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Durability of Surface Treatments and Intermediate Agents Used for Repair of a Polished Composite

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Clinical Relevance

In order to reach high composite repair strength, it is advisable to sandblast the polished composite surface with aluminum oxide. A hydrophobic intermediate agent should then be applied in order to prolong durability of the composite repair.

SUMMARY

Objective: To evaluate the effects of surface treatment and intermediate agent hydrophilicity on durability of the composite repair by means of the microtensile bond strength test (μTBS) and silver nitrate uptake (SNU) and the effects of surface treatment on composite roughness (Ra) and micromorphological features (SEM). Methods: Thirty resin composite blocks (4x6x6 mm) (Opallis, FGM) were polished after seven days and divided into three groups: no treatment (NT); roughening with a fine-grit diamond bur (DB); 50 μm aluminum oxide sandblasting (AO). A hydrophobic (Adhesive bottle, Scotchbond Multi-Purpose [SBMP]) or hydrophilic adhesive (Adper Single Bond 2 [SB]) was then applied. The same composite was used for repair. Composite-composite bonded sticks (0.6 mm²) were tested immediately ([IM) or after six months (6M) of water storage in tension (1.0 mm/minute). Two bonded sticks from each resin composite block were immersed in a 50% (w/v) solution of silver nitrate, photo-developed and analyzed by SEM. The composite specimens after surface treatments were analyzed with a contact profilometer (Ra) and SEM. Three-way repeated measures and one-way ANOVA were used to analyze data from the μTBS and Ra, respectively (α=0.05). Results: AO showed the highest composite repair strength (MPa) and

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Ra (µm) (52.7 ± 6.4; 4.1 ± 0.5), while the no treatment group (36.1 ± 6.1; 0.57 ± 0.5) showed the lowest. DB (44.1 ± 5.6; 1.5 ± 0.2) had an intermediate performance. The Ra results were confirmed by SEM images. SNU was observed only in SB specimens after 6M for all groups. Conclusions: Aluminum oxide treatment provides the highest composite repair strength, regardless of the hydrophilicity of the intermediate agent and storage period. Early signs of degradation were detected for SB after six months as silver nitrate deposits within the adhesive layer.

INTRODUCTION

A review of the literature revealed that different surface treatments have been suggested for the repair of resin composite restorations, along with varied chemical treatments to optimize bonding of the repair material to the existing composite restoration. Although promising results were obtained in these earlier studies, the tests were often performed on aged composite[1-6] or fresh composite light-cured in contact with a mylar strip or a glass plate.[7-11]

After polishing an esthetic composite restoration, the under-contour of some regions may be highlighted, thus requiring an immediate or one-week later repair. A previous study demonstrated that the interfacial bonding between layers of composites decreases as the original layer sets,[12] as well as after prolonged water storage for aging purposes.[13] An amount of 40%-50% of the unreactive methacrylate groups is present after light polymerization of composites in contact with a mylar strip, probably due to the organic-rich layer formed on the surface,[14] and this amount is reduced up to 50% after polishing, as the organic-rich layer is removed and inorganic filler particles without further bonding ability are exposed to the surface.[14] Primary bonding to these particles is not likely and probably relies on the micromechanical interlocking produced by roughening with diamond burs[15-17] or air abrasion with aluminum oxide.[1,6-7,9,18-19] Thus, the effectiveness of surface treatments for recently polished restorations should be a matter of further study.

Not only does surface treatment play a role in composite repair strength. In order to guarantee an intimate adaptation of the repair material to polished composite, an intermediate material is required, as the repairing composite does not properly wet the treated resin composite.[8,12,15,17] Hydrophilicity of the intermediate material for bonding may impair durability of the interfacial bond repair, since more hydrophilic adhesives tend to absorb more water over time;[20] however, few attempts have been made to address this issue.[21]

Therefore, the current study evaluated the effects of surface treatment and intermediate agent hydrophilicity on the immediate and durability results of the composite repair of recently polished restorations by means of the microtensile bond strength test (µTBS) and silver nitrate uptake (SNU) and the effects of surface treatment on the composite roughness (Ra) and micromorphological features.

METHODS AND MATERIALS

Thirty resin composite blocks were made by layering 2-mm thick increments of a microhybrid resin composite (Opallis, FGM Dental Products, Joinville, SC, Brazil, shade A3) in an additional silicone mold (4 mm deep x 6 mm wide x 6 mm long). Each increment was condensed with a clean plastic filling instrument to avoid contamination and was light-cured for 40 seconds (VIP, BISCO, Inc, Schaumburg, IL, USA, output: 600 mW/cm^2). The last increment was covered and compressed with a glass microscope slide in order to obtain a flat surface. Each specimen was removed from the mold and the surfaces of the composite blocks were polished with Sof-Lex Finishing and Polishing Disks (3M ESPE, St Paul, MN, USA). Coarse (150), medium-grit (360), fine-grit (600) and superfine-grit (1200) disks were employed. Each one was applied five times (two-second applications) under light pressure in only one direction. The specimens were then stored in a dark vial with saline solution at 37°C for one week.

The specimens were randomly divided into three groups according to the kind of surface treatment: Group NT (control)—no further treatment was performed on the composite surface; Group DB—roughening with a fine-grit diamond bur for 10 seconds under water cooling (#2135F, KG Sorensen, São Paulo, SP, Brazil, 46 µm mean particle size). Before the roughening procedure, the operator trained on the surface of an analytical balance to determine the equivalent manual pressure that would be placed on the surface of the resin composite (Mettler, type H6; Columbus, OH, USA). The pressure was equivalent to a mass of approximately 4.0 ± 1.0 g; Group AO: the surfaces were sandblasted with 50 µm aluminum oxide powder for 10 seconds at a working distance of 5 mm at a pressure of 5.5 Pascals (Pa) with an intraoral sandblaster (Microetcher II, Danville Engineering Inc, San Ramon, CA, USA).

For purposes of cleaning, a 35% phosphoric acid etchant (Scotchbond etchant gel, 3M ESPE, St Paul, MN, USA) was applied for 30 seconds. After water-rinsing (30 seconds) and air-drying (10 seconds), specimens from each group were randomly assigned to two subgroups according to the intermediate agents investigated: hydrophobic, non-solvated bonding group ([SBMP] Adhesive bottle, Adper Scotchbond Multi Purpose Plus, 3M ESPE) and the hydrophilic and solvated adhesive group ([SB] Adper Single Bond 2, 3M ESPE). Both adhesives were rubbed into the composite surfaces for 10 seconds. Solvent evaporation was performed using

an air-spray for 10 seconds at a distance of 5 cm in groups bonded with Adper Single Bond 2. For standardization purposes, the same procedure was performed in specimens from the SBMP group. The intermediate agents used in the current study and their chemical composition are reported in Table 1.

Table 1: *Composition of the Materials Employed in This Study*

Material	Composition
Opallis Composite (FGM Dental Products)	Bis-GMA monomers, Bis-EMA, TEGDMA, UDMA, camphorquinone, co-initiator, silanized barium-aluminum silicate glass (particle size of 0.5 µm, 79.8 wt%), pigments and silica.
Adper Single Bond 2 (3M ESPE)	Bis-GMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids, silica nanofiller (5 nm diameter silica particles, 10 wt%).
Adhesive, Adper Scotchbond Multi Purpose Plus (3M ESPE)	Adhesive bottle: Bis-GMA, HEMA and initiators.

Abbreviations: Bis-GMA: bis-phenol A di-Glycidyl Methacrylate; Bis-EMA: bis-phenol A di-Glycidyl Methacrylate ethoxylated; TEGDMA: Triethylene Glycol Ddimethacrylate, UDMA: Urethane Dimethacrylate; HEMA: Hydroxyethyl Methacrylate.

Two 2-mm increments were then placed over the treated surfaces with the same resin composite and light-cured with the same light-curing device for 40 seconds. Bonded composite-composite samples were sectioned with a slow-speed diamond saw (Isomet, Erios Prod Odont Ltd, São Paulo, SP, Brazil) under water-cooling in both the "x" and "y" directions across the bonded interface to obtain bonded sticks with a cross-sectional area approximately 0.6 mm^2. The bonded sticks from each composite-composite block were then divided for testing either immediately (IM) or after six months (6M) of water storage at 37°C.

Microtensile Testing

The actual cross-sectional area of each stick was measured with the digital caliper to the nearest 0.01 mm and recorded for subsequent calculation of the resin-dentin bond strength (Absolute Digimatic, Mitutoyo, Tokyo, Japan). Each bonded stick was attached to a jig in a universal testing machine (Kratos Dinamometros, São Paulo, SP, Brazil) with cyanoacrylate resin (Super Bonder gel, Loctite, São Paulo, SP, Brazil) and subjected to a tensile force at 1.0 mm/minute. The failure modes were evaluated at 400X (HMV-2, Shimadzu, Tokyo, Japan) and classified as cohesive (failure exclusively within the composite—C), adhesive (failure at composite-composite interface–A) or adhesive/mixed (failure at the composite-composite interface that included cohesive failure of the neighboring composite, A/M).

After analyzing the microtensile bond strength data for normality of data distribution (Kolmogorov-Smirnov test) and homogeneity of variances (Levene's test), a three-way repeated measures analysis of variance (ANOVA) was applied, with composite repair strength as the dependent variable and surface treatment, intermediate agent and storage time (IM vs 6M) as the independent factors. The storage period was considered the repeated measure. The Tukey's test was used for post-hoc comparisons at a significance level of 0.05.

Silver Nitrate Uptake (SNU)

Two bonded sticks from each composite block at each storage period were not tested in tension but coated with two layers of nail varnish applied to within 1 mm of the bonded interfaces. The specimens were rehydrated in distilled water for 10 minutes prior to immersion in the tracer solution for 24 hours. Conventional silver nitrate was prepared according to the protocol previously described by Tay and others.[22] The sticks were placed in conventional silver nitrate in darkness for 24 hours, rinsed thoroughly in distilled water and immersed in a photo-developing solution for eight hours under a fluorescent light to reduce the silver ions into metallic silver grains within voids along the bonded interface.

The specimens were wet polished using SiC paper with decreasing grit (1000, 1200, 1500, 2000, 2400) and 1 and 1/4 µm diamond paste (Buehler Ltd, Lake Bluff, IL, USA) using a polishing cloth. They were ultrasonically cleaned, air dried, mounted on aluminum stubs and sputtered with carbon (MED 010, Balzers Union, Balzers, Liechtenstein). The composite-composite interfaces were analyzed in a scanning electron microscope (JSM 6060, JEOL, Tokyo, Japan) operated in the backscattered electron model. The working distance was 8 mm and the accelerating voltage was 20 KV.

Three pictures were taken of each specimen. The first picture was taken in the center of the stick. The remaining two pictures were taken 0.1 mm to the left and right of the first one, respectively. As two sticks per specimen were evaluated and a total of five specimens were used for each experimental condition, a total of 30 images were evaluated per group. All the pictures were taken by a technician who was blinded to the experimental conditions under evaluation and all the pictures from each group were then viewed together and representative images from each experimental condition were selected.

Surface Roughness Measurement

Fifteen resin composite blocks were made by a microhybrid resin composite (Opallis, FGM Dental Products, Joinville, SC, Brazil, shade A3) in an addition silicone mold (4 mm deep x 6 mm wide x 6 mm long). Each increment (±1 mm thick) was condensed with a clean

plastic filling instrument to avoid contamination and light-cured for 40 seconds (VIP, BISCO, Inc, output: 600 mW/cm^2). The last increment was covered and compressed with a glass microscope slide in order to obtain a flat surface. Each specimen was removed from the mold, polished with Sof-Lex Pop On disks (3M ESPE) as previously reported and stored in a dark vial with saline solution at 37°C for one week.

The specimens were then divided into each of the different surface treatment groups and treated as mentioned. The surface roughness test was performed with a contact profilometer (Mitutoyo Surftest 301, Mitutoyo, Tokyo, Japan). Three successive measurements in different directions were recorded for all specimens in each group, and the average surface roughness (Ra) value was obtained. The cutoff value for surface roughness was 0.25 mm, and the sampling length for each measurement was 1.25 mm. The data were analyzed using one-way ANOVA and Tukey's test at a significance level of 0.05.

Scanning Electron Microscopy Analysis

The same specimens used in the roughness test were prepared for scanning electron microscopy (JSM 6400, JEOL, Tokyo, Japan) analysis. The specimens were sputter-coated with gold to a thickness of approximately 200Å in a vacuum evaporator. Photographs of representative areas of the polished and treated surfaces were taken at 1200x magnification.

Table 2: *Means and Standard Deviations (MPa) of Composite Repair Strength for All Experimental Conditions*

		SB		SBMP	
	(*)	IM	6M	IM	6M
Control	C	35.7 ± 4.8	41.1 ± 5.9	35.2 ± 6.9	32.5 ± 7.4
Diamond bur	B	41.2 ± 2.8	46.4 ± 3.1	46.3 ± 5.8	42.5 ± 7.2
Aluminum oxide	A	49.7 ± 4.9	52.6 ± 3.5	56.4 ± 7.4	52.1 ± 5.9

**Only the surface treatment was statistically significant (p=0.0001). Different letters indicate means statistically different.*

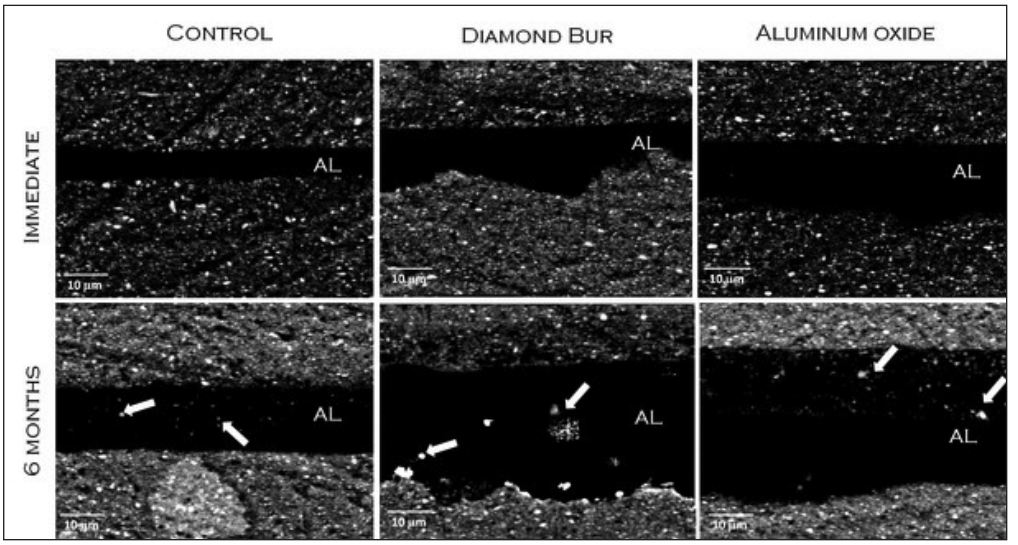

Figure 1. *Representative back-scattered SEM images of the composite-composite interfaces bonded with Adper Single Bond 2 under the experimental conditions of this study. Note the presence of spotted silver nitrate deposits (white arrows) within the adhesive layer after six months of water storage, regardless of the surface treatment.* AL–adhesive layer.

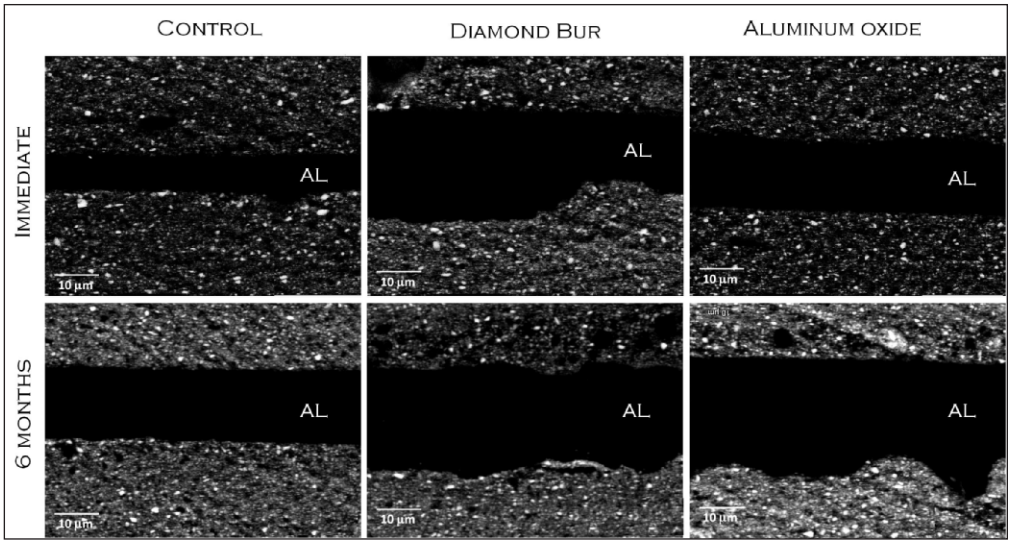

Figure 2. *Representative back-scattered SEM images of the composite-composite interfaces bonded with the bonding resin of Adper Scotchbond Multi Purpose Plus under the experimental conditions of this study. Note the absence of silver nitrate deposits within the adhesive layer.* AL–adhesive layer.

RESULTS

Microtensile Testing

Only the main factor Surface treatment was significant ($p<0.0001$). The highest composite repair strength (MPa) was observed for the aluminum oxide group (52.7 ± 6.4). The diamond bur group (44.1 ± 5.6) had an intermediate performance between the aluminum oxide and control groups. The lowest composite strength repair was observed for the control group (36.1 ± 6.1) (Table 2).

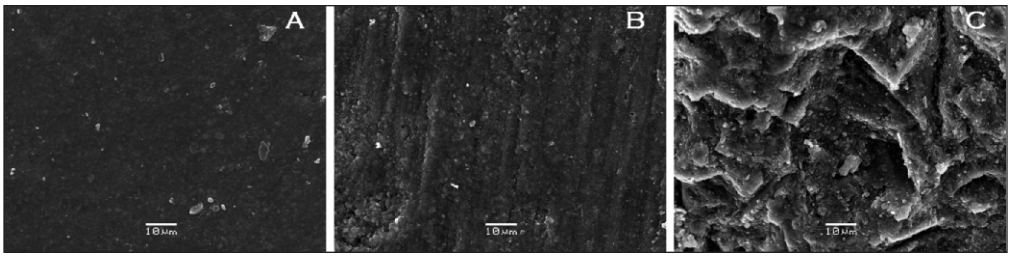

Figure 3. *Representative SEM images of the composite surfaces after different surface treatments. Observe that the surface roughness after aluminum oxide sandblasting is higher than that of the diamond bur and control groups. A= Control, B= Diamond bur c= Aluminum oxide.*

Silver Nitrate Uptake

Representative images of the SB and SBMP groups can be seen in Figures 1 and 2, respectively. The type of surface treatment did not interfere with the silver nitrate deposition. No silver nitrate deposition was seen for either adhesive in the immediate period. However, after six months of water storage, spotted silver nitrate deposits became clear for the SB groups. This finding was not observed for the SBMP adhesive.

Surface Roughness Test and SEM Analysis

One-way ANOVA showed that surface roughness was statistically significant ($p<0.0001$). The ranking of surface roughness values (Ra, µm) from the lowest to the highest were as follows: control group (0.57 ± 0.53) < diamond bur group (1.49 ± 0.19) < aluminum oxide group (4.14 ± 0.54). The results of the profilometric measurements were largely confirmed by the SEM images (Figure 3).

DISCUSSION

The results of the current investigation, in agreement with other studies,[1,7,15,18-19,23-25] confirm that surface abrasion with 50-µm aluminum oxide particles significantly improves the composite repair strength. Although the current study did not evaluate the condition of the composite to be repaired, this seems to indicate that this is of less importance as long as the composite surfaces are treated beforehand.

SEM observations of earlier studies,[7,18] as well as those from the current investigation (Figure 3), showed that aluminum oxide sandblasting is able to produce more micro-retentive features, increasing the surface area available for wetting and bonding by the adhesive resin. This may have accounted for the strongest interfacial bond achieved in this experiment following air abrasion. Previous investigators reported that air abrasion with aluminum oxide, followed by bonding agent application, can result in an intraoral repair strength that is nearly identical to the cohesive strength of the original composite.[7,23]

The composite repair strength achieved after grinding the substrate surface with a diamond bur was significantly lower than that achieved after aluminum oxide abrasion, although higher than that of the control groups (polished composite surfaces). These differences in bond strength, as compared with air-abraded specimens, are probably related to the less retentive microscopic pattern produced by the rotating instrument. In fact, the roughest composite surface was achieved after abrasion with aluminum oxide, and this may explain the superiority of this technique. Although diamond particles (in the diamond bur) and aluminum oxide powder have approximately the same particle size, probably the amount of particles of aluminum oxide sandblasted in the resin composite is higher, which accounts for the greater roughness produced by the latter. This is in line with the findings of the control group: the composite repair strength was the lowest when performed on polished surfaces. This has an important clinical implication. If repair is to be performed on a recently polished composite surface, clinicians should attempt to increase the surface area prior to the procedure.

Although the application of an intermediate agent is considered essential for higher composite repair strength for the great majority of investigators,[7,12,15,17,26] the literature lacks information about the influence of hydrophilicity of the intermediate agent on the durability of the composite repair. The results of the current study demonstrated that hydrophilicity of the intermediate agent did not affect the immediate composite repair strength and silver nitrate deposition; however, spotted silver nitrate deposits were seen in specimens bonded with the solvated, hydrophilic system (SB) after six months of water storage. Since it was already reported that the simple air drying step used under clinical conditions is not capable of removing the solvent content of the adhesive layer,[27] one could speculate that occurrence of the spotted mode of silver nitrate deposition in the hydrophilic solvated SB system could be attributed to areas of incompletely removed solvents. However, silver nitrate deposits were not present in the immediate SB specimens. As silver nitrate deposits were only observed after water storage, it is

likely that they resulted from solubility of the hydrophilic adhesive layer after contact with water and further water sorption.

The presence of entrapped solvents theoretically facilitates fluid transport into and out of the network due to high water sorption/solubility.[28] However, it seems that differences in the material polarity play a preponderant role in silver nitrate deposition. Adhesive interfaces absorb water after long-term water storage, and its amount is positively correlated with hydrophilicity of the adhesive system.[20,29] This explains why the SBMP system (hydrophobic adhesive) did not exhibit silver deposits after six months of water storage. Absorbed water molecules occupy the free volume between polymer chains and crosslinks, causing swelling of the polymer structure and lead to plasticization and softening of the resin structure.[30] After this relaxation process, unreacted monomers trapped in the polymer network are released to their surroundings, resulting in a higher solubility.[20] This might create microvoids that were likely filled with silver nitrate.

Although this finding did not result in any reduction in composite repair strength, it represented signs of early degradation and may lead to marginal discoloration and, eventually, interfacial debonding. The lack of reduction of the bond strength repair for the SB group can also be attributed to the water storage period employed. Perhaps the evaluation of such bonds for more prolonged periods of time would be able to detect reductions in the bond strength in the Adper Single Bond groups. The specimens were only stored for six months, because this is the storage period mostly used by researchers to study degradation of resin dentin bonds.[31-33]

The extent to which the results of the current investigation may be extrapolated for the clinical scenario and may affect clinical longevity of a clinical repair is yet to be addressed.

CONCLUSIONS

Aluminum oxide treatment provides the highest composite repair strength, regardless of the hydrophilicity of the intermediate agent and storage period. Early signs of degradation were detected for SB after six months as silver nitrate deposits within the adhesive layer.

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