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Effect of Surface Treatments on Microtensile Bond Strength of Repaired Aged Silorane Resin Composite

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Clinical Relevance

To obtain high repair bond strength on aged silorane composite, aluminum oxide sandblasting should be used as a surface treatment procedure. After aluminum oxide sandblasting, either silorane composite with the LS system adhesive or methacrylate composite with a methacrylate dental adhesive can be used.

SUMMARY

Objective: This laboratory study compared the repaired microtensile bond strengths of aged silorane resin composite using different surface treatments and either silorane or methacrylate resin composite. Methods: One hundred eight silorane resin composite blocks (Filtek LS) were fabricated and aged by thermocycling between 8°C and 48°C (5000 cycles). A control (solid resin composite) and four surface treatment groups (no treatment, acid treatment, aluminum oxide sandblasting, and diamond bur abrasion) were tested (N=12 blocks, 108 beams/group). Each treatment group was randomly divided in half and repaired with either silorane resin composite (LS adhesive) or methacrylate resin composite (Filtek Z250/Single Bond Plus). After 24 hours in 37°C distilled water, microtensile bond

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strength testing was performed using a nontrimming technique. Surface topography after surface treatment was analyzed using scanning electron microscopy (SEM). Failure mode was examined using optical microscopy (50×). Results: Weibull-distribution survival analysis revealed that aluminum oxide sandblasting followed by silorane or methacrylate resin composite and acid treatment with methacrylate resin composite provided insignificant differences from the control ($p>0.05$). All other groups were significantly lower than the control. Failure was primarily adhesive in all groups. Conclusion: Aluminum oxide sandblasting produced microtensile bond strength not different from the cohesive strength of silorane resin composite. After aluminum oxide sandblasting, aged silorane resin composite can be repaired with either silorane resin composite with LS system adhesive or methacrylate resin composite with methacrylate dental adhesive.

INTRODUCTION

One of the main disadvantages of methacrylate composite is polymerization shrinkage. There have been several attempts to overcome polymerization shrinkage such as using an incremental layering technique, placing a stress-absorbing liner, and changing the light-curing procedures.[1,2] Recently, a silorane-based material was introduced. Silorane composite has shown approximately 0.94 vol% to 1.5 vol% shrinkage[3–5] and comparable flexural strength to methacrylate composite.[3] A silorane molecule comes from the reaction of oxirane and siloxane molecules.[3,6,7] Whereas the polymerization process of methacrylate composite occurs via free radical polymerization,[8,9] the polymerization of silorane composite is generated by the cationic ring-opening polymerization of the oxirane molecule. The silorane composite has two main advantages: low polymerization shrinkage due to the ring-opening polymerization of the oxirane monomer and increased hydrophobicity because of the siloxane molecule.[10]

Silorane composite consists of four main components.[3,7] The filler is a combination of fine quartz particles and radiopaque yttrium fluoride. The polymer matrix is silorane, and the photoinitiator is camphorquinone and iodonium salt. The use of fine particular quartz contributes to good esthetic performance and mechanical stability. The quartz surface is modified with a silane layer to increase the hydrophobic character of the surface of the filler and act as the interface between filler and matrix, facilitating the reinforcement of the resin.

The average lifetime of composite restorations is 5.5 years to eight years, with an average annual failure rate of 2.2%.[11,12] Recurrent caries and discoloration are the main reasons for replacement in general dental practice.[13] Replacement frequently involves the removal of additional tooth structure to create new enamel bonding, leading to a larger restoration with further loss of tooth structure.[14]

Several methods of surface treatment have been widely used to establish adequate bond strength between aged and new layers of composite, including surface hydrofluoric acid etching, sandblasting with aluminum oxide particles, abrasion with a diamond bur followed by silica coating, and the use of intermediate bonding agents.[2,10,14–17] The bonding of new to aged composite mostly depends on micromechanical interaction, but chemical bonding is also to be taken into consideration.[18–20]

In a clinical situation, there may be no information about the chemical composition of the existing resin composite restorations. Understanding how to repair existing silorane restorations is critical because there is limited information regarding repair protocols of silorane composite. Based on this concern, the purpose of this study was to compare the repaired microtensile bond strength of aged silorane composite using different surface treatments and either silorane or methacrylate composite.

MATERIALS AND METHODS

One hundred eight silorane composite blocks (Filtek LS, shade A2, 3M ESPE, St Paul, MN, USA) were fabricated using a silicone mold (6 mm × 6 mm × 12 mm for the control and 6 mm × 6 mm × 6 mm for the test specimens). Each 2-mm-thick increment was placed using a plastic instrument and was cured for 40 seconds using a Demetron LC curing unit (Kerr, Orange, CA, USA) with an intensity of 600 mW/cm^2. The intensity of the LED curing light was monitored with a Cure Rite Visible Curing Light Meter (Dentsply, York, PA, USA). The last increment was covered with a Mylar strip to obtain a flat surface and to aid in removal of excess material. All specimens were polished using 320, 400, and 600 silicon carbide paper including the top surface in order to remove the excess and to make the surface perpendicular to the specimen's long axis. All specimens were cleaned in tap water for 10 minutes in an ultrasonic device to remove loose particles and stored in distilled water for 24 hours.

All specimens were aged by thermocycling (5000 cycles, 8°C to 48°C, dwell time of 30 seconds, transfer time of 10 seconds). All tested specimens were randomly divided into four surface treatment groups, and each group was divided into two subgroups (12 blocks for each subgroup) to repair with either silorane (group 1S, 2S, 3S, 4S) or methacrylate composite (group 1M, 2M, 3M, 4M; Table 1). The surface treatment procedure was performed as described below:

Group 1 (No Treatment)—No further treatment was performed.

Group 2 (Acid Treatment)—The aged specimen surfaces were etched with 35% phosphoric acid gel (Scotchbond Etchant, 3M ESPE) for 15 seconds for the group that was repaired with methacrylate composite (group 2M). The aged specimen surfaces were etched with LS System self-etch primer (LS System adhesive, 3M ESPE) for 15 seconds for the group that was repaired with silorane composite (group 2S).

Group 3 (Aluminum Oxide Sandblasting)—The resin composite surface was abraded for 10 seconds with an intraoral air abrasion unit (Microetcher II, Danville Engineering INC, San Ramon, CA, USA) from a distance of approximately 10 mm and perpendicular to the composite block using 50-μm aluminum oxide particles (Danville Engineering INC) with an air pressure of 60 psi.

Group 4 (Diamond Bur Abrasion)—The resin composite surface was roughened with a coarse-grit diamond bur for 10 seconds (No. 027, Brasseler, Savannah, GA, USA). A high-speed handpiece with a water spray was used. Before the roughening procedure, the operator was trained on the surface of an analytical balance (AE 100, Mettler-Toledo Inc, Columbus, OH, USA) to be able to replicate the manual pressure (approximately 4.0 ± 1.0 g)[21] that was placed on the composite surface.

Then, all specimens were rinsed with tap water, and excess water was removed with canned compressed oil-free air (Falcon Dust Off Air Duster, Branchburg, NJ, USA) and repaired with either silorane (Filtek LS, shade C2, with LS System adhesive) or methacrylate composite (Filtek Z250, shade A4, with Adper Single Bond Plus adhesive agent, 3M ESPE) according to the manufacturer's recommendation (as described below) using an incremental technique with a silicone mold (Table 2).

LS System Adhesive

After the surface treatment procedure, the LS System adhesive self-etch primer was applied on

Table 1: *Materials, Batch Number, and General Compositions*

Materials	Batch No.	General Composition
Filtek LS (3M ESPE)	N18197	*Filler:* Silanized quartz, yttrium fluoride 76 wt%
		Matrix: 3,4-Epoxycyclohexylethyl-cyclopolymethylsiloxane, Bis-3,4-epoxycyclohexylethylphenylmethyl-silane
Filtek Z250 (3M ESPE)	N163688	*Filler:* Zirconia/silica 85 wt%
		Matrix: Bis-GMA, a blend of UDMA and Bis-EMA
LS adhesive (3M ESPE)	N157377	*Self-etch primer:* Phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, camphorquinone and silane-treated silica filler, initiators, stabilizers
		Bond: Hydrophobic methacrylate, phosphorylated methacrylate, TEGDMA, silane-treated silica filler, initiators, stabilizers, camphorquinone
Adper Single Bond Plus adhesive (3M ESPE)	393173	*Etchant:* 35% phosphoric acid
		Bond: Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator system, methacrylate functional copolymer of polyacrylic and polyitaconic acids, silica particles

Abbreviations: Bis-GMA, bisphenol A diglycidyl ether diemthacrylate; EMA, bisphenol A polyethylene glycol diether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate, TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Table 2: *Experimental Groups and Surface Treatment Protocol*

Group	Repair Materials	Surface Treatment	Bonding Procedure
Control	—	—	—
1S	Filtek LS	No surface treatment	LS adhesive bonding
1M	Filtek Z250	No surface treatment	Single bond application
2S	Filtek LS	LS self-etch primer	LS adhesive bonding
2M	Filtek Z250	35% Phosphoric acid etching	Single bond application
3S	Filtek LS	Aluminum oxide sandblasting	LS self-etch primer + LS adhesive bonding
3M	Filtek Z250	Aluminum oxide sandblasting	35% phosphoric acid + single bond
4S	Filtek LS	Abrasion with diamond bur	LS self-etch primer + LS adhesive bonding
4M	Filtek Z250	Abrasion with diamond bur	35% phosphoric acid + single ond

the surface-treated resin composite for 15 seconds except for the no surface treatment group and the acid treatment group because the acid treatment group was previously etched from the surface treatment. Then, all etched specimens were gently dried with canned compressed oil-free air and were cured for 10 seconds. Then, LS adhesive bonding was applied on the surface of all composite surfaces, followed by gentle air drying and 10 seconds of light cure.

Adper Single Bond Plus System Adhesive

After the surface treatment procedure, 35% phosphoric acid gel (ScotchbondEtchant, 3M ESPE) was applied to the treated composite surfaces for 15 seconds except for the no surface treatment group and the acid treatment group because the acid treatment group was previously etched from the surface treatment. Then, the etched resin composites were rinsed for 10 seconds. Excess water was removed with canned compressed oil-free air. Then, Adper Single Bond Plus adhesive agent (3M ESPE) was applied in three consecutive coats for 15 seconds on all composite surfaces with gentle agitation using a fully saturated applicator, followed by air drying for five seconds to evaporate the solvent and 10 seconds of light cure.

Then, a new layer of resin composite was applied to the aged silorane composite using an incremental technique with the aid of a silicone mold. Each 2-mm-thick increment was placed using a plastic instrument and was cured for 40 seconds. The last increment was covered with a Mylar strip to obtain a flat surface and to aid in removal of excess material.

All repaired resin composite blocks were stored in 37°C distilled water for 24 hours. Then, the composite blocks were cut using a slow-speed water-cooled saw equipped with a diamond-impregnated disk (Isomet, Buehler, Lake Bluff, IL, USA) at a speed of 300 rpm, producing nine beams per block with an average area of 0.64 mm^2 (0.8 mm × 0.8 mm) for each beam. The beams located at the periphery of block were discarded.

Microtensile Bond Strength Testing

After storing in 37°C distilled water for 24 hours, the beams were placed in a notched Geraldeli jig (ODEME Biotechnology, Joacaba, SC, Brazil) for microtensile testing using cyanoacrylate resin (Loctite Superglue Control Gel, Henkel Corporation, Avon, OH, USA). All beams were loaded in tension in a universal testing machine (MTS Sintech Renew 1123, Eden Prairie, MN, USA) until fracture at a crosshead speed of 1 mm/min.

Statistical Analysis

Comparisons between the groups for differences in microtensile bond strength were performed using a

Weibull-distribution survival analysis at the 0.05 significance level.

Scanning Electron Microscopy (SEM) Analysis

Additional specimens were fabricated, aged, treated, and prepared for SEM (model JSM-5310LV, JEOL, Peabody, MA, USA). Specimens were sputter coated with gold to a thickness of approximately 200 A° in a vacuum cold sputter (Denton Vacuum Desk II cold sputter, Moorestown, NJ, USA). Micrographs were taken at 2000× and 10,000× magnification to evaluate the surface topography created by the different surface treatments.

Failure Analysis

Fracture surfaces of the repaired groups were examined using optical microscopy at 50× magnification. The type of failure was determined to be either adhesive failure (between aged and repairing composites involving the intermediate layer), cohesive failure (within the aged or repairing composite), or mixed (combination of adhesive and cohesive failures).

RESULTS

Microtensile Bond Strength

Aluminum oxide sandblasting followed by silorane or methacrylate-based resin composite (group 3S, 3M) and acid treatment with methacrylate resin composite (group 2M) provided insignificant differences from the control (*p*>0.05; Table 3). All other groups were significantly lower than the control, and no other significant difference was found among other groups.

Type of Failure

As shown in Table 4, in most tested beams, fractures developed at the composite-composite interface (81%), followed by cohesive failure (18%) and mixed failure (1%), respectively.

SEM Analysis

SEM analysis revealed different surface topography (Figure 1) of surface-treated silorane composite. Al_2O_3 sandblasting (Figure 1G,H) produced substantial surface irregularities, which could be supported by the highest microtensile bond strength. Even though self-etch primer (Figure 1C,D) and phosphoric acid (Figure 1E,F) could remove the superficial portion of the aged composite surface with some filler particles exposed, the surface roughness of the acid treatment was less obvious. In addition, at 10,000× magnification, acid treatment with phosphoric acid (Figure 1F) showed more granular-like surface topography compared with the remaining groups. No surface treatment (Figure 1A,B) and diamond bur (Figure 1I,J) abrasion showed comparable surface topography with some irregularities resulting from polishing with silicon carbide paper, which could be supported by the comparable microtensile bond strength. Moreover, diamond bur abrasion (Figure 1J) also showed exposed filler particles, but no exposed filler was observed in the no-treatment group (Figure 1B).

Table 3: *Mean, Percentage of Cohesive Strength, and p Value of Experimental Groups*

Surface Treatment	Group	Mean Bond Strength, MPa	Percentage of Cohesive Strength	*p* Value
Control	Control	62.6 + 1.6	100	—
No treatment	1S	37.2 + 1.6	59.42	0.006*
	1M	40.8 + 1.9	65.18	0.030*
Acid etch	2S	37.3 + 2.0	59.58	0.008*
	2M	44.1 + 1.8	70.45	0.070
Sandblast	3S	50.2 + 1.6	80.19	0.230
	3M	47.8 + 1.5	76.36	0.130
Abrasion	4S	39.0 + 1.7	62.30	0.014*
	4M	37.7 + 1.6	60.22	0.007*

** Statistically significant difference (p<0.05) when compared with the control. There was no other statistically significant difference among other groups.*

DISCUSSION

Microtensile bond strength was chosen in the present study because the purpose of this experiment was to compare the repair bond strength that is at the interface between aged and a newly repaired composite. The bond strength values from microtensile bond strength testing are normally from adhesive failure because of a uniform stress distribution, so the bond strength value better represents the adhesive bond strength instead of

Table 4: *The Incidence of Failure Mode (%)*

Surface Treatment	Group	Type of Failure, %			
		Adhesive	Mixed	Cohesive	Did Not Break
No treatment	1S	87	0	13	0
	1M	77	0	22	1
Acid treatment	2S	80	1	19	0
	2M	82	0	18	0
Sandblast	3S	72	2	24	2
	3M	63	1	36	0
Abrasion	4S	94	0	6	0
	4M	88	6	6	0
Total		81	1	18	0

Abbreviations: M, the silorane composite (Filtek LS) was repaired with methacrylate composite (Filtek Z250); S, the silorane composite (Filtek LS) was repaired with silorane composite (Filtek LS).

cohesive strength from cohesive material failure. In this study, the microscopic study showed most failures resulted from adhesive failures. As approximately 81% of the bond strength values were associated with adhesive failure, it can be assumed that the bond strength values are highly representative of adhesive bond strength rather than cohesive bond strength.

The result of the present study is in accordance with other composite repair studies[10,14,17,21] supporting aluminum oxide sandblasting as an effective surface treatment procedure for the repair of composite restorations. Either silorane composite repaired with silorane composite (group 3S) or repaired with methacrylate composite (group 3M) showed no significant difference in repair bond strength compared with the cohesive strength of silorane composite (control). This finding indicates that aluminum oxide sandblasting provides comparable microtensile bond strength to the solid silorane specimens.

SEM images showed that aluminum oxide sandblasting provided substantial surface roughness compared with the other groups. Rodrigues and others and Costa and others explained that a high surface roughness resulting from sandblasting produced microretentive features, enhancing the surface area available for bonding.[10,21]

An interesting result was found in the no surface treatment group (group 1S, 1M). Even though the repair bond strength was significantly lower than the control, the repair bond strength in the no surface treatment group was approximately the same as or even higher (with no significant difference) than that of the diamond bur abrasion group. Both mechanical and chemical bonding might be considered as an explanation. First, polishing the top surface of the composite blocks with silicon carbide paper prior to the aging procedure produced surface roughness for further bonding. Furthermore, SEM analysis also supported the above hypothesis. SEM images showed that no surface treatment and diamond bur abrasion presented a similar pattern of surface roughness (Figure 1). Second, as described by Padipatvuthikul and Mair,[22] the surface of aged composite might contain microdefects, which can be penetrated by the unfilled resin of the bonding agent, resulting in a micromechanical retention. Third, the solvent in the adhesive bonding may soften the surface of aged composite, creating swelling and gelation of the surface and subsurface layers.[22] The monomer in the layer of the repair material might access and cross-polymerize with the unreacted functional group of silorane, creating a chemical bond. Fourth, the aging cycles may not have been sufficient to create hydrolytic degradation on the composite surface so that the repair bond strength might have come from the remaining reactivity of the material.

The use of diamond bur abrasion on the aged silorane composite resulted in significantly weaker repair bond strengths than the cohesive strength of silorane composite regardless of the repair material used. The effect of surface abrasion with a diamond bur on the repair bond strength of aged composite is debated because some studies did not show significantly improved repair bond strength[23] while others did.[24,25] Thus, this technique provides a less predictable outcome compared with aluminum oxide sandblasting.

Acid treatment, including phosphoric acid, normally shows no effect in repair bond strength.[13,26–28] In this study, it was interesting to see quite different results from the two acid etching subgroups. When repaired with Filtek Z250 (group 2M), the 35% phosphoric acid was able to produce microtensile

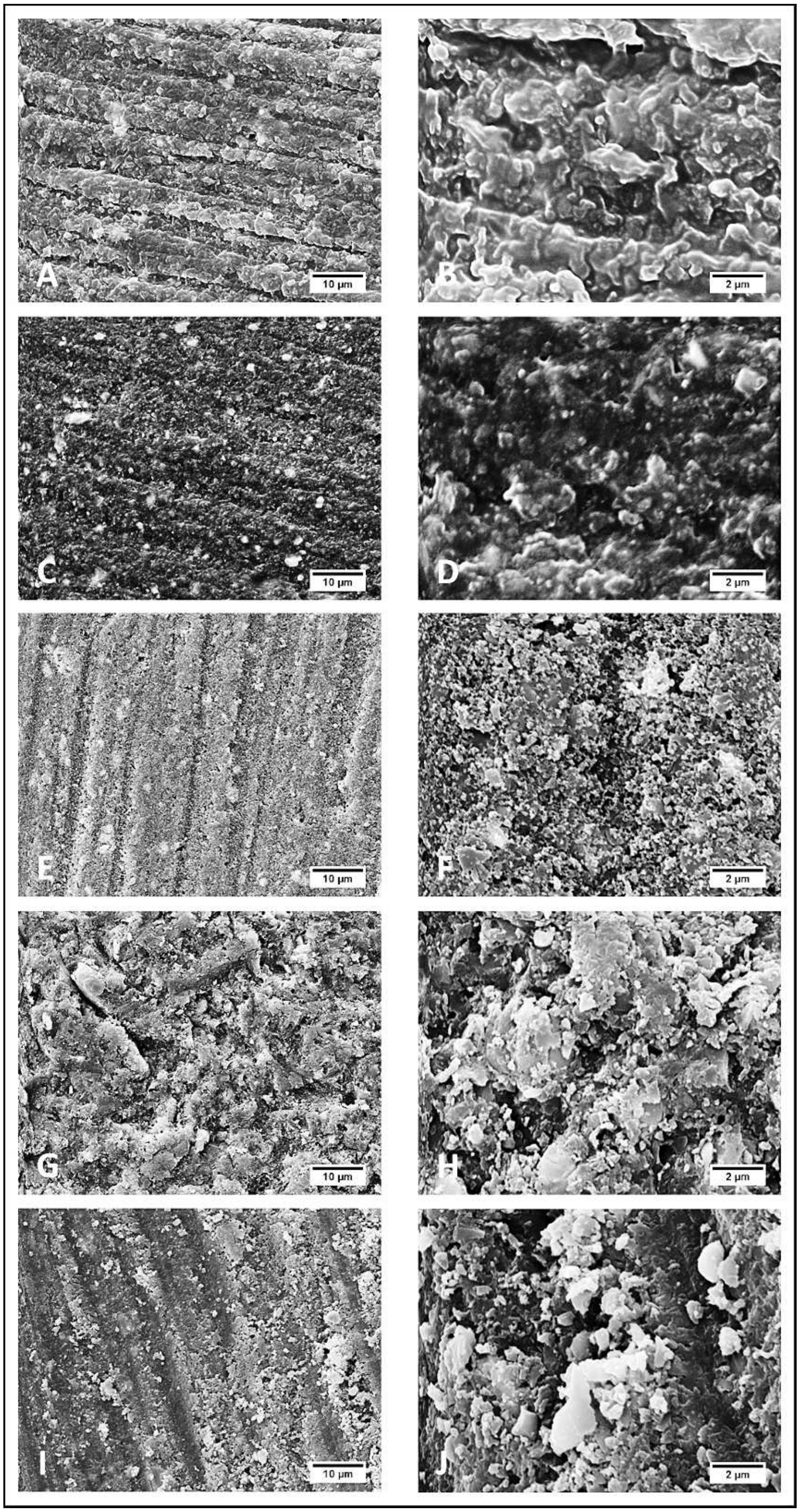

Figure 1. *The scanning electron micrographs of aged silorane composite treated with different surface treatments. (A): (2000×), (B): (10,000×): No surface treatment group; (C): (2000×), (D): (10,000×): self-etch primer; (E): (2,000×), (F): (10,000×): 35% phosphoric acid; (G): (2000×), (H): (10,000×): 50 µm Al_2O_3 sandblasting; (I): (2000X), (J): (10,000×): diamond bur abrasion. Scanning electron microscopy images showed the high surface irregularities on the silorane composite treated with aluminum oxide sandblasting. There were some exposed filler particles observed in the acid treatment groups (Figure 1C,E), Al_2O_3 sandblasting (Figure 1H), and diamond bur abrasion (Figure 1J).*

bond strength values comparable to the cohesive strength of the silorane composite. In the subgroup that was etched with LS self-etch primer and repaired with Filtek LS (group 2S), the repair bond strength was significantly lower than that of the cohesive strength of the material. Furthermore, results from subgroups 1M and 1S were interesting as well. Silorane composite repaired with methacrylate composite without surface treatment (group 1M) seemed to produce better microtensile bond strength than if silorane was used as the repair material (group 1S), even though values were not significantly different. Group 1M also resulted in higher numbers of cohesive failures, which suggested higher success in bonding of the two materials at their interfaces. From these results, methacrylate composite seems to be a better choice of repair material in the situation that an air abrasion unit is unavailable. Although the amount of bonding between methacrylate composite and the aged silorane composite without mechanical surface treatment is unknown, the chemical bonding may likely contribute to this increase in repair microtensile bond strength. In addition, with mechanically prepared surfaces, either abrasion with a diamond bur (group 4M, 4S) or abrasion with aluminum oxide air abrasion (group 3M, 3S), silorane composite seemed to work better than methacrylate composite with nominally higher repair bond strengths (even though there was no statistically significant difference). The chemical reaction between the methacrylate composite and the aged silorane composite must be further explored to explain these results.

When considering the repair material for aged silorane composite, the clinically sufficient repair bond strength is not known. It is well known that resin composite seldom fails mechanically at the junction with etched enamel. Therefore, it can be surmised that a repair microtensile bond strength that is similar to that of resin composite to etched enamel (33.8-55.6 MPa[29]) would be clinically adequate.[24] In this study, repair bond strength (37.2–50.2 MPa) was in the same range as the bond strength of resin composite to enamel. Moreover, the repair microtensile bond strength in group 3M and group 3S was comparable to the cohesive strength of the silorane resin composite. This might indicate that when using aluminum oxide sandblasting on the aged silorane composite, it can be repaired with either silorane composite with the LS system self-etch adhesive or a methacrylate composite using a methacrylate dental adhesive.

CONCLUSION

Within the limitations of this laboratory study, the following conclusions can be drawn:

1. Aluminum oxide sandblasting produced microtensile bond strength comparable to the cohesive strength of silorane composite.
2. After aluminum oxide sandblasting, aged silorane composite can be repaired with either silorane composite with LS system adhesive or methacrylate composite with methacrylate dental adhesive.

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