Effect of Different Surface Treatments on the Bond Strength of the Hybrid Ceramic Characterization Layer

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Purpose: Using the microshear bond strength (µSBS) test, this study investigated the bond strength between a hybrid ceramic and the extrinsic characterization layer after different ceramic surface treatments.

Materials and Methods: Hybrid ceramic blocks (Vita Enamic, Vita Zahnfabrik) were sectioned and randomly divided into 4 groups (N = 120) according to the surface treatment and aging (n = 15): P: polishing; E: acid etching with HF; A: aluminum oxide blasting; S: self-etching ceramic primer. The specimens were silanized, then cylinders of light-curing characterization material (Vita Enamic Stain, 1.6 mm diameter x 2 mm height) were fabricated, followed by glazing. The specimens were subsequently immersed in distilled water for 24 h and subjected to the µSBS test using a universal testing machine (load cell 0.5 mm/min, 50 kgf) or tested after thermocycling for 10,000 cycles in water (5°C–55°C). After treatment, the specimen surfaces were analyzed using SEM, with failure types defined as adhesive, predominantly adhesive, or cohesive. The data were analyzed by two-way ANOVA followed by Tukey's test ($p < 0.05$).

Results: The most frequent failure type was predominantly adhesive between ceramic and the characterization layer. There were statistically significant differences between the surface treatments ($p < 0.05$). Thermocycling did not lead to statistically signifcant different results ($p > 0.05$). For groups P and A, a sharp decrease in SBS was observed.

Conclusion: The absence of surface treatment drastically reduced the microshear bond strength between the ceramic and the characterization layer. Conditioning with 5% hydrofluoric acid for 60 s is the most suitable treatment for adhesion of the characterization layer to hybrid ceramic.

Key words: bond strength, ceramic, hybrid ceramic

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CAD/CAM materials with compositions similar to those of composite resin are becoming ever more popular due to shorter milling times and a reduced number of clinical sessions.[1,10] Of these materials, polymer-infiltrated ceramic, also known as hybrid ceramic, is being increasing used in oral rehabilitation due to its low elastic modulus (30 GPa) and Vickers hardness compared to conventional ceramic materials, which reduces the spread of cracks and fissures.[1,5,16]

Hybrid ceramics are clinically indicated for posterior teeth and ultraconservative preparations, and are available as monochromatic and polychromatic blocks for better clinical results. Although these materials are available in a wide range of colors and translucencies, it is often necessary to apply pigmentation and glaze before their application for oral rehabilitation. Whereas traditional ceramics require a firing cycle to crystallize the characterization layer (stain and glaze), hybrid ceramic does not need a firing step for this, due to its content of composite resin. The characterization layer is thus polymeric and photopolymerizable.[7]

Table 1 Brand name, manufacturer, and chemical composition of materials used in this study

Material	Brand	Manufacturer	Composition	Batch
Hybrid ceramic	Vita Enamic	Vita Zahnfabrik	SiO_2, Al_2O_3, NA_2O, K_2O, B_2O_3, ZrO_2, CaO	52460
Etching agent	Vita Adiva cera-etch	Vita Zahnfabrik	Hydrofluoridric acid 5%	77150
Ceramic primer	Vita Adiva c-prime	Vita Zahnfabrik	Solution of methacrylsilanes in ethanol	E51802797
Self-etching ceramic primer	Monobond Etch & Prime	Ivoclar Vivadent	Butanol, tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, bis(triethoxysilyl) ethane, silane methacrylate, colorant, ethanol, water	x11842
Stain	Vita enamic stain	Vita Zahnfabrik	Cristobalite, dibenzoyl peroxide, dicyclohexyl phthalate	32300
Stain liquid	Vita enamic stain liquid	Vita Zahnfabrik	Methyl methacrylate, aromatic urethanacrylate	74180
Light-cured glaze	Vita enamic glaze	Vita Zahnfabrik	Methyl methacrylate, 2-propenoic acid, reaction product with pentaerythrite, diphenyl (2,4,6-trimethylbenzoyl) phosphinoxide	73680
Al_2O_3 (air abrasion)	Aluminum oxide	Bio Art	50-µm Al_2O_3	41719

Hybrid ceramic restorations can be repaired with resin composite in the oral environment if limited delamination and fractures have occurred, saving time and facilitating handling as well as application.[15] Additionally, a surface treatment is necessary for the adhesion of the resinous material to the dental substrate, to guarantee esthetics, durability, and union.[8] Surface treatments such as conditioning with 5% hydrofluoric acid, sandblasting with aluminum oxide, and the use of silane combined with the application of a characterization layer can favor wear resistance, and can also influence in the longevity of the restoration.[7,17]

However, the literature does not contain pertinent studies on the bond strength of hybrid ceramic to the characterization layer, as determined by bond strength tests. Therefore, this study aimed to evaluate the microshear bond strength (µSBS) between the hybrid ceramic and its characterization layer, taking into account the different surface treatments performed on the hybrid ceramic surface. Two null hypotheses were posed: 1. surface treatments will not influence bond strength and 2. aging will not reduce the bond strength.

MATERIALS AND METHODS

Specimen Preparation

The materials, manufacturers, and compositions are shown in Table 1. Hybrid ceramic blocks (Vita Enamic, Vita Zahnfabrik; Bad Säckingen, Germany) were cut with a diamond disk (IsoMet 1000, Buehler; Lake Bluff, IL, USA), and all specimens were polished with sandpaper of increasingly fine grit (#400, 600 and 1200) under constant water cooling in a polishing machine (EcoMet/AutoMet 250, Buehler). Forty hybrid ceramic blocks with dimensions of 10 mm x 8 mm x 2 mm were embedded in self-curing acrylic resin (JET, Dental Articles Classic; Curitiba, Brazil). A sample power of 90% was calculated by the Minitab Software (Power Curve for General Full Fractorial; State College, PA, USA) considering a 95% two-tailed confidence interval, for a minimum of 15 samples per group. After that, the samples were randomly divided into 8 groups according to surface treatment and aging (n = 15) (Table 2). According to the manufacturers, the recommended surface treatments are acid etching or sandblasting. However, previous studies had also described polishing and the application of self-etching ceramic primer as alternative surface treatments for hybrid ceramics.[15,17]

In group P, the hybrid ceramic surface was polished with sandpaper. In Group E, the ceramic surfaces were etched with 5% hydrofluoric acid (Vita Adiva cera-etch, Vita Zahnfabrik) for 60 s followed by an ultrasonic bath in distilled water for 10 min. In group A, the specimens were air abraded with 50-µm aluminum oxide (Al_2O_3) at 1 bar pressure for 10 s, followed by an ultrasonic bath for 10 min. In group S, self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent; Schaan, Liechtenstein) was applied on the specimen surface for 20 s using a microbrush, followed by an ultrasonic bath for 5 min, and finally the stain and glaze layers were applied.

Specimens from groups P, E, and A were silanized with silane (Vita Adiva C-Prime, Vita Zahnfabrik) using a microbrush, and after 30 min the characterization layer was applied. A black stain was applied to specimens at a 1:1 powder: liquid ratio, followed by photoactivation for 6 s, after which the glaze was applied and photoactivated for 30 s by light curing (Valo LED, Ultradent; South Jordan, UT, USA) with Xtrapower configuration (3200 mW/cm^2) following the manufacturer's instructions.

Table 2 Characteristics of the study groups according to surface treatment and finishing

Groups	Surface treatments	Finishing
P	Polishing and silanization with Vita Adiva C-Prime	Stain + Glaze
E	5% HF etching with Vita Adiva cera-etch (E) for 60 s, cleaning (ultrasonic bath with distilled water for 10 min) and silanization with Vita Adiva C-Prime	Stain + Glaze
A	Sandblasting with 50-μm aluminum oxide (Al_2O_3) at 1 bar, cleaning (ultrasonic bath with distilled water for 10 min) and silanization with Vita Adiva C-Prime	Stain + Glaze
S	Self-etching ceramic primer with Monobond Etch & Prime (S) and cleaning (ultrasonic bath with distilled water for 5 min).	Stain + Glaze

Table 3 Two-way ANOVA for SBS data

	df	SS	Ms	F	p-value
Surface treatment	3	1501.21	500.404	65.58	**0.000**
Thermocycling	1	8.89	8.894	1.17	0.283
Surface treatment vs thermocycling	3	2.39	0.797	0.10	0.957
Error	112	854.61	7.630		
Total	119	2367.11			

p-value in bold indicates significant difference in SBS ($p < 0.05$). df: degrees of freedom; SS: sum of squares; Ms: mean square.

Staining and Glazing (Characterization)

Tygon tubes (Ø = 1.6 mm, height = 2 mm) were fixed over the treated ceramic surface for the fabrication of cylinders. Then, a thin layer of stain (Vita Enamic Stain liquid; Vita Zahnfabrik) and glaze (Vita Enamic Glaze, Vita Zahnfabrik) were applied to the ceramic surfaces and photoactivated.

Thermocycling (Simulated Aging)

The groups were subdivided into “immediate” and “aged”. The immediate specimens were stored in an oven (Orion culture oven 502, Fanem; São Paulo, Brazil) at 37°C for 24 h and subjected to the μSBS test. The aged specimens were submitted to thermocycling[2,11] (10,000 cycles) in a thermocycler (Biopdi thermocycler; São Paulo, Brazil), with temperatures of 5 (±1)°C and 55 (±1)°C, with 30 s of immersion in each bath and 2 s of water drainage. The literature reports thermocycling as the worst aging scenario, with 10,000 cycles representing 1 year in vivo.[12] After thermocycling, the specimens were submitted to the μSBS test.

Shear Bond Strength (SBS) Test

The samples were submitted to the SBS test with a load cell (50 KgF, 0.5 mm/min) in a universal testing machine (DL-1000, EMIC; São José dos Pinhais, Brazil). The load was applied to the base of the cylinder by an orthodontic wire (Ø = 0.2 mm) coupled to the universal testing machine. The wire was perpendicular to the surface of the characterization layer/ceramic interface.

The bond strengths were obtained using the formula: $S = F/A$, where S = adhesive strength (MPa); F = force (N); A = interfacial area (mm). With a cylinder radius of 0.8 mm, the resulting cross-sectional bonding area was 2 mm^2.

In the present study, a smaller adhesive area was used to decrease the incidence of defects incorporated at the moment of staining application, generating more reliable values of bond strength. One of the advantages of the μSBS test over the microtensile bond strength test is that it is not necessary to cut the sample after characterization, thus reducing residual stresses and technical artifacts.[9]

Failure and Data Analyses

After the SBS test, a stereomicroscope (Stereo Discovery V20, Zeiss; Göttingen, Germany) was used to analyze the fracture surface. Then, micrographs of the different failure types were taken with a scanning electron microscope (SEM; Vega3, Tescan; Bro, Czech Republic) at 15 kV and magnifications of 130X (Figs 1a to 1c) and 100X (Fig 1d). Failure modes were classified as: predominantly adhesive, in which more than 60% of the failure occurred in the adhesion zone; adhesive between ceramic and characterization-layer interface; cohesive in ceramic; cohesive in the characterization layer.[15]

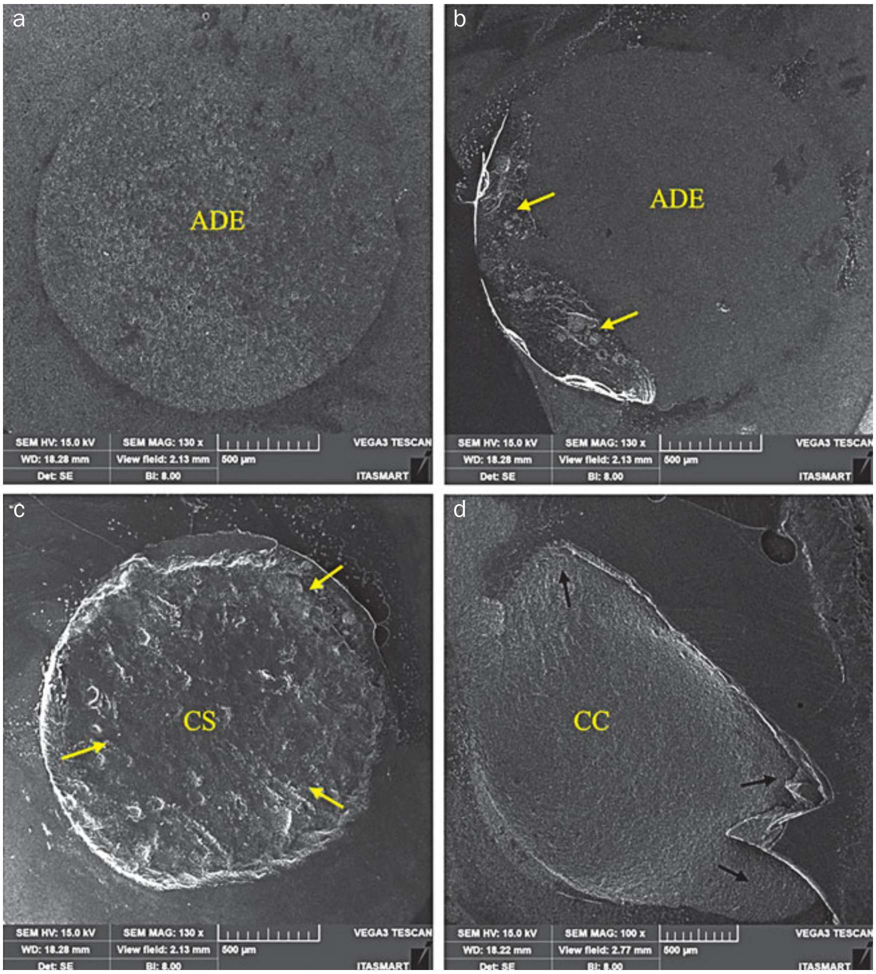

Fig 1 Micrographs of failures mode, 130X magnification a. adhesive (ADE); b. predominantly adhesive; c. cohesive characterization layer (CS); d. cohesive ceramic (CC). Yellow arrows indicate remnants of characterization layer on the surface of hybrid ceramic.

Table 4 Mean (MPa) bond strengths by group

Surface treatment	Thermocycling	Bond strengths
Polishing (P)	y	4.54 (1.2)^{D}
	n	4.99 (1.3)^{D}
Hydrofluoric etching (E)	y	13.85 (4.2)^{A}
	n	14.19 (2.3)^{A}
Aluminum oxide (A)	y	11.24 (2.8)^{AB}
	n	11.60 (2.7)^{A}
Self-etching ceramic primer (S)	y	7.11 (3.2)^{CD}
	n	8.14 (2.8)^{BC}

Different superscript letters indicate statistically significant differences. y: yes; n: no.

The means and standard deviations of the μSBS of each area were calculated and statistically analyzed. The data were normalized and then subjected to two-way ANOVA (factors: "surface treatment and "thermocycling"). Then, Tukey's test was employed to compare the means, with significance set at $\alpha = 0.05$.

Topographic Analysis

Topographic analysis of the surfaces was performed with scanning electron microscopy (Vega3, Tescan) at 15 kV and a magnification of 3000X. Representative specimens from each group were selected after observation with the same stereomicroscope as above (Stereo Discovery V20), then the surface topography of the specimens from different groups were visualized using SEM (P: surface polishing; E: surface etching with hydrofluoric acid; A: air blasting with aluminum oxide; S: surface etching with self-etching ceramic primer).

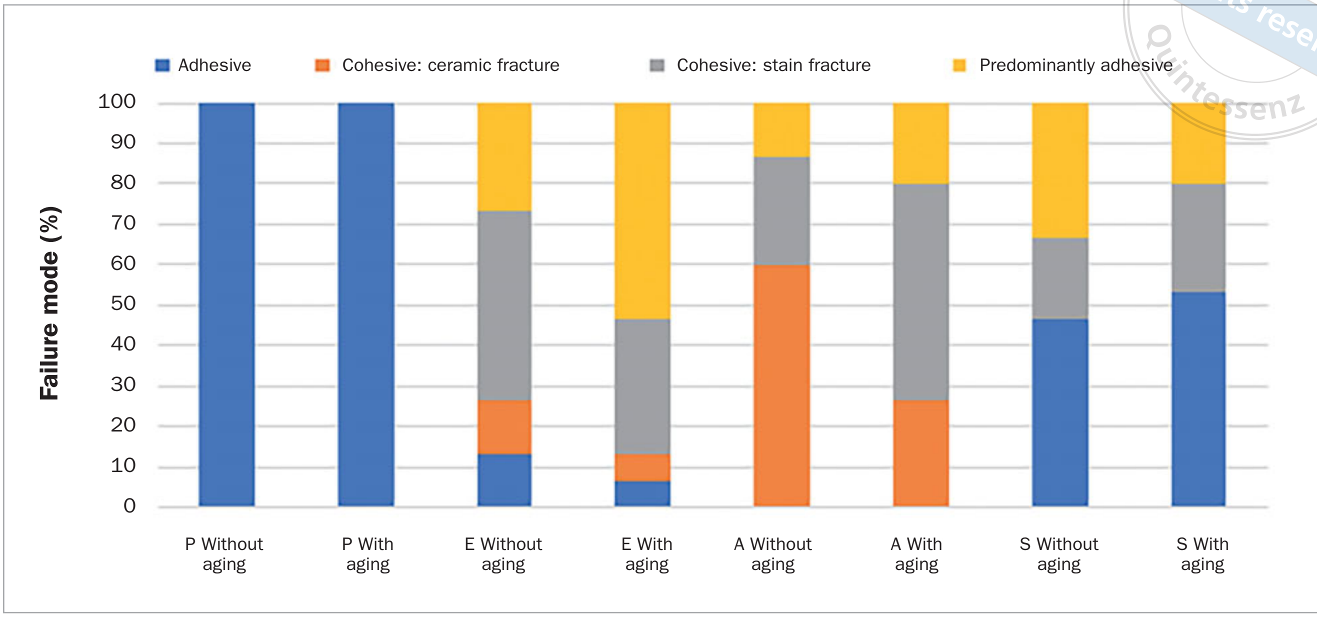

Fig 2 Bar graph of failure mode distributions for characterization layer bonded to hybrid ceramic. Failure modes: adhesive between characterization layer and ceramic hybrid; cohesive: ceramic fracture; cohesive: characterization layer fracture; predominantly adhesive between characterization layer and ceramic. P: surface polishing; E: surface oxide; S: surface etching with self-etching ceramic primer.

RESULTS

Failure Analysis and SBS Test

For surface treatments, two-way ANOVA (Table 3) showed statistically significant differences ($p = 0.000$), whereas there was no statistically significant difference for the "thermocycling" factor ($p = 0.283$). For group P, no surface treatment but polishing, the bond strength was drastically reduced, while group E had the highest bond strength both with and without thermocycling. (Table 4). For groups P and S, both before and after thermocycling, there were no cohesive failures of the ceramic. Figure 1 depicts the failure modes, and Fig 2 shows the distribution of failure modes. The most common failure modes were adhesive and cohesive in the characterization layer.

Topographic Analysis

Figure 3 shows the micrographs of the groups according to the surface treatment: a. polishing; b. surface etching with 5% HF; c. sandblasting with aluminum oxide; d. surface etching with self-etching ceramic primer. Polished specimens showed a homogeneous surface without exposure of the feldspathic ceramic network. Specimens etched with 5% HF exhibited the ceramic network after dissolution of the polymeric infiltrate. Sandblasted specimens showed slight exposure of the ceramic network, and self-etching ceramic primer-treated surfaces showed only slight degradation.

DISCUSSION

The present study evaluated the influence of different surface treatments of hybrid ceramic (polishing with silicon carbide sandpaper, 5% HF etching, sandblasting with Al_2O_3, and self-etching ceramic primer) and thermocycling, using the μSBS test to determine the bond strength between the hybrid ceramic and the characterization layer. The findings of this study showed that bond strength was not influenced by aging in water (thermocycling), leading to acceptance of the second null hypothesis. The factor responsible for lowest bond strength measure was the absence of a surface treatment on the hybrid ceramic surface, leading to rejection of the first null hypothesis. Thus, bond strength between the hybrid ceramic and characterization layer was affected only by the factor "surface treatment".

The hybrid ceramic is available as blocks for CAD-CAM processing. This block is composed of feldspar ceramic infiltrated by a polymer, which does not need a firing step in the oven after milling; only finishing and polishing are necessary.[17] Most specimens showed adhesive failures between hybrid ceramic and characterization layer. The μSBS test was chosen due to the ease of execution and also because it reveals important information regarding the adhesion of dental materials.[4,9] Other tests such as microtensile and microshear are widely used due to the smaller test area; however, the present study used an equivalent area of 2 mm^2. Thus, there was a smaller inclusion of defects in the adhesive area.

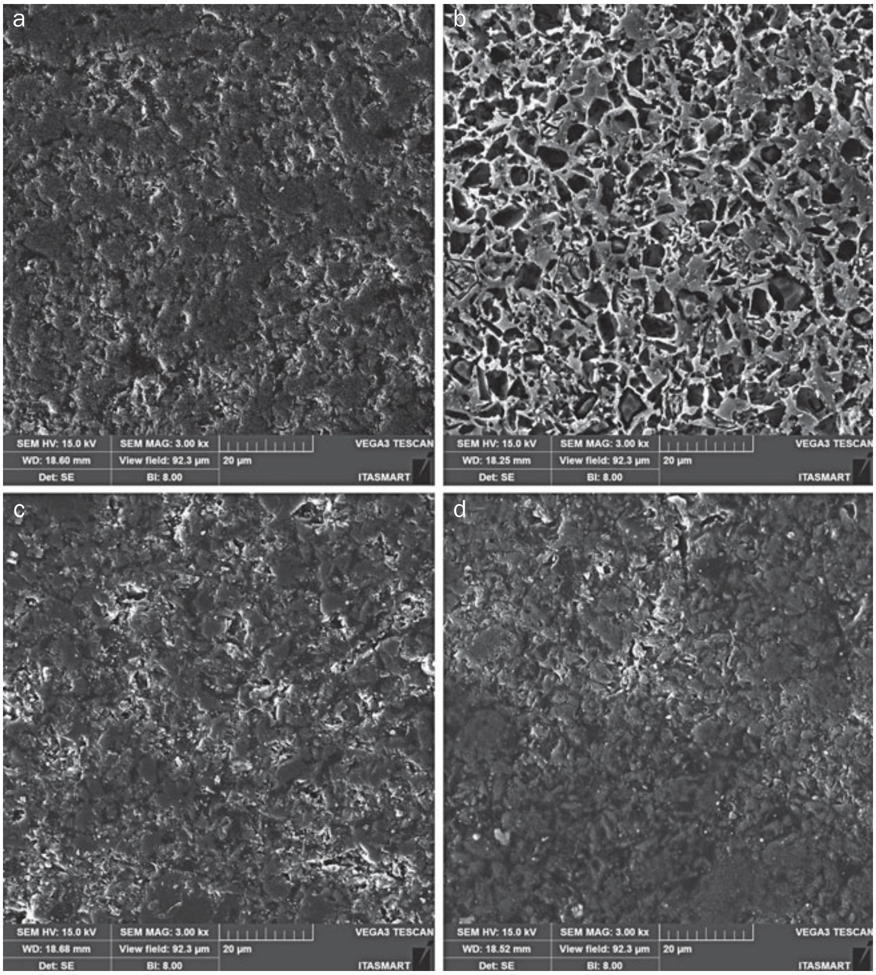

Fig 3 SE Micrographs of the groups according to the surface treatment: a. polishing; b. surface etching with 5% HF; c. sandblasting with aluminum oxide 50-µm Al_2O_3 50; d. surface etching with self-etching ceramic primer.

The topography of the hybrid ceramic surface is shown in Fig 3. This figure shows the exposured feldspar ceramic network and irregularities after removing the polymeric infiltrate. Although the polishing of the surface generates a more homogeneous layer with fewer defects, it does not favor mechanical retention, so the bond strength was lower.

Nevertheless, surface etching with 5% HF showed removal of the polymeric portion, exposing the feldspar ceramic network, which led to the highest bond strengths between the hybrid ceramic surface and the characterization layer. One reason for this is the characterization layer's low viscosity, which facilitates its penetration into the pores of the feldspar ceramic network caused by HF etching.[4,8,15]

The surfaces sandblasted with aluminum oxide showed small roughened areas and exposed feldspar ceramic network. This group showed a considerable rate of cohesive failure in ceramic, as shown in Fig 2. Although self-etching ceramic primer yielded a surface with minor defects after the etching process, the treated surface did not improve bond strength between the hybrid ceramic and the characterization layer.

Aging by thermocycling had no effect on the bond strength of the evaluated material. Any degradation at the adhesive interface can be attributed to hydrolytic degradation.[3,13] However, in the present study, the evaluated surface treatments promoted acceptable long-term bond durability against the aging process.

CONCLUSION

This hybrid ceramic required surface treatment for the adhesion of the characterization layer. It can be inferred that surface etching of the hybrid ceramic with 5% HF, followed by the application of self-etching ceramic primer, are the best surface treatments for the adhesion of the characterization layer under the conditions of the present study. Thermocycling reduced bond strength in all groups, regardless of surface treatment.

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Clinical relevance: Hybrid ceramic requires a surface treatment for the application of the extrinsic characterization layer. Among the surface treatments studied, 5% HF etching showed the highest values for bond strength between the hybrid ceramic surface and the characterization layer.