Originals Article

The Effect of Silanization and Thermocycling on the Microshear Bond Strength of Silane-containing Universal Adhesive to Aged Resin Composite

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Abstract

The purpose of this study was to evaluate the effect of silanization and thermocycling on the microshear bond strength (µSBS) of a silane-containing universal adhesive (Scotchbond Universal adhe-sive; 3M ESPE) (SU), and a 3-step etch and rinse adhesive system (Adper™ Scotchbond™ Multi-Purpose Adhesive; 3M ESPE) (SM). Seventy-two thermocycling-aged resin composite specimens were prepared and divided into four groups based on silane application. Group 1: no silane application before SM adhesive (SMN), Group 2: silane application before SM adhesive (SMS), Group 3: no silane application before SU (SUN), and Group 4: silane application before SU adhesive (SUS). Each group was randomly divided into two equal subgroups: 24 h in water storage (n=9) or 5,000-cycle thermocycling (n=9) before µSBS evaluation. The µSBS values were analyzed using Three-way ANOVA and Tukey's post hoc test (p=0.05) and the fracture modes of the fracture interface were evaluated. Threeway ANOVA indicated that thermocycling and silanization significantly affected µSBS (p<0.05), while the adhesive system did not affect µSBS (p>0.05). In the 24 h water storage subgroups, the SMS group had a significantly higher µSBS compared with that of the other groups. In contrast, in the thermocycled subgroups, there was no significant difference in µSBS between groups. In conclusion, after a 5,000-cycle thermocycling, the repair bond strengths of a silane-containing universal adhesive and a conventional etch-and-rinse adhesive were comparable.

Keywords: Aged resin composite, Microshear bond strength, Silanization, Thermocycling, Universal adhesive

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Introduction

The popularity of resin composite restorations has increased because their use can preserve tooth structure with high esthetic results. However, the oral environment and function can cause resin composites to degrade, resulting in defects, e.g. microleakage, marginal discoloration, wear, chipping, and restoration fracture.[1,2] Repairing the restoration defect rather than its total replacement is a more time-efficient and conservative option that can preserve sound tooth structure, protect the pulp from operative trauma, and reduce the risk of iatrogenic pulp exposure and adjacent tooth injury.[3-5]

Several methods have been developed to repair a resin composite defect, including surface preparation before using a resin-based adhesive, such as grinding with burs, etching with hydrofluoric acid or phosphoric acid, air abrasion, and silanization.[6-8] A silane coupling agent is normally used when placing a resin composite to promote a chemical bond between the inorganic filler particles and the organic resin matrix.[11] Thus, using a silane coupling agent can improve the repair bond strength. Studies have demonstrated that silane application enhances the repair bond strength by promoting chemical bond between the filler of old resin composite and the adhesive resin matrix.[7,9]

A universal adhesive system incorporates a silane coupling agent in the adhesive composition for simplifying the clinical steps, requiring less time, multipurpose use, and more user-friendly compared with the 3-step etch-and-rinse adhesive systems.[7,9] Many Studies have evaluated the use of silane-containing universal adhesives in repairing resin composite restorations.[7-11] However, only a few studies investigated the long-term repair bond strength of universal adhesive systems on the aged resin composite.

The purpose of this study was to evaluate the effect of silanization and thermocycling on the repair μSBS of a silane-containing universal adhesive and to compare the repair μSBS of a silane-containing universal adhesive with that of an etch-and-rinse adhesive.

Materials and Methods

Seventy two resin composite specimens (3-mm diameter and 2-mm deep) (Fig. 1B) were fabricated using resin composite (Filtek™Z350TM XT, 3M ESPE, St. Paul, MN, USA; shade A1) in epoxy resin molds (Fig. 1A) then covered with a celluloid strip (LEIBINGER™ Mylar Strip), compressed with a glass slide, and each specimen was separately light-cured using an LED light-curing unit (Demi™ Plus, Kerr Corp, Orange, CA, USA) for 40 sec. The light output intensity was ≥ 800 mW/cm^2 (evaluated by a radiometer (Kerr; Orange, CA, USA)). The prepared specimens were thermocycled for 5,000 cycles between 5 and 55°C with a 30-sec dwell time and a 10-sec transfer time to simulate 6-month clinical service.

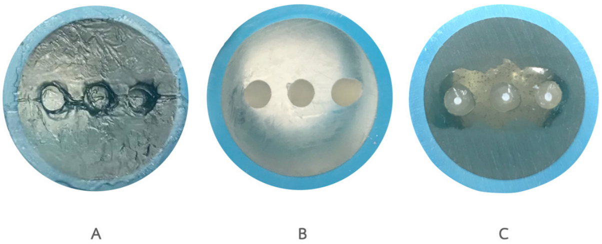

Figure 1 *Resin composite specimen preparation (A) the three epoxy resin molds size 3-mm diameter and 2-mm depth created within polyvinyl chloride (PVC) tube. (B) resin composite specimens. (C) three clear plastics tubes placed on the resin composite specimens and filled with the repair resin composite*

The specimens were polished using wet 320-grit silicon carbide paper to create a standardized surface. Thirty-two percent phosphoric acid (Scotchbond™ Etchant, 3M ESPE, USA) was applied on the specimens in a brushing motion for 15 sec, rinsed for 15 sec, and air dried. The specimens were randomly divided into 4 groups (n=18) according to the surface treatments and the adhesives used; Group 1: Adper™ Scotchbond™ Multi-Purpose Adhesive (3M ESPE, USA) (SM), Group 2: treated with RelyX™ Ceramic Primer (3M ESPE, USA) and Adper™ Scotchbond™ Multi-Purpose Adhesive (SMS), Group 3: Single Bond™ Universal Adhesive (3M ESPE, Germany) (SUN), Group 4: treated with RelyX™ Ceramic Primer and Single Bond™ Universal Adhesive (SUS). The materials were applied individually on each specimen according to the manufacturers' instructions as shown in Table 1.

Table 1 Materials, composition, and application methods used in the study (Modified from the manufacturers' Material Safety Data Sheet and instructions for use)

Materials	Composition	Application Methods
Scotchbond™ Etchant (3M ESPE, MN, USA)	Water, phosphoric acid, alcohol, thickening agent	- Apply etchant to aged resin composite surfaces for 15 sec. - Thoroughly rinse for 15 sec. - Air dry
Adper™ Scotchbond™ Multi-Purpose Adhesive (3M ESPE, MN, USA :SM)	Bis-GMA, HEMA, initiator	- Apply adhesive to the etched and dried resin composite surfaces. - Light-cure for 20 sec
Single Bond™ Universal Adhesive (3M ESPE, Germany :SU)	MDP Phosphate Monomer, Di-methacrylate resins, HEMA, Vitrebond copolymer, ethanol, water, filler, initiators, silane	- Apply adhesive to the etched and dried resin composite surfaces for 20 s - Air dry for 5 sec and light-cure for 20 sec.
Filtek™ Z350 XT (3M ESPE, MN, USA)	Bis-GMA, UDMA, TEGDMA, Bis-EMA, non-aggregated 4 to 11 nm zirconia, non-aggregated 20 nm silica and aggregated zirconia/silica cluster filler (63.3 vol%)	- Apply a 2-mm. thick layer. - Light-cure for 40 sec.
RelyX™ Ceramic Primer (3M ESPE, MN, USA)	Pre-hydrolyzed silane coupling agent, alcohol, water	- Apply RelyX Ceramic Primer with a brush on the etched and dried resin composite surfaces for 1 min. - Air dry

Abbreviations: bis-GMA (bisphenol A-glycidyl methacrylate), bis-EMA (bisphenol A diglycidyl methacrylate ethoxylated, HEMA (hydroxyethylmethacrylate), MDP (methacryloxydecyl phosphate). TEGDMA (triethylene glycol dimethacrylate), UDMA (urethane dimethacrylate)

After surface treatment and adhesive application, a clear cylindrical plastic tube, 0.7 mm in-ternal diameter x 1.0 mm high (Tygon tubing, Norton Performance Plastic Co, USA) was placed on the resin composite specimen and the adhesive was light-cured for 20 sec. The light output intensity was ≥ 800 mW/cm2. After curing, each tube was filled with nanohybrid resin composite shade A4 (Fil-tek™ Z350XT, 3M ESPE, USA) and light-cured for 40 sec (Fig. 1C).

The specimens were stored in water at 37°C for 24 h. The plastic tubes were removed and specimens were evaluated using 2.5x magnification loupes (ZEISS EyeMag Smart, Carl Zeiss Meditec AG, Germany) to evaluate the integrity of the old and new resin composite interface. Any specimens with interfacial gaps, bubbles, or any defect were excluded from the study.

For each group, half of the specimens (n=9) were subjected to the μSBS test immediately after removing the tubes. The other half of the specimens were subjected to a 5,000-cycle thermocycling between 5°C and 55°C. The dwell time and transfer time were 30 sec and 10 sec, respectively.

The specimens were mounted on a universal testing machine (EZ-S; Shimadzu, Japan), and a 0.4-mm thick blade was placed parallel and adjacent to the old and new resin composite interface and specimens were tested to failure at a crosshead speed of 0.5 mm/min. The mode of failure was analyzed using a stereomicroscope at 45x magnification and categorized as adhesive failure; failure at the bonding interface, cohesive failure; failure in either the substrate or repair resin composite or mixed failure; failures in both adhesive and resin composite.

Statistical analysis

The μSBS data were analyzed by Three-way ANOVA and Tukey's post hoc test (*p*=0.05) using IBM SPSS Statistics Subscription, Build 1.0.0.1508 (SPSS Inc., Chicago, IL).

Results

The μSBS test results are presented in Table 2. Three-way ANOVA indicated that thermocycling and silanization significantly affected the μSBS ($p<0.001$ and $p=0.001$, respectively). In contrast, the adhesive system did not affect μSBS ($p=0.750$). There was no interaction between the adhesive system, silanization, and thermocycling ($p=0.121$).

After 24-h water storage, the SMS group had a significantly higher μSBS compared with that of the other groups ($p<0.05$). In the thermocycled subgroup, the SUS group demonstrated a higher μSBS than that of the other groups; however, the difference was not significant.

Considering the effect of thermocycling within the same group, only the μSBS of the non-thermocycled SMS group was significantly higher than that of its thermocycled counterpart ($p<0.05$). In contrast, μSBSs between the other non-thermocycled groups and their respective thermocycled groups were not significantly different.

Considering the effect of silanization on each adhesive system, silanization significantly increased the μSBS in the conventional adhesive group ($p<0.05$). However, silanization in the silane-containing adhesive did not significantly increase the μSBS.

The failure modes are shown in Figure 2. Adhesive failure was predominantly found in all groups, with a few mixed failures, but no cohesive failure was seen.

Table 2 *μSBS values (mean ± SD)*

Group	Abbreviation	μSBS ± SD (MPa) 24 h water storage	μSBS ± SD (MPa) 5,000-thermocycled
Scotchbond™ Multi-Purpose Adhesive	SMN	12.91 ± 3.92 ^{a,A}	7.82 ± 1.69 ^{c,A}
Silane + Scotchbond™ Multi-Purpose Adhesive	SMS	23.72 ± 8.89 ^{b,B}	12.21 ± 2.34 ^{c,C}
Single Bond™ Universal Adhesive	SUN	14.79 ± 6.35 ^{a,D}	12.92 ± 4.55 ^{c,D}
Silane + Single Bond™ Universal Adhesive	SUS	14.44 ± 2.39 ^{a,E}	13.10 ± 2.72 ^{c,E}

Different letters indicate a significant difference between groups (p>0.05); UPPERCASE letters indicate a significant difference in each row (significantly different μSBS between 24 h water storage and 5,000-thermocycled of the same group) and lowercase letters indicate significant differences in each column (significantly different μSBS among the groups after 24-hour water storage or 5,000-cycle thermocycling)

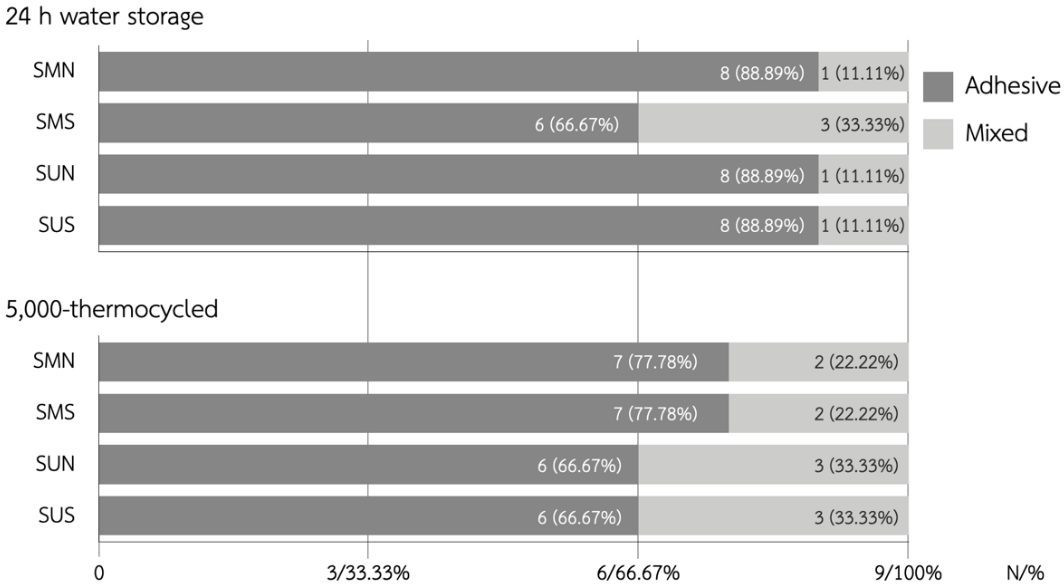

Figure 2 *The mode of failure (n/%)*

Discussion

In the present study, the μSBS test was selected to overcome the limitations of the microtensile bond strength test, which has the difficulty in specimen preparation and the high number of pre-test failures.[12] Moreover, sample preparation method for the μSBS test generates lower stress at the bond interface before testing, except for the mold removal.[13-15] Consistent with this, the present study had no pre-test failures. Furthermore, adhesive failure was the predominant failure mode which corresponds with the suggestion that adhesive failures indicate that using the μSBS test is valid.[16]

The bond between two layers of resin composite occurs between unreacted methacrylate groups (c=c) in each layer in the oxygen-inhibited layer.[8,17] However, after polishing and being in the oral environment, resin composite restorations lack the free radicals and unreacted methacrylate groups needed to bond with the repair resin composite.[18] Thus, aging the resin composite before bonding with the repair resin composite is important to simulate the clinical situation. In the present study, thermocycling was used to age the resin composite as described in the previous study.[19]

Bonding between a restoration and a repair resin composite requires free radicals and unreacted methacrylate groups, which are not present on restoration that has been in the oral cavity for some times. Therefore, surface preparation to expose the filler particles and create surface irregularities on the restoration is crucial for a successful repair.[8,18] Wet grinding with 320-grit sandpaper, which is comparable to a fine diamond bur, was chosen to establish uniform repair surfaces and to expose the filler particles.[7] The surface was then cleaned with phosphoric acid[20] and applied with adhesive to increase wettability and create micromechanical retention via the penetrated and polymerized resin into the prepared surface.[8,21]

In addition to forming an effective bond with the original resin composite, the survival of the repair restoration is an important clinical parameter to be evaluated. Thus, thermocycling at 5,000 cycles, which is equal to 6-month clinical service[22], was selected to induce thermal stress and hydrolytic degradation of the old and new resin composite interface. This artificial aging process allows the long-term clinical bonding performance to be evaluated *in vitro.*

The 3-step etch-and-rinse adhesive was used as the control group due to favorable laboratory and clinical results.[15] The results of the present study indicated that the repair bond strength of the silane-containing universal adhesive was not significantly different from that of the conventional etch-and-rinse adhesive in the non-thermocycled and the thermocycled groups. The μSBS of the adhesives evaluated in this study were not significantly different between the non-thermocycled and the thermocycled groups, except for the SMS group. Silanization significantly increased the μSBS in only the non-thermocycled etch-and-rinse adhesive group.

This study revealed that the repair bond strength of a silane-containing universal adhesive was comparable to that of a conventional adhesive. These results might be due to additional chemical bonding between the exposed filler particles and the silane coupling agent and the 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer in the silane-containing universal adhesive. According to the manufacturer, the resin composite used in this study contains 78.5% by weight (63.3% by volume) inorganic fillers, which are a combination of non-agglomerated/non-aggregated silica filler, non-agglomerated/non-aggregated zirconia filler and aggregated zirconia/silica clusters. Prior studies demonstrated that silica fillers chemically reacted with a silane coupling agent[7,8], whereas zirconia fillers chemically bonded to 10-MDP.[23,24] These reactions may improve the repair bond strength of a silane-containing universal adhesive.

Studies have demonstrated that silanization improves the repair bond strength of the conventional methacrylate resin adhesive between the aged and repair resin composite.[7,8,11] Silane coupling agents are bifunctional molecules that contain silanol groups that react with the exposed inorganic filler particles in the resin composite and organofunctional groups that react and co-polymerize with the methacrylate groups of the adhesive.[9,24] The silane-coated surface is reactive and forms covalent bonds with the adhesive. Moreover, silane increases the wettability of the aged resin composite surface, which improves the infiltration of the resin adhesive into surface irregularities.[6,7,9,18] Corresponding with the results of the present study, silanization significantly increased the repair bond strength of a conventional adhesive when tested immediately and tended to increase after aging because the silanol groups form a covalent bond with the exposed inorganic filler, i.e. silica filler.[24] In contrast, silani-zation did not significantly improve the repair bond strength of a silane-containing universal adhesive in the non-thermocycled and the thermocycled groups. These findings are in line with those of prior studies.[24] This may be because the exposed silica filler particles can interact with the silane and be insufficient in number to form an additional chemical bond with the silane in a silane-containing universal adhesive.[18] However, some studies have found that silanization improves the repair bond strength of a silane-containing universal adhesive.[7,8,25] These discrepancies are accounted for by the fact that the effect of silanization on a silane-containing adhesive varies depending on the surface treatment and silane composition.[8]

Although 5,000-thermocycles greatly reduced the μSBS of a conventional adhesive, the μSBS of the silane-containing adhesive was only slightly reduced. This could be due to the positive effect of the stable chemical bonds between the 10-MDP in the silane-containing universal adhesive and the exposed zirconia filler.[23,24] Conversely, the reduced μSBS in the conventional adhesive group may result from bond degradation over time because it contains only Bis-GMA and HEMA, which do not promote a stable chemical bond as 10-MDP.[9] Moreover, a prior study demonstrated that the bond between phosphate esters, i.e. 10-MDP and the zirconia surface hydroxyl groups, was more hydrolytically stable compared with the bond between silane coupling agents and old resin composite.[7,9,27]

Incorporating 10-MDP into an adhesive might have a positive effect on the repair bond strength of a resin composite with zirconia filler because 10-MDP forms a chemical bond with oxide groups, such as SiO_2, Al_2O_3, and ZrO.[21,27] Therefore, 10-MDP in the silane-containing universal adhesive may improve the repair bond strength of a resin composite containing silica or alumina. However, the surface preparation and the type of adhesive and silane coupling agent used can also affect the repair bond strength. Furthermore, several studies demonstrated that different resin composite compositions, such as the type and amount of fillers, also influence the repair bond strength.[21,28] Future studies evaluating various resin composites are needed to determine the effect of 10-MDP in a silane-containing universal adhesive on the repair bond strength.

The present study has some limitations. The sample size in this study is small. The larger the sample size, the more power the study would be. In addition, the *in vitro* results of our study might not correlate with clinical outcomes. Furthermore, the type of adhesive and silane coupling agent used could have an effect on the repair bond strength. The present study evaluated only one silane-containing universal adhesive and one resin composite; thus the results may not be applicable to other similar materials. Further investigations are needed to establish a standard resin composite repair protocol. However, based on our results, the examined silane-containing universal adhesive is a viable choice for resin composite repair.

Conclusion

Within the limitations of this in vitro study, several conclusions can be drawn. The repair bond strength of a silane-containing universal adhesive is comparable to that of an etch-and-rinse adhesive. Silanization increases the repair bond strength of an etch-and-rinse adhesive, but does not affect that of the silane-containing universal adhesive. Aging using 5,000 thermocycles has no effect on the repair bond strength of the silane-containing universal adhesive on aged resin composite.

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Conflicts of Interest

There are no conflicts of interest declared by the authors. The funder was not involved in any part of the research process.

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