



## Evaluating Various Preparation Protocols on the Shear Bond Strength of Repaired Composite

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### ABSTRACT

**Introduction:** The aim of this study is to evaluate the effect of different combinations of various surface treatments on the shear bond strength (SBS) of repaired composite resin.

**Materials and methods:** A total of 122 composite samples were prepared from Filtek Z350 XT. Samples were light cured and stored for 6 weeks. Surface treatment of old composite was done in five groups: Group I: bur roughening + phosphoric acid etching, group II: bur roughening + hydrofluoric acid etching + silane coupling agent, group III: air abrasion + phosphoric acid etching, group IV: air abrasion + phosphoric acid etching + silane coupling agent, group V: air abrasion + hydrofluoric acid etching + silane coupling agent. Bonding agent was applied to all surface-treated old composites and light cured. The fresh composite resin was bonded to treated surfaces and cured and stored in water at 37°C for 6 weeks. Shear bond strength was measured by a universal testing machine.

**Results:** Shear bond strength values of all groups were not statistically significant except for group V, which showed statistically significant higher SBS than group III.

**Conclusion:** Techniques with readily available materials at the clinic can attain similar SBS to more elaborate technique involving potentially hazardous materials.

**Keywords:** Bond, Composite, Etching, Repair, Silane.

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### INTRODUCTION

Resin-based composite restorations are the most utilized dental filling material for restoration of both anterior and posterior teeth in dental practice nowadays. No matter how well those materials are adhesively bonded to tooth structure, they are subjected to different degenerative changes within the oral cavity.<sup>1</sup> Approximately 50% of resin-based composite restorations are replaced after 5 years of service, and the main reasons are secondary caries, marginal staining, marginal defects, marginal or body fracture, discoloration, degradation and loss of anatomical form, unsatisfactory shade, and painful symptoms.<sup>2</sup>

Traditionally, replacement was the ideal approach to treat defective composite restorations; however, repairing composites offers an alternative and more conservative approach where restorations are partly still serviceable.<sup>3</sup> Repairing serviceable restorations is gaining wider acceptance than replacing them regarding the modern concept of minimally invasive dentistry, where evidence supports localized repair rather than replace the entire restoration.<sup>4</sup> Repairing composite restoration may be considered the treatment of choice for surface discoloration of existing restorations, small areas of recurrent caries along the margin of an otherwise sound composite restoration, or when complete removal of a very large composite restoration would unnecessarily jeopardize the health of a tooth, as well as laboratory fabricated (indirect) resin composite repair.<sup>5</sup> However, because the repair procedure may result in weaker restorations, therefore, successful resin repair requires the development of an adequate interfacial bond between old and new resin composites.<sup>6</sup>

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According to Lewis et al,<sup>7</sup> the efficiency of the repair is related to the magnitude of the bond strength obtained at that interface. The bond strength between increments of composite should be equal to the cohesive strength of the material. If the composite has been contaminated, polished, processed in a laboratory (indirect composite restorations), or aged, the adhesion to a new composite is reduced to 25 to 80% of the original cohesive strength.<sup>8-12</sup>

The air-inhibited layer on the surface and the high degree of conversion with low degree of unreacted carbon double bond would translate into a limited number of unsaturated double bonds. The latter is necessary for ensuring chemical bonds between the fresh and aged composite.<sup>5,13</sup> Several studies have reported that surface treatment promoting mechanical interlocking, surface wetting, and chemical bonding will enhance the repair bond significantly with the new composite.<sup>6,8,9,14-16</sup>

Accordingly, repair studies addressed several different techniques to improve the bond strength of composite repair through surface roughening with burs, airborne particle abrasion with aluminum oxide particles with or without silanated silica coating, acid etching with phosphoric or hydrofluoric acid, silane coupling agent application, and using various resin-based adhesive systems.<sup>5,8,10,11,14,15,17-19</sup> Studies investigating surface preparation with either bur roughening or air abrasion have yielded significant increased bond strength when air abrasion was used as a microretentive method on the surface to be repaired.<sup>8,9,13,15,17,19-22</sup> Etching with hydrofluoric acid was reported with better bonding strengths compared with etching with phosphoric acid.<sup>23-26</sup> Silanization can enhance bonding of chemically aged postcured composite with fresh composite. Silanization proved to give better bonding results.<sup>5,15,26-28</sup> Intermediate adhesives and bonding agents would improve the strength of the repaired composite and enhance bonding significantly between aged composites and fresh composite layers.<sup>14,16,18,19,29,30</sup>

However, not all general practitioners have additional tools, such as chairside air abrasion or silica coating devices. Phosphoric acid, silane, and bonding agents are found in most practices. Moreover, diamond finishing burs and acid etching with phosphoric acid as surface treatment in repair procedure are the most common repair approach taught by European and North American dental schools.<sup>31,32</sup>

The aim of this study was to advocate the best clinically effective composite repair protocol. Bur roughening, air abrasion, hydrofluoric acid etching, and phosphoric acid etching with or without silanization before bonding composite were investigated.

The null hypothesis stipulated that there is no difference in the shear bond strength (SBS) of the repaired composite restoration between bur roughening and air abrasion and between hydrofluoric acid and phosphoric acid treatments with or without silanization.

## MATERIALS AND METHODS

A total of 122 composite samples were prepared from Filtek Z350 XT (3M ESPE, St. Paul, MN, USA). Each composite sample was made in a cylindrical mold of 8 mm height and 9 mm diameter, and all molds were made of addition silicone vinyl polysiloxane duplicating material (Elite Double, Zhermack, Ohlmuhle, Germany). Each mold was filled with a double 2 mm layer of A2 dentin shade by means of a plastic instrument. Each layer was light polymerized with the tip of light curing unit (Starlight Pro, Mectron, Carasco, Italy) for 40 seconds. Each sample was then light cured from all sides for additional 40 seconds after removing it from the mold. The light output was calibrated according to the manufacturer's instructions. The light curing tube was kept in contact with the composite surface to ensure adequate curing at a 90° to the top surface.

All samples were kept dry for 24 hours; samples were then divided into control samples (n = 12) and test samples (n = 110). The test samples were prepared under water with a high-speed bullet shape fine diamond finishing (yellow-labeled) bur (Dia-Tessin, Vanetti SA, Gordevio, Switzerland). Then, samples were polished with low-speed green and pink Soflex (3M ESPE, St. Paul, MN, USA) finishing disks.

After polishing, each sample was rinsed for 15 seconds, and all samples (control and test) were stored in distilled water at 37°C for 6 weeks.

Test samples were randomly distributed into five groups (n = 22) for repair using the following methods as shown in Table 1.

*Group I:* Composite surfaces were roughened in five strokes with high-speed bullet shape rough diamond

**Table 1:** Distribution of testing groups according to treatment methods

Groups number	Surface roughening	Acid treatment	Silanization	Bonding
I	Rough diamond bur	32% phosphoric acid	–	Bonding agent
II	Rough diamond bur	9.5% hydrofluoric acid	Silane coupling agent	Bonding agent
III	Air abrasion	32% phosphoric acid	–	Bonding agent
IV	Air abrasion	32% phosphoric acid	Silane coupling agent	Bonding agent
V	Air abrasion	9.5% hydrofluoric acid	Silane coupling agent	Bonding agent

(black labeled) bur (Dia-Tessin, Vanetti SA, Gordevio, Switzerland) under water; 32% phosphoric acid gel (Scotchbond Universal Etchant, 3M ESPE, St. Paul, MN, USA) was applied to the composite surface for 30 seconds with brushing with a microbrush. The acid was rinsed for 15 seconds and dried for 15 seconds. Bonding agent (Adper Single Bond 2, 3M ESPE, St. Paul, MN, USA) was applied in two coats with a microbrush with 5 seconds waiting time and then light cured for 20 seconds.

*Group II:* Composite surfaces were roughened in five strokes with a high-speed bullet shape rough diamond bur; 9.5% hydrofluoric acid solution (porcelain etch, Vista, Racine, USA) was applied to the composite surface for 60 seconds with brushing using a microbrush. The acid was rinsed for 15 seconds and dried for 15 seconds. Silane coupling agent (RelyX Ceramic Primer, 3M ESPE, St. Paul, MN, USA) was applied to the etched composite surface and allowed to dry for 60 seconds. Finally, the bonding agent was applied on the silanated surface in two coats with a microbrush. Wait 5 seconds and light cure for 20 seconds.

*Group III:* Composite surfaces were air abraded for 10 seconds using 50  $\mu\text{m}$  aluminum oxide abrasive with a chairside abrasion unit (MicroEtcher, Danville Engineering, Danville, CA, USA), operating at a pressure of 60 psi at a 10 mm distance and 90° to composite surface. The samples were then rinsed for 10 seconds and dried for 5 seconds; 32% phosphoric acid gel was applied to the composite surface for 30 seconds with brushing using a microbrush. The acid was rinsed for 15 seconds and dried for 15 seconds. The bonding agent was applied in two coats with a microbrush. Wait 5 seconds and light cure for 20 seconds.

*Group IV:* Composite surfaces were air abraded for 10 seconds using 50  $\mu\text{m}$  aluminum oxide abrasive with a chairside abrasion unit, operating at a pressure of 60 psi at a 10 mm distance and 90° to composite surface, rinsed for 10 seconds and dried for 5 seconds; 32% phosphoric acid gel was applied to the composite surface for 30 seconds with brushing using a microbrush. The acid was rinsed for 15 seconds and dried for 15 seconds. Silane coupling agent was applied to the etched composite surface and allowed to dry for 60 seconds. Finally, the bonding agent was applied on the silanated surface in two coats with a microbrush. Wait 5 seconds and light cure for 20 seconds.

*Group V:* Composite surfaces were air abraded for 10 seconds using 50  $\mu\text{m}$  aluminum oxide abrasive with a chairside abrasion unit, operating at a pressure of 60 psi at a 10 mm distance and 90° to composite surface, rinsed for 10 seconds and dried for 5 seconds; 9.5% hydrofluoric acid solution was applied to the composite surface for 60 seconds with brushing using a microbrush. The acid was rinsed for 15 seconds and dried for 15 seconds. Silane

coupling agent was applied to the etched composite surface and allowed to dry for 60 seconds. The bonding agent was applied in two coats with a microbrush. Wait 5 seconds and light cure for 20 seconds.

All the treated samples were inserted in their molds, and fresh Filtek Z350 XT composite layer (A2 enamel shade) of 2 mm thickness was condensed over each prepared surface. A different shade was chosen for the repairing composite to enable visual identification and orientation of the repair interface during SBS testing. After light polymerizing for 40 seconds, another 2 mm layer of composite was applied and cured for 40 seconds. Each sample was then light cured from all sides for additional 40 seconds after removing it from the mold. Test samples were kept dry for 24 hours, and then they were stored in distilled water at 37°C for 2 weeks.

The samples were mounted in the jig of the universal testing machine (JINAN material testing machine, Jinan, China) (Fig. 1). Shearing force of 0.5 mm/minute at failure was recorded by a person blind to the samples. Shear bond strength was calculated by dividing the failure force by the cross-sectional area of the samples.

Data were analyzed using one-way analysis of variance (ANOVA); normality test, i.e., Kolmogorov–Smirnov test; and Bonferroni *post hoc* test using Statistical Package for the Social Sciences (SPSS) statistical software. A confidence level of 95% was selected to determine statistical significance.

## RESULTS

The initial loss of bonding SBS values was found to range between 5.08 and 17.76 MPa. The mean SBS values of each group are presented in Table 2. The initial loss of bonding and complete debonding SBS results for tested groups are presented in Graph 1. Data obtained were analyzed using SPSS, version 20 (Illinois, USA) at  $p < 0.05$  significance level. Normality test, “Kolmogorov–Smirnov

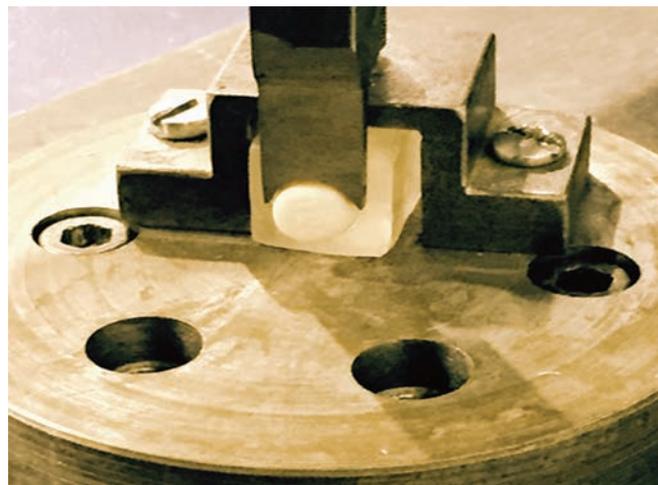


Fig. 1: Sample loaded in the jig of the SBS testing machine

**Table 2:** Mean (SD) values of SBS at initial loss of bonding for all tested groups

	<i>n</i>	Mean SBS (SD) in MPa
Control	12	9.63 (3.3)
Bur, PA, No SCA	22	8.01 (2.9)
Bur, HA, SCA	22	8.57 (3.5)
AA, PA, No SCA	22	7.00 (3.2)
AA, PA, SCA	22	9.11 (2.4)
AA, HA, SCA	22	11.10 (3.0)

SD: Standard deviation; PA: phosphoric acid; SCA: silane coupling agent; HA: hydrofluoric acid; AA: air abrasion

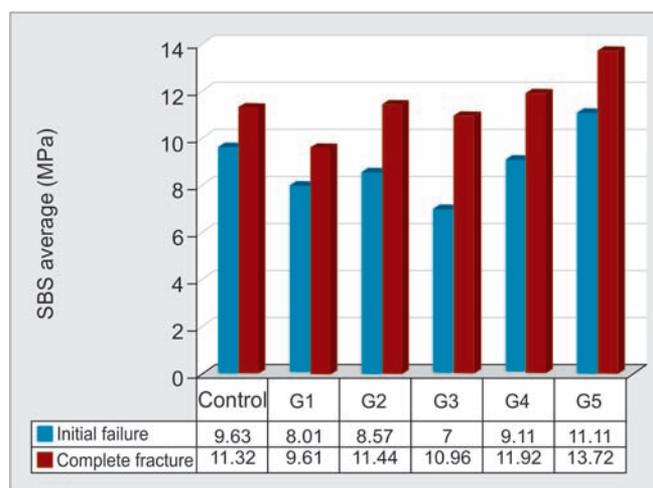
test," indicated that SBS values are normally distributed. Hence, the SBS values were analyzed using one-way ANOVA test and Bonferroni *post hoc* test. The SBS values of all groups were not statistically significant except for group V, which showed statistically significant higher SBS than group III ( $p = 0.000$ ).

When observed under stereomicroscope, three types of failure were noticed: Cohesive (Fig. 2A), adhesive (Fig. 2B), and mixed fractures (Fig. 2C). Samples treated with phosphoric acid were found to primarily fail on the adhesive bond between the two composite layers. Groups treated with hydrofluoric acid had mixed failure.

Kruskal–Wallis test showed statistically significant difference between the two groups treated with hydrofluoric acid. Mann–Whitney test confirmed the difference to occur between composite specimens treated either with silane coupling agent or without.

## DISCUSSION

When repairing old composite restorations, surface pretreatment of the old composite has two purposes: To remove the superficial layer altered by the saliva exposing a clean higher energy composite surface and to increase the surface area through the creation of surface irregularities.<sup>33</sup> Bonding between old and new composite may occur by three distinct mechanisms: (1) Through a chemical bonding with the organic matrix, (2) through a chemical bonding with the exposed filler particles, and

**Graph 1:** Shear bond strength values at initial and complete failure (MPa)

(3) through micromechanical retention to the treated surface.<sup>14</sup>

Five different surface treatment strategies were employed in this study. Two different mechanical treatments were done for the surface roughening (air abrasion and bur roughening) and two chemical treatments (acid etching with either phosphoric acid or hydrofluoric acid) with and without silanization. The results of this study showed the mean SBS value of  $9.63 \pm 3.3$  MPa in the control group, which was nontreated resin composite samples. They were not subjected to any surface treatment to obtain the value on which the repaired composite resin restoration should not decrease.<sup>11,18,34</sup> The low SBS values for the control and test groups are suggested to be due to the long storage time in distilled water at  $37^\circ\text{C}$  for 6 weeks. This aging method affects negatively the SBS values due to water absorption, leaching of unreacted monomers, swelling of the matrix, and degradation of the matrix filler interface directly by hydrolysis.<sup>35</sup>

Suggested surface treatments in the current study have no statistically significant effect on the SBS between composite layers ( $p > 0.05$ ) at baseline (except for group V). These findings indicate that clinically oriented repair



**Figs 2A to C:** (A) Representative sample from group II with cohesive failure in the composite substrate (8 $\times$ ); (B) representative sample from group III with adhesive failure at the interface between aged and fresh composite (8 $\times$ ); and (C) representative sample from group V with mixed failure partially at the interface and partially in the substrate (8 $\times$ )

techniques (bur roughening, acid etching, silane coupling agent, and bonding) will provide similar SBS values to the control group. These results suggest that it is not necessary to purchase any additional armamentaria in dental practice, such as chairside air abrasion devices making repairs simple, efficient, and cost-effective.<sup>16,18</sup>

In this study, only group V which was treated by air abrasion with hydrofluoric acid etching and silane coupling agent with bonding had higher mean SBS value than group V. The other groups had comparable mean SBS values to the control group. Chemical treatment of the surface by 37% phosphoric acid yielded insignificant increase in bond strength when compared with the control group. The results of this study revealed that acid pretreatment did not significantly change the morphological pattern of the aged composite surface and its action was limited to superficial cleaning effect of the composite surface as reported by other studies.<sup>10,11,34,36</sup> Readily available phosphoric acid might suffice to use intraorally rather than using the potentially hazardous hydrofluoric acid to provide similar SBS values to the control group.

Moreover, the results of this study did not find a difference in the SBS between samples repaired with bur roughening and those repaired with air abrasion. Although those findings are in agreement with some previous studies,<sup>11,16,29,37</sup> they do not support the results of other former studies.<sup>19,22,33,34,38</sup> These studies found that surface treatment with  $Al_2O_3$  powder yielded the highest repair shear and tensile bond strength (TBS) that nearly reach the cohesive bond strength of the original composite. It appears that micromechanical interlocking is the least important factor in strengthening SBS for repaired composite in comparison to chemical treatments (etching, silanization, and bonding). Silane coupling agent application has been shown to increase SBS in composite repairs.<sup>5,15,35,38</sup> The bifunctional molecule of the silane coupling agent bonds the inorganic filler particles of the resin with the methacrylate of the adhesive system, and increases the wettability of the adhesive system to infiltrate into the irregularities of the treated composite surface.<sup>35</sup>

Hydrofluoric acid with silane coupling agent after air abrasion (group V) has provided the highest SBS both at initial and complete debonding. These results are consistent with other studies that reported high TBS values for repaired laboratory composites with hydrofluoric acid, or air abrasion with  $Al_2O_3$  particles, both with resin/silane primer, with values ranging from 32.9 to 39.6 MPa.<sup>38</sup>

These results indicate that hydrofluoric acid and air abrasion were able to achieve better mechanical interlocking between the two composite layers when compared with groups treated with phosphoric acid and bur roughening. Silane coupling agent application also appeared to

play an active role in providing proper chemical bonding between the two layers of composite. Although this was not reflected on the SBS values at baseline, this might have an impact on aged repaired composites subjected to thermocycling and cyclic loading.

Shear tests lead to nonhomogeneous stress distribution in the bonded interface, which eventually causes erroneous interpretation of the results due to the failure occurring in the substrate rather than the adhesive zone.<sup>35</sup> Nevertheless, in our study, it was necessary to assess the mode of failure because there were no significant differences between the SBS values among all the test groups (except group V).

Etching procedures are used to facilitate bonding because it removes surfaces debris and creates a porous surface. This porosity leads to enhance the retentive bond between the resin and tooth. In this study, the groups treated with hydrofluoric acid produced cohesive and mixed debonding in comparison to the adhesive debonding with phosphoric acid. The cohesive and mixed mode of failure observed under the stereomicroscope for groups II and V confirms the stronger effect of hydrofluoric acid in comparison to phosphoric acid.<sup>23-26</sup> Although this effect was not clear in the SBS values, it was noticed from the adhesive failure observed for the phosphoric-treated surfaces. It is thought that hydrofluoric acid produces an aggressive effect on the surface containing silica fillers (this is based on the affinity of fluoride to silicon) because composite contains silica fillers. Hydrofluoric acid attacks the silica phase of composite, producing a retentive surface for micromechanical bonding.<sup>28</sup>

## CONCLUSION

Within the confines of this study, we concluded the following:

- Air abrasion did not provide higher repair SBS than bur roughening the repaired surface.
- Acid etching with phosphoric acid provided similar repair SBS, but different failure patterns to those observed with hydrofluoric acid etch.

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