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Effect of surface treatments on repair strength, roughness and morphology in aged metal-free crowns

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Aim: The roughness and micromorphology of various surface treatments in aged metal-free crowns and the bond strength of these crowns repaired with composite resin (CR) was evaluated in vitro. Methods: A CR core build-up was confectioned in 60 premolars and prepared for metal-free crowns. Prepared teeth were molded with the addition of silicone, and the laboratory ceromer/ fiber-reinforced crowns (SR Adoro/Fibrex Lab) were fabricated. Subsequently, the crowns were cemented and artificially aged in a mechanical fatigue device (1.2 X 10⁶ cycles), then divided into 4 groups (n = 15) according to the surface treatment: 1) phosphoric acid etching (PA); 2) PA + silane application; 3) roughening with a diamond bur + PA; and 4) sandblasting with Al₂O₃ + PA. After the treatments, the crowns (n = 2) were qualitatively analyzed by scanning electron microscope (SEM) and surface roughness (n = 5) was analyzed before and after the surface treatment (Ra parameter). The remaining crowns (n = 8) received standard repair with an adhesive system (Tetric N-Bond) and a nanohybrid CR (Tetric N-Ceram), and the microshear bond strength (SBS) test was performed (0.5 mm/min). Roughness and SBS data were analyzed by one- and two-way ANOVA, respectively, as well as Tukey's post-test (α = 0.05). Results: Sandblasting with Al₂O₂ + PA resulted in the highest final roughness and SBS values. The lowest results were observed in the PA group, whereas the silane and diamond bur groups showed intermediate values. Conclusion: It may be concluded that indirect ceromer crowns sandblasted with aluminum oxide prior to PA etching promote increased roughness surface and bond strength values.

Keywords: Ceramics. Composite resins. Electron microscope tomography. Shear strength. Surface properties.

Introduction

Indirect restorations, also known as "ceromer," "polymeric glass porcelain," or "second-generation laboratory CR," are widely used in clinical practice because they minimize the adverse effects of direct restorations, such as polymerization shrinkage¹, poor marginal adaptation, and postoperative sensitivity² In addition, they can provide better standards of translucency and can be low-cost alternatives to all-ceramic restorations³.

Although indirect resins possess high mechanical strength, these restorations are subject to fractures as any other material. This type of failure should be carefully evaluated to define the best treatment. Clinically, the affected crowns can be classified according to the extent of the fracture. A fracture can be minimal (e.g., cracks) or extensive (e.g., displacement of more than half of the crown)^{4,5}.

Corroborating *in vitro* studies⁶⁻⁸, clinical studies show that most cases of crown fractures are repairable^{9,10}. This is advantageous because complete replacement of indirect restorations may present more disadvantages than advantages, such as the treatment complexity and expense¹¹. With the evolution of adhesive techniques, adhesive repair has been widely used and can be considered beneficial, allowing good longevity in this type of dental restoration^{12,13}.

For proper repair, the surface of the indirect restoration should be subjected to a pre-treatment to create micromechanical retention with the repair material¹⁴. In the available literature, several surface treatments techniques are described for the repair of composites. Roughening with diamond burs, sandblasting with aluminum oxide, conditioning with hydrofluoric acid etching or PA etching, and silanization are the most frequently reported^{11,15,16}.

The current literature presents several studies comparing different surface treatments; however, the best pre-treatment technique still generates controversial results¹⁷⁻¹⁹. Thus, the present study aimed to evaluate the surface roughness, morphology, and repair strength of aged indirect resin restorations with SEM, microshear bond strength test, and digital roughness meter. The tested hypothesis was that differences would exist in morphology, surface roughness, and bond strength after various surface treatments.

Material and Methods

Sixty extracted human mandibular premolars, with the protocol number 1871/10 from the research ethics committee of the State University of Ponta Grossa (Brazil), were stored in distilled water at 4°C and used within 6 months after extraction. To be included in the study sample, teeth should be sound, without cracks, and not submitted to previous endodontic treatment. Teeth were transversally sectioned 2 mm above the cement-enamel junction using a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) and received a standardized endodontic treatment.

After 1 week, the root canals were prepared to receive glass fiber posts (Whitepost DC # 0.5, FGM, Joinville, SC, Brazil), which were cemented with the Excite DSC (Ivoclar-Vivadent, Schaan, Liechtenstein) adhesive system and Variolink II (Ivoclar-Vivadent) resin cement in accordance with the manufacturer's instructions. After the post-luting procedures, cores were built-up with a nanohybrid CR (Tetric N-Ceram, Ivoclar-Vivadent). An incremental technique was used to place the CR, and each 2 mm increment was light cured for 20 s.

Indirect composite crowns cementation

The composite cores were prepared to receive a full indirect composite restoration using a high-speed hand piece under water cooling. In all roots a ferrule was made in the coronal ending with 2.0 mm height, 1.2 mm depth, and 1.5 mm occlusal reduction.

Full indirect composite restorations were fabricated with the SR Adoro (Ivoclar-Vivadent) restorative system reinforced by fibers (Fibrex-Lab Coronal, Angelus, Londrina, PR, Brazil). After fitting and adjustment, the restorations were adhesively cemented with Excite DSC and Variolink II according to the manufacturers' recommendations.

Teeth were then embedded in acrylic resin (Duralay, Reliance, Worth, IL, USA) and periodontal ligament was simulated using a polyether impression material (Impregum[™] Soft, 3M ESPE, St Paul, MN, USA), according to the method described by Soares et al. 2005².

Mechanical aging

To increase the study's reliability²⁰, the samples were subjected to mechanical fatigue in a controlled chewing simulator (Elquip, São Carlos, SP, Brazil). The samples were placed at the base of a material-fatigue-testing machine at a 90° angle in relation to the horizontal plane and were subjected to repetitive impacts directed on the occlusal surface of the crown. A lower force of 40 N (to avoid possible fractures) at a frequency of 2 Hz was applied for 1.2 X 10⁶ cycles, which represents 5 years of clinical service^{21,22}. During the cycles, the samples were kept at 37°C in relative humidity.

Surface treatments of the indirect restorations and experimental groups

The specimens were then randomly divided into 4 groups, according to the surface treatments. Each treatment was performed on a square delimited area (3 mm x 3 mm) on the buccal surface of each crown. In the PA group, the buccal surfaces of the indirect CR were treated with 35% PA for 2 min according the manufacture's recommendation, washed for 2 min with distilled water, and gently air dried for 5 s at 2 cm.

For the silane group—after PA treatment as reported above—a silane coupling agent (Prosil, FGM, Joinvile, SC, Brazil) was applied for 1 min with a disposable applicator, and the surface was dried with compressed air for 5 s at 2 cm.

The buccal surfaces of the diamond bur group were roughened with a diamond bur (# 3195, KG Sorensen, São Paulo, SP, Brazil) using a high-speed hand piece under water cooling for 5 s, with weak movements and minimal wear. Then, the surface was conditioned with PA as reported in the first group.

For the sandblasting group, the surfaces were sandblasted with 50 μ m Al₂O₃ (Microblaster Standard Model, Bio-Art, São Carlos, SP, Brazil) for 10 s and then conditioned with PA as reported in the first group.

Surface roughness test

After mechanical aging, the initial roughness (IR) of five random buccal surface restorations per group was obtained with a digital roughness meter (Mitutoyo Surftest-301, Mitutoyo-Kawasaki, Kanagawa, Japan). Three measures were performed on each specimen, and the arithmetic mean was obtained from these values. The mean represents the IR. Surface roughness reading was performed using the Ra parameter (μ m) and the ISO 2001 measuring profile²³, a 0.25 mm cut-off, 1.25 mm in length and 0.1 mm/s speed. Afterward, the specimens were submitted to the abovementioned surface treatments and stored at 37°C in artificial saliva, simulating oral condition. After 48 h of the surface treatments procedures, we measured the final roughness (FR) in the same way as the initial evaluation.

SEM analysis

Two restorations per group were prepared for the SEM (SSX – 550; Shimadzu, Tokyo, Japan). The surfaces were sputter coated with gold in a vacuum evaporator (Belzers SCD 050 SputterCoater, Bal-Tec, Germany) and photomicrographs of representative areas were taken at 1.000x magnification.

Bond strength test

After surface treatment, eight crowns per group were submitted to microshear bond strength test. For this purpose, one coat of the adhesive system (Tetric N-Bond, IvoclarVivadent) was applied on the delimited area (3 mm x 3 mm) of the treated buccal surfaces and then gently air dried for 5 s and light-cured for 10 s (Table 1).

Three Tygon tubes, approximately 0.75 mm in diameter and 1 mm high, were used for each crown. The tubes were positioned on the flattest areas of the treated buccal surface (3 mm x 3 mm) of the indirect restorations, filled with CR (Tetric N-Ceram, IvoclarVivadent), and individually photoactivated for 40 s. Each light-cured specimen was protected with aluminum strip to afford protection from additional polymerization, as well as the unpolymerized specimens. All specimens were checked with an optical microscope (OLYMPUS-BX 51, Olympus, Tokyo, Japan) at 10x magnification to discard any specimens with air bubbles or evident gaps at the interface.

Material (Manufacturer)	Composition	Instructions for use
Tetric N-Bond (Ivoclar Vivadent)	Phosphoric acid acrylate, HEMA, BisGMA, urethane dimethacrylate, ethanol, film-forming agent, catalysts and stabilizers.	Apply a thick layer of Tetric N-Bond for at least 10 seconds. Remove excess material and the solvent by a gentle stream of air and light-cure for 10 seconds.
Tetric N-Ceram (Ivoclar Vivadent)	Dimethacrylates (19-20 wt.%); barium glass, ytterbium trifluoride, mixed oxide, copolymers (80-81 wt.%); additives, catalysts, stabilizers and pigments are additional contents (< 1 wt.%). The total content of inorganic fillers is 55–57 vol.%. The particle size of inorganic fillers is between 40 nm and 3000 nm	Apply Tetric N-Ceram in layers of max. 2 mm or 1. Polymerize each layer individually for 40 seconds.

Table 1. Manufacturer, composition and instructions for each material used in the study.

All light-curing procedures of this study were performed with a LED light-curing device (Radii Plus, SDI Limited, Victoria, Australia) using a 1200 mW/cm² power density. The specimens were mounted in acrylic resin and placed in a universal testing machine (Kratos, São Paulo, SP, Brazil), and a microshear force was applied using a shearing blade as close as possible to the adhesive interface. The load was applied to the interface at a crosshead speed of 1 mm/min until failure, and the bond strength values were recorded in MPa.

Statistical analysis

Before running parametric statistical analysis, we tested whether the assumptions of normality of the data and equality of variances were valid, using the Shapiro-Wilk and Barlett tests at an alpha of 5%. The data from surface roughness and bond strength were statistically analyzed by one- and two-way ANOVA, respectively, and Tukey's test was used for pairwise comparisons at a 5% significance level.

All calculations were performed using SPSS® statistical software (Statistical Package for the Social Sciences, version 21.0 Mac, SPSS Inc., Chicago, IL, USA).

Results

The means and standard deviations of surface roughness (Ra parameter) and microshear bond strength values (MPa) for the experimental groups are demonstrated in Table 2.

In relation to the surface roughness, two-way ANOVA showed that the cross-product interaction between the factors time and experimental groups were statistically significant (p < 0.001). At baseline, all groups were statistically similar (p > 0.05). Roughness increased significantly after the treatments in all groups (p < 0.001). The final roughness was higher in the sandblasting group and lower in the PA group, whereas the silane and diamond bur groups showed intermediate values.

For the microshear bond strength, one-way ANOVA showed significant statistical differences between the experimental groups (p < 0.0001). The lowest repair strength was observed for the PA group and the highest was observed in the sandblasting group. The silane and diamond bur groups were statistically similar and had an intermediate performance.

In the SEM images (Figure 1), the diamond bur and sandblasting groups showed very irregular surfaces. However, they differed in the direction of the grooves and depres-

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Experimental Groups –	Baseline	Post treatment	 Snear bond strength
Phosphoric acid	0.24 ± 0.08 ^E	0.42 ± 0.14 ^D	9.4 ± 3.1 ^c
Silane	0.21 ± 0.07 ^E	0.64 ± 0.15 °	20.3 ± 6.1 ^в
Diamond bur	0.25 ± 0.07 ^E	0.86 ± 0.11 ^в	18.3 ± 4.9 ^в
Sandblasting	0.21 ± 0.09 ^E	1.28 ± 0.13 ^A	37.1 ± 8.7 ^A

Table 2. Mean values \pm standard deviation of roughness (Ra parameter) and microshear bond strength (MPa) for each experimental group (*).

* Comparisons are valid just for the same property. Distinct letters show significant differences (p < 0.05).



Figure 1. Images of the surface roughness obtained by the SEM (x 1,000). *A: phosphoric acid group; B: silane group; C: diamond bur group: D: sandblasting group.*

sions. Grooves are unidirectional in the diamond bur group, probably resulting from the direction of the bur roughening, whereas in the sandblasting group they do not follow a pattern due to the abrasion of aluminum oxide particles on the surface.

Discussion

Fatigue studies can mimic the effect of mechanical and thermal cycles, as well as a wet oral environment²⁴. Mechanical aging better reproduces the clinical reality, because failures and fractures in indirect CR restorations occur only after years of clinical service¹³. Although previous studies have already investigated different surface treatment techniques for the repair of indirect restorations²⁵⁻²⁷, most did not sim-

ulate three important clinical features found in this study's protocol: cementation of a fiber post to stabilize the final restoration, simulation of the periodontal ligament, and simulation of mechanical aging.

In a clinical scenario, the core and post are placed to retain the final restoration in endodontically treated teeth, improving their integrity²⁸. The presence of the periodontal ligament and tooth-supporting structures partially absorb the masticatory loads; therefore, studies that did not simulate these structures may have obtained unreliable values²¹. Finally, post-retained indirect crowns are subject to repetitive ordinary chewing forces over time, as well as other environmental challenging factors²⁹. Thus, mechanical aging is essential to simulate closely the clinical conditions to which these indirect restorations are subjected. In this study, all specimens were submitted to 1.2 X 10⁶ cycles of mechanical fatigue, which is commonly assumed to correspond to 5 years of clinical service²².

In this study, the results showed that air abrasion with aluminum oxide promoted the highest bond strength values^{30,31} and higher roughness. Although some authors have reported that pre-treatment with diamond burs can yield higher repair strengths than air abrasion with aluminum oxide^{32,33}, other studies^{30,34,35} have shown the opposite, with results similar to our observations.

The higher surface roughness produced by aluminum air abrasion increases the surface area and wetting for the adhesive penetration^{36,37}, which may have yielded the highest bond strength values. This positive correlation between increased surface roughness and improved repair strength has already been demonstrated in other studies^{38 39}.

Indeed, the bond repair strength observed in the PA etching group might be lowest because this procedure produced the lowest surface roughening on the aged resin surface. Previous studies have demonstrated that acid etching alone is not enough to guarantee adequate repair strength⁴⁰.

Although surface treatment with silane does not generate the roughest surfaces, this group presented intermediate bond strength values, similar to that achieved by the asperization with diamond bur. The chemical bond produced by this bifunctional molecule between the inorganic particles of glassy substrates (silica filler particles) and the adhesive CR matrix¹⁹ probably compensated for the reduced surface area. This bonding agent has a general chemical structure, $R'Si(OR)_3$, where R' is the organ functional group (typically a methacrylate) that reacts to the adhesive system or the composite cement, creating a covalent bond after polymerization. The alkyl group (R) is hydrolyzed to a silanol (SiOH), creating a covalent bond with the inorganic silicon particles⁴¹.

This study has some limitations, due to which not all clinical conditions could be reproduced. In addition, only a resin cement was used, the chewing forces of an occlusion were not applied in the mechanical aging, and the treatments were performed on a flat surface rather than a cusp area. In summary, the results of the present study demonstrated that aged indirect CRs should be pre-treated with aluminum oxide + PA prior to repair to increase the surface roughness and consequently the bond strength repair. Thus, the tested hypothesis was accepted.

Sandblasting aged indirect resin restorations with aluminum oxide prior to PA etching increases the surface roughness and repair bond strength values.

Conclusions

Sandblasting aged indirect resin restorations with aluminum oxide prior to PA etching increases the surface roughness and repair bond strength values.

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