

# An *In Vitro* Assessment of the Effects of Three Surface Treatments on Repair Bond Strength of Aged Composites

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## Clinical Relevance

To achieve a durable composite-composite bond, it is highly recommended that besides selective grinding of the surface to be repaired, specific combinations of silane primer and bonding agent should be used, without the need for acid etching of the composite surface.

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## SUMMARY

**Objective:** This study evaluated the effectiveness of three surface treatments on repair shear bond strength (SBS) of aged composites.

**Methods:** A total of 120 cylindrical samples made of a micro-hybrid composite (Clearfil AP-X) were randomly assigned to one control and three experimental groups (n=30) after water storage (3 weeks). All experimental groups included surface roughening with diamond burs. Subsequent treatments were provided as follows: group 1—only (self-etching) bonding; group 2—silane and bonding; and group 3—phosphoric acid etching, silane, and bonding. The composites were repaired with the same brand and were aged (water storage [48 hours] and thermocycling [2000 cycles]). Each group was divided into two subgroups (each, n=15): new—water storage at 37°C for one week; old—water storage for six months. The

SBS was tested. The fracture mode was assessed under 40× magnification.

**Results:** Mean SBS values (MPa) for the study subgroups were as follows: control (new:  $3.38 \pm 1.6$ ; old:  $1 \pm 0.76$ ), group 1 (new:  $27.3 \pm 1.8$ ; old:  $25.7 \pm 1.9$ ), group 2 (new:  $59.1 \pm 7.9$ ; old:  $50.8 \pm 4.6$ ), and group 3 (new:  $48.5 \pm 8.6$ ; old:  $39 \pm 3.5$ ). Significant influence of the conditioning method and the duration of water storage was observed ( $p < 0.01$  [two-way analysis of variance {ANOVA}]). The SBS of all groups were significantly different (all  $p$  values  $< 0.01$  [Tukey]). Longer water storage time significantly reduced repair bond strength in all experimental groups ( $p < 0.01$ ). Although the control group and group 1 showed approximately 100% and 75% adhesive failures, respectively, groups 2 and 3, respectively, demonstrated about 75% mixed and cohesive failures. Weibull analysis showed that groups 2 and 1 had the lowest and highest probabilities of failure among the experimental groups, respectively.

**Conclusion:** All experimental groups produced acceptable SBS levels; however, use of silane and bonding systems showed the most superior results. Acid etching reduced the SBS.

## INTRODUCTION

The use of composite resins in restorative dentistry is well established. Despite recent improvements, the restoration may exhibit fracture, staining, or other defects, which can lead to clinical problems.<sup>1-4</sup> Treatment choices consist of repair and total replacement. Repair of a restoration with small defects is a conservative treatment because complete removal would lead to larger cavities with further loss of tooth substance.<sup>5</sup> Therefore, restoration replacement may result in unnecessary removal of previously etched enamel.<sup>6</sup> In addition, according to several clinical studies, repair is an acceptable alternative to replacement of composite restorations.<sup>7,8</sup>

Bonding between two composite layers is accomplished by the presence of an oxygen-inhibited unpolymerized layer of resin.<sup>5,9</sup> In the mouth, absorption of water by diffusion through the resin phase of composites will affect the ability of the new composite to adhere to the aged composite, because the number of available unsaturated double bonds diminishes with aging.<sup>10</sup> The shear bond strength (SBS) of fresh composite to the composite aged in water or contaminated with artificial saliva has been studied previously.<sup>11-13</sup> In these studies, various

methods have been suggested for establishing satisfactory SBS levels between the existing composite and the fresh one: mechanical grinding, acid etching, resin coating, or a combination of these treatments. The resins used in the previous studies were mainly bis-GMA and urethane dimethacrylate resins diluted with triethylene glycol dimethacrylate. Compared with no treatment, mechanical treatments increased the SBS, but greater strength was achieved by using an immediate bonding layer.<sup>11-13</sup> Different studies showed that acid etching with phosphoric acid had little effect on bond strength.<sup>11</sup> Li found that resin-to-resin bond strength could be increased if the composite was primed with acetone, which is a component of many dentin bonding systems.<sup>14</sup>

Several studies have shown that use of a silane agent or an intermediate bonding agent, or roughening of the surface of the aged composite, may considerably enhance the SBS of the repair bond,<sup>15-21</sup> although some authors have suggested that silane application might be unnecessary when a bonding agent is going to be used.<sup>15,16</sup> Nevertheless, some repair system kits composed of silane agents, monomers, and self-etching water-based primers and bonding agents have shown appropriate results compared with one-step primers or intermediate resins.<sup>22</sup>

This study attempted to comparatively assess the efficacy of three surface treatments provided by a commercial composite repair kit in increasing the SBS of a fresh layer of composite bonded to an aged composite.<sup>23</sup>

## MATERIALS AND METHODS

Brand names, chemical compositions, and batch numbers of the materials used in this study are listed in Table 1.

### Sample Preparation

A total of 120 cylindrical samples made of a micro-hybrid composite (Clearfil AP-X, Kuraray, Japan) were fabricated according to the manufacturer's instructions using a polyethylene mold 6 mm high with an internal diameter of 5 mm. Composite increments (2 mm thin) were light-cured (Coltolux 75, Coltene/Whaledent, Cuyahoga Falls, OH, USA) vertically from a 1 mm distance for 40 seconds. Light intensity was calibrated at 800 mW/cm<sup>2</sup> with a radiometer (Optilux Radiometer Model 100, SDS Kerr, Danbury, CT, USA) after curing of each of five specimens. After polymerization, the polyethylene molds were gently removed.

Table 1: Brand Names, Chemical Compositions, and Batch Numbers of Materials Used in This Study<sup>a</sup>

Material	Brand	Chemical Composition	Batch Number
Primer	Clearfil SE Bond Primer	MDP, HEMA, hydrophilic dimethacrylates, photoinitiator, water	00522A
Bonding agent	Clearfil SE Bond Adhesive	MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, silanated colloidal silica, photoinitiator, water	00736A
Silane	Clearfil Porcelain Bond Activator	Bisphenol a polyethoxy dimethacrylate, 3-methacryloxypropyl trimethoxy silane ( $\gamma$ -MPS)	00167B
Etchant	K-Etchant	40% H <sub>3</sub> PO <sub>4</sub>	0355B
Composite resin	Clearfil AP-X Shade A3	Micro-hybrid composite consisting mainly of high-density fine barium glass particles (85% of composite weight), plus silanated barium glass, silica, colloidal silica, bis-GMA, TEGDMA, photoinitiator	01065A

<sup>a</sup> All materials were manufactured by Kuraray Medical, Tokyo, Japan.

## Aging

To age the composite, the substrates were placed in distilled water and stored in an oven (WTE Binder, Tuttlingen, Germany) at 37°C for three weeks.

## Surface Treatments of the Aged Composite

Specimens were randomly divided into four groups (each, n=30) (Table 2):

- **Control:** No surface treatments were performed on control specimens.
- **Group 1 (Self-etching Bonding):** Composite surfaces were roughened for 20 seconds with a diamond bur (No. 881-012, Diatech Dental AG, Swiss Dental Instruments, Charleston, SC, USA) attached to a water-spraying high-speed handpiece (KaVo K9, Handpiece Type 950, KaVo, Biberach,

Germany). A new set of burs was used after every five preparations. Afterward, the available primer (Table 1) was applied to the composite surface for 20 seconds (using an applicator sponge) and was gently air thinned. The bonding agent (Table 1) was applied to the surface (with an applicator) and light-cured for 20 seconds.

- **Group 2 (Silane Bonding):** Composite surfaces were roughened as described previously. Then the primer and the silane (Table 1) were blended 1:1, applied to the composite surface for five seconds, and air thinned. Afterward, the bonding agent was applied to the surface and light-cured for 20 seconds.
- **Group 3 (Etching/Silane Bonding):** After surface roughening with diamond burs, the surface was etched (40% phosphoric acid, Table 1) for five seconds and then was rinsed and dried thoroughly,

Table 2: Surface Treatment Protocols for Materials Used in This Study<sup>a</sup>

Groups	Surface Roughening	Phosphoric Acid Etching	Silane Application	Priming	Bonding
Control	-	-	-	-	-
1 Bonding	•	-	-	•	•
2 Silane/Bonding	•	-	•	•	•
3 Etching/S/B	•	•	•	•	•

<sup>a</sup> Bullet, present; Hyphen, absent; S/B, Silane/bonding.

according to the manufacturer's instructions. Afterward, the primer, the silane, and the bonding agent were applied as described in group 2.

### Repairing the Aged Composite

After the previously described surface treatments were applied with the use of a translucent polyethylene mold 3 mm in diameter and 6 mm high, fresh composite resin (Clearfil AP-X) was placed by the same operator on the conditioned flat surface of the aged composite cylinder, and then was vertically photopolymerized (40 seconds) in three increments of not more than 2 mm.

### Aging the Repaired Composite Setup

After polymerization, specimens were removed from the mold and were stored in distilled water placed in an oven (WTE binder) at 37°C for 48 hours; they then were thermocycled (Dorsa, Behsaz, Iran) for 2000 cycles at between  $5 \pm 2^\circ\text{C}$  and  $55 \pm 2^\circ\text{C}$  with a dwell time of 30 seconds and a transfer time of five seconds.

After thermocycling, to consider the stability of composite repairs in water, the specimens in each group were randomly assigned to two subgroups (each,  $n=15$ ) (subgroups *new* and *old*). Before the SBS was tested, specimens in the subgroups labeled *new* were stored in distilled water in an oven (WTE binder) at 37°C for one week, while specimens in the subgroup *old* were stored in the same conditions (37°C) for six months.

### Repair SBS

A shearing rod attached to a universal testing machine (Zwick Model 1494, Zwick GmbH & Co KG, Ulm, Germany) exerted the shear force to the samples (parallel to the flat bonding area) at a crosshead speed of 0.5 mm/min until fracture (Figure 1). The SBS was calculated in megapascals (MPa) by dividing the fracture load (Newton) by the repair surface area ( $7.065 \text{ mm}^2$ ). A stereomicroscope (Olympus SZX-12, Olympus, Tokyo, Japan) was used to visualize the fractured surfaces of specimens at 40× magnification to determine the fracture type (adhesive [at the intermediate layer], cohesive [within the old or the repairing resin composite], or mixed [a combination of cohesive/adhesive failure modes]).

### Statistical Analysis

Descriptive statistics were calculated. Data were analyzed using two-way analysis of variance (ANOVA) with focus on factors of treatment type and

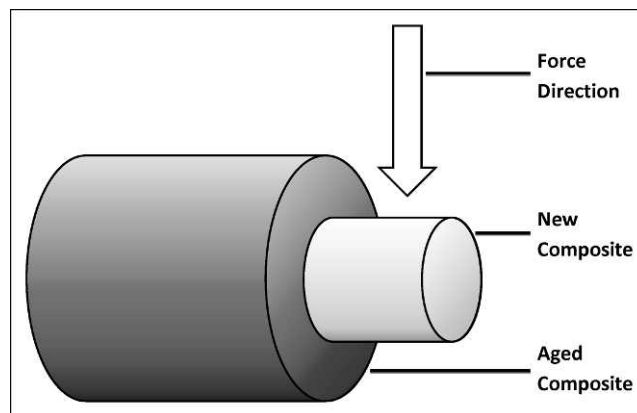


Figure 1. Schematic illustration of the SBS test setup.

storage time, and the Tukey post hoc test was completed. The level of significance was set at 0.05. In addition, Weibull analysis of data pertaining to the experimental groups was carried out, and stress rates at 5% and 90% fracture probabilities were predicted.

## RESULTS

Descriptive statistics are presented in Table 3 and Figure 2. According to two-way ANOVA, significant differences between groups were evident in terms of surface treatment and water storage ( $p<0.01$ ). The Tukey post hoc test showed that mean SBS values in all experimental groups were significantly higher than those of the control group ( $p<0.01$ ).

After one week of water storage, groups employing silane and the bonding agent (groups 2 and 3) showed repair SBS values that were significantly higher than those of the control group and group 1, according to the Tukey test ( $p<0.05$ ; Figure 2). After six months of water storage, mean SBS of groups 3 and 4 was again significantly greater than that of the control group and group 1 ( $p<0.01$ ). The SBS of group 2 was significantly higher than that of the other groups ( $p<0.01$ ) both before and after long-term water storage. SBS values of the *old* subgroups (six months of water storage) in all four groups were significantly lower ( $p<0.01$ ) than SBS values of the *new* subgroups (one week of water storage) in the same groups.

### Reliability of Bond Strength

Results for Weibull analysis are presented in Table 4 and Figure 3. Characteristic bond strengths and stresses at 5% and 90% failure probabilities for group 2 were higher than those of the other groups. Weibull analysis gives the Weibull modulus charac-

Table 3: Mean Shear Bond Strength (MPa) and Frequency Distributions (Percentage) of Sites of Bond Failure Through the Composite Repair									
Groups	Subgroups	Mean, MPa	SD	CV, %	Mean, 95% CI		Bond Failure Sites, %		
					Lower	Upper	Adhesive	Mixed	Cohesive
Control	New	3.38	1.60	47.5	2.57	4.19	100	—	—
	Old	1	0.76	76.4	0.62	1.38	100	—	—
Bonding	New	27.3	1.8	6.6	26.39	28.21	75	25	—
	Old	25.7	1.9	7.3	24.74	26.66	75	25	—
Silane/Bonding	New	59.1	7.9	13.3	55.1	63.1	—	25	75
	Old	50.8	4.6	9.1	48.47	53.13	—	25	75
Etching/Silane/Bonding	New	48.49	8.6	17.7	44.14	52.84	10	75	15
	Old	39	3.5	9	37.23	40.77	—	75	25
Abbreviations: CI, confidence interval; CV, coefficient of variation; SD, standard deviation.									

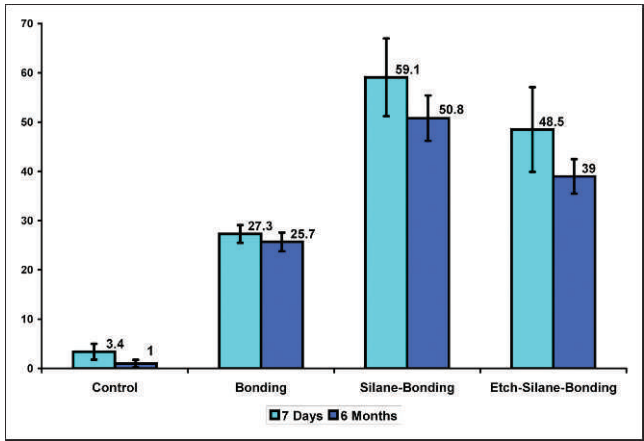


Figure 2. Mean (and SD) of the SBS (MPa) of different surface treatments.

teristic. At 90% probability of failure, bond strength was lower in the *old* subgroups compared with the *new* subgroups. High Weibull moduli were obtained in the groups, including silane application (groups 2 and 3), after six months of water storage (Table 4, Figure 3).

Site of Bond Failure

The mode of failure was predominantly adhesive for the control group and group 1. Group 2 tended to

exhibit cohesive failure inside the aged composite material. In group 3, the mode of failure was mainly mixed. The mode of failure for each group remained unchanged after six months of water storage (Table 3).

DISCUSSION

Repair of a restoration would always be preferable to its replacement. However, there is the risk that repair may weaken the restoration. Several composite repair studies have shown wide variation of interfacial repair bond strengths from 25% to 80% of the cohesive strength of respective substrate materials.<sup>24-28</sup> In this study, three different conditioning concepts were selected. The first step in all three methods involved mechanical surface preparation followed by application of the chemical component. One group was based on both mechanical surface roughening and phosphoric acid application to create micromechanical retention; the other two systems used only diamond bur roughening. The repair strength required for a satisfactory composite repair *in vivo* has not been carefully investigated by longitudinal clinical studies. In contrast, resin-enamel bond strength has been investigated and is reported to be of the order of 15 to 30 MPa.<sup>17,29</sup> It seems that repair bond strength similar to composite-etched enamel bond strength would be clinically



Table 4: Weibull Analysis of Experimental Groups

Groups	Subgroups	m	r <sup>2</sup>	$\sigma_{0.05}$ , MPa	$\sigma_{0.9}$ , MPa
Bonding	New	21.4	0.93	23.97	28.63
	Old	13.16	0.96	21.4	28.36
Silane/Bonding	New	7.24	0.97	41.67	70.46
	Old	17.46	0.93	43.84	54.51
Etching/ Silane/Bonding	New	5.16	0.94	29.03	60.67
	Old	11.07	0.97	31.15	43.92

Abbreviations: m, Weibull modulus; r<sup>2</sup>, correlation coefficient;  $\sigma_{0.05}$  and  $\sigma_{0.9}$  stress levels at 5% and 90% fracture probabilities, respectively.

acceptable.<sup>30</sup> Some other studies showed that composite repair SBS rates greater than 18 MPa or 20 to 25 MPa are clinically acceptable for survival in occlusal function.<sup>31-33</sup> Subsequently, results of the present study would suggest that methods using a silane and a self-etching bonding system would produce adequate repair bond strengths. In the present study, samples in group 2 (silane bonding) were roughened, followed by use of a primer and a silane. This group showed the highest SBS of all repaired specimens. However, results of group 1 (bonding) with the lowest bond strength were as well within the acceptable range.<sup>31-33</sup> In this study, the significantly lowest SBS was found in the control group with no treatment; this resulted in a significant decrease in bond strength after long-term storage.

### Aging

When a restoration is restored in the oral cavity for a long time, this means that it has been subjected to humidity, and therefore its free radicals have been eliminated.<sup>34</sup> In this study, to stimulate the aging process, composite resins were stored in 37°C distilled water for three weeks.<sup>21,22,35</sup> This approach was based on a study of Pillar and others, who found that water storage for less than 28 days did not affect fracture resistance of the composite.<sup>36</sup> In this study, boiling the specimens was not used as a method of stimulating the aging process, because Brendeke and Ozcan showed that microcracks and filler dissolution will happen following the boiling process.<sup>37</sup> All repaired specimens were placed in water and thermocycled to simulate the stress placed on

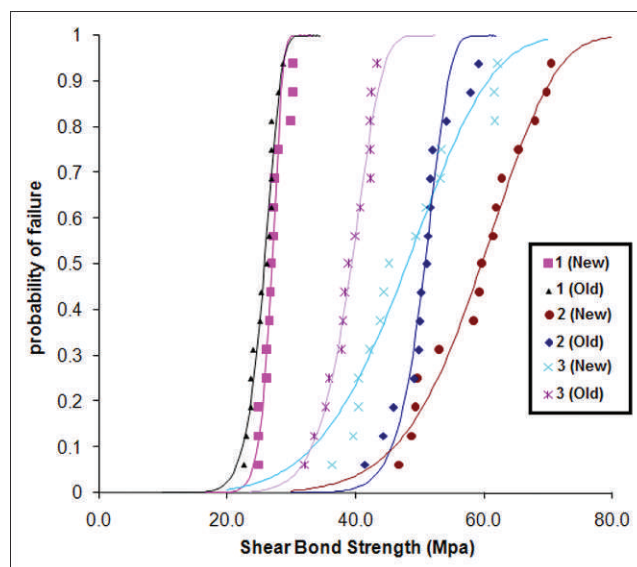


Figure 3. Plot of probability of failure vs the SBS (MPa) of different surface treatments.

interfacial bonds.<sup>38</sup> After thermocycling, to consider the stability of composite repairs in water, half of the specimens were stored in distilled water for a period of six months.

### Surface Roughening

In the experimental groups, all aged composites were roughened by diamond burs to obtain a higher bond before any additional treatments were provided. The phenomenon that aged composites roughened with a diamond bur might exhibit higher bond strengths was shown by Bonstein and others.<sup>21</sup> Also, Bouschlicher and others found that surface roughening is necessary for creating macro and micro retention. They showed that increasing exposed filler particles besides providing silane treatment could produce a higher SBS.<sup>20</sup> Crumpler and others concluded that surface roughness might enhance the ability of fresh composite to interlock mechanically into the substrate, because increased surface area is available for micro-mechanical bonding.<sup>39</sup> Söderholm and Roberts went further and concluded that surface abrasion was the single most important factor during composite repair.<sup>6</sup>

### Treatment With 40% Phosphoric Acid Etchant

In this study, group 2 (silane bonding without etching) showed the highest SBS before and after long-term storage. This value was statistically significantly different compared with that of group 3 (etching/silane bonding). Also, significant differences in the bond strengths of these two groups were

detected after six months of storage. Previous studies showed no increase in bond strength when repaired composites were treated with phosphoric acid.<sup>11,19,21,40,41</sup> Also, Fawzy and others reported that acid etching exercises provide only a superficial cleaning effect, which removes debris and grinding dust from the resin-based composite surface.<sup>42</sup> Not only was the application of phosphoric acid etchant ineffective in this study, it also had a negative effect on the SBS (compared with group 2). These differences might be attributed in part to the manufacturing and chemical composition details and the quality of different brands used in these studies.<sup>15</sup>

### Effects of Silane and Bonding System

Application of silane and the primer followed by the bonding resin showed the highest SBS values after short-term or long-term water storage times; this finding was in agreement with the findings of several previous studies.<sup>20,22,43,44</sup> Both groups that provided silane treatment had higher SBS values when compared with groups lacking the silane application. This may be due to the formation of siloxane bonds between fillers and the polymer matrix. These results supported the findings of several previous studies showing that the application of silane and unfilled resins can improve bond strength,<sup>22,36,41,45-47</sup> and contrasted with the results of some other studies reporting that the addition of silane to the bonding system might not significantly improve bond strength.<sup>15,16</sup> Controversy might be rooted in the different protocols and materials employed in these studies.

However, acid etching of the composite surface contributed to reduction of repair SBS in the group containing silane. In addition, Weibull analysis showed that treatment of the aged composites with silane and the bonding agent without acid etching produced the most reliable bond strength.

### Extended Water Storage

In this study, a trend of decreasing bond strength was noted when repaired specimens were stored for six months in distilled water in all groups. This phenomenon was dominant in groups treated with the silane coupling agent and the bonding resin. These groups exhibited less difference in SBS when compared with group 1 (bonding). This phenomenon is consistent with the findings of Söderholm and others, who showed that water diffusion might weaken the bonded area.<sup>48</sup> The adhesive/silane system has potential water uptake caused by the presence of hydroxyl, carboxyl, and phosphate

groups; this may cause the monomer to be more hydrophilic, and hence more prone to water sorption. This leads to reduction of the mechanical properties of the polymer.<sup>49</sup>

### Technique Sensitivity

Weibull analysis provides information about the variability of results, reflecting the structural reliability of materials or bonded assemblies.<sup>26,50,51</sup> The Weibull modulus ( $m$ ) is an experimental constant related to the defect size distribution.<sup>52</sup> The probability of failure, calculated from the Weibull distribution, has been considered a suitable alternative for analyzing SBS data and may be useful in selecting techniques or materials that are less technique sensitive.<sup>53-55</sup> In this study, the lowest probability of failure was obtained in the groups repaired with silane and the bonding agent, and the highest probability of failure was noted in the group treated with the bonding agent only, both before and after long-term storage. However, the bond strengths of all experimental groups fell within the acceptable range for bearing occlusal load.

### Fracture Type

In general, failure modes indicate that those groups with high bond strengths exhibit cohesive failure inside the composite. However, low bond strength groups tend to exhibit adhesive failure rather than cohesive failure. Fractures within the composite resin (cohesive failure) seem to be more appropriate for bearing occlusal loads.<sup>20</sup> In the present study, failure mode analysis revealed that the SBS of group 2 (silane bonding) was significantly different from that of acid-etched composites. No difference in failure modes was noted after six months of aging in three groups, but in the acid-etched group, a slight improvement in the failure mode was not significant.

However, it should be noted that it cannot be certainly determined using a light microscope whether failure was truly adhesive. Therefore, more mixed failures than those observed may have occurred, and results should be cautiously interpreted.

### Limitations

This study was limited by some factors. Findings regarding a specific brand should be cautiously generalized to other brands.<sup>15</sup> However, consistency between the results of this study and those of several other studies might confirm the advantage of silane

usage regardless of the brand used. Moreover, during long-term water storage, specimens were not subjected to any occlusal loadings. Finally, surface roughening was performed in all experimental groups. Future studies should include some experimental groups in which the effects of surface roughening without subsequent chemical treatments could be observed. In addition, some control groups should be included, in which only the effects of chemical treatments (without surface roughening) could be assessed. These measures exceeded the available time and budget projected for this study but might be considered in further studies.

### CONCLUSIONS

Within the limitations of this *in vitro* study, the following points could be concluded:

1. Treating the surface of the aged composite is necessary when fresh composite is added. With no treatment, nearly no bond exists between the two materials.
2. All surface treatments studied here showed acceptable results.
3. Use of a silane bonding agent before application of the adhesive after mechanical grinding produced the best results.
4. Acid etching of the composite surface might negatively affect the SBS and hence is not recommended.
5. Long-term water storage considerably decreased the repair SBS, regardless of the treatment used.

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