Original Article

Effects of three surface conditioning techniques on repair bond strength of nanohybrid and nanofilled composites

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ABSTRACT

Background: Repair bond strength of different composite resins has been assessed in few studies. In addition, reports on the efficacy of surface treatments are debated. Therefore, this *in vitro* study was conducted to evaluate the effect of three surface treatments on two nanocomposites versus a microhybrid composite.
Materials and Methods: In this experimental study, 135 composite blocks (45 specimens per composite) of microhybrid (Filtek Supreme Z250, 3M ESPE, USA), nanohybrid (Filtek Supreme XT, 3M ESPE), and nanofilled (Filtek Supreme Z350, 3M ESPE) were thermocycled (5000 rounds) and then surface roughened (except in a control group of 9 specimens of three composite types). Each composite type was divided into three subgroups of surface treatments: (1) Bur abrading and phosphoric acid (PA) etching, (2) sandblasting and PA etching, and (3) hydrofluoric etching and silane application ($n = 15 \times 9$, complying with ISO TR11405). Composite blocks were repaired with the same composite type but of a different color. Microtensile bond strength and modes of failure were analyzed statistically using two-way analyses of variance, Tukey and Chi-square tests ($\alpha = 0.05$).
Results: There were significant differences between three composite resins ($P < 0.0001$) and treatment techniques ($P < 0.0001$). Their interaction was nonsignificant ($P = 0.228$). The difference between nanofilled and nanohybrid was not significant. However, the microhybrid composite showed a significantly higher bond strength (Tukey $P < 0.05$). Sandblasting was significantly superior to the other two methods, which were not different from each other.
Conclusion: Within the limitations of this *in vitro* study, it seems that microhybrid composite might have higher repair strengths than two evaluated nanocomposites. Among the assessed preparation techniques, sandblasting followed by PA etching might produce the highest bond strength.

Key Words: Acid etching, air abrasion, composite resins, dental, dental adhesives, tensile strength

INTRODUCTION

Staining, fracture, or departures can clinically compromise composite restorations.[1,2] A questionable composite restoration can either be completely changed with a new restoration or be repaired.[2] A full replacement is the most frequent practice; however, it is over-treatment since it might deteriorate dental/pulpal tissues, remove intact structures and etched enamel, and enlarge the cavities.[3] Hence, restoration

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repair seems preferable over total replacement.[2,3] Nonetheless, repair might weaken the restoration's retention potential.[2,4,5]

The adhesion between fresh and old composite surfaces is achieved by a layer of oxygen-inhibited nonpolymerized resin.[2,6,7] Aging and water sorption might compromise the bond strength by removing this unpolymerized film or reducing the unsaturated double carbon-carbon bonds.[2,6,8] The prognosis of this bond depends on multiple factors including old composite's surface properties as well as applied surface treatments.[3,9-12] A variety of techniques are suggested to increase the composite-to-composite bond. These methods (including irrigating, disk/bur abrading, sandblasting, etching, or the application of silane/bonding agents) attempt to alter the composite surface topology.[2,7,10,12,13] Other important factors determining the surface characteristics of a composite resin are the composition and ratio of fillers.[3] Having a high proportion of filler particles, nanocomposites are claimed to have promising physicomechanical properties. However, despite their broadening usage as esthetic materials, their repair bond strengths are not assessed except in a few studies.[3,5] Furthermore, the studied surface treatments for improving repair bond strength of composite-to-composite are highly controversial:[14] Some researchers have found promising effects using hydrofluoric (HF) acid, roughening with a bur, or sandblasting.[4,5,11] On the other hand, some studies have failed to show a proper influence of these methods.[1,15]

In view of the above-mentioned shortcomings and controversies and considering that results of a specific brand of a material cannot be necessarily generalized to other brands of the same material.[7] This study was conducted to assess the repair bond strength and mode of failure of three composites (two different nanocomposite types in comparison to a microhybrid composite), all under the effect of three different surface treatments. Since shear bond strength is not as reliable as microtensile bond strength (μTBS), we used the microtensile technique in this study.[3,16]

MATERIALS AND METHODS

This *in vitro* experimental study was performed on 144 composite specimens (9 experimental groups of 15 specimens each, and a control group of 9 specimens). The size of groups was in accordance with the ISO TR 11,405 standard.[17] All the experimental operations except the laboratory works were performed by a single operator. The laboratory works were performed by experienced laboratory experts. The materials used in this study are summarized in Table 1.

Preparation of experimental composite blocks
A total of 135 composite blocks were fabricated from the B1 color of three different composite materials (45 specimens per composite type) mentioned in Table 1.

The composite blocks (4 mm high and 8 mm in diameter) were molded using stainless steel cylinders. The metal cylinders were incrementally filled with composite layers, from the bottom to the top [Figure 1]. Each composite increment was 2 mm thick. After placing each layer, it was light-cured (40 s) vertically from a 1-mm distance, using a light-emitting diode unit (Demetron II, Kerr, Italy). After curing each composite block, the light-curing unit was calibrated at 600 mW/cm². To create a smooth composite surface, after placing the final layer of composite and before curing it, a transparent Mylar matrix strip was placed on the surface [Figure 1], and

Table 1: Used materials, along with their brands, manufacturers, and countries of origin

Material	Brand	Manufacturer	Country
Microhybrid composite	Filtek Supreme Z250	3M ESPE	USA
Nanohybrid composite	Filtek Supreme XT	3M ESPE	USA
Nanofilled composite	Filtek Supreme Z350	3M ESPE	USA
Enamel-bonding agent	Margin-Bond	Colten	Switzerland
Phosphoric acid	3M ESPE	3M ESPE	USA
HF acid	Pulpdent	Pulpdent	USA
Two-part silane-coupling agent	Bis-Silane Porcelain Primer	Bisco	USA
Self-cure transparent resin	Acropars	Acropars	Iran
Nail polish	Jordana	Jordana	USA
Glue	Mitreapel	Beta Kimya San, Vetic AS	Turkey

HF: Hydrofluoric

the composite was light-cured.[18] After polymerization, the molds were gently removed.[2]

Preparation of control group

Similar to the above procedures, 9 composite blocks were fabricated incrementally from the above mentioned 3 composite types (each control subgroup contained 3 specimens). The only difference between the experimental and control composite blocks was that control stainless steel cylinders were of 8 mm high.

The control composite blocks were built up in four increments of 2 mm each [Figure 2]. Each layer was light-cured (40 s) vertically from a 1 mm distance. The light-curing unit was calibrated at 600 mW/cm², after curing each composite block. In this group, no transparent matrix strip was used. The 8 mm high control stainless steel templates were later used for repairing the experimental composites as well.

Thermocycling

All experimental specimens were thermocycled (Vafaei Industrial, Iran) for 5000 rounds between 5°C and 55°C temperatures (dwell time = 30 s, transfer time = 10 s). The control specimens were not thermocycled.

Surface treatments of aged experimental composites

Each experimental composite group (*n* = 45) was randomly divided into three subgroups of 15 specimens each. Each experimental subgroup was subjected to one of the following three surface treatments. The control specimens were not surface-abraded.
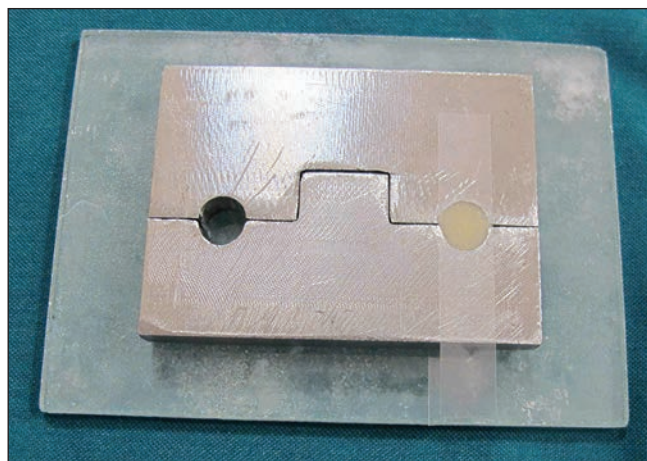
Subgroup A (sandblast-phosphoric acid etching)

The smooth and aged surface of each specimen was sandblasted for 10 s. This was done by spraying 50 μm aluminum oxide particles from a 5 mm distance under the air pressure of 60-100 PSI. The nozzle of the sandblaster device (Microsandblaster, Dento Prep, RØNVIG, Dental Mfg, Denmark) was held perpendicular to the composite surface. Subsequently, each composite block was etched by means of 35% phosphoric acid (PA) [Table 1] for 30 s. Finally, they were rinsed (30 s) and dried (10 s) from a 5 mm distance using an oil-free air syringe.[16]

Subgroup B (diamond bur abrading-phosphoric acid etching)

Aged composite surfaces were abraded for 10 s with a cylindrical diamond bur with coarse particles (D&Z, Germany) attached to a high-speed water-spraying handpiece. After preparing every 5 specimens, used burs were replaced with new ones. Afterward, composite blocks were etched using 35% PA [Table 1] for 30 s. Finally, they were rinsed (30 s) and air-dried (10 s) from a 5 mm distance.[16]

Subgroup C (hydrofluoric etching-silane bonding)

Each aged composite surface was etched using 9.6% HF [Table 1] for 60 s. It was then air–water–sprayed (20 s) and air-dried (10 s) from a 5 mm distance. Afterward, a two-part silane-coupling agent [Table 1] was applied to the etched surface according to the manufacturer's instructions.

Repairing the aged and conditioned experimental composites

The 4 mm-high experimental blocks were mounted at the bottom of the 8 mm-high control template cylinder. An enamel-bonding agent [Table 1] was applied to the abraded surface of the aged composite block. Afterward, 2 mm thick layers of fresh
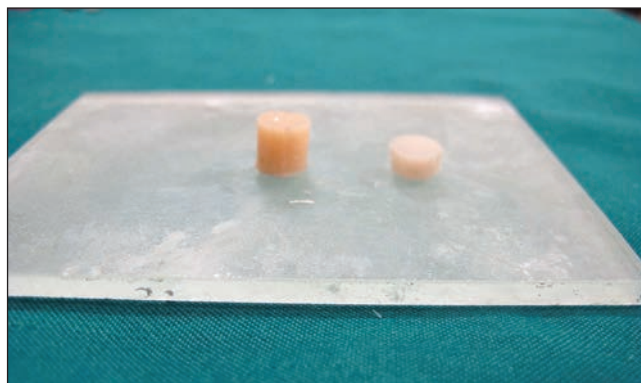

Figure 1: A filled template is shown on the right, covered with a Mylar matrix.

Figure 2: A control composite block (left) versus an experimental one (right).

composite were placed incrementally, within the 8 mm high control mold. Each increment was then vertically photopolymerized (40 s) as stated above.[2] This way, a new same-size block of fresh composite was built up on the abraded surface of the aged composite. The type and brand of the fresh composite were matched with the type and brand of the aged composite (i.e., microhybrid composite [Filtek Supreme Z250], nanohybrid [Filtek Supreme XT], and nanofilled [Filtek Supreme Z350]). However, the color of fresh composite was deliberately selected as A4 to be distinguishable from the aged composite (B1).

Aging the repaired experimental composites

All experimental specimens (which had been thermocycled, surface-abraded, and repaired before), were stored in normal saline (24 h) at room temperature.[2]

Preparing micron samples

Merging in acrylic resin

Since the composite specimens were small and light, they could not be conveniently cut by a high-speed disk cutter (Nemofanavaran Pars, Mashhad, Iran), as the disk would throw them out instead of preparing them. Therefore, they were first modified temporarily to a heavier and bulkier specimen with squared angles, so that the disk cutter would not throw them out. This was done by adding transparent acrylic resin [Table 1] to the composite: First, a layer of red formaldehyde-free nail polish [Table 1] was applied to all composite surfaces. When the nail polish was dried, the self-curable transparent acrylic resin was poured into a cubic plastic template of 10 mm height, 15 mm width, and 25 mm length. The composite cylinder was placed in the acrylic resin. Once the resin was cured, the block was removed from the plastic template and subjected to cutting machine [Figure 3].[19-22]

Trimming

A high-speed disk saw [2000-3500 rounds/min, Table 1] was used to trim the composite cylinders into rods of 1 mm^2 × 1 mm^2 cross-sections. The disk saw was 300-μm thick and of 110-mm diameter [Figure 3].[19-22] The device carved composite rods (8 mm length × 1 mm height × 1 mm width) out of the blocks of composite merged in transparent acrylic resin.[19-22]

Microtensile assessment

The 1 mm^2 × 1 mm^2 composite rods were mounted symmetrically from their both ends on the plates

Figure 3: The composite specimen (red) merged in transparent acrylic resin.

of a tensile testing machine (Micro Tensile Tester, Bisco, USA), using glue [Table 1 and Figure 4]. The test machine applied the tensile force at 0.5 mm/min speed to the both ends of the composite blocks. The necessary force to debond each composite rod was recorded. The debonding force was divided by the surface area size of the rod base (1 mm^2) to calculate the μTBS in μTBS (MPa). Rods detached from any of their glued ends (instead of being debonded) would be excluded. This happened in the case of six composite rods, two of which were replaced with new rods.

Mode of failure

A stereomicroscope (Olympus SZX-12, Olympus, Japan) was used to assess the debonded surfaces at ×8 magnification to determine the failure mode. The failure mode was defined as "adhesive" failure, if the detachment had been occurred through the junction of the composites. Cohesive failure meant a breakage through one of the composites (fresh or aged). Mixed failure meant a combination of both types.[2]

Statistical analysis

Descriptive statistics and frequency distributions were calculated for μTBS and mode of failure. The findings regarding the μTBS values were analyzed using one-way and two-way analyses of variance (ANOVA), Tukey's *post-hoc*, Mann–Whitney U-test, and independent-samples *t*-test. The modes of failure were analyzed using Chi-square test. The level of significance was set at 0.05.

RESULTS

Four of the specimens in one of the subgroups (subgroup C of nanofilled composite [Z350 XT]) were

failed (debonded before exerting tensile force) and excluded. The µTBS results are presented in Table 2.

The two-way ANOVA showed significant differences between the bond strengths of three composites ($F = 55.320$, $P = 0.0000$) and between three surface treatments ($F = 17.609$, $P = 0.0000$). However, the interaction of the variables treatment and composite was not significant ($F = 1.43$, $P = 0.228$), meaning that the effect of treatments was similar in the case of all tested composites.

The Tukey's *post-hoc* test showed that the difference between nanofilled (Z350 XT) and nanohybrid

(Z250 XT) composites was not significant; however, microhybrid (Z250) composite showed significantly better results compared to above-mentioned composites [Table 3]. The efficacy of two treatments surface roughening with diamond burs and HF + silane application was similar. However, sandblasting was significantly superior to the other two methods [Table 3].

The one-way ANOVA showed a significant difference between the subgroups ($F = 18.53$, $P = 0.0000$). The Tukey's *post-hoc* test showed significant differences in 19 out of 36 pairwise comparisons with the highest difference seen between etched nanofilled composite (Z350 XT) and sandblasted microhybrid (Z250). The lowest difference was observed between etched nanofilled composite and etched nanohybrid composite. The Mann–Whitney U-test was used to compare the positive control groups of each composite with the three corresponding subgroups of the same composite. The independent-samples *t*-test was used to compare the whole control groups (all composites combined) with each type of composite in the control group. The independent-samples *t*-test was also used to compare surface treatments with the control [Table 2].

The mixed mode of failure was the common type of failure in most of subgroups and groups. However, according to the Chi-square test, the comparisons were mostly nonsignificant [Table 4]. Among the surface treatments, only surface roughening by diamond burs resulted in a significantly higher rate of mixed failure type [Table 4]. There was no significant difference between the modes of failure of three composite types ($P = 0.708$). However, a significant overall difference was observed between the modes of failure of three surface treatments ($P = 0.035$), indicating a shift in the modes of failure of diamond bur and acid etching/silane application toward mixed failures [Table 4].

Table 2: Descriptive statistics for the µTBSs (MPa) in all the subgroups

Composite	Treatment	*n*	Mean	SD	95% CI
Nanofilled (Z350 XT)	Bur	15	15.671	7.126	12.49-18.85
	Sandblast	15	22.538	8.972	19.36-25.72
	HF etching	11	13.595[a]	4.070	9.88-17.31
Microhybrid (Z250)	Bur	15	29.557[b]	5.029	26.38-32.74
	Sandblast	15	32.392[c]	7.771	29.21-35.57
	HF etching	15	28.923[d]	7.326	25.74-32.10
Nanohybrid (Z250 XT)	Bur	15	18.349[e]	4.629	15.17-21.53
	Sandblast	15	25.176[f]	5.019	22.00-28.36
	HF etching	15	14.481[g]	2.841	11.30-17.66
Nanofilled	All	41	17.63[h]	8.065	15.08-20.17
Microhybrid	All	45	30.29	6.831	28.24-32.34
Nanohybrid	All	45	19.34[i]	6.115	17.5-21.17
All	Bur	45	21.19[j]	8.248	18.71-23.67
All	Sandblast	45	26.70[k]	8.401	24.18-29.23
All	HF etching	41	19.53[l]	8.836	16.74-22.32
Nanofilled	– (Control)	3	24.92[a]	5.57	11.08-38.75
Microhybrid	– (Control)	3	46.79[b,c,d]	11.22	18.92-74.66
Nanohybrid	– (Control)	3	35.46[e,f,g]	5.93	20.73-50.19
All	– (Control)	9	35.72[h,i,j,k,l]	11.74	26.7-44.75

Each superscript letter marks the elements of a statistically significant pairwise comparison. The used tests for pairwise comparisons were Mann–Whitney U-test (for the letters "a to g"), and *t*-test (for the letters "h to i"). SD: Standard deviation; CI: Confidence interval; µTBSs: Microtensile bond strengths; HF: Hydrofluoric

Table 3: Results of the Tukey test for pairwise comparisons between three composite types and pairwise comparisons between three surface treatments

Compared groups		Mean difference (MPa)	SE	*P*	95% CI	
					Low	Up
Nanofilled (Z350 XT)	Microhybrid (Z250)	−12.66	1.34	0.0000	−15.85	−9.48
Nanofilled (Z350 XT)	Nanohybrid (Z250 XT)	−1.71	1.34	0.4133	−4.90	1.48
Microhybrid (Z250)	Nanohybrid (Z250 XT)	10.96	1.31	0.0000	7.84	14.07
Diamond bur	Sandblast	−5.51	1.31	0.0001	−8.62	−2.40
Diamond bur	HF etching	1.67	1.34	0.4320	−1.52	4.85
Sandblast	HF etching	7.18	1.34	0.0000	3.99	10.36

SE: Standard error; CI: Confidence interval; HF: Hydrofluoric

Figure 4: The tensile test machine.

Table 4: Net distributions of modes of failure and statistical comparisons of modes of failure adhesive versus mixed, using Chi-square test

Composite	Treatment	*n*	Adhesive	Mixed	*P*
Nanofilled (Z350 XT)	Diamond bur	15	3	12	0.020
	Sandblast	15	10	5	0.197
	HF etching	11	6	5	0.763
Microhybrid (Z250)	Diamond bur	15	5	10	0.197
	Sandblast	15	6	9	0.439
	HF etching	15	6	9	0.439
Nanohybrid (Z250 XT)	Diamond bur	15	5	10	0.197
	Sandblast	15	9	6	0.439
	HF etching	15	4	11	0.071
Nanofilled	All	41	19	22	0.639
Microhybrid	All	45	17	28	0.101
Nanohybrid	All	45	18	27	0.180
All	Diamond bur	45	13	32	0.005
All	Sandblast	45	25	20	0.456
All	HF etching	41	16	25	0.160

HF: Hydrofluoric

DISCUSSION

The results of this study showed that the tested nanocomposites were not superior, and the microhybrid composite had a higher bond strength in comparison with nanohybrid and nanofilled composites, which had similar TBS rates. This pattern was visible both in the experimental and control groups. Microhybrid was the only composite in this study with an insignificant difference with control. Sandblasting was the most useful method, followed by two similarly ineffective methods of surface roughening by diamond burs and etching/silane application. In addition, this order of treatment efficacy was the same for all the tested composites. Compared to the cohesive strength of corresponding substrates, the interfacial repair bond strength had declined mostly in a significant way, which confirmed the previous results reporting bond strengths between 25% and 80% of control.[2,4,5]

Aging can cause water infiltration into the resin and into the junction of fillers and matrix, deteriorate composite matrix by hydrolytic degradation of the silane film over fillers or matrix swelling and also remove its free radicals by water sorption and thermal stresses.[2,3,8,14] A substantial portion of the composite-to-composite bond is chemical and introduced by monomers in the oxygen-inhibited layer of the cured composite and monomers of the fresh composite.[6-8] Surface roughening is necessary or perhaps the most important factor for improving the repair bond strength because of creating micro- and macro-interlocking and broadening the surface.[2,6-8,10,12,13,18,23] Moreover, shaving a layer of resin may expose a rough and fresh surface, which might improve the bond strength.[7,13] However, the bond strength did not increase up to the control levels in the present research. This might be due to the lack of oxygen-inhibited coating and the small amount of free monomers and photoinitiators in deeper layers of aged composite, which are now exposed.[7,23] Therefore, although this viscose coating consists of unpolymerized molecules that may produce covalent interfacial bonds,[8,12] the bonding ability of this layer never compares to fresh composites, as its free monomers and photoinitiators are reduced.[7,8,23] Moreover, water sorption might swell the matrix and/or degrade the silane layer on fillers.[3,8] Furthermore, thermal fluctuations of thermocycling might produce microfractures in the resin or through its interface with fillers.[3,25] This aging method was used in this study, since thermocycling for 5000 cycles might be more effective than acid citric storage or water boiling.[11,14]

Repair bond strength necessary for an acceptable composite repair *in vivo* are not investigated. However, the strength of satisfactory resin-to-enamel bonds might be between 15 and 30 MPa.[2,26] Perhaps, repair bond strengths similar to composite-to-enamel bond strengths might be acceptable in clinical conditions.[2,27] According to certain authors, composite repair bond strengths need to be over 18 MPa to be clinically satisfactory.[2,26] Most of the assessed groups in this study produced repair bond strengths comparable or higher than the suggested minimum. Nanofilled and nanohybrid composites did not show significant differences. This was contrasting to another study which found nanofilled composite

of better repair retention.[28] A study compared two different nanohybrid composites with a microhybrid composite.[3] One of nanohybrid materials showed results similar to microhybrid, while the other one showed slightly better results.[3] It seems that besides numerous influencing factors, the type and brand of composites might be the case.[2,7]

In the present study, three surface conditioning approaches were used. Two methods depended on an initial mechanical surface roughening followed by acid etching using PA. The other one relied on two chemical methods (etching using HF and then applying silane). The highest bond strength values in our study belonged to the group subjected to sandblasting followed by acid etching with PA. The effect might be mostly due to the sandblasting, since the other method (diamond bur application that had the acid etching step as well) did not provide similarly appropriate bond strengths. Moreover, PA etching alone has not been successful in increasing repair bond strength; it might only be a superficial cleanser.[1,2,18] Many studies have shown promising effects for silane application which might be related to new siloxane bonds created between resin and fillers.[2,9,10,13,15,26] In this study, however, the silane application resulted in the weakest bond strengths and was the only group that failed to increase the bond strength to 20 MPa or higher. Few researches as well-reported that the addition of silane to the bonding system might not significantly improve bond strength, or even might reduce it.[2,7,13] Methodological differences such as used brands and their qualities and compositions might contribute to the controversy.[7] The conclusive finding was the superiority of sandblasting followed by PA etching compared to the other methods, regardless of composite groups. In the nanofilled group, sandblasting was the only subgroup among all subgroups that was not significantly lower than control. This result was in line with previous studies showing aluminum oxide sandblasting as the best approach to regain proper bond strengths.[29] Nevertheless, from a clinical perspective, HF application and sandblasting are not as safe and convenient as surface roughening with diamond burs.[14] Contradictory results have been reported with the use of diamond burs for preparing composite surfaces prior to bonding.[9,12,14,15,18] The findings of the present study should be evaluated further using negative control groups, in which thermocycling and composite repair would have been done however without any surface conditioning.

Cohesive failures are fractures within the composite matrix and suggest a powerful adhesive bond between two composites, being greater than the cohesive bond within each of the composites. Therefore, they indicate acceptable clinical outcomes for a restoration repair.[1-3] On the other hand, samples with low bond strengths are likely to depart at the junction of two composites (adhesive failure) which imply lower success of repair bond or more robust structure of composite matrices. No pure cohesive failures were observed in this study. The composite types did not show a significant effect on mode of failure. However, surface roughening with diamond burs improved the mode of failure to the cohesive end. It should be taken into consideration that adhesive failures are not easily detected under light microscopy, and the results should be carefully interpreted since a greater number of mixed failures might have happened.[2]

CONCLUSION

Microhybrid composite might have a better repair bond strength compared with nanocomposites, while nanofilled and nanohybrid types are less likely different from one another. The assessed surface conditioning techniques might not recover bond strengths up to control levels. Sandblasting followed by PA etching might produce the highest repair bond strengths.

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Conflicts of interest
The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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