Effect of Three Surface Treatments on the Adhesive Properties of Indirect Composite Restorations

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Purpose: To evaluate the effect of different surface treatments of composite resin blocks on the adhesive properties of indirect composite restorations. The null hypothesis tested was that none of the performed surface treatments would produce greater bond strength.

Materials and Methods: The crowns of 80 extracted molars were transversally sectioned next to the pulp to expose flat, deep dentin surfaces. Eighty-eight cylindrical composite specimens measuring 3.5 mm in diameter and 10 mm in height were prepared and randomly divided into 4 groups (CG, HFSiG, SaG, SaSiG), which respectively received the following treatments: control (CG): etching with 9.5% HF acid gel and application of a silane (HFSiG); sandblasting (SaG) with 50- μ m Al₂O₃ from a distance of 10 mm at a pressure of 2.5 bars for 10 s; combination of sandblasting and silanization procedures (SaSiG). Two composite specimens of each group were analyzed with SEM, while the remaining twenty cylindrical specimen were bonded to dentin samples using a two-step adhesive system and a thin layer of composite. After 24 h storage and 5000 thermocycles, all specimens were loaded to failure under tension in a universal testing machine. The mean differences of each group were analyzed with the Kruskal-Wallis test, while multiple comparisons were made using the Ryan-Einot-Gabriel-Welsch Range test. P-values less than 0.05 were considered to be statistically significant in all tests. The fracture pattern of bonded specimens was also evaluated by SEM.

Results: SEM analysis showed morphological changes in each group. The mean values (in MPa) of TBS (\pm SD) for groups CG, HFSiG, SaG, and SaSiG were 11.17 \pm 3.48, 10.81 \pm 5.19, 16.51 \pm 3.45 and 16.55 \pm 3.16, respectively. Statistical analysis showed that the bond strength was significantly affected by surface treatment (p < 0.001). Multiple comparison analysis identified statistically significant differences for CG and HFSiG vs SaG and SaSiG (p < 0.05), while no significant differences were found for the comparisons CG vs HFSiG and SaG vs SaSiG (p > 0.05). Only a few adhesive failures were recorded (CG: 0.5%; SaG: 0.4%; HFSiG: 0.5%; SaSiG: 0.7%). The null hypothesis was rejected.

Conclusion: Composite surface treatments are important for adhesion of indirect composite restorations. Roughening the composite area of adhesion, sandblasting, or both sandblasting and silanizing can provide statistically significant additional resistance to tensile load. Hydrofluoric acid etching with silane treatment did not reveal significant changes in tensile bond strength. These findings suggest that sandblasting treatment was the main factor responsible in improving the retentive properties of indirect composite restorations.

Keywords: tensile bond strength, composite, surface treatments.

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The use of composite resin for dental restorations has increased with the improvement of the bonding systems, curing systems, and mechanical-physical properties of the resin systems. The recently developed resin composites are superior to the earlier versions in regard to wear resistance

and color stability. However, the main shortcomings – eg, polymerization shrinkage – still remain.^{8,13} Polymerization shrinkage, ranging from 1.5% to 3% of the total material volume,⁹ is a major problem in adhesive filling techniques. The contraction produces stresses which can exceed the cohesive and adhesive strengths of the restorative materials.¹³ In posterior cavities, the mass to be polymerized is so large that the shrinkage forces win out, especially where cervical margins are located in dentin, producing marginal defects and gaps despite careful application.¹⁰ This promotes microleakage, which can cause secondary caries, pulpal irritation, postoperative sensitivity, and marginal discoloration.⁵

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For larger restorations, it is possible to say that inlays are better alternatives than direct resin composite fillings.³⁹ Postcuring at a high temperature resulted in a greater stress relaxation and conversion compared to the directly placed, only light-cured composite;^{14,32} the decreased stress on the bonding surface resulted in an improved bond and seal.^{16,50} Moreover, the wear resistance,^{19,52} physical properties,^{34,49} and color stability^{51,11} of indirect composite restoratives have been improved. Recently, several new indirect composite restorative systems claim to be successful for occlusal restorations. These new microhybrid composites are characterized by a filler:matrix ratio that is significantly greater than that of the preceding generation.³⁰

However, in indirect restorations, the bond strength between (a) the resinous base and luting composite and (b) the luting composite and inlay resulting from micromechanical retention or copolymerization was found to be critical,³⁶ especially after restoration postcuring. If these interfaces were weak components of the restoration, they would have significant consequences. For indirectly fabricated restorations, the weakest part of the restoration is the resin luting agent layer exposed at the margin.^{24,38} Some articles have discussed surface treating indirect resin composites^{20,43,44} and porcelain^{26,27} with sandblasting or silane agents to improve bond strength between the resin luting agent and cured resin composite or porcelain. A number of techniques have also been proposed to improve the bond strength of composite repair, ie, roughening, etching the substrate surfaces with acidulated phosphate fluoride, HF acid gel, airborne particle abrasion, or using adhesive resins.^{1,7,21,25,33} However, the effect of composite surface treatment on the adhesive properties of indirect restorations is not entirely clear. While several researchers found that surface roughness of the composite was an important factor in developing high repair bond strength,^{25,37} others reported that bond strength decreased when the surface was ground or roughened.^{7,21} Despite the hazards of HF acid gel, etching the surface of indirect composite restorations with this acid followed by the application of a silane coupling agent is a wellknown and recommended method to increase bond strength, while not much importance has been given to sandblasting treatments.

The purpose of this in vitro study was to evaluate the effect of three surface conditioning methods (sandblasting, silanization after hydrofluoric acid etching, or both together) on the adhesive properties of indirect composite restorations and to identify whether an optimal method exists. SEM analysis of the treated composite substrates was also performed in order to evaluate morphological changes before adhesion, and the fracture pattern of bonded specimens after tensile testing.

MATERIALS AND METHODS

Specimen Preparation

Eighty freshly extracted human third molars were selected and stored in an aqueous solution of 0.5% chloramine T at 4° C until the start of the experiment. The inclusion criteria were absence of carious lesions and restorations. Each crown was sectioned perpendicular to its longitudinal axis at 2 mm from the cementoenamel junction using a slow-speed diamond saw (Micromet M, Remet; Casalecchio di Reno, Bologna, Italy) under copious water spray, in order to expose a flat surface of deep dentin next to the pulp. Each surface was then ground with 180-grit silicon carbide (SiC) paper under running water for 30 s to produce and standardize the smear layer thickness on the dentin surface. The bonding surfaces were then examined under a stereomicroscope (Nikon SMZ10; Tokyo, Japan) to ensure that they were free of residual enamel.

Parallel to this, 88 composite specimens were obtained by placing the microhybrid composite Enamel-Plus HFO UD3 (Micerium, Avegno; Genova, Italy) inside translucent polyethylene cylindrical molds with an inner diameter of 3.5 mm and height of 10 mm. Each mold was put on a glass surface and then filled with composite resin. The composite was placed in the mold in a few 2-mm-thick increments, following the layering technique. Each increment was light cured for 40 s (XL 3000, 3M; St Paul, MN, USA) with a 450mW/cm² output. The procedure resulted in cylindrical specimens of composite resin measuring 3.5 mm in diameter and 10 mm in height, producing for all samples an equal area of adhesion of 9.6 mm². Before testing, the polyethylene molds were gently removed from the test samples, which were then subjected to an additional cycle of polymerization in an oven for composites at 70°C for 10 min (Bulb PlusT-Micerium, Avegno; Genova, Italy). All composite surfaces were then ground with 600-grit silicon carbide (SiC) paper under running water for 30 s to expose filler particles. Subsequently, the cylindrical composite specimens were randomly assigned to one of the 4 conditioning methods (n = 22 for each group).

Surface Conditioning Methods

The surface treatments performed in each group were as follows:

- 1. Control group (CG): No further surface treatment was applied to this group.
- 2. Silanized group (HFSiG): The adhesive substrates were etched with 9.5% HF acid gel (Porcelain Prep-Kit, Pulpdent; Watertown, MA, USA) for 60 s. They were rinsed under running tap water for 30 s and dried with compressed oil-free air for 30 s. Dry-Rite agent (Porcelain Prep-Kit, Pulpdent) was applied with an eyedropper to dry the surfaces. A silane agent (Porcelain Prep-Kit, batch number 060202) was then applied to the composite specimens, allowed to evaporate for 3 min, and air dried for 30 s in accordance with the manufacturer's recommendations.
- 3. Sandblasted group (SaG): Air-borne particle abrasion with 50- μ m Al₂O₃ (Korox, Bego; Bremen, Germany) was applied using an intraoral air-abrasion device (Micerium, Avegno; Genova, Italy). The tip of the microetcher was kept 5 cm away from the surface of each specimen and applied for 10 s at 2.0 bar pressure. All specimens were then rinsed under running tap water to remove the debris.
- 4. Sandblasted and silanized group (SaSiG): Composite samples were sandblasted as described for SaG. Silane solution then was applied as described for HFSiG.



Fig 1 Dentin sample embedded in acrylic (top); composite specimen (left) and its polyethylene mold (right); insulating tape with the hole to limit the bonding area (bottom).



Fig 2 Test specimen aligned on universal testing machine for tensile bond strength test.

SEM Analysis

In an additional experiment before adhesion procedures, the surface of two substrates of each group was examined using a scanning electron microscope (SEM) (LEO 435 vp LEO Electron Microscopy; Cambrige, UK). Areas representing the average roughness of topographical contours of each composite specimen were viewed and photographed at original magnifications of 500X and 1000X. All samples were first sputter coated with gold (Emitech K550, Emitech; Ashford, Kent, UK), vacuum-packed in argon for 2 min and in 25 mA to obtain a uniform stratum of gold powder of 100 A, and then observed with the SEM.

Bonding Procedures and Tensile Bond Strength (TBS) Test

All the bonding procedures were carried out in accordance with the manufacturer's instructions by the same operator throughout the experiments.

Before bonding, composite inlays were randomly assigned to dentin samples and their total thickness was first recorded with a digital micrometer. Each tooth was embedded in acrylic resin and the dentin substrate was then etched with 37% phosphoric acid gel (EnaEtch, Micerium, Avegno; Genova, Italy) for 30 s, thoroughly rinsed for 30 s and lightly blot dried, leaving the dentin visibly moist. The two-step adhesive system used for this study (Enabond, HFO, Micerium, Avegno) was applied with a microbrush to etched dentin, gently dried to evaporate the solvent and light cured for 40 s (XL 3000, 3M; St Paul, MN, USA) with a 450-mW/cm² output. Before cementation and in order to limit the bonding area, a small piece of insulating tape with a central hole was placed on the dentin surface. Tape was perforated with a modified Ainsworth rubber-dam punch machine to create holes 3.5 mm in diameter (Fig 1). Then, a thin

layer of composite (EnamelPlus, UD2, Micerium, Avegno) was used as cement and placed over the dentin substrates. Subsequently, the composite specimens (3.5 mm diameter and 10 mm height) were bonded on.

Upon placement of the adhesive composite, the bonded assembly was held centrally between the two measuring arms of the vertically positioned digital micrometer. A load pressure of about 5 N was applied on the cylindrical composite specimens in order to standardize and simulate clinical conditions of inlay cementation. This pressure was repeated three times for 5 s each at intervals of 15 s. The composite film thickness was kept at approximately 100 µm. The micrometer arms were slowly adjusted to produce a reading that was 100 μ m (mean) thicker than that initially recorded for the respective dentin specimen and composite inlay. During the dwell time and before the composite resin luting agent completely polymerized, every excess was carefully removed with a thin instrument used to place composite fillings. The adhesive interface was light cured (XL 3000, 3M; 450 mW/cm² output) under a load of 5 N from 4 directions for 40 s, for a total exposure time of 160 s. After 24 h of storage in distilled water at 37°C, all specimens were thermocycled (Thermocycler 2000, Heto-Holten A/S; Allerod, Denmark) for 5000 cycles between 4°C and 55°C, with a dwell time of 15 s. The transfer time from one bath to the other was 2 s. All the above procedures resulted in experimental specimens of composite-bonded dentin that were tested until failure in tension using a Universal Testing Machine (Lloyd LR3OK, Lloyd Instruments; Fareham, UK). To exclude uncontrollable later forces due to misalignment, parallelism between resin blocks and cylindrical composite specimens was obtained by using a parallel device (CL-MF2002S, Heraeus Kulzer; Hanau, Germany). The two extremities of each

Table 1 Experimental results in MPa

	CG	HFSiG	SaG	SaSiG
Mean	11.17	10.81	16.51	16.55
± SD	3.48	5.19	3.45	3.16

Table 2 Statistical analysis

	CG vs	CG vs	CG vs	SaG vs	SaG vs	HFSiG vs
	HFSiG	SaG	SaSiG	HFSiG	SaSiG	SaSiG
Statistical significance	NS	p < 0.05	p < 0.05	p < 0.05	NS	p < 0.05

 $\label{eq:GG} CG = \mbox{control group; } HFSiG = \mbox{silanized group; } SaG = \mbox{sandblasted and} \\ silanized group; NS = \mbox{not statistically significant, } p > 0.05.$

Table 3 Percent of failure type by group

Failure type in %	CG	HFSiG	SaG	SaSiG
(a) adhesive: (composite/cement) (b) cohesive:	74.1	70.1	10.6	11.3
(in cement) (c) adhesive:	5.9	5.9	29.4	23.5
(cement/adhesive) (d) adhesive:	19.5	23.5	59.6	64.5
(dentin/adhesive)	0.5	0.5	0.4	0.7

experimental specimen were hooked to the dynamometer clamps in order to allow a perfect longitudinal axis between dentin and composite substrates during tensile loading (Fig 2). Finally, the tensile bond test was performed with a precharge of 0.5 N at a crosshead speed of 0.5 mm/min and 50 kgf load cell. The force (N) at failure was recorded and the tensile bond strength values (MPa) were calculated from the peak load at failure divided by the specimen surface area. The fractured surfaces of all bonded specimens were then inspected under SEM (LEO 435 vp LEO Electron Microscopy) to define the type of failures. Failure modes were categorized as: (a) adhesive failure along the composite/cement interface; (b) cohesive failure within the resin cement; (c) adhesive failure along the cement/adhesive interface; and (d) adhesive failure between the dentin surface and the adhesive. The fractional area of each failure mode in a fractured beam was determined from the SEM micrographs using image analysis software. The area occupied by each failure mode in one group was first determined and expressed as a percentage of the total bonding surface area of that particular group. The fractional areas of the four failure modes in a fractured beam were then converted to percentage surface areas for that beam.

Statistical Analysis

Data were expressed as mean and standard deviation for each group analyzed (CG, HFSiG, SaG and SaSiG). Statistical analysis was performed using SPSS Advanced Statistical 11.5 software for Windows (SPSS; Chicago, IL, USA). The differences in means of each group were analyzed by the Kruskal-Wallis test. Bond strength was taken as the dependent variable while the independent factor was the type of surface treatment. Multiple comparisons were made by Ryan-Einot-Gabriel-Welsch Range test (Q-test). P-values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

SEM analysis revealed significant morphological changes of all group specimens according the surface treatment performed. TBS test results are shown in Table 1 and Fig 3. Means values of TBS and standard deviation (\pm SD) in MPa for the groups CG, HFSiG, SaG, and SaSiG were 11.17 \pm 3.48, 10.81 \pm 5.19, 16.51 \pm 3.45 and 16.55 \pm 3.16 respectively. The overall difference of the four groups analyzed with the Kruskal-Wallis test revealed that the TBS was significantly affected by the surface treatment (p < 0.001) (Table 2). The Ryan-Einot-Gabriel-Welsch Range test for multiple comparisons showed that the SaG and SaSiG specimens had significantly higher bond strengths than CG and HFSiG (p < 0.05). No statistically significant differences were found for the groups CG vs HFSiG and SaG vs SaSiG (p >



Fig 3 Mean values of bond strength for each group analyzed. Groups with different letters are statistically significantly different. (p < 0.05; Ryan-Einot-Gabriel-Welsch Range test). Vertical lines represent the standard deviations.

0.05). This result suggests that sandblasting treatment was the main reason why SaSiG had higher bond strength, while silane treatment alone did not produce significant changes in tensile bond strength. The failure modes of bonded specimens are shown in Table 3. A very low percentage of adhesive failures to dentin was recorded in every group (CG: 0.5%; HFSiG: 0.5%; SaG: 0.4%; SaSiG: 0.7%). Differences in the percentages of composite/resin cement interface adhesive failures were found (CG: 74.1%; HFSiG: 70.1%; SaG: 10.6%; SaSiG: 11.3%).

DISCUSSION

Many factors can influence the bonding performance of adhesive systems to dentin, such as dentin substrate, testing procedures, and handling of materials. Although the use of composite materials in restorative dentistry has increased dramatically in recent years, fractures and failures can still occur. The bond strength between increments of composite is equal to the cohesive strength of the material.^{4,28} However, if the composite is contaminated, polished, processed in a laboratory (indirect composite restorations), or aged, the adhesion to a new composite is reduced by 25% to 80% of the original cohesive strength.^{1,3,48} Various methods have been reported to improve the reactivity of highly converted composites. These methods include: acid etching,44 air abrasion.^{3,6} and the use of solvents and silanes.⁴¹ However, there seems to be no consensus in the literature regarding the best conditioning method to improve adhesive properties of indirect composite restorations, although a number of techniques have been proposed to improve the bond strength of ceramic inlays to dentin, eg, through roughening, etching the substrate surfaces with acidulated phosphate fluoride, using solvents and silanes, HF acid gel, airborne particle abrasion, thermocycling, or using different kinds of resin cement.^{22,26,31,35,46} Roughening and cleaning inlay surfaces through different treatments represent important steps to improve micromechanical interlocking and chemical bonding to resin.^{40,45}



Fig 4 Composite surface ground with 600-grit silicon carbide paper, shown as control. SEM, 500X.



Fig 5 Composite surface ground with 600-grit silicon carbide paper, shown as control. SEM, 1000X.

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Fig 6 SEM imagae, 500X magnification. HF-etched and silanized composite resin surfaces of adhesion. Specimen shows a moderate amount of surface relief with the presence of pores.



Fig 8 SEM image, 500X magnification. Sandblasted composite resin surface of adhesion. Specimen shows the highest surface relief with severe undercuts and presence of grooves.



Fig 10 SEM image, 500X magnification. Sandblasted and silanized composite resin surface of adhesion. Speciment shows morphological changes with undercuts, grooves, and some pores. A thick silane layer is evident.



Fig 7 SEM image, 1000X magnification. HF-etchaed and silanized composite resin surfaces of adhesion. Specimen shows a moderate amount of surface relief with the presence of pores.



Fig 9 SEM image, 1000X magnification. Sandblasted composite resin surface of adhesion. Specimen shows the highest surface relief with severe undercuts and presence of grooves.



Fig 11 SEM image, 1000X magnification. Sandblasted and silanized composite resin surface of adhesion. Speciment shows morphological changes with undercuts, grooves, and some pores. A thick silane layer is evident.

The aim of this study was then to evaluate the efficacy of different mechanical and chemical procedures used to improve the bond strength of highly polymerized microhybrid composites. The composite surface treatments performed in this study were selected on the basis of the best conditioning methods suggested by previous studies to improve ceramic bond strength to dentin. The results of this study showed that the composite-dentin bond strength was significantly affected by surface treatments. Airborne particle abrasion with 50- μ m Al₂O₃ proved to be the most effective and reliable surface treatment. The bond strengths resulting from sandblasting treatments performed in groups SaG and SaSiG were statistically higher than any other treatment performed in groups HFSiG and CG.

SEM evaluation was also performed on composite surfaces of the control (Figs 4 and 5) and treated groups (Figs 6 to 11). This analysis revealed that HF acid gel pretreatment followed by silane application (HFSiG) dissolves the filler component of composites and produces a moderate amount of surface relief with the presence of pores (Figs 6 and 7). However, these changes did not improve bond strength significantly. On the other hand, groups treated with 50- μ m Al₂O₃ (SaG) exhibited the highest surface relief with severe undercuts and presence of groves (Figs 8 and 9). The same topographic aspect was also observed in sandblasted and silanized specimens (SaSiG) with the additional presence of some pores. A thick silane layer was evident (Figs 10 and 11). The results of this study suggest that sandblasting treatment creates irregularities on composite surfaces which facilitate micromechanical interlocking, significantly improving the adhesive properties of indirect restorations. These findings, however, are not in line with the results of a previously published study.²⁹ The different results obtained in that study are probably due to the use of different materials, different hydrofluoric acid concentrations and etching times, and different microetching pressures and particles. Martin et al²⁹ examined the effects of various surface treatments on the repaired strength of heat-treated composites and concluded that surface treatments did not provide a significant improvement of bond strength when compared to the control group. Surface treatment with air abrasion resulted in the strongest repairs, while surface treatment with phosphoric acid and hydrofluoric acid resulted in the weakest repairs. This study's divergent results are probably due to the use of different parameters of surface treatment. Martin et al^{29} used a flow of 50- $\!\mu m$ aluminum oxide particles at a pressure of 60 psi and a distance of 1 cm, but they did not specify the microetching time. Moreover, hydrofluoric acid was applied for 2 min, and no silane agent was subsequently applied on composite surfaces.

Although the effect of composite surface treatments on bond strength to resin cements has been investigated in several studies, 1,7,21,25,29,33,37 there is no consensus on the results obtained through different procedures. Moreover, previous studies evaluated the three composite treatments performed in this study but did not compare their effects.

Although silane coupling agents are reported to be adhesion promoters capable of forming chemical bonds with organic and inorganic surfaces, ^{47,53} it appears from the findings presented that silane treatment did not significantly im-

prove the bond strength to dentin of hydrofluoric-acid-etched composite inlays. In contrast, application of a silane coupling agent to pretreated ceramic surfaces provides a chemical covalent hydrogen bond, and is a major factor for a sufficient resin bond to silica-based ceramics.^{2,18} Silanes are bifunctional molecules that bond silicon dioxide with OH groups on the ceramic surface. They also have a degradable functional group that copolymerizes with the resin's organic matrix.⁴²

Several studies have reported that, after silane application, bonding to the resin occurs by an additional polymerization reaction between methacrylate groups of the matrix resin and the silane molecules during composite curing.^{15,23} However, this study showed that the application of silane on hydrofluoric-acid-etched composite inlays was not able to significantly improve bond strength. The HFSiG behavior is difficult to explain. A limitation of the study is that it is not known whether the HFSiG bond strength is due to hydrofluoric acid etching or to the silanization procedure. It is possible that after etching, no filler particles are left for silane to react with. Moreover, one important property of silane is to increase the wettability of a material by making the surface hydrophobic. By not wetting the surface of the composite specimens with a low-viscosity material (eg, unfilled resin), it is not known if the composite resin cement was able to flow into the crevices created by hydrofluoric acid (HFSiG) or air abrasion (SaG). It cannot be ascertained whether, due to its viscosity, the composite cement profitted from the better wettability. On the other hand, other variables may be investigated to explain the different reports obtained in the various studies. Storage time and thermocycling treatments are also reported to be able to significantly influence bonding performance between composite to dentin when all the other conditions were taken as standard.^{12,17} Despite all the above considerations, it is possible to conclude that sandblasting treatment was the most effective treatment in this study with regard to tensile bond strength. Although SEM evaluation of the surface samples showed topographic changes such as pores after silanized surface treatments, sandblasting treatment with 50-µm Al₂O₃ produced slight surface scratches and thus the greatest surface relief with severe undercuts, which are important to create interlocking of composite resin to inlay restorations when bonded to dentin. Mean values and statistical analysis revealed a stronger bond for the sandblasted groups compared to the control and silanized treatment methods. In the sandblasted groups (SaG and SaSiG), the SEM fractographic analysis revealed more cohesive fractures within the resin cement and cohesive failures along the cement/adhesive interface, respectively categorized as failure modes (b) and (c), and less adhesive failure along the composite/cement interface (a) than those found in the other groups (CG and HFSiG).

CONCLUSION

These results confirm that sandblasting treatment was the main factor responsible in improving the mechanical retention of indirect composite restorations. Moreover, adhesive failures along the dentin surface were revealed only in some areas of all the observed tested groups. This shows that the bond strength between the adhesive system and dentin is always a reliable and predictable parameter, which did not vary in this study.

Based on the results of the present study, it can be recommended that clinicians always roughen and clean the inlay surface through sandblasting before composite inlay cementation.

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Clinical relevance: The use of a sandblasting procedure is an effective surface pretreatment for the cementation of the indirect composite restoration tested.