Repair Bond Strength of Aged Composite Resins Using Different Surface Treatment Protocols

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This study evaluated shear bond strength (SBS) of thermally aged composite resins repaired using different surface protocols. Four-hundred composite resin samples were made using the following materials (100 samples per material): Filtek Z350XT (FXT); Spectra Smart (SSM); IPS Empress Direct (EDI); and Forma (FOR). Each group’s samples were then divided into 10 groups (n = 10 samples per group): G1: no surface treatment; G2: phosphoric acid-etching + universal-adhesive (PU); G3: surface roughening + PU (RPU); G4: RPU + silane (RPSU); G5: surface roughening + hydrofluoric acid-etching + universal adhesive (RHU); G6: RHU + silane (RHSU); G7: dry sandblast + PU (DsPU); G8: DsPU + silane (DsPSU); G9: wet sandblast + PU (WsPU); and G10: WsPU + silane (WsPSU). G1 was freshly repaired, and G2 to G10 were thermally aged before repair. Specimens were tested for SBS, and the failure type was observed with a magnifying loupe. Representative images were obtained using a scanning electronic microscope. Data were analyzed by two-way analysis of variance and Tukey post hoc tests (P = .05). Differences were detected among different surface treatments and among different composite resins with equal surface treatments (P < .05). SBS means ranged from 10.48 (FOR:G2) to 20.70 (FXT:G7). The highest SBS values were seen in G7 to G10 (P > .05), while lowest values were generally observed for G2. G1 showed higher results compared to G2 (P < .05), except for EDI (P > .05). Most failures corresponded with cohesive type. In general, thermally aged composite resin presented a decreased repair bond strength potential when no additional surface treatment was applied. Sandblasting improved the SBS of repaired aged composite resins. Int J Periodontics Restorative Dent 2023;43:e53–e60. doi: 10.11607/prd.5026

In an era of conservative dentistry, maintaining function of a restoration for as long as possible is considered paramount for the clinical success of treatments. Continuously replacing a composite resin restoration might cause excessive wear of sound tooth structure,¹ and repairing restorations may increase its longevity.² Nevertheless, for a composite resin repair to be successful, a durable bond must be obtained within the old restoration and the new resin material.³ Different methods have been proposed to achieve macromechanical or micromechanical retention and/or chemical adhesion.⁴ Such techniques relate to the application of phosphoric acid,⁵ hydrofluoric acid,⁶ airborne-particle abrasion,⁷ preparation using burs⁸ or abrasive papers⁸,⁹ silanes,¹,³ and universal adhesives,³ among others. Although the use of phosphoric acid is effective for promoting retention on enamel and dentin tissues, its efficiency is limited to old composite resin restorations, only cleaning the surface to be repaired.¹⁰ On the other hand, hydrofluoric acid is capable of dissolving glass filler particles, but its efficiency depends on the type of composite resin used.⁶ Regarding airborne-particle abrasion and bur preparation, their main function is to promote increased surface roughness and free surface energy.¹ Finally, silanes promote a chemical bond

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between the resin matrix and filler particles by increasing the wettability of the surface that will receive the adhesive material. The challenge of repairs is the lack of free radicals available for adhesion with the new material and the water absorption over time. Moreover, chemical degradation of the old composite resins and leaching of compounds decrease the reactivity of the material, and thus the previously described methods must be taken into account in order to prepare the surface and increase bonding. Other factors that can affect resin repair are the experience, knowledge, and judgment of the clinician; no consensus exists regarding a definitive standardized clinical protocol.

This study evaluates the effect of different types of repair protocols on the shear bond strength (SBS) of different composite resin types, both freshly prepared and after undergoing thermal cycling. The null hypotheses of this study were as follows: (1) No significant difference in SBS values would be observed between different composite resin repair protocols after aging; and (2) composite resins would behave equally when a resin repair protocol is performed.

### Materials and Methods

#### Preparation of Samples

A total of 400 cylinders (7-mm diameter, 2-mm height) were made using four different composite resins (n = 100 cylinders per resin): Filtek Z350 XT (FXT) by 3M ESPE, shade A2E; Spectra Smart (SSM) by Dentsply Sirona, shade A2 Universal; IPS Empress Direct (EDI) by Ivoclar Vivadent, shade A2E; and Forma (FOR) by Ultradent, shade A2. The sample size was determined according to a previous study. The compositions of the resin materials used in this experiment are listed in Table 1.

Resin cylinders were made in a single increment of composite resin and were photoactivated with a light-curing unit (Bluephase Style 20i, Ivoclar Vivadent) on high mode (1,200 mW/cm²); this was verified every 5 specimens by a Bluephase Meter II radiometer (Ivoclar Vivadent) for 20 seconds, with the light probe tip positioned at a 90-degree angle to the surface. Then, the resin cylinders were fixed into autopolymerizing polymethylmethacrylate resin (Inlay Pattern Resin, clear, Dur-O-Lay) using polyvinyl chloride molds supported on a plastic tray, with

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**Table 1 Composition of the Composite Resins and Adhesive Used**

<table>
<thead>
<tr>
<th>Product</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Batch no.</th>
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<tbody>
<tr>
<td>Filtek Z350 XT (shade A2E)</td>
<td>3M ESPE</td>
<td>Bis-GMA, UDMA, TEGDMA, and Bis-EMA, a combination of non-agglomerated/non-aggregated silica filler, non-agglomerated/non-aggregated zirconia filler, and aggregated zirconia/silica cluster filler (comprised of silica and zirconia particles). Filler size: 4–20 nm. Filler content: 72.5 wt%</td>
<td>N845129</td>
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<tr>
<td>Spectra Smart (shade A2 Universal)</td>
<td>Dentsply Sirona</td>
<td>Bis-GMA, TEGDMA, CQ, EDAB, and BABG and BAFG glass with nano-sized silicon dioxide filler. Filler size: 0.02–3.0 µm (average: 0.7 µm). Filler content: 75–77 wt%</td>
<td>312287J</td>
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<tr>
<td>IPS Empress Direct (shade A2E)</td>
<td>Ivoclar Vivadent</td>
<td>UDMA, TCDD, Bis-GMA, barium glass fillers, Ba-Al-fluorosilicate, glass, prepolymer, mixed oxide, catalysts, stabilizers and pigments. Filler size: 0.4–0.7 µm (barium glass), 1–10 µm (prepolymer); 150 nm (mixed oxide). Filler content: 78.1 wt%</td>
<td>X15428</td>
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<tr>
<td>Forma (shade A2)</td>
<td>Ultradent</td>
<td>Bis-GMA, TEGDMA, Bis-EMA, and UDMA, zirconia/silica and barium glass. Filler size: 0.7 µm (average). Filler content: 67 wt%</td>
<td>DC5DW</td>
</tr>
<tr>
<td>Single Bond Universal</td>
<td>3M ESPE</td>
<td>MDP phosphate monomer, HEMA, dimethacrylate resins, ethanol, vitrebond copolymer, silane, water, fillers, ethanol, initiators.</td>
<td>643243</td>
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</table>

BABG = bariumaluminoborosilicate; BAFG = bariumaluminofluoroborosilicate; Bis-EMA = bisphenol A ethoxylated dimethacrylate; Bis-GMA = bisphenol A diglycidyl methacrylate; CQ = camphoroquinone; EDAB = organic amine ethyl-4-dimethylaminobenzoate; HEMA = 2-hydroxyethyl methacrylate; MDP = methacryloyloxoydecyldihydrogen phosphate; TCDD = tricyclodecane dimethanol dimethacrylate; TEGDMA = triethylene glycol dimethacrylate; UDMA = urethane dimethacrylate.
6-mm perforations where the resin cylinder was centered (with the propose of protecting the testing surface), and fixed with double-sided bonding tape (3M ESPE) with perforations of equal diameter.

Afterwards, fixed cylinders in each material group were randomly assigned to one of 10 different intervention groups (n = 10 cylinders per intervention per material; Table 2). Samples were immersed into distilled water and stored in a hot-air oven (Air Forced Laboratory Incubator, MRC) at 37°C for 24 hours, except for the first group (positive control), where an increment of composite resin was layered over the fresh composite resin samples, with the oxygen inhibited layer still intact, 5 minutes after fabrication. The specimens in the remaining groups were thermally aged, with 5,000 thermal cycles (5°C to 55°C, 30-second dwell time) performed using a thermal cycling machine (fabricated and donated by the Civil Engineering Department of the Universidad de los Andes). The repair surface treatment protocols were as follows: (1) G1: no surface treatment (positive control); (2) G2: phosphoric acid-etching (Condac 37, FGM) + universal adhesive (Single Bond Universal, 3M ESPE) (PU); (3) G3: surface roughening + PU (RPU); (4) G4: RPU + silane (Prosil, FGM) (RPSU); (5) G5: surface roughening + hydrofluoric acid-etching (Condac Porcelana, FGM) + universal adhesive (RHU); (6) G6: RHU + silane (RHSU); (7) G7: dry sandblast + PU (DsPU); (8) G8: DsPU + silane (DsPSU); (9) G9: wet sandblast + PU (WsPU); and (10) G10: WsPU + silane (WsPSU). A graphical description of each group is shown in Fig 1.

The same universal adhesive (U) system was used for the groups, following manufacturer instructions. Surface roughening was performed (R) using abrasive paper (silicon carbide paper; SiC Paper #320, Struers) and a grinding machine (LaboPol-6, Struers) at 600 rpm under water irrigation, standardizing pressure using polyvinyl siloxane matrix. Sandblasting, both wet (Ws) and dry (Ds), was performed with 50-μm alumina-oxide (Al₂O₃) particles for 10 seconds, keeping a 10-mm distance from the treated surface, using an intraoral sandblaster device (Dental Air Polisher AP-H, Woodpecker) attached to conventional dental unit;
when Ws was performed, the water irrigation system was activated. The device tip was positioned at a 90-degree angle to the testing surface. Orthophosphoric (P) and hydrofluoric (H) acid etching were performed for 20 seconds, then the samples were rinsed with water and dried. Silanization (S) was performed with a microbrush, and the samples were left to dry until the surface had an opaque appearance.

Resin repair was performed with a Bonding Mold Insert and a Bonding Clamp (Ultradent) with the same composite used to fabricate the resin cylinder, photoactivated as previously described. All specimens were immersed into distilled water and stored in a hot air oven (Air Forced Laboratory Incubator) at 37ºC for 24 hours.

**SBS Test**

Repaired resin cylinders were subjected to SBS testing using a Microtensile Tester (Bisco); load was applied with a speed of 0.5 mm/minute until failure. SBS data were converted to MPa (N/mm²) and analyzed with Shapiro-Wilk test for normality. Due to normal data distribution, two-way analysis of variance and Tukey post hoc tests were performed for 20 seconds, then the samples were rinsed with water and dried. Silanization (S) was performed with a microbrush, and the samples were left to dry until the surface had an opaque appearance.

Resin repair was performed with a Bonding Mold Insert and a Bonding Clamp (Ultradent) with the same composite used to fabricate the resin cylinder, photoactivated as previously described. All specimens were immersed into distilled water and stored in a hot air oven (Air Forced Laboratory Incubator) at 37ºC for 24 hours.

**Results**

Regarding the effects of different surface repair treatments on SBS of different conventional composite resins (Table 2, Fig 2), statistical analysis revealed significant differences within groups, both regarding the same composite resin within different surface treatments (P < .05) and different composite resins within the same surface treatment (P < .05). SBS means ranged from 10.48 (FOR:G2) to 20.70 (FXT:G7). Generally, G1 (positive control group) showed significantly higher SBS values (P < .05) compared to other surface treated groups, except for EDI.

In general, for all composite resins, surface repair treatment protocols from G7, G8, G9, and G10 showed the highest SBS results (P < .05), while the lowest SBS values were observed for G2, which was significantly different (P < .05) compared to other surface treated groups, except for EDI.

When different composites were compared, no significant differences were observed among them for G2, G5, G7, G8, G9, and G10 surface treatment protocols (P > .05). For the other groups, different composite resins behaved differently. For G1, FOR and SSM showed the highest SBS values after repairing, both significantly different than FXT and EDI (P < .05). For G3, the highest SBS values were

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**Table 2** **Mean Shear Bond Strength by Group**

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<tbody>
<tr>
<td>Filtek Z350 XT (FXT)</td>
<td>13.67 ± 2.48</td>
<td>10.99 ± 1.16</td>
<td>17.29 ± 2.49</td>
<td>13.60 ± 1.66</td>
<td>15.53 ± 3.24</td>
<td>15.73 ± 1.21</td>
<td>20.70 ± 1.31</td>
<td>19.11 ± 1.12</td>
<td>18.68 ± 0.71</td>
<td>20.15 ± 1.02</td>
</tr>
<tr>
<td>Spectra Smart (SSM)</td>
<td>18.26 ± 1.80</td>
<td>11.06 ± 1.01</td>
<td>16.38 ± 1.42</td>
<td>16.23 ± 1.24</td>
<td>14.54 ± 1.17</td>
<td>17.85 ± 1.71</td>
<td>18.60 ± 0.97</td>
<td>18.45 ± 0.92</td>
<td>17.34 ± 1.19</td>
<td>19.13 ± 1.73</td>
</tr>
<tr>
<td>IPS Empress Direct (EDI)</td>
<td>12.43 ± 1.70</td>
<td>11.31 ± 0.96</td>
<td>13.33 ± 1.04</td>
<td>13.96 ± 1.03</td>
<td>16.63 ± 1.78</td>
<td>17.32 ± 1.04</td>
<td>20.37 ± 0.94</td>
<td>18.34 ± 1.38</td>
<td>17.63 ± 1.09</td>
<td>18.38 ± 1.11</td>
</tr>
<tr>
<td>Forma (FOR)</td>
<td>17.44 ± 2.54</td>
<td>10.48 ± 1.09</td>
<td>14.58 ± 1.32</td>
<td>16.32 ± 1.90</td>
<td>15.88 ± 0.74</td>
<td>18.33 ± 0.98</td>
<td>19.00 ± 1.10</td>
<td>17.86 ± 1.18</td>
<td>18.91 ± 0.85</td>
<td>18.61 ± 1.08</td>
</tr>
</tbody>
</table>

Ds = dry sandblasting; H = hydrofluoric acid-etching; P = phosphoric acid-etching; R = roughening; S = silane; U = universal adhesive; Ws = wet sandblasting.

G1 is the positive control and G2 is the negative control.

Data are presented in MPa as mean ± SD. Letters indicate significantly significant differences (P < .05). Uppercase letters indicate differences by row (composite resin). Lowercase letters indicate differences by column (group).

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observed for FXT, which were significantly different compared to EDI and FOR \( (P < .05) \) and were similar to SSM \( (P = 1.000) \). For G4, SSM and FOR showed the highest SBS values, similar to EDI \( (P > .05) \), but significantly greater than FXT \( (P < .05) \). Lastly, for G6, the highest SBS was observed for FOR, similar to SSM and EDI \( (P > .05) \) but significantly greater than FXT \( (P < .05) \), which in turn was also similar when compared with SSM and EDI \( (P > .05) \).

Regarding the distribution of failure types (Fig 3), most were cohesive failures (failure inside the composite resin), followed by adhesive failure, which showed higher percentages when \( H \) was used to perform the surface treatment. Mixed failures were only observed on SSM G2 and G3 samples and on FOR G5 samples. Representative SEM images were taken of each failure type after SBS testing (Fig 4).

**Discussion**

This study evaluated differences in SBS values between different composite repair protocols after aging, and between different composite resins when the same resin repair was performed. The null hypotheses were rejected, as significant differences \( (P < .05) \) were found between protocols and composite resins.

Composite repair success is dependent upon achieving a functional restoration with a lasting adhesion at the newly created interface.\(^2\) Dealing with a union affected by the degradation of the composite in oral conditions\(^15\) could reduce the radical activity of monomer functional groups of the restorative material.\(^16\) In the present study, in order to simulate this degradation, samples from groups G2 to G10 were thermally cycled,\(^17\) using time and temperatures \( (5,000 \) thermal cycles, \( 5^\circ C \) to \( 55^\circ C \) \) that correspond to about 6 months of aging.\(^18\) The reduced radical activity of the aged group with only orthophosphoric acid surface treatment (G2, negative control) could explain the differences detected,\(^12\) as different composite resin repair protocols showed different SBS results after aging.

SBS means ranged from 10.48 (FOR:G2) to 20.70 (FXT:G7), similar to other studies.\(^16\) In almost all composite groups, G2 showed the lowest SBS values, which were significantly lower than G1 (positive control) with the exception of EDI samples, where no significant
differences were detected. In addition, G1’s increased repair bond strength potential can be explained by the cohesive strength of the composite resin, given that all the specimens from this group presented cohesive failure, agreeing with the results from other studies. When evaluating failure type of all samples, adhesive strength surpassed the cohesive strength of the composite resins, as it corresponded to cohesive fracture in most cases; this was followed by adhesive failure, where H generally seemed to reduce adhesive strength, as higher percentages of adhesive failure were observed when H was used to perform the surface treatment. Mixed failures were only observed on SSM G2 and G3 samples and on FOR G5 samples.

In order to increase the durable bond established between aged and new composite materials, macromechanical, micromechanical, and chemical surface treatments are recommended. When performing resin repair surface treatments, acid etching is commonly used to promote a cleansing effect in the aged composite resin and because it
contributes to adhesion when dental substrate is present. All aged groups underwent acid etching (P or H), combined with U. In clinical conditions, U and P are used for adhesion to enamel and dentin and for resin repair protocols. Because different chemical compositions of adhesives may affect SBS values, all aged groups were repaired using the same adhesive system. However, chemical surface treatment with H can also have a micromechanical effect on the composite resin surface, depending on its composition. In the present study, a specific action of H alone on SBS values could not be detected, but it did not seem to produce a significant improvement in SBS values with the exception of EDI samples, in which G5 and G6 presented higher SBS values than G3 and G4, probably due to their high content of barium glass fillers, which could have made the samples more reactive to H. In the present study, it was decided not to compare the influence of H in sandblasted groups, which would have made the resin repair protocols more complex, and the study intended to focus on building an effective and simplified protocol; also, surface treatment with H has its limitations, as resin repair is executed intraorally and H could potentially damage the soft tissues.

In vitro studies have shown favorable results using silane, before adhesive system application, over the aged composite resin surface. Silane application increases the methacrylate groups’ reactivity of the repaired resin and improves the wettability of the adhesive system. Some of the present surface treatment protocols included silane, showing significantly higher values only for SSM and FOR when applied in G6 samples (RHSU), compared to G5 samples (RHU). For G4 (RPSU), significant differences were not detected when compared with G3 (RPU) and among sandblasted groups.

Microretention on the repaired composite resin surface provides a greater contact area for bonding on the aged composite surface. In the present study, Ds and Ws promoted better micromechanical retention than R and showed the best SBS values, which is in accordance with other studies. Moreover, the bonding to exposed filler particles present in sandblasted groups could have been improved by the silane present in the composition of U used. No significant differences were detected between Ds and Ws samples with the exception of EDI, where the SBS of G7 samples (DsPU) was significantly higher than G9 samples (WsPU). No references were found in the literature comparing these two sandblasting methods. Regarding R, better SBS performance was observed when combined with other treatments, as seen in SSM:G6 (RHSU; results equal to sandblasted groups), EDI:G5 (RHU) and G6 (results similar to sandblasted groups); and FOR:G6 (results equal to sandblasted groups).

It must be noted that in the present in vitro study, the composite resin used to create the cylinders was known in advance, allowing the repair to be performed with the same composite used to make the sample tested; usually, this valuable information is unknown in most clinical cases, thus being a limitation in this study. Future studies should focus on performing resin repairs with a different composite resin than the one used for fabricating the samples. Despite the several studies that have been performed to evaluate resin repair protocols, there still is not a standardized technique to repair all composite resin due to the different reactions of these materials to certain repairing protocols. The present results suggest that micromechanical surface treatment with sandblasting treatments, either wet or dry, is highly indicated for any resin repair protocols.

Conclusions

The results of the present study conclude that: (1) Aged composite resin without sandblasting or surface-roughing treatment may decrease the repair bond strength potential compared to fresh composite resin; (2) micromechanical surface treatment protocols using sandblasting (with 50-μm Al₂O₃ particles), either wet or dry, improve the SBS of aged, repaired composite resin when combined with chemical surface treatment; (3) SBS differences among composite resins were detected only when the same surface treatment protocol was applied without including sandblasting; and (4) sandblasting is highly indicated when a composite resin repair is going to be performed, even if old composite resin composition is unknown.
Acknowledgments

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