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Effect of Surface Treatment, Silane, and Universal Adhesive on Microshear Bond Strength of Nanofilled Composite Repairs

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Clinical Relevance

The use of a silane-containing universal adhesive eliminates the need to apply silane as a separate step in the clinical protocol for composite repair.

SUMMARY

Purpose: The aim of this study was to evaluate the effect of surface treatment and universal adhesive on the microshear bond strength of nanoparticle composite repairs.

Methods: One hundred and forty-four specimens were built with a nanofilled composite (Filtek Supreme Ultra, 3M ESPE). The surfaces of all the specimens were polished with SiC paper and stored in distilled water at 37°C for 14 days. Half of the specimens were then air abraded with Al_2O_3 particles and cleaned with phosphoric acid. Polished specimens (P) and polished and air-abraded specimens (A), respectively, were randomly divided into two sets of six groups (n=12) according to the following treatments: hydrophobic adhesive only (PH and AH, respectively), silane and hydrophobic adhesive (PCH, ACH), methacryloyloxydecyl dihydrogen phosphate (MDP)–containing silane and hydrophobic adhesive (PMH, AMH), universal adhesive only (PU, AU), silane and universal adhesive (PCU, ACU), and MDP-containing silane and universal adhesive (PMU, AMU). A cylinder with the same composite resin (1.1-mm diameter) was bonded to the treated surfaces to simulate the repair. After 48 hours, the specimens were subjected to microshear testing in a universal testing machine. The failure area was analyzed under an optical microscope at 50× magnification to identify the failure type, and the data were analyzed by three-way analysis of variance and the Games-Howell test (α=0.05).

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Results: The variables "surface treatment" and "adhesive" showed statistically significant differences for $p<0.05$. The highest mean shear bond strength was found in the ACU group but was not statistically different from the means for the other air-abraded groups except AH. All the polished groups except PU showed statistically significant differences compared with the air-abraded groups. The PU group had the highest mean among the polished groups. Cohesive failure was the most frequent failure mode in the air-abraded specimens, while mixed failure was the most common mode in the polished specimens.

Conclusions: While air abrasion with Al_2O_3 particles increased the repair bond strength of the nanoparticle composite, the use of MDP-containing silane did not lead to a statistically significant increase in bond strength. Silane-containing universal adhesive on its own was as effective as any combination of silane and adhesive, particularly when applied on air-abraded surfaces.

INTRODUCTION

Composite resins are now routinely used in dentistry because of their aesthetic and adhesive characteristics and their superior ability to preserve sound tooth structure.[1-3] However, conditions in the oral environment, such as temperature and pH changes, diet, and other factors, may cause resin composites to degrade.[4,5] *In vivo* degradation can lead to discoloration, microleakage, wear, chipping, and even fracture of the restoration.[6] Nevertheless, if the defects are minor, such as loss of anatomical shape and extrinsic discoloration, the restoration may not require total replacement.[7] In such situations, repair of the restoration is more appropriate, as it is minimally invasive and a fast, simple procedure.[8] Furthermore, composite repair has been reported to be as effective as total replacement and to cause less tooth structure removal, increasing restoration longevity and reducing the cost of treatment.[7-11]

The success of the repair procedure in composites depends on several factors, such as the characteristics of the surface, the wettability of the chemical bonding agents, and the chemical composition of the composite.[10,12,13] A rough surface is crucial in composite repair and can be achieved mechanically by the use of highly abrasive diamond burs, air abrasion with aluminum oxide particles, or chemically by hydrofluoric acid etching.[14] In addition to micromechanical retention, a chemical bond is desirable to enhance the bond strength of composite repairs. Dental composites usually consist of filler particles, such as silica, lithium aluminum silicates, hydroxyapatite, and boron silicates. The application of a silane agent can increase the bonding between the fillers and the organic resin matrix.[15-19] However, as some commercially available composites contain zirconia particles in addition to the traditional glass and ceramic fillers, further studies are required into the role of silane in the repair of these materials.

Adhesives also seem to exert an influence on composite repair bond strength. Their role is to increase the wettability of the mechanically treated, silanized surface.[20] Recently, a new category of adhesives called multimode or universal adhesives was launched. These contain a phosphate monomer (methacryloyloxydecyl dihydrogen phosphate [MDP]) and silane in addition to conventional functional monomers.[21] However, there is a dearth of studies in the literature on the impact of such adhesives on composite repair bond strength.[22,23]

This study therefore sought to analyze the effect of a universal adhesive used with and without a conventional and an MDP-containing silane agent on the repair bond strength of a silica/zirconia nanofilled composite. The null hypothesis was that there would not be any differences between the treatments when they were used to repair a nanofilled composite.

METHODS AND MATERIALS

A total of 144 truncated cone-shaped specimens (7.5-mm top diameter; 4.5-mm bottom diameter; 3-mm thickness) were built with an A2 enamel shade nanofilled resin composite (Filtek Supreme Ultra, 3M ESPE, St Paul, MN, USA). The specimens were built using a split metal mold placed between Mylar strips and two glass plates. The composites were packed into the mold in two increments and light cured for 20 seconds each with an LED light-curing unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) at a light intensity of 800 to 1000 mW/cm^2. The intensity was measured with a radiometer after every five specimens cured.

The specimens were then embedded in self-cured acrylic resin in a plastic ring with at least 1 mm of their surface exposed. All the specimens were ground sequentially with 400- and 600-grit SiC paper under water cooling and then kept in distilled water at 37°C for 14 days.

Table 1: *Materials Used in the Study*

Materials	Manufacturer	Composition
Heliobond	Ivoclar Vivadent (Schaan, Liechtenstein)	Bis-GMA; TEGDMA; catalysts and stabilizers
Scotchbond Universal Adhesive	3M ESPE (St Paul, MN, USA)	Bis-GMA; HEMA; decamethylene dimethacrylate; ethanol; water; silane treated silica; 1,10-decanediol and phosphorous oxide; copolymer of acrylic and itaconic acid; dimethylaminobenzoate; camphorquinone; (dimethylamino) ethyl methacrylate; methyl ethyl ketone
Ceramic Primer	3M ESPE	Methacryloxypropyl trimethoxysilane; water; ethyl alcohol 3-(trimetoxysilyl methacrylate)
Monobond Plus	Ivoclar Vivadent	3-(trimethoxysilyl) propyl methacrylate; methacrylated phosphoric acid ester
Filtek Supreme Ultra Restorative Composite (A2 enamel shade)	3M ESPE	Silane-treated ceramic; silane-treated silica; UDMA; bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA; silane-treated zirconia; polyethylene glycol dimethacrylate; TEGDMA; 2,6-di-tert-butyl-p-cresol

Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Half of the specimens were air abraded for 10 seconds with 50-μm Al_2O_3 particles 10 mm from the surface under a pressure of 4 bar. The surfaces of all the specimens were then etched with 37% phosphoric acid for 30 seconds, washed with air/water spray for 60 seconds, and dried with a blast of air for 60 seconds.

Double-sided adhesive tape with a central orifice 1.2 mm in diameter was used to limit the bonding area on the composite surfaces that were to be repaired in all the specimens. Polished specimens (P) and polished and air-abraded specimens (A), respectively, were randomly divided into two sets of six groups (n=12) according to the bonding procedure. The materials used in the study and their respective compositions are shown in Table 1.

The specimens in groups AH and PH were treated only with a layer of hydrophobic adhesive (Heliobond, Ivoclar Vivadent) applied on the air-abraded or polished surface, respectively, and light cured for 20 seconds. Groups ACH and PCH were treated with a conventional silane (Ceramic Primer, 3M ESPE) for 60 seconds, and groups AMH and PMH were treated with MDP-containing silane (Monobond Plus, Ivoclar Vivadent) for the same amount of time. After application of the silane, an air jet was used to evaporate the solvent, and the same hydrophobic adhesive was applied as described above.

The specimens in groups AU and PU were treated with a layer of universal adhesive (Scotchbond Universal Adhesive, 3M ESPE) applied on the air-abraded or polished surface, respectively; air-dried for 20 seconds for the solvent to evaporate; and light cured for the same time. Groups ACU and PCU were treated with a conventional silane (Ceramic Primer, 3M ESPE), and groups AMU and PMU were treated with MDP-containing silane (Monobond Plus, Ivoclar Vivadent) for 60 seconds. After the silane had been applied, the solvent was evaporated with an air jet, and the same universal adhesive was applied as described above.

The composite repair was carried out by inserting the same composite into transparent tubes (1.1-mm diameter and 1 mm high) placed on the treated surfaces. The repair composite was light cured with the same LED-curing unit for 40 seconds, and the specimens were stored in distilled water at 37°C for 48 hours, after which the plastic tubes and tapes were removed carefully using a scalpel blade to expose the cylindrical composite repairs. These were then analyzed under an optical microscope (Olympus BX60, Olympus Corp, Tokyo, Japan) at 50× magnification to identify any interfacial flaws, gaps, bubbles, or other defects. Specimens with defects were excluded from the study. Microshear bond strength testing was performed in a universal testing machine (EMIC 2000, Instron, Illinois Tool Works Inc, Norwood, MA, USA) using a metal blade positioned at the repair interface at a crosshead speed of 0.5 mm/min until failure occurred.

The failure area was examined under an optical microscope (Olympus BX60, Olympus Corp) at 50× magnification to identify the failure type, which was classified as adhesive (adhesive interface), cohesive in the original composite resin, cohesive in the repair composite resin, or mixed (more than one type).

The data were analyzed descriptively to obtain the mean and standard deviation for each group. Three-way analysis of variance, Tukey Honestly Significant Difference and Games-Howell tests were performed to detect statistical differences between the variables and to compare the groups. All tests were performed

Table 2: *Mean ± Standard Deviation of Microshear Bond Strength (MPa) of the Study Groups*[a]

Group	Surface Treatment	Silane	Adhesive	Bond Strength	Significant Differences
AH	Al_2O_3 air abrasion	—	Heliobond	16.45 ± 4.04	BCD
ACH		Ceramic Primer	Heliobond	20.82 ± 4.89	AB
AMH		Monobond Plus	Heliobond	18.63 ± 3.66	AB
AU	Al_2O_3 air abrasion	—	SB Universal	19.58 ± 4.32	AB
ACU		Ceramic Primer	SB Universal	20.91 ± 4.23	A
AMU		Monobond Plus	SB Universal	17.46 ± 2.95	ABC
PH	Polishing	—	Heliobond	8.44 ± 2.14	F
PCH		Ceramic Primer	Heliobond	13.29 ± 2.87	DE
PMH		Monobond Plus	Heliobond	12.43 ± 2.54	DE
PU	Polishing	—	SB Universal	15.05 ± 3.75	BCD
PCU		Ceramic Primer	SB Universal	13.31 ± 3.88	CDE
PMU		Monobond Plus	SB Universal	10.79 ± 2.43	EF

[a] *Groups connected by the same letter in the same column are not significantly different* ($p < 0.05$).

at a 5% significance level using the SPSS 20.0 statistical package (IBM Inc, Chicago, IL, USA).

RESULTS

The results for microshear bond strength are shown in Table 2 and Figure 1. The differences between the variables "adhesive" and "surface treatment" were statistically significant, as was the interaction between "adhesive" and "silane."

Air-abraded specimens treated with a conventional silane and universal adhesive (ACH) had the highest repair bond strength in the study. However, among the air-abraded specimens, this result was statistically different from the result only for specimens treated with hydrophobic adhesive alone (group AH).

In contrast, the polished specimens treated with hydrophobic adhesive (group PH) had the lowest

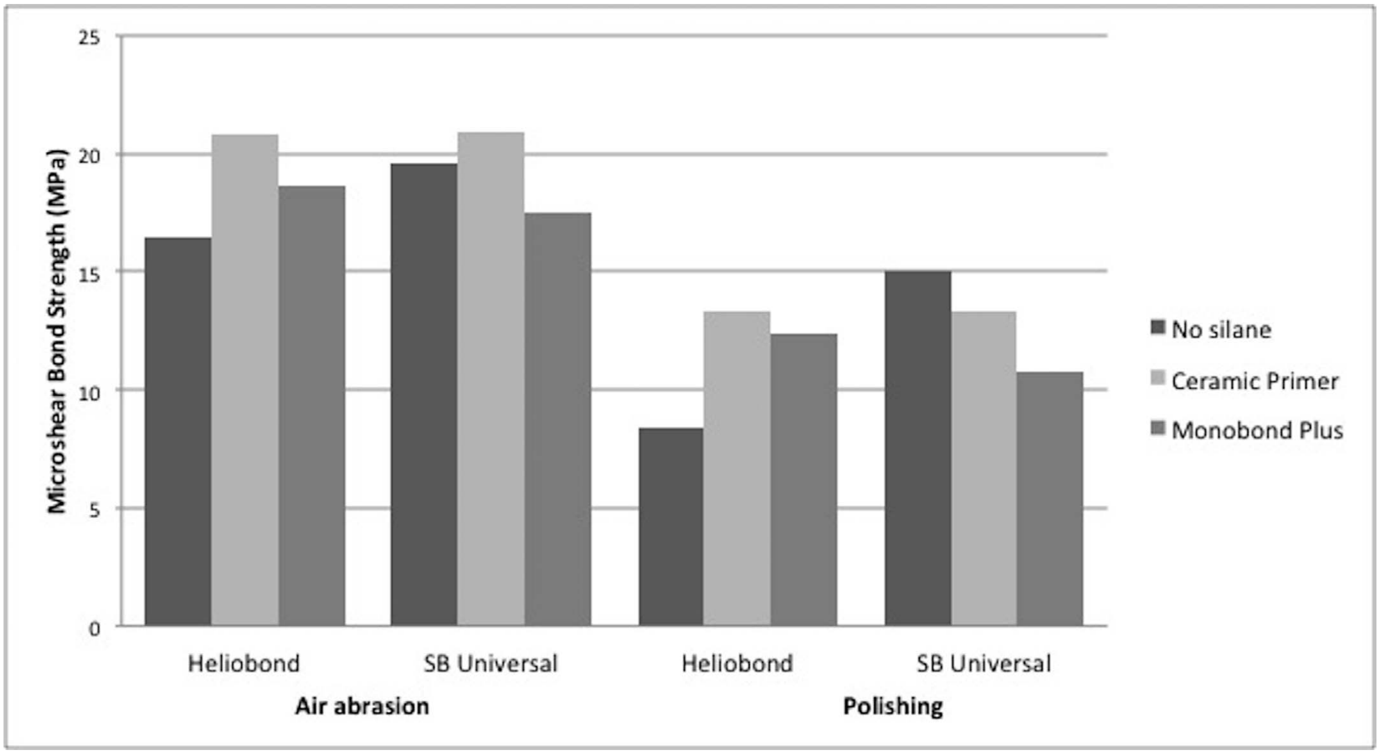

Figure 1. *Microshear bond strength (MPa) of the repaired groups according to surface treatment and type of silane.*

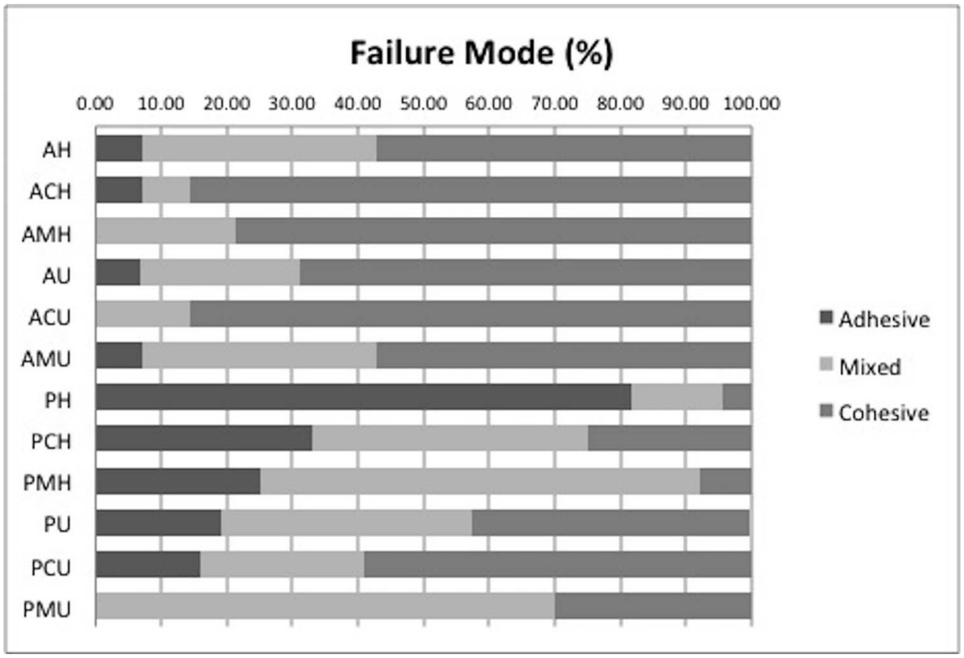

Figure 2. *Failure modes (%) of the evaluated groups.*

repair bond strength, although this result was not statistically different from the result for the group that received the same surface treatment followed by treatment with phosphate-containing silane and universal adhesive (PMU). In general, the polished specimens had lower shear bond strength than the air-abraded specimens. Interestingly, the results for bond strength in the PU group (polished surfaces treated with universal adhesive) were not statistically different from those for the air-abraded groups treated with hydrophobic adhesive (AH) alone, with hydrophobic adhesive and one type of silane (ACH and AMH), with universal adhesive alone (AU), or with universal adhesive and the phosphate-containing silane (AMU).

The failure modes of the specimens are shown in Figure 2. While most of the failures in the air-abraded specimens were cohesive in the composite repair, most of the failures were mixed in the polished specimens. In group PH (polished specimens treated with the hydrophobic adhesive), over 80% of the failures were adhesive.

DISCUSSION

This study evaluated the effect of different surface treatments and bonding materials on the bond strength of repairs in a nanofilled composite. The null hypothesis was rejected, as significant differences were found between the groups.

Filtek Supreme Ultra, the composite used in this study, is a nanocomposite with fillers that are a combination of nonagglomerated/nonaggregated 20-nm silica filler, nonagglomerated/nonaggregated 4- to 11-nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20-nm silica and 4- to 11-nm zirconia particles). The filler loading for nontranslucent shades is about 78.5% by weight and 63.3% by volume.[24] The inorganic portion consists of 60% to 80% ceramic and 1% to 10% silica/zirconia by weight. A previous study using the same composite found similar bond strengths when the oxygen-inhibited layer was maintained and when silane was applied before the adhesive in immediate repairs.[25] In the present study, bond strength was higher when conventional or MDP-containing silane was applied before the hydrophobic adhesive than when only the adhesive was applied. These results are probably due to the presence of exposed particles after air abrasion and the consequent good chemical bond between the resin matrix of the composite repair and exposed particles in the original resin.

Several studies have shown that application of a silane agent increases the wettability of the surface to be repaired, promoting a chemical bond between the resin matrix and the silica or glass filler particles.[25-29] Silane coupling agents are bifunctional, as they have not only a nonhydrolyzable functional group with a double carbon bond that reacts and can polymerize with monomers containing double bonds in the organic matrix of the resin composite but also hydrolyzable alkoxy groups that react with hydroxyl groups in the inorganic surface of silica-based materials forming oxygen bridges.[28]

Because of its highly crystalline, inert nature, zirconia can bond to resin-based materials to only a limited degree.[30] Different surface treatments, cleaning methods, and ceramic primers have been investigated in an attempt to overcome this limitation. The surface treatments most frequently used to improve mechanical retention to zirconia ceramic substrates are air abrasion with aluminum oxide particles and silica coating.[30-33] Recently, some studies have demonstrated that phosphate monomer–containing silanes can bond chemically to zirconia better than a conventional silane coupling agent.[31,32,34,35] Attia and Kern[33] compared the so-called universal primer Monobond Plus with a conventional silane applied to air-abraded or silica-coated zirconia ceramic. They used different cleaning methods and reported significantly higher long-term resin bonding to zirconia ceramic with the universal primer than with the conventional silane. However, to our knowledge, the present study is the first to investigate the efficacy of a phosphate monomer–containing silane in composite repairs. Our results show that Monobond Plus failed to produce a repair with higher bond strength than the conventional silane Ceramic Bond. This can be attributed to the low zirconia content of the nanofilled composite used here, around 10%.

The universal adhesive Scotchbond Universal contains, in addition to conventional methacrylate monomers, 10-MDP, a functional monomer known for its ability to bond chemically to calcium and make the adhesive interface more resistant to biodegradation.[36] Phosphate esters can also bond directly to the surface hydroxyl groups of non–silica-containing ceramics, such as zirconia, and enhance the hydrolytic stability of bonding more than silane coupling agents.[35] Furthermore, Scotchbond Universal adhesive contains prehydrolyzed silane, claimed by the manufacturer to be stable up to at least one year in storage.[21] A universal adhesive was investigated as a bonding agent for the repair of a nanofilled composite in a recent study in which air abrasion resulted in similar flexural bond strength to that achieved using phosphoric acid etching and unrepaired specimens (controls).[22] In another study, application of a universal adhesive was as effective as a conventional silane before application of a bonding agent in the repair of composites aged for three months and six years.[23] Our results show that the effectiveness of the universal adhesive was independent of the type of silane but that the repair bond strength increased when this type of adhesive was used with air abrasion as the surface treatment, probably because this resulted in a greater proportion of exposed ceramic fillers at the surface of the composite, increasing the surface area for bonding.

The use of an adhesive resin between the original and the repair composite increases the wettability of the surface as the resin penetrates and polymerizes into the prepared surface creating micromechanical retention.[6,7] A study comparing repair with and without application of adhesives in different composites concluded that their use increases bond strength for both immediate repairs and repairs of aged composites.[37] Since the aim of the present study was to evaluate the effect of different silanes and of air abrasion surface treatment, all specimens received an adhesive layer before the repair. However, the features of both adhesives evaluated are quite different in terms of viscosity, composition, and application mode. Although Heliobond is a Bis-GMA/TEGDMA–based material that is not expected to promote chemical bonding, air abrasion of the composite surface before Heliobond was applied resulted in greater repair bond strength.

After repair, the composite specimens in the present study were aged by storing them in distilled water for 14 days. Maximum water absorption occurs in the first week of storage, resulting in leaching of components and plasticization of the resin matrix.[38] Although some studies on repair bond strength use thermocycling as the aging process,[10,27,37] most use storage in water.[14,22,25,26,29] The latter method is sufficient to simulate clinical aging, as it causes water to be absorbed and consequent reduction of unreacted methacrylate carbon double bonds, which is required for the chemical bond with the repairing composite.[25]

While the most common test to evaluate the bonding effectiveness of composite repairs is the shear bond strength test,[7,25,23,27,37] this has frequently been criticized for its inhomogeneous stress distribution along the interface.[39] In the present study, air-abraded specimens had a higher percentage of cohesive fractures than polished specimens. This may be attributable to difficulties positioning the metal blade exactly at the specimen's interface, leading to dislocation of the stress toward the original composite. Adhesive failure was more common in the PH group, in which only the hydrophobic adhesive was used, resulting in the lowest bond strength in the study.

Composite repair is a minimally invasive technique that preserves sound tooth structure and increases the longevity of restorations. However, the surface treatment and chemical bonding between the new and existing (aged) composite must be maximized to ensure an effective repair. Additional studies with different composites, bonding agents, and surface treatments should be performed to improve this technique and raise awareness of this treatment option among clinicians.

CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions can be drawn. While air abrasion with Al_2O_3 particles increased the repair bond strength of the nanoparticle composite, the use of MDP-containing silane did not affect the results. The application of a silane-containing universal adhesive alone was as effective as any of the silane and adhesive combinations tested. The use of this material seems to be promising for the simplification of nanofilled composite repair.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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