Effect of Different Surface Treatments and Repair Materials on Shear Bond Strength of Bulk-Fill Resin Composite

Alyaa A. Soliman, Rabab Mehesen, Ashraf I. Ali

Abstract:

Objective: To evaluate the effect of different surface treatments on shear bond strength (SBS) between bulk-fill resin composite (BFRC) and bulk-fill flowable resin composite (BFFRC) or conventional flowable resin composite (CFRC).

Materials and Methods: Sixteen blocks were prepared from Tetric N-Ceram BFRC, aged by thermo-cycling, and then stored in artificial saliva for 6 months. The blocks were divided into 4 groups (n=4) according to the surface treatment; Group 1: roughened by coarse discs, Group 2: roughened by coarse discs and silanization, Group 3: roughened by air abrasion, and Group 4: roughened by air abrasion and silanization. Each group was subdivided into 2 subgroups (n=2) according to the used repair restorative system; subgroup A repaired with Tetric N-Bond universal adhesive/ Tetric N-Flow BFRC, and subgroup B was repaired with Prime&Bond universal adhesive /Spectra ST flow CFRC. The repair RC was placed on the RC blocks using tygon tubes and cured. The specimens were tested for SBS using a universal testing machine.

Results: Regarding the effect of different surface treatments for both repair restorative systems, there was a significant difference (P<0.001). Regarding the effect of silane, there was no significant difference (p= 1.00). There was no significant difference between repair with CFRC or BFFRC (P =0.679).

Conclusions: Surface treatment by air abrasion followed by silane and universal adhesive can be attempted clinically for the repair of aged BF restoration. The aged BFRC could be effectively repaired with the same bulk-fill or conventional RC if proper repair protocol was used.

Introduction:

Tooth restoration with resin composite (RC) materials is widely used due to its esthetic properties, adhesion, and adequate mechanical properties. During clinical service, dynamic variations in pH and temperature in the oral cavity lead to the degradation of RC materials. These changes can occur in various phenomena including micro-leakage, discoloration, wear, or fracture, and may lead to the replacement of the restoration. Total replacement may weaken the tooth structure, take a long time, and harm the dental pulp. Therefore, repairing the RC materials is an option to conserve tooth structure and reduce intervention.

The adhesion between old and new RC depends on the presence of the oxygen inhibition layer. This layer includes unreacted monomers, which can copolymerize with the newly added RC. The absence of this layer is the most difficult aspect of RC repair process. So, the aged RC needs to be activated mechanically and chemically to improve the adhesion of repaired RC, however, there is no agreement on the technique or materials that should be applied for the repair process.

To simulate intra-oral conditions, laboratory aging of RC is necessary. Different methods have been used for this purpose, including thermo-cycling, artificial saliva storage, immersion in distilled water, boiling, or even in vitro exposure to citric acid and sodium chloride solutions. Thermo-cycling continues to be one of the most commonly used methods of RCs aging, and the storage in artificial saliva simulates intraoral degradation of RC restorations.

The shear bond strength (SBS) test is simple in vitro test for determining the bond strengths of materials having a high surface area (usually 3–6 mm in diameter). In addition, in vitro bond strength tests are important for evaluating the performance of an adhesive system and can be correlated to the clinical situation. It was reported that loading of the specimens under shear may be considered clinically more significant than tensile or flexural loading as it produces elements of shear, tensile and compressive stresses that commonly occur during chewing.

Conventional tooth restoration with RCs is generally done with layering or incremental technique. Each layer should be about 2 mm thick to achieve complete polymerization. New improved RC properties such as Bulk-fill resin composite have been developed. It is often used due to its increased depth of cure from 2 to 4 mm and less polymerization shrinkage. These features can save time and provide adequate strength for application. There is limited research about the repair potential and effectiveness of repair techniques of bulk-fill resin composites (BFRCs). So, this current study aimed to investigate the SBS of an artificially aged BFRC repaired with bulk-fill flowable resin composite (BFFRC) and conventional flowable resin composite (CFRC) by using different surface treatment techniques. The null hypothesis of this study was; that there was no significant difference in shear bond strength of repaired bulk fill resin composite using different surface treatment techniques. Also, there was no significant difference in using different resin composite repair materials.

Null hypotheses

The following null hypotheses were tested; there was no significant difference in shear bond strength of repaired...
bulk fill resin composite using different surface treatment techniques. Also, there was no significant difference in shear bond strength of repaired bulk fill resin composite using different resin composite repair materials.

**Materials and Methods:**

Three different RC restorative materials were used in this study; Tetric N-Ceram Bulk Fill, Tetric N-Flow Bulk Fill, and Spectra ST flow. The flowable resin composites were bonded with their corresponding universal adhesive, Silano silane coupling agent was used also.

**Specimens preparation:**

Sixteen blocks were prepared from bulk-fill resin composite (Tetric N-Ceram, Ivoclar Vivadent, Schaan, Liechtenstein) using a stainless steel mold with dimensions of 25 mm length, 4 mm width, and 4mm height. The mold was placed on a glass slide then the RC was packed into the mold, in one increment using a clean non-stick titanium coated applicator. After that, RC surface was covered with a Mylar strip and compressed with compressed air. The top surface of the blocks was polished using supra-fine flexible discs with abrasive particles size 8µm (TOR VM Ltd, Moscow, Russia) in a slow-speed handpiece. The blocks were subjected to a thermo-cycling aging protocol between temperatures 5-55°C with 15 sec dwell time and transfer time of 5 sec in distilled water for 500 cycles using a thermo-cycling machine (ROBOTA, Alexandria, Egypt). Then, the blocks were stored in artificial saliva, for 6 months at37°C using an incubator (BTC, BioTech Company, Cairo, Egypt) till the time of testing. The artificial saliva was changed periodically every 5 days.

**Grouping, surface treatment, and repair procedures of the specimens:**

All the blocks were randomly divided into four groups equally (n=4) according to the surface treatment: Group 1: in which the blocks had surface treatment of roughening by coarse discs with abrasive size 70-90 µm (TOR VM Ltd, Moscow, Russia). Group 2; in which the blocks had surface treatment of roughening by coarse flexible disc and silanization (Silano silane). Group 3; in which the blocks had surface treatment by air abrasion device (Bio-art Micro Jato, Bio-art Equipamentos Odontologicos LTDA, Brazil). Group 4; in which the blocks had surface treatment by air abrasion device and silanization. After that, each group was subdivided into two subgroups (n=2) according to the repair restorative system used. Subgroup A was repaired with BFFRC and bonded with its corresponding universal adhesive (Tetric N-Flow Bulk Fill /Tetric N-Bond universal adhesive). Subgroup B was repaired with CFRC and bonded with its corresponding universal adhesive (Spectra ST flow/Prime&Bond universal adhesive).

For group 1, the block surface (unmarked) was roughened using coarse discs with abrasive size 70-90µm (TOR VM Ltd, Moscow, Russia). The discs were mounted on a slow-speed handpiece (Bien Air Dental, Bienne, Switzerland) at 20,000 rpm. A new disc was used for each block to ensure adequate equal roughness. Then, the blocks were cleaned by the etching gel, which was applied on the surfaces of the block for 15 sec, rinsed with water for another 15 sec, and dried with compressed air to remove debris of resin composite from the mechanical preparation. After that, the blocks of subgroup A were repaired with (Tetric N-Flow Bulk Fill / Tetric N-Bond universal adhesive). Tetric N-Bond universal adhesive was applied and agitated for 20 sec, the adhesive was dispersed with compressed air until a glossy firm layer was obtained and light-cured for 10 sec. The block was attached to five tygon tubes (2mm height and 3mm internal diameter). A low viscosity RC (Tetric N-Flow BF) was injected into the tygon tube till full filling. Each tube was light-cured for 10 sec from the top side according to the manufacturer's instructions. After the polymerization, the tubes around the RC cylinders were removed using a scalpel blade, each cylinder was light-cured again for 10 sec from each side according to the manufacturer's instructions. The specimens were stored in distilled water at 37°C for 24 h in an incubator until they were tested. The blocks of subgroup B were repaired with (Spectra ST flow / Prime&Bond universal adhesive). The Prime&Bond universal adhesive was applied and agitated for 20sec, dried with gentle air drying for approximately 5 sec to evaporate the solvent, and light-cured for 10 sec. The block was attached to 5 tygon tubes, composite inserted, and cured as mentioned in subgroup1.

For group 2, all the steps were the same as that of group1, and before the application of the universal adhesive, the silane coupling agent was applied according to the manufacturer’s instructions. One layer was applied and left for 1 minute, and another layer was applied and dried lightly with air. Then, the adhesive and repair materials were applied as mentioned before. For group 3, the blocks were sandblasted using an air abrasion device (Bio-art Micro Jato, Bio-art Equipamentos Odontologicos LTDA, Brazil) that uses a 50 µm sized aluminum oxide particles stream (Al2O3). It was applied perpendicular to the surface for 10 sec under 2 bar pressure for each block. The nozzle tip was placed 10 mm far from the block surface in slow motions. After that, the block was cleaned with etching gel (Scotchbond™ Universal Etchant, 3M ESPE), which was applied to the surfaces of block for 15 sec, rinsed with water for another 15 sec, and dried with compressed air. After that, they were rinsed with...
water for 20 sec, air-dried for 10 sec. The blocks were bonded and restored as mentioned in the group 1. For group 4, the blocks were sand-blasted as in group 3, followed by the application of silane coupling agent (Silano), universal adhesive, and repair materials as mentioned before in group 2.

Shear Bond Strength test:

The shear bond strength was determined after additional storage in distilled water at room temperature for 24h. The specimens were fixed on the lower stationary part of a universal testing machine (Instron, model 3345, England) using a specially designed metallic jig. The used shear force was applied by a chisel-shaped loading device and was parallel to the adhesive interface until failure occurred. This load was applied to the adhesive interface, as close as possible to the surface of the substrate at a cross-head speed of 0.5 mm/min.²⁴

Statistical analysis:

All the data of this study were collected, tabulated, and statistically analyzed using version 22 of IBM SPSS Statistics software for Windows (IBM SPSS Corporation 2013, Armonk, NY). After testing the normality using Shapiro–Wilk test, quantitative variables were summarized as mean ±SD for normally distributed data, and median, range for non-normally distributed ones. For normally distributed data, a student t-test was used to compare 2 independent Groups, and a one-way ANOVA test was used to compare more than 2 independent Groups with a Post Hoc Tukey test to detect pair-wise comparison. Mann-Whitney test was used to compare 2 or more independent Groups for non-normally distributed data. The significance of the results was judged at P-value ≤ 0.05.

Results:

For comparing the effect of different surface treatments for both repair restorative systems, a one-way ANOVA test was used. The mean shear bond strength was the highest for group 4 (113.79 ± 4.79 MPa), followed by group 3 (83.01 ± 3.79 MPa), then group 1 (76.89 ± 2.92 MPa), and the lowest value was for group 2 (64.66 ± 2.54 MPa). There was a statistically significant difference between them (P<0.001) as shown in, (Table 1).

For testing the effect of silane on results, Mann–Whitney U test was used to compare silane-containing and non-containing groups. Median shear bond strength was 87.5 MPa for groups with silane (2 and 4) which was higher than the group without silane (1 and 3) which was 79.8 MPa. There was no statistically significant difference (P= 1.00) as shown in,(Table 2).

By comparing median shear bond strengths of two flowable resin composites repair restorative systems by Mann Whitney U test, there was no statistically significant difference irrespective of the surface treatment used. Median shear bond strength was higher for conventional than bulk repair flowable resin restorative systems (80 MPa and 79.8 MPa respectively) as shown in, (Table 3).

<table>
<thead>
<tr>
<th>Group</th>
<th>Shear bond strength</th>
<th>Mean ±SD</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Roughened by coarse discs</td>
<td>76.89 ± 2.92</td>
<td>F=334.6</td>
</tr>
<tr>
<td>2</td>
<td>Roughened by coarse discs and silanization</td>
<td>64.66 ± 2.54</td>
<td>P&lt;0.001*</td>
</tr>
<tr>
<td>3</td>
<td>Roughened by air abrasion</td>
<td>83.01 ± 3.79</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Roughened by air abrasion and silanization</td>
<td>113.79 ± 4.79</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Shear bond strength</th>
<th>Median</th>
<th>Range</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>With silane (Group 2+4)</td>
<td>87.5</td>
<td>61.1 – 123.3</td>
<td>p= 1.00</td>
</tr>
<tr>
<td>Without silane (Group 1+3)</td>
<td>79.8</td>
<td>72.3 – 88.2</td>
<td></td>
</tr>
</tbody>
</table>

Table 3: Comparison of shear bond strength in MPa for different flowable resin composites within different surface treatment techniques

<table>
<thead>
<tr>
<th>Shear bond strength</th>
<th>Median</th>
<th>Range</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional</td>
<td>80</td>
<td>57 – 129.3</td>
<td>P =0.679</td>
</tr>
<tr>
<td>Bulk</td>
<td>79.8</td>
<td>61.9 -121.1</td>
<td></td>
</tr>
</tbody>
</table>

Discussion:

In reparative dentistry, the most critical aspect of effective RC repair treatments is ensuring reliable adherence to the RC restoration. Several techniques are used to improve the adhesion between old and new RC but there is no agreement on the procedure or materials that should be used for the repair process. Thus, it was essential to estimate the effect of different surface treatments and different RC repair materials on the repair of aged bulk-fill RC restorations. In this study, coarse discs with abrasive size 70-90µm were...
used to correspond to the roughness obtained by medium grain diamond bur. These coarse discs were used for macro mechanical treatment because of their availability, technical simplicity, and demonstrated success in the repair process, while micro mechanical treatment using 50 µm aluminum trioxide particles (Al2O3) is more common and has been established to increase repair bond strength.

In addition to mechanical treatment, chemical treatment which includes adhesive resin material and silane coupling agent was used. Mechanical treatments aim to improve micromechanical retention between the old and new RCs, whereas chemical treatments aim to improve chemical adhesion. The universal adhesives were used to reduce repair time, technique sensitivity, and the number of steps required. Also, each RC was utilized with its opposite adhesive during the repair procedure to acquire the best possible results from the material, as recommended by the manufacturers. Bulk-fill resin composite was employed in this study to make placing specimens easier and faster. Also, one of the most important factors influencing the success of the repair is the used type of repair resin, and the repair processes should be performed with the same RC as the original restoration. The aging procedure was performed to simulate the clinical environment by thermo-cycling and storage in artificial saliva at 37°C for 6 months to simulate the RC degradation that occurs in the oral environment.

The null hypothesis of this study that there was no significant difference between different surface treatment techniques and repair materials was rejected. The current study showed that the group roughened by air abrasion device and bonded with silane and universal adhesive showed the highest SBS values with a significant difference from the other tested groups. Several studies suggested that Al2O3 air abrasion followed by application of a silane and bonding agent as effective pretreatment methods during repair procedures. It could be explained that sandblasting with 50 µm sized aluminum oxide particles produces a more roughened RC surface and more micro retentive surface area, thus improving the surface area available for bonding. Also, the silane has the chemical ability to bond with filler particles of the aged RC. In addition, silane has higher surface wettability, facilitating infiltration of the adhesive into surface irregularities resulting from the removal of the filler particles, and thus facilitate higher repair bond strength. This was in agreement with Ahmadizenouz et al., and Kuşdemir et al., who reported that air abrasion is recommended before repairing an existing RC restoration to achieve success full repair treatment. Also, Souza et al., reported that Sandblasting with Al2O3 followed by application of silane layer produced high bond strength after RC aging. On the other hand, this study was in disagreement with Eren et al., who found that the effect of abrasive tip and air abrasion on repair bond strength was similar. The discrepancy between the two studies might be due to the different methodologies as well as the different RC types that were used. The microstructure, abrasion resistance, and composition of the materials are all factors that influence the effect of air abrasion on the surface.

The results of this study showed that the roughening by coarse discs followed by silane and bonding with universal adhesive had the lowest bond strength. This could be explained by the roughening with coarse discs tending to produce a very irregular surface which may be not retentive ideally leading to a decrease in the surface area available for mechanical interlocking with the adhesive resin and the repair RC. Also, the thick intermediate layer created by the application of silane and adhesive reduced micromechanical bonding of aged RC. This result was in disagreement with Aquino et al., who found that using of silane is essential when the abrasive tip was used for mechanical preparation but not when Al2O3 air abrasion was used. The discrepancy between the two studies might be due to the chemical interaction between silane and RC is affected by the amount of accessible silica on the surface, this effect may vary depending on the type of repaired RC. The Filtek BFRC employed in their study contained around 1-10 percent by weight silane-treated silica, which may have increased the repair bond strength of the group roughened by the abrasive tip. Unlike the Tetric N-Ceram BFRC was used in this study, which did not include silica.

The results of groups treated with silane had a higher median SBS value than groups treated without silane with no statistically significant difference. The failure of silane coupling agents to predictably increase the bond between new and old RCs when compared to bonding agents could indicate that mechanical interlocking is the most important factor contributing to repair bond strength, and silane appears to be an augmenting factor for RC repair.

These results were in agreement with Akgül et al., who reported that Silanization did not improve the repair bond strength. Also, Michelotti et al., found that using a separate silane coupling agent before applying the universal adhesive did not increase repair bond strength.

The null hypothesis that there was no significant difference between repair materials was accepted. The results of groups repaired with the CFRC had a higher median SBS value than groups repaired with the BFFRC with no statistically significant difference. This may be related to the fluidity of CFRC, which promotes surface wetting, and manipulation simplicity decreases air bubbles formation, and so improves bond strength. These results were in agreement with Akgül et al., who concluded that the repair bond strength of the aged BFRC was not significantly affected by the type of repair RC when the same surface-treatment methods were applied. Also, Ayar et al., reported that aged BFRC might be properly repaired using BF or CRCs.
Conclusions:
Surface treatment by air abrasion followed by application of silane and universal adhesive can be attempted clinically for the repair of aged bulk filament composite restoration. The aged bulk fill could be effectively repaired with the same bulk fill or conventional resin composite if proper repair protocol was used.

References:
8. Souza MO; Leitune VC; Rodrigues SB; Samuel SM; Collares FM. One-year aging effects on microtensile bond strength of composite and repairs with different surface treatments. Braz Oral Res. 2017;31:1-7.
17. Kupradit P; Anuntasirichinda S; Kanpitaya B; Chareonwichienchai C. Shear Bond Strength of Bulk-fill Resin Composite after Bur and Air Abrasion Surface Treatments. CM Dent J. 2021;42(2):75-82.
22. Passos SP; Özcan M; Vanderlei AD; Leite FP; Kimpara ET; Bottino MA. Bond strength durability of direct and indirect composite systems following surface conditioning for repair. J Adhes Dent. 2007;9(5):443-447.


28. Fornazari IA; Wille I; Meda EM; Brum RT; Souza EM. Effect of Surface Treatment on Microshear Bond Strength of Nanofilled Composite Repairs. Oper Dent. 2017;42(4):367-374.


34. Mendes LT; Loomans BA; Opdam NJ; Silva C; Casagrande L; Lenzi TL. Silane coupling agents are beneficial for resin composite repair: a systematic review and meta-analysis of in vitro studies. J Adhes Dent. 2020;22(5):443-453.


38. Kaneko M; Caldas RA; Feitosa VP; Consani RLX; Schneider LFJ; Bacchi A. Influence of surface treatments to repair recent fillings of silorane-and methacrylate-based composites. J Conserv Dent. 2015;18(3):242-246.

