMA or MDP (which are used in universal adhesives) in silane solution significantly decreased its contact angle, reduced surface wettability, and chemical reaction of silane with ceramics.[34,35] Yoshihara *et al.* demonstrated that in the Single Bond Universal, the silane molecule is not stable when mixed with acidic components of adhesive and continuously hydrolyzed during storage.[36] It was shown that silane in Clearfil Universal Bond caused a structural network that entraps water and ethanol and complicates their movement. Therefore, solvent removal is harder in Clearfil Universal Bond due to its high viscosity.[37]

In the present study, there was no statistically significant difference among groups in terms of fracture pattern. The fracture pattern in the shear test is partly due to the test mechanics and the stress distribution during force application and does not necessarily show bond durability. The difference in fracture pattern may be related to differences in mechanical properties of adhesives and differences in the interface characteristics formed in adhesives.[38]

CONCLUSION

1. The delayed bond strength of Single Bond 2 was significantly better than two studied universal adhesives (Single Bond Universal and Clearfil Universal Bond)
2. Application of silane had no effect on the immediate and delayed bond strength of Single Bond 2 and Single Bond Universal
3. Application of silane significantly decreased the delayed bond strength of Clearfil Universal Bond
4. There was no significant difference between the immediate and delayed bond strength of all adhesives (with or without silane application)
5. There was no significant difference between the groups in terms of fracture pattern.

Conflicts of interest
The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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