

# Does use of silane-containing universal adhesive eliminate the need for silane application in direct composite repair?

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**Abstract:** This *in vitro* study aimed to evaluate the effect of a silane-containing universal adhesive used with or without a silane agent on the repair bond strength between aged and new composites. Forty nanohybrid composite resin blocks were stored in distilled water for 14 d and thermo-cycled. Sandpaper ground, etched, and rinsed specimens were randomly assigned into four experimental groups: silane + two-step etch-and-rinse adhesive system, two-step etch-and-rinse adhesive system, silane + silane-containing universal adhesive system, and silane-containing universal adhesive system. Blocks were repaired using the same composite. After 24 h of water storage, the blocks were sectioned and bonded sticks were submitted to microtensile testing. Ten unaged, non-repaired composite blocks were used as a reference group to evaluate the cohesive strength of the composite. Two-way ANOVA and Tukey's tests were used to analyze average  $\mu$ TBS. One-way ANOVA and Dunnett post-hoc tests were used to compare the cohesive strength values and bond strength obtained in the repaired groups ( $\alpha = 0.05$ ). The  $\mu$ TBS values were higher for the silane-containing universal adhesive compared to the two-step etch-and-rinse adhesive system ( $p = 0.002$ ). Silane application improved the repair bond strength ( $p = 0.03$ ). The repair bond strength ranged from 39.3 to 65.8% of the cohesive strength of the reference group. Using universal silane-containing adhesive improved the repair bond strength of composite resin compared to two-step etch-and-rinse adhesive. However, it still required prior application of a silane agent for best direct composite resin repair outcomes.

**Keywords:** Tensile Strength; Dental Restoration Repair; Dental Restoration, Permanent.

## Introduction

The annual failure rate of composite resin restorations varies from 1 to 5%<sup>1</sup> in permanent teeth and 1.7 to 12.9%<sup>2</sup> in primary dentition. Basically, the management of defective restorations includes two options: replacement or repair of the restoration.<sup>3</sup> Although clinical criteria for evaluating direct restorations have been proposed,<sup>4</sup> it is not clear which clinical situations one must choose repair or replacement of defective restorations.<sup>5,6</sup> The general consensus tends toward restoration repair given its numerous advantages, such as preservation of dental structure and reduction of



treatment costs.<sup>3</sup> On the other hand, re-restoring teeth can require more complex restorative procedures that raise the risk of initiating a re-intervention cascade, known as the restorative cycle.<sup>7</sup> Although most dentists claim to perform repairs, and this approach has been adopted by most dental schools, the proportion of truly repaired restorations is still low.<sup>8</sup> Repair may increase the survival of restorations placed in primary and permanent teeth,<sup>9,10</sup> but there is no gold standard protocol or materials established for treating aged composite surfaces before repair.<sup>11</sup>

Successful repair procedures require a durable bond between the old restoration and the new composite resin. New composite may be adhered to aged composite through micromechanical interlocking to irregularities in the prepared surface and through chemical bonding to the filler particles and the organic matrix.<sup>12,13,14</sup> Bonding agents improve the chemical bonds between the old and new materials. Silanes increase surface wetting, thereby enhancing diffusion of the bonding agent into the substrate.<sup>15</sup> Silane coupling agents may also form covalent bonds with filler particles and co-polymerize with the methacrylate groups present in repair material.<sup>16</sup> A recent systematic review showed that application of silane coupling agents and adhesives play a role in improving repair bond strength when physical treatments, such as burs, were applied to the aged composite.<sup>12</sup>

Silane agents have been directly incorporated into adhesive systems. Use of a silane-containing universal adhesive could eliminate the need to apply silane as a separate step during the clinical protocol for composite repair. Evidence about effect of a silane-containing universal adhesive associated or not with a silane agent on the repair bond strength of composite is limited and the results are contradictory.<sup>17,18</sup> Therefore, the current study sought to evaluate the effect of a silane-containing universal adhesive used with or without a silane agent on the repair bond strength between aged and new composites. The hypothesis was that use of a silane-containing universal adhesive would eliminate the silane application for direct composite repair.

## Methodology

A silane coupling agent (RelyX Ceramic Primer, 3M Oral Care, St. Paul, USA) and two adhesive systems were tested: a two-step etch-and-rinse adhesive system (Adper Single Bond Plus, 3M Oral Care, St. Paul, USA) and a silane-containing universal adhesive system (Scotchbond Universal Adhesive, 3M Oral Care, St. Paul, USA). A1E and A3B shades of the nanohybrid composite resin (Filtek Z350 XT, 3M Oral Care, St. Paul, USA) were used in order to differentiate between the aged and new composite resin. A detailed description of the materials is presented in Table 1.

**Table 1.** Composition and application mode of the materials tested.

RelyX Ceramic Primer (3M Oral Care, St. Paul, USA) #Batch number 1720700505	Methacryloxypropyl trimethoxysilane; water; ethyl alcohol 3-(trimethoxysilyl methacrylate)	Apply one coat of silane  Gently air dry for 5 s
Scotchbond Universal Adhesive (3M Oral Care, St. Paul, USA) #Batch number 1809600708	Etchant: 37% phosphoric acid, MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	Apply the adhesive for 20 s with vigorous agitation Gentle air thin for 5 s Light-cure for 10 s
Adper Single Bond Plus (3M Oral Care, St. Paul, USA) #Batch number 1812300361	Etchant : 37% phosphoric acid  HEMA, water, ethanol, Bis-GMA, dimethacrylates, amines, metacrylate-functional copolymer of polyacrylic and polyitaconic acids, 10% by weight of 5 nanometer-diameter spherical silica particles	Apply 2 consecutive coats of adhesive for 15 s with gentle agitation  Gently air dry for 5 s  Light-cure for 10 s
Z350 XT A1E e A3B Shades (3M Oral Care, St. Paul,, USA) #Batch numbers 1729300455, 1732800739	Bis-GMA, UDMA, TEGDMA, Bis-EMA, non-agglomerated/ non-aggregated 20 nm silica filler, non-agglomerated/ non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler	Insert the composite in 2 mm increments  Light-cure for 20 s

MDP: 10-methacryloyloxydecyl-dihydrogen-phosphate; Bis-GMA: bisphenyl-glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-EMA: ethoxylated bisphenol-A dimethacrylate; UDMA: urethane dimethacrylate

### Preparation of aged composite blocks

Forty blocks of nanohybrid composite resin (A1E shade) measuring 8 x 8 mm in depth and width and 4 mm in height were fabricated using a metallic mold (8 x 8 x 8 mm). The mold was fixed on a glass slab. Composite resin was packed into the mold in two increments that were each light cured for 20 s with a light-emitting diode curing unit (Radii-cal; SDI, Victoria, AUS) with a light output of at least 1,250 mW/cm<sup>2</sup>. Light intensity output was monitored with a Demetron Curing Radiometer (Kerr, Orange, USA). The composite was carefully condensed with a clean filling instrument in order to avoid contamination and void entrapment. After setting, composites were gently removed from the mold and the thickness of each block was confirmed with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan). The specimens were stored in distilled water at 37°C for 14 d<sup>19</sup> prior to aging. The blocks were further aged by thermal cycling 5,000 times between 5 and 55°C, with a dwell time of 20 s and transfer time of 3 s.<sup>19</sup> The aged specimen surfaces were wet-ground with 320-grit silicon carbide grinding paper for 5 s to remove the superficial resin-rich layer and create standardized repair surfaces.<sup>19,20</sup> All specimen surfaces were then etched with 37% phosphoric acid for 30 s, washed with air/water spray for 60 s, and dried with a blast of air for 60 s.<sup>18</sup>

### Bonding procedures

The 40 aged blocks were randomly assigned (Random Allocation software, version 1.0, Iran) into four experimental repair protocol groups (n = 10): silane + two-step etch-and-rinse adhesive system, two-step etch-and-rinse adhesive system, silane + silane-containing universal adhesive system, and silane-containing universal adhesive system. All materials were applied according to the manufacturer's recommendations (Table 1). The aged composite blocks were carefully placed over the original mold and then repaired using nanohybrid composite resin (A3B shade). Resin was applied in two incremental layers that were each light cured for 20 s following the same protocol as the aged specimens. This process resulted in 8-mm high specimens. Upon removal from the mold, the specimen surfaces covered by the mold were further cured for 20 s. Specimens were stored

in distilled water at 37°C for 24 h. A single trained operator carried out all procedures.

### Microtensile bond strength ( $\mu$ TBS)

To guarantee that the testing machine operator was blinded, each composite block was numbered according to the randomization sequence. Blocks were sectioned into sticks with a cross-sectional area of approximately 0.8 mm<sup>2</sup> using a water-cooled diamond saw in a cutting machine (Isomet, Buehler, Lake Bluff, USA). Approximately 40 sticks were obtained for each block. The sticks were carefully examined with a stereomicroscope (HMV-2, Shimadzu Corp., Kyoto, Japan) at 40× magnification. Those with interfacial flaws, gaps, bubbles, or other defects were discarded. The cross-sectional area of each stick was measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) to calculate the bond strength values, measured in MPa. Pretesting failures were not observed. The bonded sticks were attached to a universal testing machine for microtensile testing (EZ-SX series, Shimadzu Corp., Kyoto, Japan) with cyanoacrylate and tested at a crosshead speed of 1mm/min. The  $\mu$ TBS, measured in MPa, was obtained by dividing the load at failure (N) by the cross-sectional area (mm<sup>2</sup>) of each stick.

### Failure mode

The fracture surfaces were examined under a stereomicroscope (HMV-2, Shimadzu Corp., Kyoto, Japan) at 40× magnification to determine whether the failure mode was adhesive (failure between restorative material and bonding agent or between bonding agent and repair composite) or cohesive (failure exclusively within the aged or new composite resin). A examiner blind to experimental groups evaluated the failure mode.

### Cohesive strength of non-aged composite – reference group

Ten blocks of nanohybrid composite resin (A1E shade) measuring 8x8x8 mm were fabricated using a metallic mold. The mold was fixed on a glass slab. Composite was packed into the mold in four increments that were each light cured for 20 s with

a light-emitting diode curing unit (Radii-cal; SDI, Victoria, AUS). After inserting the last increment, the Mylar strip was pressed down over the mold for 30 s and the specimen was light cured through the strip. The thickness of each specimen was verified with a digital caliper. Specimens were stored in distilled water at 37°C for 24 h then prepared for the  $\mu$ TBS test. Figure summarizes the experimental design.

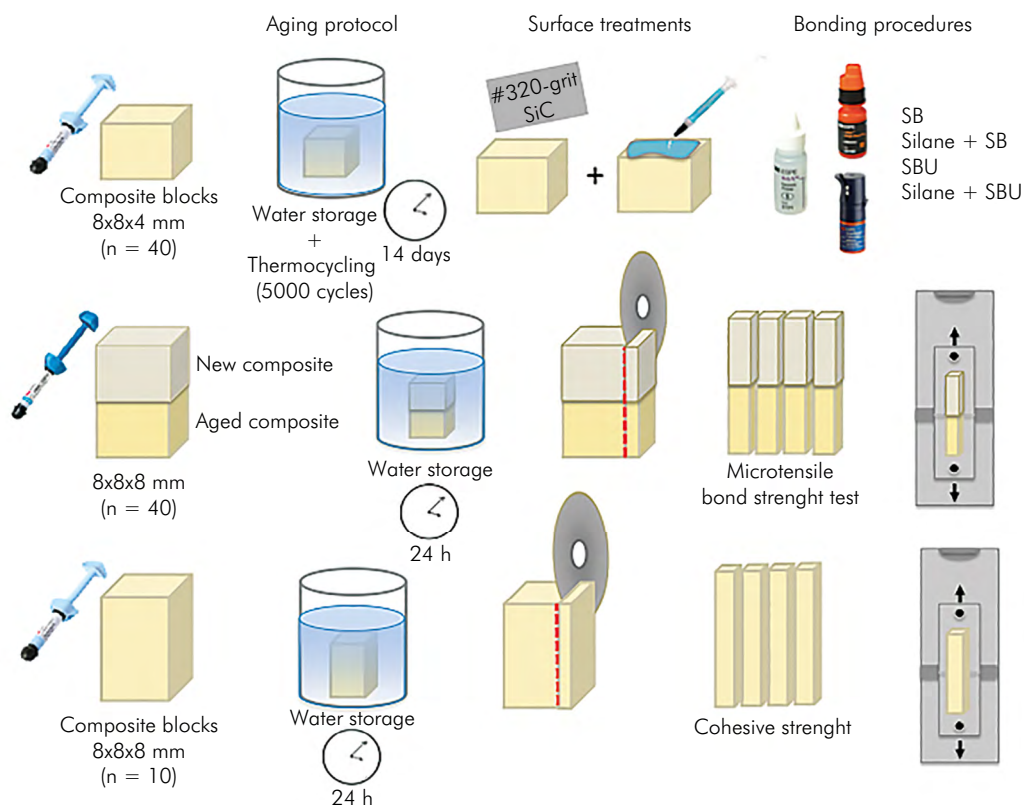
### Statistical analysis

The experimental unit was the resin block. Thus, the  $\mu$ TBS values from every stick from the same block were averaged for statistical analysis. Sticks with cohesive failures from the repaired groups were not included in the analysis. The  $\mu$ TBS mean for each test group represents the mean of the ten blocks used per group. The ten blocks sample size was previously estimated using the following parameters: 80% power, a coefficient of variation of 20%, and assuming a two-sided 5% significance level for comparisons.

Normal data distribution was confirmed using a Kolmogorov-Smirnov test. The  $\mu$ TBS means of the repaired groups were analyzed by two-way ANOVA and Tukey's *post-hoc* tests. One-way ANOVA and Dunnett *post-hoc* tests were used to compare the cohesive strength values and bond strength obtained in the repaired groups. The significance level was set at  $p < 0.05$ . Statistical analyses were performed using Minitab18 software (Minitab Inc., State College, USA).

### Results

The  $\mu$ TBS means, standard deviations, and distribution of the failure mode for all experimental groups are shown in Table 2. Cohesive strength values of non-aged specimens were higher than bond strength obtained in all repaired groups ( $p < 0.01$ ). Repair bond strength ranged of 39.3% to 65.8% of cohesive strength of the reference group.



SB: Adper Single Bond Plus, SBU: Scotchbond Universal Adhesive.

**Figure.** Experimental design of the study.

**Table 2.** The microtensile bond strength means (MPa), standard deviations, and distribution of the failure mode for all experimental groups

Experimental groups	Bond strength	% in relation to cohesive strength values	Failure mode		
			Adhesive	Cohesive (old composite)	Cohesive (new composite)
Adper Single Bond Plus	27.5 ± 10.5 <sup>B</sup>	39.3%	90.6%	4.9%	4.5%
Silane + Adper Single Bond Plus	35.7 ± 3.7 <sup>B</sup>	51.1%	87.7%	7.3%	5.0%
Scotchbond Universal Adhesive	38.7 ± 10.5 <sup>B</sup>	55.4%	93.7%	4.3%	2.0%
Silane + Scotchbond Universal Adhesive	46.0 ± 8.4 <sup>B</sup>	65.8%	83.0%	10.0%	7.0%
Cohesive strength – Reference group	69.9 ± 17.8 <sup>A</sup>				

\*Different capital superscript letters indicate statistically significance differences between cohesive strength values and bond strength values of the repaired groups ( $p < 0.05$ ).

Main factors “adhesive system” ( $p = 0.002$ ) and “silane coupling agent” ( $p = 0.03$ ) were statistically significant. On the other hand, the cross-product interaction “adhesive system *vs.* silane coupling agent” was not statistically significant ( $p = 0.85$ ).

The  $\mu$ TBS values were higher for the silane-containing universal adhesive compared to the two-step etch-and-rinse adhesive system. Previous silane application improved the repair bond strength, irrespective of the adhesive system (Table 3).

## Discussion

Both silane coupling agents and adhesive systems appeared to establish an adequate bond strength between the aged composite and the new composite.<sup>12</sup> Considering the “universal application” idea behind these contemporary all-in-one adhesives, use of a silane-containing universal adhesive for composite repair would simplify the clinical protocol, thereby reducing chair time and operator errors. In the current study, silane-containing universal adhesive used with previous acid etching produced higher repair bond strength values compared to the two-step etch-and-rinse adhesive system.

Composite surfaces aged *in vitro* show superficial dissolution and increased surface roughness, which may contribute to mechanical entanglement of the

adhesive systems.<sup>21</sup> Additionally, Scotchbond Universal Adhesive contains 10-MDP, a functional monomer that can chemically bond to zirconia surface.<sup>22</sup> Considering the zirconia content of fillers in Z350 XT, the 10-MDP monomer may help to promote repair bond strength by providing additional chemical bonding. However, applying silane separately improved the repair bond strength, irrespective of the adhesive. Therefore, the tested hypothesis was rejected.

Removing the superficial layer from an old composite and roughening it with a diamond bur is necessary to obtain micromechanical retention. In laboratory studies, the standardized surface roughness is obtained using 320-grit silicon carbide grinding paper, which simulates the roughness obtained with a medium diamond bur.<sup>19,20</sup> This physical treatment can dissolve or remove the polymer matrix covering the glass fibers or particles and create a state where silane coupling agents can interact with silica. Degradation of dental composites during storage can also break filler-polymer bonds, allowing for surface loss of glass particles.<sup>23</sup> Although there is currently no consensus on an aging method that can completely imitate clinical conditions, the current study chose to age the composite resin using water storage for 14 d followed by 5,000 thermocycles.<sup>19</sup>

Silane coupling agents promote chemical bonding by forming siloxane bonds between silicate-containing

**Table 3.** The microtensile bond strength means (MPa) and respective standard deviations considering the main factors

Adhesive system	Bond strength	Silane coupling agent	Bond strength
Adper Single Bond Plus	31.6 ± 10.0 <sup>B</sup>	With silane	40.8 ± 9.8 <sup>A</sup>
Scotchbond Universal Adhesive	42.3 ± 7.4 <sup>A</sup>	Without silane	33.1 ± 9.3 <sup>B</sup>

Different capital superscript letters indicate statistically significant differences for each factor, separately ( $p < 0.05$ ).

filler particles exposed on the repair surface and the resin matrix of a fresh resin layer.<sup>24</sup> Additionally, silanes have greater surface wettability, facilitating adhesive penetration into surface defects<sup>25</sup> and improving the repair bond strength. Scotchbond Universal adhesive contains prehydrolyzed silane that the manufacturer claims is stable up to at least one year in storage. However, the amount of silane in its composition is not reported by the manufacturer and may be not sufficient to improve the repair bond strength.

A previous study<sup>17</sup> also found that silane surface treatment improved  $\mu$ TBS of a silane-containing universal adhesive before composite placement. Conversely, a other study<sup>18</sup> reported that silane-containing universal adhesive alone was as effective as any combination of silane and adhesive. This contradictory finding may be due to methodological differences related to bond strength test (microshear versus microtensile) and type of composite resin (nanofilled versus nanohybrid). Differently of the composite resin Z350XT, Filtek Supreme Ultra Restorative composite (3M Oral Care) contains silane-treated ceramic, silane-treated silica and silane-treated zirconia. It has been reported that incorporation of silanized filler particles in the resin matrix improves the physical and mechanical properties of resin composites in terms of mechanical strength and hydrolytic stability<sup>26</sup> and it may have an influence in the surface treatment for repair.

Repair bond strength is measured as the maximum force prior to specimen fracture. If a large percentage of specimens are cohesively fractured, few conclusions can be drawn regarding repair bond strength because bond strength is usually lower than cohesive strength. The majority of failures observed in the current study across all experimental groups were adhesive. We used the cohesive strength of composites that were not aged as a reference for the desired or optimal repair strength. The cohesive strength of new material is unrealistic in aged specimens because composites gradually lose strength as they age.<sup>19</sup> The repair bond strength

for each substrate material ranged from 39.3 to 65.8% of the cohesive strength of the reference group. The silane paired with silane-containing universal adhesive repair protocol resulted in the bond strength that was closest to the cohesive strength of the reference group. Thus, the probability of failures at the composite–repair interface such as fractures could be minimized. It should be emphasized that the repair procedure for direct composites involves a dental structure in most clinical scenarios. Scotchbond Universal Adhesive shows satisfactory bonding to enamel and dentin.<sup>27</sup> Therefore, bonding between a silane-containing universal adhesive and dental substrate, which usually involves repairing a restoration, could help minimize the need for prior silane application to the surface composite. The likelihood of obtaining a chemical bond to a composite substrate slowly decreases over time due to post-curing and water uptake. This leads to hydrolysis of available double bonds and few carboxyl groups for chemical bonding to a new composite.<sup>28</sup> Furthermore, one-bottle prehydrolyzed silane solutions, such as RelyX Ceramic Primer, have a relatively short shelf life and gradually become less reactive after the bottle has been opened, thereby preventing optimal adhesion<sup>29</sup>. Future studies evaluating the use of silane as a pretreatment or incorporating it with the adhesive to enhance repair durability are necessary in order to recommend a universally applicable repair protocol. It is important to note that the current results are limited to the materials used in this study and may not apply to other silane-containing universal adhesives.

## Conclusions

Under these study conditions, use of a universal silane-containing adhesive improved the repair bond strength of composite resin compared to a two-step etch-and-rinse adhesive. However, it still required prior application of a silane agent for best direct composite resin repair outcomes.

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