Original Research
Subseção

# Bond strength between a polymer-infiltrated ceramic network and a composite for repair: effect of several ceramic surface treatments

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**Abstract:** The effects of several ceramic surface treatments on bond strength of a polymer-infiltrated ceramic network and resin composite as repair material were evaluated. CAD-CAM blocks of a polymer-infiltrated ceramic network (Vita Enamic) were sliced and subjected to aging process, followed by embedding in acrylic resin.The bonding/repair area was treated as follows (n = 30): C– without treatment; UA– universal adhesive application; FM– 10% hydrofluoric acid and silane application; OM– airborne-particle abrasion with aluminum oxide and silane application; RP– tribochemical silica coating; and CA– surface grinding and application of universal adhesive. Composite resin cylinders were made on the treated surface. Specimens from each group were assigned randomly to two subgroups (n = 15) considering storage condition: Baseline (shear tests after 48 hours) or Storage (tests after 6 months under distilled water). The treated surfaces were analyzed by goniometry, roughness, and SEM. Two-way ANOVA and 1-way ANOVA were applied to analyze the bond data and roughness / contact angle data, respectively, followed by Tukey's test ($\alpha$ = 5%). Surface treatments and storage conditions affected bond strengths ($p < 0.01$). Surface grinding (CA) followed by universal adhesive promoted the highest value of bond strength (14.5 ± 4.8 MPa for baseline, 8.5 ± 3.4 MPa for storage) and the roughest ceramic surface. Grinding with silicon carbide paper (simulating diamond bur) followed by the application of a universal adhesive system is the best option for repairing fractures of the polymer-infiltrated ceramic network.

**Keywords:** Dental Ceramics; Adhesion; Conditioning; Ageing; Fracture.

## Introduction

Metal-free restorations in modern dentistry include new categories of ceramic materials, such as the hybrid ceramics for CAD-CAM technology.[1,2] Polymer-infiltrated ceramic network (also known as hybrid ceramics) combine the properties of composites and ceramics. One of the advantages is the decrease in the propagation of cracks, which change directions and deflect the polymeric material.[3]

Vita Enamic (Vita Zahnfabrik, Bad Säckingen, Germany) is a hybrid ceramic, composed of 86% of an inorganic matrix of feldspathic ceramic

and 14% of a polymer, which interpenetrates the ceramic network.[2,4] This ceramic was developed with the purpose of mimicking the dental structures; it presents mechanical properties similar to teeth, such as the elastic modulus and the Vickers hardness, thus causing less wear of the occluding teeth when compared to other ceramic categories.[3,5,6]

Although the combination of the crystalline matrix and the polymeric material results in a decrease in crack propagation, this ceramic still presents a low elastic modulus and inert biaxial flexural strength when compared to other ceramic systems,[2] thus clinical fractures might occur. Ceramic fractures are related to several factors including: dental conditions, such as secondary caries; parafunctional habits; trauma; internal defects of the material, such as micro-porosities; defects induced by occlusal adjustment; and stress concentrations.[7,8] The repair of fractures with composite resin[9] is sometimes performed, however, the urgency for a new restoration and size and location of the fracture[8] should be taken into account. Repairing procedures have the advantage of saving time and resources,[10] reducing microbial adhesion to the fracture and, consequently, preserving the dental remnant.[7]

*In vitro* studies suggest some surface treatments to improve the bond strength between ceramics and composite resin repairs, including surface grinding using diamond bur and silane application,[11] surface etching with 40% phosphoric acid[12] associated with the application of an adhesive system,[13] surface etching with 9.5% hydrofluoric acid followed by the silanization and application of adhesive, air-abrasion with alumina particles or tribochemical silica coating,[14] and the use of a universal adhesive.[13,15]

However, reports in the literature on the best approach and longevity of repairs in hybrid ceramics are scarce; therefore, the adhesion of composite resin repairs to the polymer-infiltrated ceramic network substrate remains to be determined. The objective of the present study was to evaluate the effect of several surface treatments of a hybrid ceramic on bond strength to a composite resin. The surface roughness and contact angle measurements were performed as well. The null hypotheses of this study were: 1) surface treatments will not influence bond strength; 2) storage time will not reduce bond strength.

## Methodology

### Microshear test

CAD-CAM blocks made of a polymer-infiltrated ceramic network (Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany) were cut with a diamond disk (IsoMet 1000, Buehler, Lake Bluff, USA) under cooling, and polished using # 400, 600, 800 grit silicon carbide papers (EcoMet/AutoMet 250, Buehler). Subsequently, the samples were submitted to thermal-cycling (6000 cycles: 5–55°C; cyclic tester # 521-6D, Ethik Technology, Vargem Grande Paulista, Brazil). The aged samples were embedded in polyvinyl chloride (PVC) with chemically activated acrylic resin (TDV, Pomerode, Brazil), leaving the bonding/repairing site exposed; samples were randomly allocated into 6 experimental groups (n = 30), according to the surface treatment:

**C-** (Negative Control) – No treatment. The adhesive area was delimited (Ø = 3 mm) with adhesive tape (Scotch® Super 33+ Vinyl Electrical Tap, Scotch, 3M, Campinas, Brazil).

**UA-** (Single Bond Universal) – Delimitation of bonding area; active application of universal adhesive (Single Bond Universal, 3M ESPE, Seefeld, Germany), for 10 seconds; application of oil-free air-drying for 5 seconds; photo-activation with LED light for 10 seconds (1200 mW / cm²; Radii-cal, SDI, Victoria, Australia).

**FM-** (Positive Control - etching with hydrofluoric acid 10% + Monobond Plus) – Delimitation of the adhesive area; etching with 10% hydrofluoric acid (Condacporcelana, FGM, Joinvile, Brazil) for 60 seconds; washing with air-water spray for 60 seconds; air-drying for 5 seconds; active application of silane (Monobond Plus, Ivoclar-Vivadent, Schaan, Liechtenstein) on the ceramic surface for 60 seconds, waiting 60 seconds for evaporation of the silane solvent followed by a light jet of air for 5 seconds.

**OM-** (Aluminum oxide + Monobond Plus) – Airborne abrasion with 45 μm aluminum oxide particles (Wilson, Polidental, Cotia, Brazil) as follows: 10 mm distance from the tip of the blasting device to the sample, 2.5 bar pressure, 90° angulation, for 10 seconds; cleaning of the specimens in ultrasonic bath in distilled water for 5 min; delimitation of the

adhesive area; application of the silane (Monobond Plus) as described for FM group;

**RP-** (Tribochemical silica coating) – Air abrasion with 30 μm silica-coated aluminum oxide particles (Rocatec Soft, 3M ESPE) using the same parameters for OM group; cleaning of the samples in a ultrasonic bath for 5 minutes in isopropyl alcohol; delimitation of the adhesive area; active application of the silane agent (RelyX ceramic primer, 3M ESPE) for 20 seconds, waiting for 5 minutes for volatilization of the ethanol;

**CA-** (Grinding + adhesive) – grinding of the surface with a # 180 μm silicon carbide paper (Norton Saint-Gobain, São Paulo, Brazil) for 20 seconds, simulating the grinding with a coarse diamond bur (181 μm grit)[16,17]; cleaning of the samples in ultrasonic bath for 5 minutes in distilled water; delimitation of the bonding area; active application of the Single Bond Universal adhesive system (3M ESPE) for 10 seconds, followed by a light air jet for 5 seconds, and photo-activation (1200 mW / cm²) for 10 seconds (Radii-cal, SDI, Victoria, Australia).

For the C, UA, and FM groups, the bonding area was delimited (Ø = 3 mm) before the adhesive procedures, using adhesive tape (Scotch® Super 33 + ™ Vinyl Electrical Tap, Scotch, Campinas, Brazil), while for the OM, RP, and CA groups, delimitation was performed after surface treatment.

After surface treatment, composite resin cylinders were made (Filtek Z 350, 3M ESPE). For this, Tygons® tubes (Ø = 3 mm, height = 3 mm) were fixed on the bonding area and filled with 2 increments of composite resin of 1.5 mm thickness. Each increment was photo-activated for 20 seconds (Radii-cal, SDI, Victoria, Australia).

Samples from each group were randomly assigned into 2 subgroups (n = 15), considering the storage conditions before shear tests: *Baseline* – samples were stored in distilled water at 37°C in an incubator (Fanem, São Paulo, Brazil) for 48h; *Storage* – samples were stored in distilled water at 37°C in a bacteriological stove for 6 months and the distilled water was renewed once of week (Estufa 520, Fanem, São Paulo, Brazil).

For bond shear tests, every sample was coupled with a device in a universal testing machine (DL1000, EMIC, São José dos Pinhais, Brasil). The load (crosshead speed of 1 mm/min) was applied as close as possible to the interface through a wire with 0.25 mm diameter, until failure occurred. Bond strength (MPa) was calculated by dividing the maximum load (N) by the bonding surface area of the resin cement ($mm^2$). The bonding area (A in $mm^2$) was the same for all samples and was calculated by the equation: $A = \pi \times (r)^2$, where $\pi = 3.14$ and $r = 1.5$ mm; *i.e.*, $A = 3.14 \times (1.5)^2 = 7.06$ $mm^2$.

The tested samples were first analyzed by stereomicroscope under 12× magnification (Discovery V20, Carl-Zeiss, Gottingen, Germany) and representative failures were observed under Scanning Electron Microscope (Inspect S 50 - FEI Company, Brno, Czech Republic). Additional topographic analyses were performed with high vacuum at 15–25 kV, 5.0 spot and magnification of 1000× and 3000×.

Failures were classified as adhesive and cohesive as follows: adhesive at ceramic and composite resin interface; cohesive, in composite resin only; cohesive, in ceramic only; predominantly adhesive when more than 60% of the failure occurred at the adhesion zone.

## Contact angle measurements

Three samples of each group were randomly selected after surface treatment; in each sample, 5 readings were performed. The contact angle was measured by means of an optical tensiometer (TL 1000 - Theta Lite, Attension, Lichfield, Staffordshire, UK) by the sessile drop technique, wherein one drop of distilled water is deposited on the surface of the sample using a syringe (Gastight Syringes # 1001-1 mL, Hamilton, Reno, USA). After 10 seconds, the drop spreads and a series of 30 images per second is recorded by a camera for 20 seconds. The "OneAttension" software (BiolinScientific, Lichfield and Staffordshire, UK) calculates the mean contact angle values for each sample.

## Surface topography

The conditioned samples were analyzed in terms of roughness analysis (Ra), in a digital optical profilometer (Wyko NT 1100, Veeco, Plainview, NY, USA), linked to the Wyko Vision 32 (Wyko, Veeco). Five readings were performed in each sample, totaling 15 readings per experimental group.

### Data analysis

Two-way ANOVA and Tukey's tests ($\alpha = 5\%$) were used to analyze the bond strength data, while 1-way ANOVA and Tukey's tests were applied for wettability and surface roughness data. The statistical tests were performed using Statistix 8.0" software.

## Results

### Bond strength

The control group was withdrawn from the statistical analysis because their bond values were close to zero.

Two-way ANOVA showed that both factors, surface treatment ($p = 0.01$) and storage ($p = 0.01$), influenced the results of bond strength.

The CA group, for both baseline and storage conditions, had the highest bond strength (Table 1). The bond strength values of the groups had a significant reduction after storage (Table 1). The main type of failure was adhesive at the hybrid ceramic and composite resin interface (Figure 1 and Table 2).

### Contact angle

One-way ANOVA demonstrated that surface treatments influenced the results of contact angle

**Table 1.** Mean values (in MPa) and standard deviations (SD) of shear bond strength data from Tukey's test.

| Groups | Storage time (Mean ± SD0 | |
|---|---|---|
| | 48 hours | 6 months |
| CA | 14.53 ± 4.79$^{A}$ | 8.49 ± 3.43$^{BC}$ |
| UA | 9.89 ± 3.45$^{B}$ | 1.83 ± 1.27$^{DE}$ |
| FM (+ control) | 9.96 ± 3.50$^{B}$ | 1.73 ± 2.45$^{E}$ |
| OM | 4.98 ± 4.14$^{CDE}$ | 5.61 ± 3.00$^{CD}$ |
| RP | 5.53 ± 2.97$^{CDE}$ | 3.98 ± 2.84$^{DE}$ |

Different capital letters indicate statistically significant differences between groups.

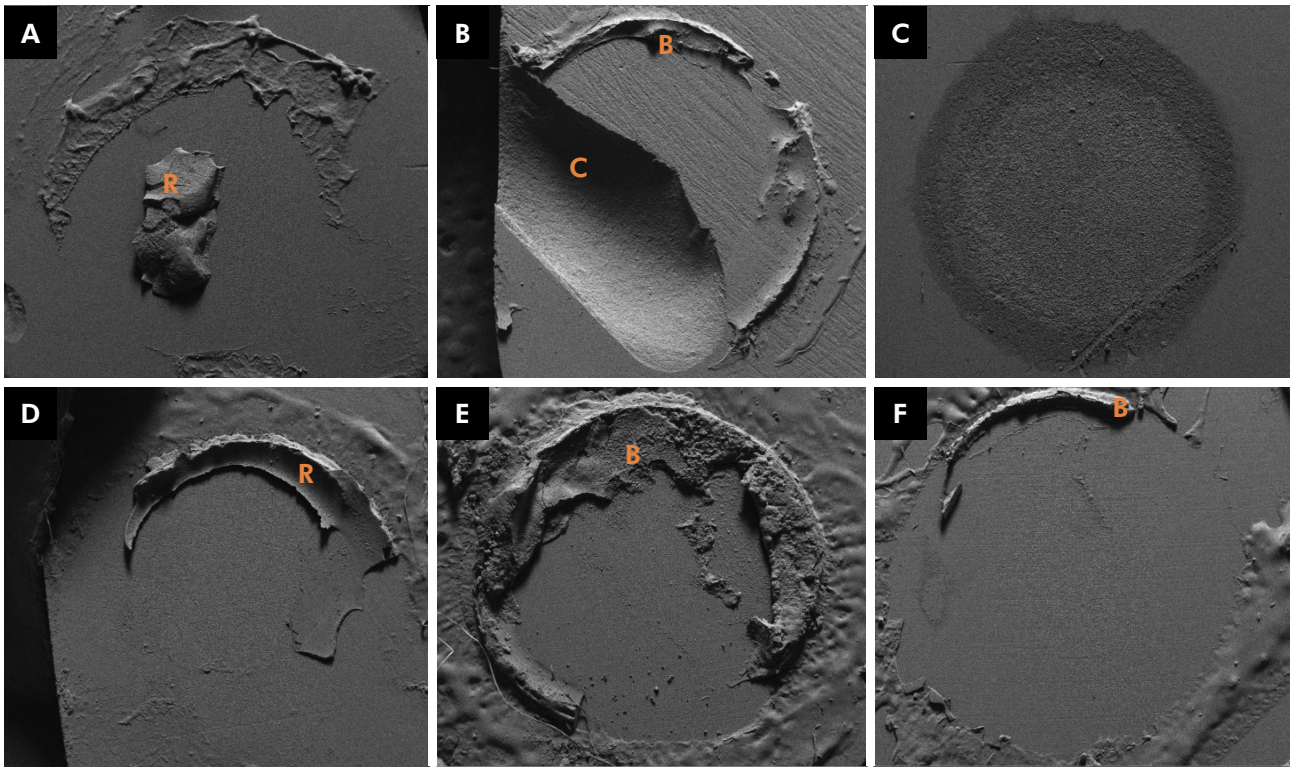


**Figure 1.** Micrographs of failures mode (70x). A) Cohesive Resin; B) Cohesive Ceramic; C) Adhesive; D) Mixed with Resin remaining; E) Cohesive Bonding Agent; F) Mixed with Bonding Agent remaining. (R), (C) and (B) are the resin, ceramic and bonding agent remaining, respectively.

**Table 2.** Failure modes of each surface treatment after 48 hours and 6 months of storage.

| Groups | 48 hours | | | | 6 months | | | |
|---|---|---|---|---|---|---|---|---|
| | Adhesive | Pred. adhesive | Cohesive resin | Cohesive ceramic | Adhesive | Pred. adhesive | Cohesive resin | Cohesive ceramic |
| C (- control) | 15 | 0 | 0 | 0 | 15 | 0 | 0 | 0 |
| OM | 15 | 0 | 0 | 0 | 15 | 0 | 0 | 0 |
| RP | 15 | 0 | 0 | 0 | 15 | 0 | 0 | 0 |
| UA | 11 | 4 | 0 | 0 | 6 | 9 | 0 | 0 |
| CA | 5 | 1 | 0 | 9 | 13 | 1 | 1 | 0 |
| FM (+ control) | 13 | 2 | 0 | 0 | 14 | 1 | 0 | 0 |

**Table 3.** Mean values (in °) and standard deviations (SD) of contact angle data from Tukey's test.

| Ceramic surface treatments | Mean ± SD |
|---|---|
| No treatment | 75.18 ± 17.06$^{A}$ |
| Oxide aluminum | 56.90 ± 16.42$^{B}$ |
| Rocatec | 54.65 ± 4.40$^{BC}$ |
| Single Bond Universal | 48.47 ± 6.20$^{BC}$ |
| Carbide | 43.07 ± 4.27$^{CD}$ |
| HF | 32.13 ± 7.99$^{DE}$ |
| Carbide + Single Bond Universal | 35.58 ± 13.69$^{DE}$ |
| Rocatec + RelyX Ceramic Primer | 26.88 ± 10.36$^{E}$ |
| HF + Monobond Plus | 2.27 ± 4.44$^{F}$ |
| Oxide aluminum + Monobond Plus | 0.21 ± 0.82$^{F}$ |

*Different capital letters indicate statistically significant differences.

**Table 4.** Mean values ($\mu$m) and standard deviations (SD) of roughness data from Tukey's test.

| Ceramic surface treatments | Mean ± SD |
|---|---|
| No treatment | 0.56 ± 0.05$^{A}$ |
| Carbide | 1.82 ± 0.23$^{B}$ |
| HF | 1.50 ± 0.13$^{C}$ |
| Oxide aluminum | 1.02 ± 0.15$^{D}$ |
| Rocatec | 0.97 ± 0.08$^{D}$ |

*Different capital letters indicate statistically significant differences.

($p < 0.0001$). Surface treatments reduced contact angle values when compared to the control group. When the silane or adhesive were applied, the contact angle were reduced more drastically (Table 3).

### Roughness and surface topography

One-way ANOVA showed that surface treatments influenced roughness results ($p < 0.0001$). Grinding with silicon carbide paper promoted the highest roughness values (Ra) (Table 4).

Surface topography analysis showed that hydrofluoric acid etching removed glass matrix particles from the ceramic creating surface retentions, whereas the treatment with carbide grinding promoted micro-retentions, maintaining the glass matrix and the polymer (Figure 2).

## Discussion

This study showed that grinding with silicon carbide paper (simulating grinding with coarse diamond bur)[16,17] followed by the application of an universal adhesive system as pre-treatment for repairing a hybrid ceramic with composite resin promoted the highest bond strength values compared to the other tested treatments. Thus, the null hypotheses were not accepted. The ceramic-resin bond improvement by grinding had been already reported by Elsaka,[11], Duzyol et al.[18] and Güngör et al.,[19] however, to the authors' knowledge, that repair method had not been reported for hybrid ceramic yet – our findings corroborate the effect of grinding.

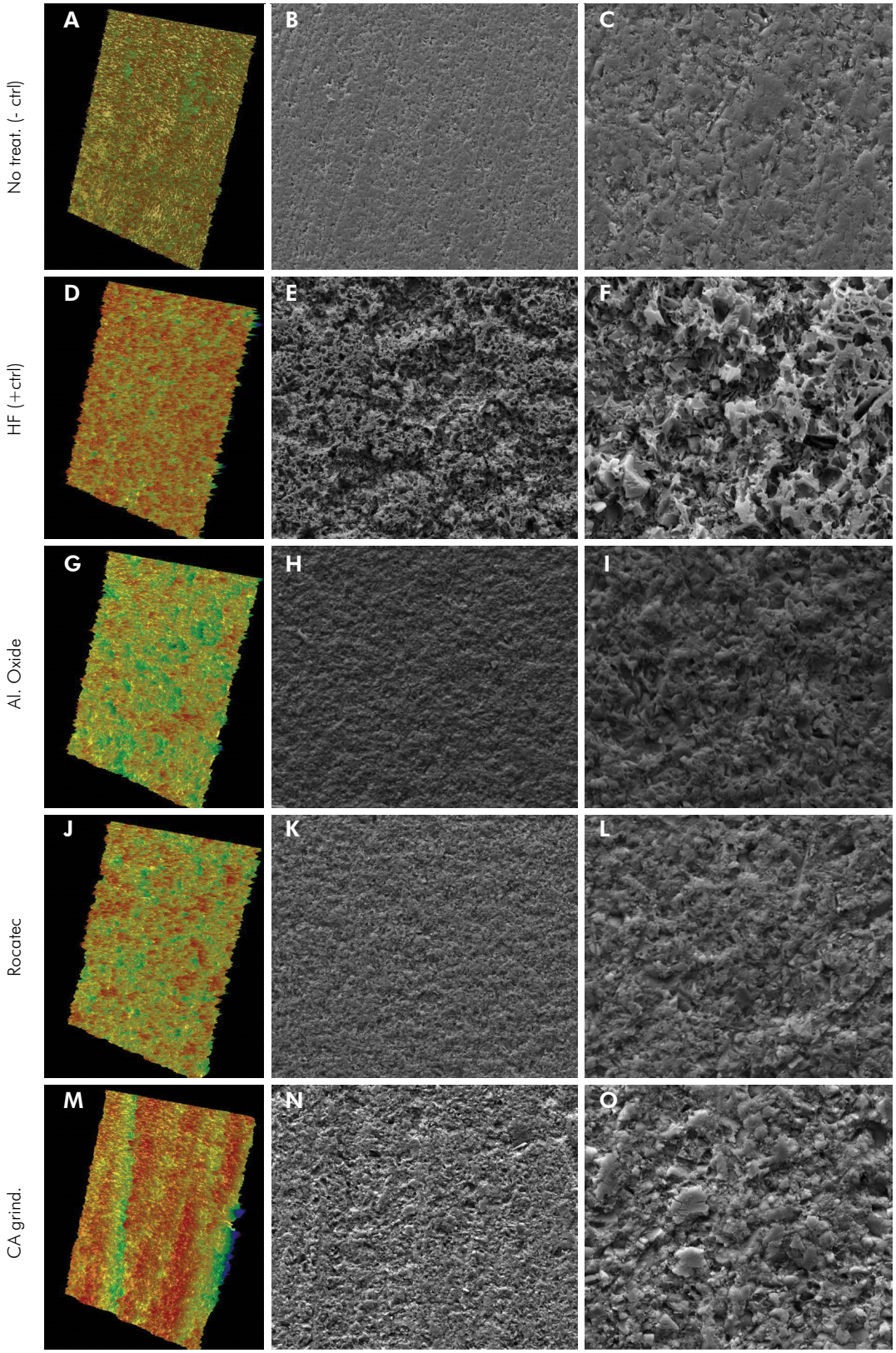


**Figure 2.** 3D profilometry images (A, D, G, J, M) and micrographs 1000x (B, E, H , K N), 3000x (C, F, I L, O) of the hybrid ceramic after surface treatments. The carbide ground specimen looks more irregular than others groups. The HF treated specimen shows another irregular standard surface which is related to a different type of ceramic (glass matrix) removal .

Grinding and application of universal adhesive before repairing has the following advantages: accessibility and ease of execution,[18,19] creation of retentions in the ceramic surface (increase in roughness), and the interpenetration of the adhesive in these retentions to form a siloxane bond between the fillers and the polymer matrix[20]. According to the literature[21] and our findings (Table 3 and Figure 1), grinding promotes the highest values of roughness, favoring the interpenetration of the bonding agent in the micro-mechanical retentions,[22] consequently increasing bond strength.

The use of a MDP-based adhesive system (Single Bond Universal, 3M ESPE) contributed to the higher values of bond strength between ceramics and the repair resins,[23] due to the phosphoric acid groups (MDP) of the polymer infiltrating in the ceramic network.[24] Even though the CA group (grinding and application of adhesive) showed a decrease in bond strength after storage, the other groups had either very low bond values or an extreme bond reduction due to hydrolytic degradation.[25] Consequently, the CA group had the highest bond strength after storage for 6 months.

Thus, mechanical treatment of the surface without the use of a bonding agent does not appear to improve the wettability of the studied materials, suggesting that the use of silane or adhesive agents could improve bond strength. Our results showed that the adhesive agent led to a decrease in the contact angle when compared to mechanical surface treatments without adhesive application;[26] the decrease of the surface tension led to a better adhesion between the substrates. In this study, it was observed that the hydrofluoric acid etching + silane presented the lowest value of contact angle (greater wettability), owing to the micro-retentions thus increase of surface energy.[11] The analysis also showed that silane effectively infiltrated the micro-retentions, but its cohesive strength was weakened after a certain time in water, leading to a decrease in bond strength after six months.

The silane coupling agent contains a bifunctional molecule, which can bind covalently to silicon dioxide and copolymerize with the organic matrix of hybrid ceramic.[27] For Schwenter et al. (2017), when a silane coupling agent was used on the etched and polymer-infiltrated ceramic, higher values of shear bond strength could be observed.[24] The authors explain the increase in shear bond strength by the additional chemical linkage of the silane coupling agents to the silicate ceramic phase of the polymer-infiltrated ceramic.[24,27] Interestingly, both the universal adhesive and the silane contain MDP, a well-known component for ceramicbonding[21]. However, the decrease in bond strength observed in the present study confirms the tendency for hydrolysis. Similarly, Schwenter et al.[24] affirmed that the hydrofluoric acid etching + silane had an extreme reduction of the bond strength (Table 1), probably due to insufficient topographic changes.

HF presents harmful characteristics such as the potential for systemic intoxication, can cause eye lesions, and can irritate soft tissues.[28,29]. Moreover, HF removes the glass matrix, conserving only the polymer component,[11,21] as observed in Figure 2. On the other hand, the other treatments only create a rough surface, maintaining both the glass matrix and the polymer. Thus, the polymer alone at the interfaces could lead to weaker bond strengths. Probably, this is the main difference between the acid-etched hybrid material and conventional feldspar-based ceramic, as the latter presents only topographical changes and no second phase materials that could affect bond properties. [21]

No surface treatment of the hybrid material investigated by us promoted bond strength stability (baseline vs storage), denoting that more studies should be conducted. Our study indicated that the storage regimen used could have induced a strong hydrolytic degradation[25] of the interfaces and materials due to the small bond area. Thus, the findings should be considered with caution.

## Conclusions

Surface grinding followed by universal adhesive application promoted the highest adhesion, and it appears to be the best method for repairing hybrid ceramics. However, the tested surface treatments did not lead to bond stability after 6 months in water, demonstrating that new conditioning methods for hybrid materials should be investigated.

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