ผลจากการเตรียมพื้นผิวต่อความแข็งแรงยึดติดหลังการซ่อมแซมของนาโนคอมโพสิต

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บทคัดย่อ

การวิจัยนี้มีจุดประสงค์เพื่อศึกษาความแข็งแรงยึดติดหลังการซ่อมแซมระหว่างคอมโพสิตใหม่และคอมโพสิตที่ผ่านการจำลองการใช้งาน และเพื่อศึกษาผลของการเตรียมพื้นผิวแบบต่างๆ ต่อความแข็งแรงยึดติดหลังการซ่อมแซมของคอมโพสิต ชิ้นทดสอบคอมโพสิตทรงกระบอก (เส้นผ่านศูนย์กลาง 5 มม X 4 มม) จำนวน 90 ชิ้น ถูกเตรียมและเก็บในน้ำปราศจากประจุที่อุณหภูมิ 37 องศาเซลเซียส เป็นเวลา 180 วัน และแบ่งออกเป็น 9 กลุ่ม (กลุ่มละ 10 ชิ้น) ซึ่งได้รับการเตรียมพื้นผิวด้วยวิธีการต่างๆ กันก่อนทำการยึดติดกับคอมโพสิตใหม่ดังนี้ กลุ่มที่ 1: ทำผิวให้ขรุขระด้วยหัวกรอเพชรความหยาบปานกลาง กลุ่มที่ 2: ทำผิวให้ขรุขระด้วยผงอะลูมินัมออกไซด์ กลุ่มที่ 3: ทำผิวให้ขรุขระด้วยหัวกรอเพชรและทาสารยึดติดแอดเปอร์สกอตช์บอนด์เอสอี กลุ่มที่ 4: ทำผิวให้ขรุขระด้วยผงอะลูมินัมออกไซด์และทาสารยึดติดแอดเปอร์สกอตช์บอนด์เอสอี กลุ่มที่ 5: ทาสารยึดติดแอดเปอร์สกอตช์บอนด์เอสอี กลุ่มที่ 6: ทำผิวให้ขรุขระด้วยหัวกรอเพชรและทาสารยึดติดแอดเปอร์สกอตช์บอนด์มัลติเพอร์เพอร์ส กลุ่มที่ 7: ทำผิวให้ขรุขระด้วยผงอะลูมินัมออกไซด์และทาสารยึดติดแอดเปอร์สกอตช์บอนด์มัลติเพอร์เพอร์ส กลุ่มที่ 8: ทาสารยึดติดแอดเปอร์สกอตช์บอนด์มัลติเพอร์เพอร์ส กลุ่มที่ 9: ไม่ทำการเตรียมผิวคอมโพสิต และกลุ่มที่ 10: เป็นกลุ่มควบคุมโดยเตรียมชิ้นทดสอบจากคอมโพสิตเป็นทรงกระบอก (เส้นผ่านศูนย์กลาง 5 มม X 8 มม) ชิ้นทดสอบทั้งหมดถูกเก็บไว้ในน้ำปราศจากประจุเป็นเวลา 24 ชม. ก่อนนำไปทดสอบหาความแข็งแรงเฉือนของการยึดติดในเครื่องทดสอบสากล ผลการทดลองได้รับการวิเคราะห์ทางสถิติด้วยการทดสอบความแปรปรวนแบบทางเดียว และการทดสอบเชฟเฟ่ที่ระดับความเชื่อมั่น 95% การเตรียมพื้นผิวให้ค่าความแข็งของการยึดติดเรียงตามลำดับจากมากไปน้อยดังนี้ กลุ่มที่ 3 > กลุ่มที่ 4 > กลุ่มที่ 6 > กลุ่มที่ 7 > กลุ่มที่ 5 > กลุ่มที่ 8 โดยกลุ่มที่ 1 2 และ 9 ไม่เกิดการยึดติดระหว่างคอมโพสิตเก่าและใหม่ ผลจากการศึกษานี้แสดงให้เห็นว่าควรต้องทำการเตรียมพื้นผิวของคอมโพสิตเก่าให้เกิดความขรุขระและต้องทาสารยึดติดเสมอก่อนทำการยึดติดกับชั้นคอมโพสิตใหม่ ในการศึกษานี้การทำพื้นผิวให้ขรุขระด้วยหัวกรอเพชรและทาสารยึดติดแอดเปอร์สกอตช์บอนด์เอสอีให้ค่าความแข็งแรงเฉือนของการยึดติดสูงสุด และมีค่าใกล้เคียงกับค่าความแข็งแรงดั้งเดิมของวัสดุ

คำสำคัญ : การซ่อมแซม การเตรียมพื้นผิว ความแข็งแรงยึดติด เรซินคอมโพสิต สารยึดติด

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Effect of Surface Treatments on Repair Bond Strength of Nanocomposite

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Abstract:

Objectives of this study were to investigate the repair bond strength of fresh composite to aged composite and the effect of different surface treatments on the bond strength. Ninety cylindrical samples (diameter of 5mm X 4 mm) of composite (Filtek® Supreme XT, 3M ESPE, USA) were prepared and kept in de-ionized water at 37^{O}C for 180 days. The samples were divided into 9 groups (n =10), each group was subjected to one of surface treatments before adding fresh composite; Group 1 roughened with medium-grit diamond bur, Group 2 air-abraded with aluminum oxide particles, Group 3 roughened with medium-grit diamond bur + Adper Scotchbond SE (3M ESPE, USA), Group 4 air-abraded with aluminum oxide + Adper Scotchbond SE, Group 5 Adper Scotchbond SE, Group 6 roughened with medium-grit diamond bur + Adper Scotchbond Multipurpose (3M ESPE,USA), Group 7 air-abraded with aluminum oxide + Adper Scotchbond Multipurpose, Group 8 Adper Scotchbond Multipurpose, Group 9 No surface treatment. Additionally, 10 cylindrical samples (diameter of 5mm X 8 mm) were fabricated as a control group. All samples were kept in deionized water for 24 hours before subjected to shear bond strength test in a universal testing machine. The results were analyzed by One-way ANOVA and Scheffe's test (p = 0.05). Treatment that resulted in the highest bond strengths was group 3 followed by group 4, group 6, group 7, group 5, and group 8 respectively. The group treated with medium-grit diamond bur or aluminum oxide air-abrasion alone and the group without treatment did not produce an adequate bond and resulted in pre-test failure. This study shows that it is essential to treat the surface of aged composite restoration by roughening and applying bonding agent before repairing with fresh material. Surface roughening with a medium-grit diamond bur together with the use of Adper Scotchbond SE adhesive can give optimal bond strength comparable to the original strength of the material.

Key words: Bonding, Bond strength, Repair, Resin composite, Surface treatment

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Introduction

Advances in adhesive technologies have strongly influenced current concepts of restorative dentistry. Application of resin-based composite restoration does not require mechanical retention, therefore enabling minimal invasive treatment by conserving healthy dental tissues. However, dynamic conditions in the oral environment, such as pH changes and temperature alteration may degrade resin composite and lead to failure of the restorations such as discoloration, microleakage, wear, fracture, and ultimately require replacement [1,2]. Total replacement of the failed restoration is the most common procedure in daily dental practice [3] although this may be regarded as over-treatment since in most cases a large portion of the restoration is clinically and radiographically intact. Complete removal of the restoration leads to unnecessary loss of sound tooth structure and sometimes repeated injuries of the pulp [3-5]. For this reason, composite restoration repair 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. Similarly to the treatment of a laboratory fabricated (indirect) resin composite repair, because there is a need to create the strongest possible bond of resin cement to a previously polymerized composite [1,6,7]. Occasionally there is a need for cementing a porcelain veneer on a previously cured composite restoration, so bond strength of resin cement to previously cured composite is a significant matter [6,7]. Successful resin composite repair requires development of an adequate interfacial bond between the old and new composites [8,2]. Bond strength of incrementally built composite up on fresh, uncontaminated or unprepared composite resin is similar to cohesive strength of the material [9]. However, there is a possibility that repair may lead to an unacceptably weak restoration. This potential problem has been investigated in several composite resin repair studies that have shown a wide variation in interfacial repair bond strengths equal 25-80% of the cohesive strength of the composite [10-13]. Because of lack of air-inhibited layer on surface, the degree of un-reacted carbon double bond is lower and chemical bonding between fresh and aged composite is therefore not reliable [14,15]. For this reason, some methods such as hydrofluoric acid etching, micro etching with air abrasion, use of coarse burs, silicon paper, green carborandum stone, and silane application have been suggested [16-23]. The use of an adhesive agent was found to play an important role in the repair bond [10,24]. While surface roughness promotes mechanical interlocking, the adhesive agent enhances surface wetting and chemical bonding with the new composite [24].

Self-etching adhesive systems were developed to simplify bonding procedure [8,25]. It was found in a previous study that the repair of aged resin composites seems to be feasible with the use of self-etching systems, they simplify the adhesion, eliminate the sensitivity of the technique, and can be used to condition both the surrounding tooth and the composite to be repaired in one procedure which is more practical [8,23]. Although the literature presents several comparative studies, there is no consensus about the benefit of composite surface abrasion over the application of intermediate adhesive resins, and there was no enough information to reach consensus about proper composite repairing method and type of adhesives on its repair bond strength. The objectives of this study were to investigate the repair bond strength of composite to composite bond after aging and to investigate the effect of different surface treatments on the composite's repair bond strength.

Materials and methods

The materials used in this study are listed in Table 1 and were used strictly according to the manufacturer's recommendations. Ninety cylindrical samples (diameter of 5mm X 4 mm) (Figure 1) of composite (Filtek® Supreme XT, 3M ESPE, USA) were prepared and kept in deionized water at 37°C for 180 days. Test samples were randomly distributed into 9 groups (n=10) for repair using the following methods:

Table 1 Materials used in this study

Materials/ Manufacturer	Composition
Filtek® Supreme XT (3M ESPE, St. Paul, MN, USA)	**Resin:** Bis-GMA, UDMA, TEGDMA, Bis-EMA(6), PEGDMA **Fillers:** combination of 20 nm silica filler, 4-11 nm zirconia filler, and aggregated zirconia/silica cluster filler **Filler loading:** 72.5% by weight (55.6% by volume)
Adper® Scotchbond SE (3M ESPE, St. Paul, MN, USA)	**Primer (liquid A):** Water, HEMA, surfactant, pink colorant **Adhesive (liquid B):** UDMA, TEGDMA, TMPTMA (hydrophobic trimethacrylate), HEMA phosphates, MHP, Bonded zirconia nanofiller, Camphorquinone initiator system
Adper® Scotchbond Multipurpose (3M ESPE, St. Paul, MN, USA)	**Conditioner:** 35% H3PO4, silica thickened **Primer:** HEMA, polyalkenoic acid copolymer, water, ethanol **Adhesive:** Bis-GMA, HEMA

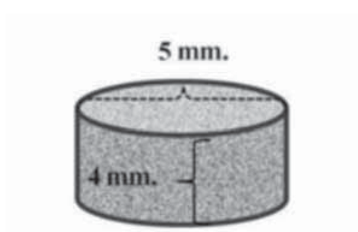

Figure 1. Composite sample dimension (Ø 5 mm, 4 mm thickness)

Group 1: Composite surfaces were roughened in 5 strokes at high speed with constant water spray with a medium-grit diamond bur (No: 856-014, ONCE diamonds, Dentsply, USA)(Figure 2). A new diamond bur was used for each 4 samples.

Figure 2. Taper Round End 856-014 Medium, ONCE Diamonds, Dentsply, USA

Group 2: Composite surfaces were air-abraded with 50µm aluminum oxide particles using a microetcher IIA (Danville, CA.)(Figure 3) operating at 3 bar pressure at a 5mm distance and 90° to composite surface for 7 seconds.

Figure 3. Microetcher IIA, Intraoral sandblaster and 50 micron aluminum oxide powder (Danville, CA.)

Group 3: Composite surfaces were roughened in 5 strokes at high speed with constant water spray with a medium-grit diamond bur. A new diamond bur was used for each 4 samples. Then Adper Scotchbond SE was applied according to the manufacturer's instructions (apply Primer A with micro-brush for 20 seconds, lightly blow with compress air for 10 seconds then apply Primer B with micro-brush using rubbing motion for 20 seconds, lightly blow with compressed air for 10 seconds, and apply a thin layer of Primer B again before light-curing for 20 seconds).

Group 4: Composite surfaces were air-abraded with 50µm aluminum oxide particles using a microetcher IIA operating at 3 bar pressure at a 5mm distance and 90° to composite surface for 7 seconds. Then Adper Scotchbond SE was applied as for Group 3.

Group 5: Adper Scotchbond SE was applied as for Group 3.

Group 6: Composite surfaces were roughened in 5 strokes at high speed with constant water spray with a medium-grit diamond bur. A new diamond bur was used for each 4 samples. Then 37% phosphoric acid was applied for 15 seconds, rinsed for 10 seconds and dried with compressed air for 10 seconds, then Adper Scotchbond Multipurpose was applied.

Group 7: Composite surfaces were air-abraded with 50μm aluminum oxide particles using a microetcher IIA operating at 3 bar pressure at a 5mm distance and 90^{o} to composite surface for 7 seconds. Then 37% phosphoric acid was applied for 15s, rinsed for 10 seconds and dried with compressed air for 10 seconds, then Adper Scotchbond Multipurpose was applied.

Group 8: Phosphoric acid (37%) was applied for 15s, rinsed for 10 seconds and dried with compressed air for 10 seconds, then Adper Scotchbond Multipurpose was applied.

Group 9: No surface treatment.

Additionally, 10 cylindrical samples (diameter of 5mm X 8 mm) were fabricated as a control group. The samples were kept in deionized water for 24 hours before subjected to shear bond strength test in a universal testing machine using crosshead speed of 0.5 mm/ min. (Figure 4) The results were analyzed by One-way ANOVA and Scheffe's test (p = 0.05).

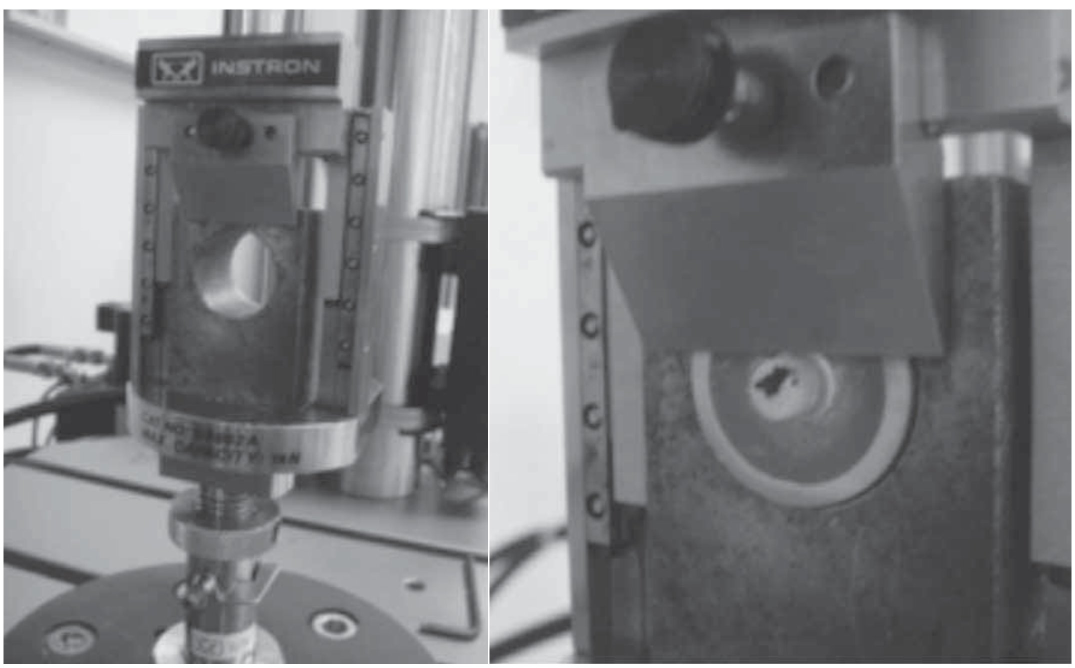

Figure 4. Specimen setup in the Universal Testing Machine. The anvil is positioned at the interface between old and fresh composite (the old composite is embedded inside acrylic resin).

Results

The mean and standard deviation of shear bond strength data are illustrated in table 2. The highest bond strength was found for control group (unrepaired composite) followed by Group 3 (abrasion with diamond bur and Scothbond SE), and Scheffe's test showed no significant difference between the two. The groups treated with Scotchbond SE, with or without surface abrasion, consistently produced greater bond strength when compared to the groups treated with Scotchbond Multipurpose. The groups treated with an adhesive alone (group 5 and 8) gave significantly lower bond strength compared to the groups treated with surface abrasion and adhesive (group 3,4 6 and 7). The group treated with diamond bur or aluminum oxide particles alone and the group without treatment did not produce an adequate bond and resulted in pre-test failure during the sample preparation process.

Table 2 The mean and standard deviation of shear bond strength for each experimental group

Experiment Group	Surface Treatment	Mean Shear Bond Strength and Standard Deviation (MPa)
1	Roughening with Medium-grit diamond bur	-
2	Air-abrading with 50μm aluminum oxide particles	-
3	Roughening with Medium-grit diamond bur + Scotchbond SE	21.7 ± 5.3^{a}
4	Air-abrading with 50μm aluminum oxide particles + Scotchbond SE	18.5 ± 5.6^{b}
5	Scotchbond SE	16.2 ± 1.2^{c}
6	Roughening with Medium-grit diamond bur + Scotchbond Multipurpose	18.1 ± 4.1^{b}
7	Air-abrading with 50μm aluminum oxide particles + Scotchbond Multipurpose	17.5 ± 2.9^{b}
8	Scotchbond Multipurpose	14.9 ± 2.5^{c}
9	No surface treatment	-
10	Control	22.4 ± 3.2^{a}

(The groups with similar superscript have no significant difference at $p < 0.05$)

Discussion

The objectives of this study were to investigate the repair bond strength of composite to composite bond after aging and to investigate the effect of different surface treatments and bonding systems on the repair bond strength. Many problems exist when repairing aged composite resin restorations. Firstly, because there is no air-inhibited layer and degree of conversion is high at the composite surface [26,27] and secondly, because of leaching of non-reacted monomers even though in minor amounts [28] there is a reduction in number of unsaturated double bonds for producing the initial and secondary bonds between the new and old composites. Also with increasing polymerization, there is a decrease in solubility and permeability of polymer [29], therefore, a roughened surface and micro-mechanical bonding is needed for composite repair. Increasing the surface roughness thus provides better mechanical interlocking and increases the probability of finding residual free carbon bonds through the surface layer of composite [1, 30]. Recent studies have found that air abrasion is effective in roughening the aged composite surface prior to bonding [18,19] and several studies have shown that the use of an intermediate bonding agent enhances the repair bond significantly [10, 24, 31].

In the present study 8 different surface treatment methods were evaluated and the repair shear bond strength results were compared with the bulk shear strength of the control group. The test was based on a shear bond strength model originally reported by Crumpler et al [32], and the specimen preparation procedure was adopted from Teixeira et al [23]. The specimens were relatively easy to fabricate using the alignment fixture during the materials' handling procedures and subsequent curing.

The repair strength required for a satisfactory composite repair in vivo has been investigated and there are a few reports on this subject. In addition, the bond strength of composite to etched enamel has been extensively investigated and is reported to be about 15-30 MPa [10,14,15]. It is known that composites seldom fail mechanically at the junction with etched enamel and it can therefore be assumed that a similar repair bond strength to that of composite to etched enamel would be clinically adequate. Measured shear bond strengths of repaired composites were in the typical range (15-25 MPa) of resin bonds to enamel and dentin [33-35], although some studies reported slightly higher values (25-38 MPa) [24,36] which may be related to the type of composites and bonding systems tested. Different aging durations and surface treatment protocols can also influence the repair bond strength [18,32,36]. However, it has been concluded that roughening of the composite surface together with the use of an intermediate bonding agent should produce acceptable bond strength [11,18,24,32].

The results of this study revealed that by roughening composite surface with medium-grit diamond followed by Scotchbond SE adhesive (Group 3), the bond strength close to the bulk shear strength of the composite as found in the control group (Group 10) could be achieved. Other treatments yielded lower repair bond strengths between 14.9 -18.5 MPa, which are considered acceptable when compare to the previously mentioned bond strength of composite to etched enamel (15-30 MPa).

Analysis with One-way ANOVA indicates significant differences between surface treatment with diamond bur and air-abrasion with 50 micron aluminum oxide particles. This is because diamond bur roughening creates micro-retentive features as well as micro-retention and this may have differentially exposed more filler particles than air abrasion methods. This result is however in contrast with the study of Kupiec who did not find significant differences in surface treatment with diamond bur and abrasion with 50μm aluminum oxide particles after 24 hours aging [24].

In this study, the aged composite surface treated with self-etching adhesive Scotchbond SE alone produced an average repair bond strength of 72.3% of the bulk shear strength of intact resin composite (control group). Interestingly, additional surface treatment with either medium-grid diamond bur roughening or air-abrasion with 50μm aluminum oxide particles can significantly increased the repair bond strength of this adhesive to 96.9% and 82.6% of the cohesive strength of intact resin composite respectively.

Two-step self-etching adhesives have been designed to simplify the bonding procedures by eliminating a separate acid-etching step. Previous studies also reported high bond strength when aged restorations were repaired with self-etching system [23,37]. It was explained that the high performance of self-etching systems in repairing resin composites has been attributed to the repetitive brushing motion used to apply self-etching systems, which may positively influence the re-bonding procedure positively by facilitating penetration of the solvent and monomer into the surface [23]. The higher repair bond strengths of the self-etching adhesive system compared to the etch and rinse adhesive system in this study may also be attributed to the differences in their solvents and monomers. Scotchbond Multipurpose contains Bis-GMA and HEMA resin which are more viscous than UDMA, TEGDMA, and TMPTMA found in Scotchbond SE. This could lead to a thicker adhesive layer that may decrease the bond strength.

In this study, the groups treated by roughening with medium-grit diamond bur alone, air-abrading with 50μm aluminum oxide particles alone, and the group without surface treatment had resulted in pre-test failure. Most failures occurred during the sample preparation process, when removing the repaired samples from the mold. These results emphasize an important role of intermediate bonding agent during resin composite repair because after aging in a humid environment, the water saturation of the resin composite was accomplished and the monomer functional group's radical activity was diminished [4]. Therefore the surfaces of the aged resin composites need to be refreshed by some new monomers. The use of an intermediate resin can enhance the repair bond strength by promoting chemical coupling to the resin matrix, bonding to the exposed fillers, or micromechanical retention through monomer penetration into the matrix microcracks [38-39]. The present study did not examine failure modes and fracture surfaces of the specimens. However, to understand the true mechanism of adhesion during composite repair, it is necessary to look at the microscopic details of the interaction between the bonding materials and the resin matrix of the aged composite [2, 23]. Further studies of the failure mode and fractography of the repaired composite surface are therefore suggested.

Conclusion

Regarding the results obtained from this study, it is essential to treat the surface of aged composite restoration by surface roughening and applying bonding agent before repairing with fresh material. Aged composite's surface roughened with medium-grit diamond bur alone or air-abraded with 50µm aluminum oxide particles alone, and aged composite's surface without any treatment were not successful in the resin composite repair process, whereas aged composite surface roughened with a medium-grit diamond bur together with the use of Adper Scotchbond SE adhesive can give optimal bond strength comparable to the original strength of the material.

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