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Effect of silane and temperature on the bond strength between glass ceramic and resin cement

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ABSTRACT

The aim of this study was to evaluate the effect of silane and temperature, as well as a new combined etch-and-prime product, on the bond strength between glass ceramic and resin cement. Thirty-five ceramic discs (12 mm × 2 mm) were randomly assigned to seven groups (n = 5) according to surface treatment: 1 – Sandblasted, 2 – Sandblasted + silane, 3 – Sandblasted + silane + heating (60 °C), 4 – Hydrofluoric acid (HF), 5 – HF + silane, 6 – HF + silane + heating (60 °C), and 7 – Etch & prime. Resin cement was applied in cylindrical molds (0.75 × 0.50 mm) at five locations on each disc and photoactivated. The specimens were stored in distilled water at 37 °C for 24 h. Microshear testing was performed in a universal testing machine at 0.5 mm/min. Data were analyzed using one-way ANOVA and Tukey's post-hoc test (α = 0.05). HF + silane at 23 °C and 60 °C showed the highest bond strength, without significant differences from etch & prime, which did not differ from HF without silane. HF without silane was statistically like sandblasted + heating. Sandblasted + silane at 23 °C and sandblasted without silane presented the lowest values, without differences between them. SEM analysis revealed distinct surface morphologies according to treatment. Within the limitations of this study, heating silane after HF etching did not significantly increase bond strength compared to room temperature silane, while etch & prime performed similarly to conventional HF + silane protocols.

INTRODUCTION

Some dental ceramic has a high degree of crystallization to enhance their mechanical properties (Fu *et al*., 2020; Zarone *et al*., 2019). Pressable lithium disilicate-based glass ceramic has become one of the most used ceramic materials in dentistry especially because this material offers high mechanical properties, is adhesive to the dental structure with the use of an appropriated cement and provides a pleasant esthetic result (Zhang *et al*., 2023). This material is based on a partially crystallized glass obtained from the controlled growth and nucleation of crystals within the glassy phase. It can be defined as a ceramic composed of one glassy phase and at least one crystalline phase (Fu *et al*., 2020). It has been shown excellent clinical results and is indicated for use in veneers, inlays, onlays, full crowns, and three-unit bridges for the anterior and premolar region (Aslan *et al*., 2019; Teichmann *et al*., 2017).

However, the intaglio surface of lithium disilicate ceramics must be adequately treated to promote reliable bonding with the resin cement. Two main adhesive procedures are commonly used, namely mechanical adhesion promoted by hydrofluoric acid (HF) etching or sandblasting of the ceramic surface by means of airborne particles, and chemical bonding promoted by a silane agent (Alrabeah *et al*., 2024; Wille *et al*., 2022). HF etching dissolves the glassy phase, creating micro-porosities (5–20 µm deep) and increasing the contact area, which promotes micromechanical interlocking with the resin cement (Bruzzi *et al*., 2017). After the reaction, the surface is washed out and irregularities are created. Silane application then enhances both surface energy and chemical bond strength (Islam *et al*., 2025). Mechanical retention in an etched surface enables the adhesive to interlock with the irregularities. Sandblasting the surface with airborne particle abrasion with aluminum oxide particles also creates a rough ceramic surface, increasing mechanical retention; however, this method may be less effective and can potentially affect flexural strength when compared to HF etching (Lankes *et al*., 2024).

After removal from the investment, pressable glass-ceramic restorations are typically sandblasted, thereby creating a rough surface that provides inherent mechanical retention (Lu *et al*., 2024). The standardize the ceramic surfaces with silicon carbide (SiC) paper and diamond pastes is a protocol used in prior investigations to eliminate preexisting microroughness that could influence bond-strength measurements (Brum *et al*., 2011). Such standardization produces a flat, polished ceramic surface, which allows the effects of hydrofluoric acid conditioning and 50 µm alumina airborne-particle abrasion to be evaluated independently (Brum *et al*., 2011). However, a completely polished surface does not reflect actual clinical conditions; thus, the used sandblasted surfaces as the control, in line with common laboratory practice (Alrabeah *et al*., 2024).

By the time of the cementation procedure, the practitioner must decide whether to keep the intaglio surface as received from the laboratory or to etch it with hydrofluoric acid (Alrabeah *et al*., 2024). The literature remains conflicting regarding whether sandblasting or etching provides superior bond strength (Alrabeah *et al*., 2024; Silva *et al*., 2021). Similarly, although it is well established that a silane-coupling agent must be applied to the glass-ceramic surface, there is still debate on whether heating the silane improves its interaction with resin cement (Abduljabbar *et al*., 2016; Yanakiev *et al*., 2021). It is known that the reactive component is a methacrylate-functional silane—a bifunctional molecule that chemically bonds to the silicon dioxide on the ceramic surface and, through its methacrylate end, copolymerizes with the resin matrix during curing (Matinlinna *et al*., 2004). In addition to chemical bonding, the silane agent enhances the surface energy and wettability of the hydrophilic ceramic surface, facilitating better resin cement and adaptation of the organic composite (Thiranukoon *et al*., 2025).

Recently the Ivoclar-Vivadent company released a product called Monobond Etch & Prime which is commercially available in single bottle. The manufacturer claims that this product allows etching and silanization of the glass ceramic surface in one step. It contains a trimethoxypropyl methacrylate for silanization and a new polyfluoride for the etching step. In the manufacturer guide there is an explanation saying that etching creates a roughness pattern which is less pronounced than with hydrofluoric gel but as efficient for bonding (Ivoclar Vivadent AG, 2023a). Of course, that on the eyes of the manufacturer the product works perfectly and besides that, the efficiency of the product is shown by some bond strength tests. However, as a new material it needs to be evaluated and compared to the traditional methods so far proved. Moreover, inconsistencies in the literature regarding the optimal use of silane and hydrofluoric acid, combined with the introduction of new surface treatment agents, highlight the need for further investigation. Considering the divergences, the aim of this study was to evaluate the effect of silane and temperature on the bond strength between glass ceramic and resin cement. The null hypothesis was that the surface treatment, silane application and heating procedures would not interfere in the bond strength.

MATERIALS AND METHODS

This study was an in vitro experimental design, and the Figure 1 provides an overview of the experimental workflow, illustrating the main steps and procedures performed in this study. Table 1 summarizes the main materials used, including their commercial names, manufacturers, and respective specifications.

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Figure 1 – Schematic representation of the experimental workflow. Lithium disilicate ceramic discs (12 mm × 2 mm) were fabricated (step 1) and subjected to different surface treatments (step 2). Resin cement cylinders were applied and light-cured on the treated ceramic surfaces (step 3). Specimens were then tested for microshear bond strength (step 4), followed by surface characterization using scanning electron microscopy (SEM) (step 5)

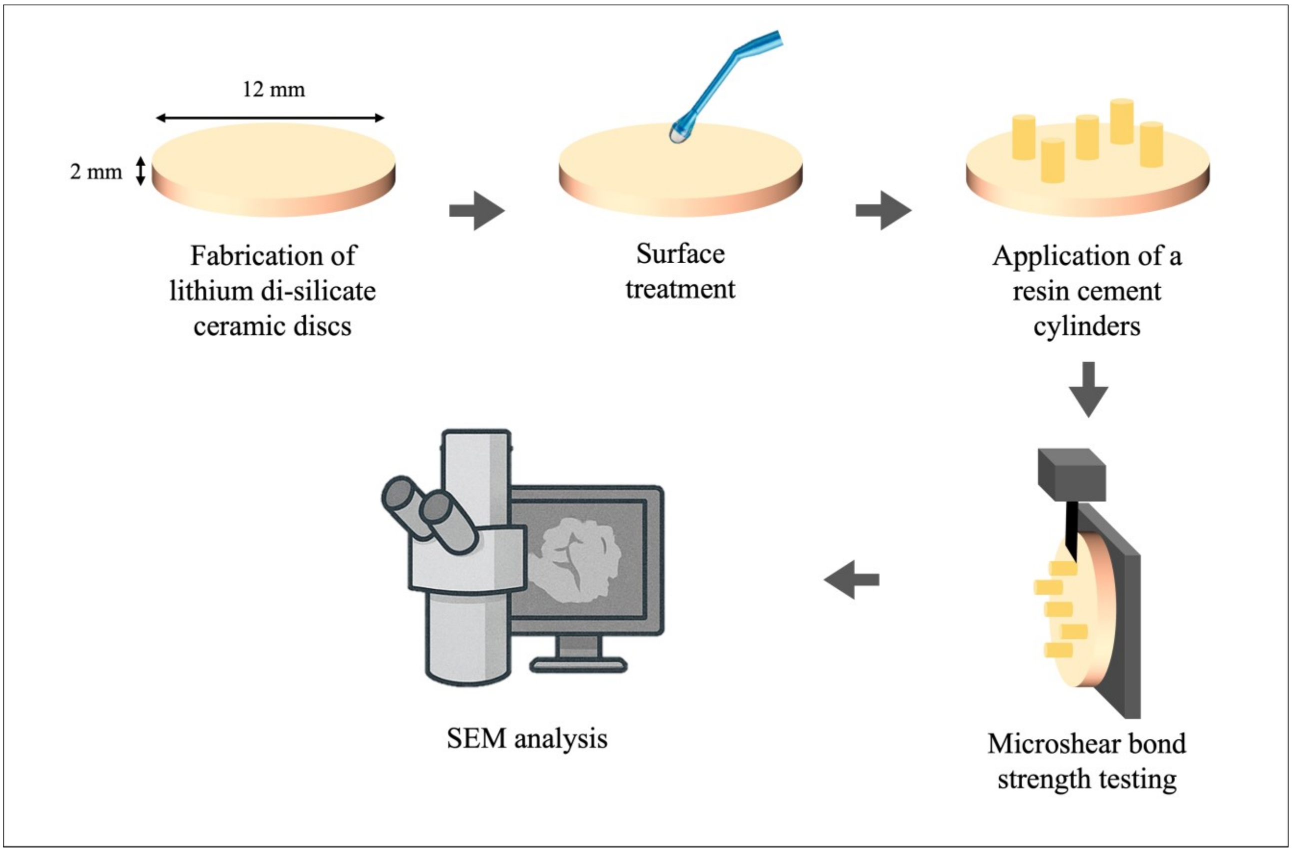

Table 1 – Commercial names, manufacturers, functions, and main compositions of the materials and equipment used in the present study

Commercial name / description	Manufacturer (City, State, Country)	Main composition*
IPS e.max Press HT D3	Ivoclar Vivadent, Schaan, Liechtenstein	Lithium disilicate ($Li_2Si_2O_5$)
IPS PressVest Speed	Ivoclar Vivadent, Schaan, Liechtenstein	Phosphate-based investment
Monobond N	Ivoclar Vivadent, Schaan, Liechtenstein	Methacrylates, functional silane, solvents
Hydrofluoric acid 10%	Dentsply, York, PA, USA	10% hydrofluoric acid gel
Monobond Etch & Prime	Ivoclar Vivadent, Schaan, Liechtenstein	Polyfluoridic acid, trimethoxypropyl methacrylate
Variolink Esthetic	Ivoclar Vivadent, Schaan, Liechtenstein	Resin matrix (methacrylates), inorganic fillers

*Compositions are generic and based on typical manufacturer information; they can be replaced with the exact technical data from the product's safety or technical data sheet (TDS).

Specimens' preparation

Thirty-five di-silicate glass ceramic (e.max Press HT D3, Ivoclar Vivadent) discs with 12 mm in diameter and 2 mm thickness were fabricated by the hot isostatic pressing (HIP) throughout the lost wax technique. The procedure started by producing wax discs with the dimensions above mentioned which were invested with phosphate-based material (IPS PressVest Speed, Ivoclar Vivadent), and the wax was eliminated in an automatic furnace (EDG 3000, EDG, São Carlos, SP, Brasil) at 850 °C for one hour. The IPS e.max Press ceramic ingots were pressed into the molds in an automatic press furnace (EP 3000, Ivoclar Vivadent). After cooling, the specimens were divested with airborne particle of 90 µm Renfert Basic Master (Renfert, Hilzingen) with 6 bars to remove the rough investment, and 2 bars of pressure to remove the investment close to the ceramic. Following, the sprues were removed using a diamond blade (006, Brassler).

After, the ceramic discs were divided into seven groups (n = 5) as following:

- Group 1: Sandblasted W/S - No additional treatment.
- Group 2: Sandblasted Sil – two silane (Monobond N – Ivoclar Vivadent) layers were applied and allowed to dry for 60s.
- Group 3: Sandblasted Sil 60 °C – two silane layers were applied and heated for 1 min at 60 °C each layer.

Hydrofluoric acid 10% (Hydrofluoric acid 10% - Dentsply) was applied for 20 s and the groups were as following:

- Group 4: HF W/S – No silane application.
- Group 5: HF Sil – Two silane layers was applied.
- Group 6: HF Sil 60° C – Two silane layers were applied followed by heating at 60 °C for 1 min for each layer.
- Group 7: Etch & Prime - A product that content acid and silane combined was applied with a microbrush, agitated it into the surface for 20s and allowed it to react for another 40s following the manufacturer's instructions.

Cementation procedures

After surface treatment, the resin cement (Variolink Estethic, Ivoclar Vivadent) was used to fill an iris that was cut from microbore Tygon tubing (TYG-030; Small Parts Inc, Miami Lakes, FL, USA) with an internal diameter and height of approximately 0.75 and 0.50 mm, respectively. The Tygon tubing was cut in a lathe using a stainless-steel no. 11 scalpel blade (Scalpel SS, Miltex Instruments Co, Rietheim-Weilheim, Germany). The Tygon tubing containing resin luting agent was put on the ceramic surface and photocured using a LED unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) with a power density of 900 mW/cm2. In this manner, each ceramic surface was bonded at 5 different locations with the resin cylinders. The assembly of ceramic/resin luting agent was stored at room temperature (23 °C) for ten min before the Tygon tubing removing procedure, then the specimens were immersed in distilled at 37 °C for 24 hours before the microshear test.

Microshear bond strength testing

The microshear bond strength test was carried out in a universal testing machine (EMIC DL3000, São José dos Pinhais, Brazil) using a 50 Kgf load cell at a cross speed of 0.5 mm/min. An especial device was attached to the universal testing machine and a stainless-steel blade was loaded on the interface between the resin cement cylinder and the ceramic surface until the bond failure.

Scanning electron microscopy

Representative fractured specimens (bonding area fractured) and interfaces were gold coated with a sputter coater (Balzers-SCD 050; Balzers Union Aktiengeselischaft Fürstentun, Liechtentein) for 180 seconds at 40 mA. They were then mounted on coded brass stubs and examined using electron microscopy (LEO 435 VP; Cambridge, England) operated at 20 kV, by the same operator.

Statistical Analysis

Bond strength (MPa) data were summarized as mean ± SD per disc (mean of five cylinders). Normality (Shapiro–Wilk) and homoscedasticity (Levene) were verified. One-way ANOVA compared the seven groups ($\alpha = 0.05$), followed by Tukey's post-hoc test. Effect sizes (η^2) and 95% CIs were reported where appropriate. Analyses were performed in BioEstat 5.0 (Instituto Mamirauá, Tefé, Brazil).

RESULTS

Microshear bond strength

As presented in Table 2, hydrofluoric acid (HF) treatment followed by silane application at both 23 °C and 60 °C yielded the highest bond strength mean values. However, these groups were not statistically different from the Etch & Prime group ($p > 0.05$). The Etch & Prime group also showed no statistical difference compared to HF without silane application.

Table 2 – Mean and standard deviation (SD) bond strength in MPa of all groups

Surface treatment	Mean (SD)
HF Sil	30.60 (4.13) A
Sand Sil	16.20 (3.28) D
HF Sil 60º C	28.92 (3.08) A
Sand Sil 60º C	22.44 (5.46) C
HF W/S	24.72 (6.34) BC
Sand W/S	13.93 (3.09) D
Etch-prime	26.96 (4.11) AB

Note: HF = Hydrofluoric acid; Sil = silane; Sand = sandblasted; HF W/S = no silane application; Sand W/S = no additional treatment. Same capital letters within in column do not have statistical differences.

The HF without silane group, in turn, exhibited similar bond strength to the sandblasted and heated silane group. The lowest mean bond strengths were observed for the sandblasted 23° C silane and sandblasted no-silane groups, which did not differ significantly from each other ($p > 0.05$). Overall, treatments involving HF and silane tended to produce higher bond strengths than sandblasting protocols, regardless of heating, while the Etch & Prime self-etching ceramic primer achieved intermediate performance.

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Scanning electron microscope

The SEM images showed that the interface between the resin cement and the glass ceramic was continuous, without voids or failures, for the HF-treated groups with two heated silane layers and two non-heated silane layers (Figure 2), as well as for the sandblasted surfaces with two heated or non-heated silane layers (Figure 3). In contrast, the sandblasted group without silane presented a visible gap between the ceramic and resin cement (Figure 4C). The Etch & Prime group demonstrated a continuous and homogeneous interface without voids or defects (Figure 5). Regarding the fractured interface, all groups showed predominantly adhesive failures located in the center of the bonded areas, with minor remnants of resin cement remaining on the ceramic surface.

Figure 2 – SEM images. (A) Interface between two heated silane layers on HF-etched ceramic (white arrows) (×3500). (B) Corresponding fractured bonded area (×150). (C) Interface between two silane layers on HF-etched ceramic (white arrows) (×3500). (D) Corresponding fractured bonded area (×150)

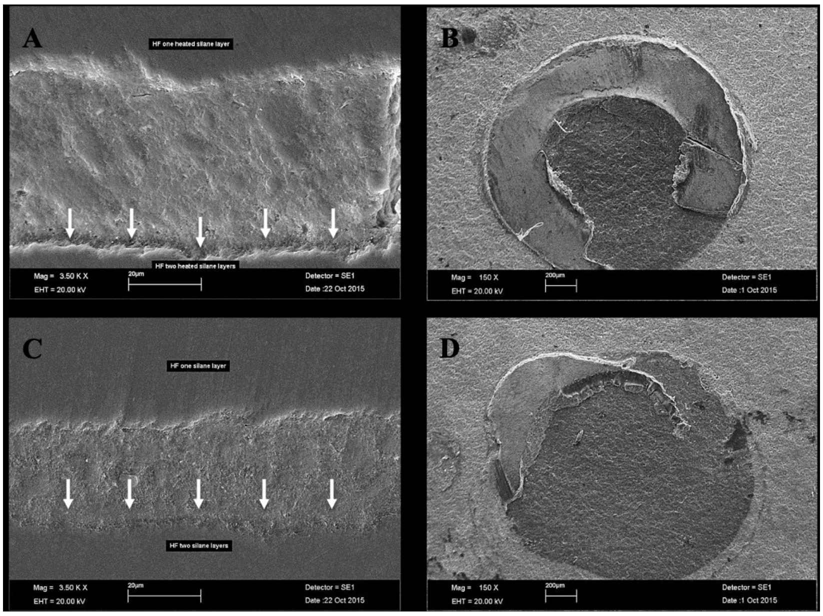

Figure 3 – SEM images. (A) Interface between two heated silane layers on sandblasted ceramic (white arrows) (×3500). (B) Corresponding fractured bonded area (×150). (C) Interface between two silane layers on sandblasted ceramic (white arrows) (×3500). (D) Corresponding fractured bonded area (×150)

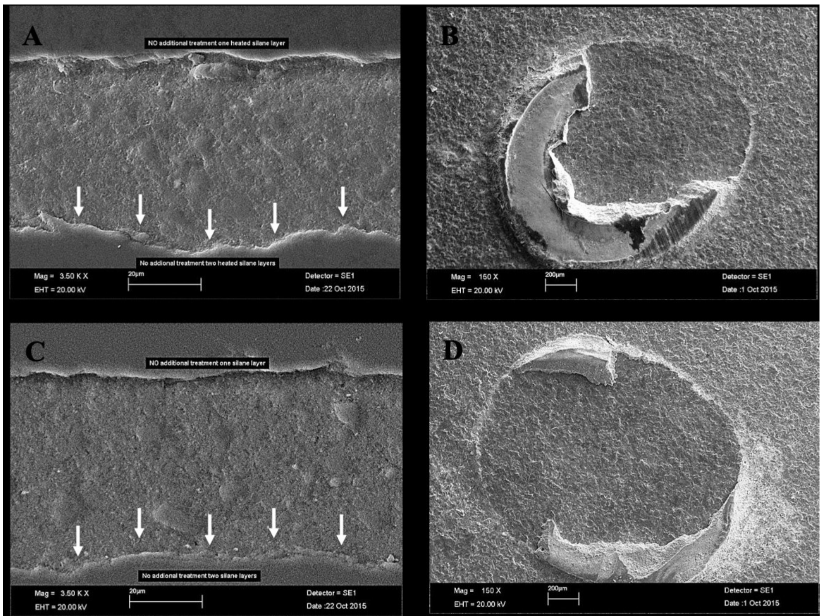

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Figure 4 – S M images. (A) Interface between HF-etched ceramic without silane (white arrows) (×3500). (B) Corresponding fractured bonded area (×150). (C) Interface between sandblasted ceramic without silane, showing a visible interfacial gap (white arrows) (×3500). (D) Corresponding fractured bonded area (×150)

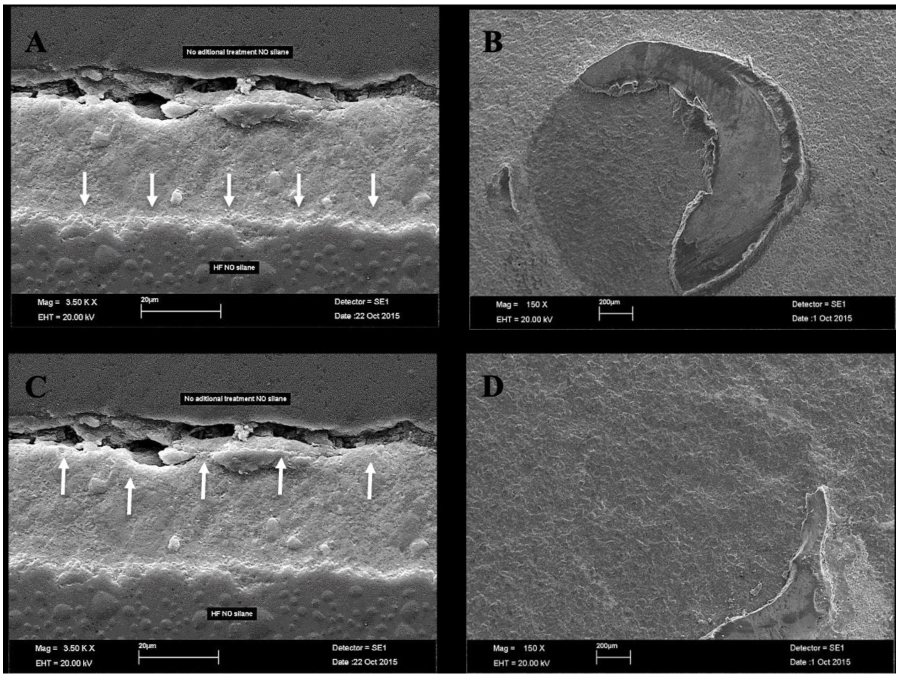
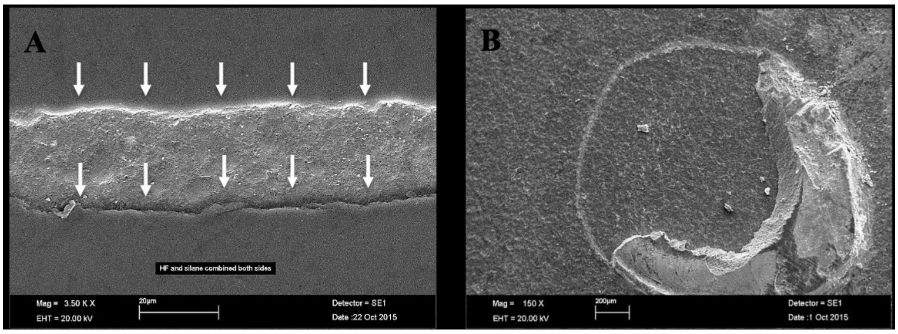


Figure 5 – SEM images. (A) Interface of the Etch & Prime group showing a continuous and defect-free bonding interface (white arrows) (×3500). (B) Corresponding fractured bonded area (×150)



DISCUSSION

The null hypothesis that the surface treatment, silane application, and heating procedures would not interfere in the bond strength was rejected. As shown in Table 2, there were significant differences among the different groups' treatments. The groups treated with hydrofluoric acid exhibited the highest bond strength results. This can be explained by the fact that hydrofluoric acid dissolves the glassy matrix and secondary crystalline phases, exposing lithium disilicate crystals and creating micro-retentive irregularities that increase the surface area (Sundheld *et al.*, 2015). The presence of these irregularities allows the bonding agent to flow and polymerize within them, resulting in an effective micromechanical interlocking between the resin bonding agent and the glass ceramic (Levartovsky *et al.*, 2021).

Sandblasting treatment resulted in lower bond strength compared to the hydrofluoric acid groups, regardless of heating or silane application. Airborne-particle abrasion with aluminum oxide particles has been shown to increase surface irregularities compared to a polished surface, enhancing micromechanical interlocking (Conner *et al.*, 2025). Even though we did not have a polished surface group, the literature is clear to show that. However, compared to the hydrofluoric acid groups, airborne particle abrasion tends to result in a more flattened and less retentive surface (Lankes *et al*., 2024). Borges *et al*. (2003) reported that airborne particle abrasion using aluminum oxide particles modified the ceramic surface but that the irregularities were shallow and superficial compared to hydrofluoric treatment. Of course, these surface treatments will be applied prior to the future cementation procedure, and even though there are divergences in results from many studies, most of them agree with the present data that hydrofluoric acid is more effective. This does not mean that sandblasting should be excluded—protocols such as simply washing the restoration in an ultrasonic bath upon its return from the laboratory may suffice in some cases—but given its simplicity, HF acid etching remains strongly recommended to enhance bond strength.

One observation that can be stated is that the heating of the silane agent resulted in the highest bond strength in the sandblasted group when compared to the same treatment without heating. Sandblasting produces a rough surface, but it is not as retentive as that created by hydrofluoric acid. The heating step may have removed the outer layers of the silane, allowing the interaction between the ceramic and resin cement to be more efficient (Abduljabbar *et al*., 2016; Yanakiev *et al*., 2021). On the other hand, this effect was not observed in the HF groups, likely because the surface etched by HF acid is much more retentive compared to the sandblasted. Thus, the interlocking is so effective that the outer-layer removal of the silane agent did not cause any effect (Alrabeah *et al*., 2024).

On the other hand, the etching and silanization of the glass ceramic in one step is very attractive because it simplifies the procedure and may decrease the operator variability of the treatment. According to the manufacturer's instructions (Ivoclar Vivadent, 2025b), the product should be applied with a microbrush and rubbed for 20 s, left on the surface for a further 40 s to allow the ammonium polyfluoride to etch the ceramic surface, rinsed thoroughly with water, and then air-dried for 10 s.This rinsing step removes polyfluoride and other byproducts, enabling effective chemical bonding of a thin silane layer (Vandewalle *et al*., 2018). In the present study, this protocol was strictly followed, and the bond strength results and SEM observations were comparable to the highest-performing groups (HF + silane), suggesting the new material may be reliable within the limitation of this study, which was conducted under static testing conditions. Thus, more studies with fatigue and aging tests need to be conducted before a definitive conclusion can be stated.

Another observation is that, compared with the traditional treatment of hydrofluoric acid etching followed by silane application, the combined etch-and-prime product requires only marginally different clinical steps and application time. The manufacturer argues that hydrofluoric acid poses greater risks than the combined product because it is highly corrosive and toxic, capable of causing deep tissue burns, systemic electrolyte disturbances, and potentially fatal metabolic effects even at dilute concentrations (Bajraktarova-Valjakova *et al*., 2018). The fluoride ion penetrates deeply, binds calcium and magnesium, and can lead to hypocalcemia, arrhythmias, and liquefactive necrosis (Lippert *et al*., 2020). That said, despite these hazards, hydrofluoric acid has been safely utilized in dentistry for decades—provided that strict safety protocols (e.g., isolation, neutralization, gloves, eye protection, proper ventilation) are rigorously followed (Türp *et al*., 2024). The combined etch-and-prime approach appears promising and may perform comparably to traditional methods in static tests; however, further fatigue and aging studies are needed before drawing definitive conclusions.

CONCLUSION

Acid with silane at 23º C and 60º C showed the highest bond strength, but did not present statistically significant differences compared to the etch & prime product that did not differ statistically from the hydrofluoric without silane. Hydrofluoric without silane did not differ from sandblasted and heated. Sandblasted with silane at 23º C and sandblasted without silane presented the lowest bond strength, but they did not differ from each other.

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