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## CASO CLÍNICO

Dr Daniel Hiramatsu

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# Bond strength between a lithium disilicate ceramic processed by different methods and a resin cement under different ceramic surface treatments

**ABSTRACT: Objective:** The aim of the present study was to evaluate the effect of different surface treatments and the processing of a lithium disilicate ceramic on bond strength and interfacial characteristics with a photo-activated resin cement. **Methods:** 20 pressed blocks (IPS e.max Press) and 20 blocks using the CAD / CAM technique (IPS e.max CAD) (10-mm diameter x 1-mm thickness) were made. Each type of processing was divided into four groups (n = 5), according to the type of surface treatment. There were eight experimental groups, in total: NT = without surface treatment; HFS = 10% hydrofluoric acid (HF) and silane application; HFU = HF 10% and universal adhesive; and EP = primer application. After preparation, the samples were stored in distilled water for 24 hours at 37 °C, and submitted to the bond strength test. The bond strength values, in MPa, were analyzed by Student's t-test, Kruskal-Wallis and Student-Newman-Keuls tests ($p > 0.05$). Scanning electron microscopy (SEM) images were performed for qualitative analysis of the fracture pattern after bond strength testing, and specimens were fabricated for cement/ceramic interface analysis. **Results and Conclusion:** The HFU

(16.8 ± 3.51 MPa) and EP (12.9 ± 3.05 MPa) treatments presented the best bond strength values for pressed ceramics and statistically better than HFU and EP of CAD / CAM ceramics. Among the CAD / CAM ceramics, the best values were presented by the HFS treatment (8.17 ± 4.81 MPa), which is statistically similar to the pressed HFS (5.92 ± 3.51 MPa). Only the NT groups presented gaps and SEM adhesive fracture pattern in the ceramic / cement interface. The other groups presented a mixed fracture pattern, without significant gaps in the ceramic / cement interface. The HFU and EP treatments were the best for pressed ceramics and HFS and HFU for CAD / CAM ceramics. SEM images showed no significant differences between surface treatment types, except NTs. **KEYWORDS:** Ceramic. Resin cements. Shear bond strength. CAD-CAM.

## INTRODUCTION

The use of dental ceramics is the reality of many dentist surgeons in clinical practice. These materials have the function of restoring totally or partially the loss of dental structure. Ceramics were for a long time exclusively used on a metallic infrastructure, but with the advent of reinforced ceramics and adhesion to dental structure, they are currently applied without the metal,[1] and there is sufficient scientific evidence showing their longevity.[2,3,4]

Among the various types of ceramics stand out the ceramics based on lithium disilicate, which consist of quartz, lithium dioxide, phosphoric oxide, alumina, potassium oxide and other components. Seventy percent of its content consists of lithium disilicate crystals, which provide mechanical resistance and favorable esthetic. Restorations of this type of material can be done in a pressed manner or by the technique of Computer Aided Design/Computer Assisted Manufacturing (CAD/CAM) and can be indicated in several cases as laminates, partial or total crowns, fixed prosthesis, single or multiple crowns.[5-8]

The preparation of pressed ceramics requires a variety of laboratory and clinical processes for the manufacturing of indirect restorations such as confection plaster model, troquelization, waxing, casting, finishing and polishing.[9] On the other hand, the CAD/CAM method consists of fewer steps, such as digitizing the plaster model (when not using the oral scanner), fabrication the restoration using specific software, machining the ceramic block, finishing and polishing.[10]In particular to the CAD/CAM method the finishing phase is characterized by the complete crystallization of the ceramic restoration. At this stage the ceramic is subjected to temperatures of 850 ° C, in its own furnace, and from 40% crystallization to 70%.[11]

Lithium disilicate ceramics exhibit excellent mechanical properties, both pressed and CAD / CAM processes. There is a similarity of fracture resistance of this type of ceramic, manufactured by both methods and present a correlation of similarity in the marginal adaptation.[12,13] These materials also show superior fracture toughness when compared to other types of materials, such as composite resins, hybrid ceramics, feldspathic ceramics and leucite-reinforced ceramics.[14,15,16]

This type of material is adhesively bonded to the tooth structure. In this way, procedures and materials are used to treat the ceramic based on lithium disilicate and thus to result in this bond. Conventionally, these ceramics are treated with hydrofluoric acid (HF), which conditions the glassy part of the ceramic, creating surface irregularities. The ceramic is then coated with a bonding agent, silane, capable of interacting with the inorganic part of the ceramic and the organic part of the cementing agent.[6,17,18]

Recently materials have been introduced that promise to simplify these clinical steps in one step. We can cite as examples of these materials, universal adhesives and ceramic primers, which condition and silanize the structure simultaneously, manufacturers claim their efficiency by reducing errors from multiple steps.[19,20,21]

In view of the above, several studies have already been carried out, but without a categorical proof of the superiority of efficacy of these simplified materials in the surface treatment process of the ceramics. It is observed the lack of studies that verify the action of the types of surface treatments in ceramics CAD/CAM and pressed in a single study. In this way, the objective of this work was to evaluate the effect of different surface treatments and the processing of the ceramic based on lithium disilicate in the bond strength and in the ceramic/ cement interfacial characteristics. The study was carried out under the following hypotheses: 1) the different types of surface treatment do not influence the values of bond strength between ceramic and resin cement; and 2) ceramic manufacturing methods do not influence the adhesive strength values between ceramic and resin cement.

## MATERIALS AND METHODS

Twenty ceramic samples with 10 mm diameter and 1 mm thickness were fabricated for e.max Press (Ivoclar Vivadent, Schaan, Liechtenstein), and twenty samples with 10 mm by 15 mm for 1 mm thickness were fabricated for e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein). Table 1 describes the materials used in the study and Table 2, gives the manufacturer's instructions.

**Table 1:** Description of the materials used.

| MATERIAL | COMPOSITION | MANUFACTURER |
|---|---|---|
| IPS e.max Press | $SiO_2$, $Li_2O$, $K_2O$, MgO, ZnO, $Al_2O_3$, $P_2O_5$ and other oxides. | Ivoclar Vivadent, Schaan, Liechtenstein |
| IPS e.max CAD | $SiO_2$, $Li_2O$, $K_2O$, MgO, $Al_2O_3$, $P_2O_5$ and other oxides. | Ivoclar Vivadent, Schaan, Liechtenstein |
| Variolink Esthetic | Organic matrix: urethane dimethacrylate and other methacrylate monomers. Inorganic matrix: ytterbium trifluoride and mixed spheroid oxide. Initiators, stabilizers and pigments. | Ivoclar Vivadent, Schaan, Liechtenstein |
| Condac porcelana | 10% Fluoridric Acid, water, thickener, surfactant and colorant. | FGM, Joinville-SC, Brazil |
| Monobond N | Alcoholic solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate. | Ivoclar Vivadent, Schaan, Liechtenstein |
| Single Bond Universal | 2-hydroxyethylmethacrylate, Bisphenol A diglycidyletherdimethacrylate (BisGMA), Decamethylenedimethacrylate, Ethanol, Silanetreatedsilica, Water, 1,10-Decanediol phosphatemethacrylate, Acryliccopolymeranditaconicacid, Caforquinone, N, N-Dimethylbenzocaine. | 3M ESPE, St Paul, MN, USA |
| Monobond Etch&Prime | Aqueous alcoholic solution of ammonium polyfluoride, silane methacrylate and dye. | Ivoclar Vivadent, Schaan, Liechtenstein |

**Table 2:** Application steps of surface treatment procedures.

| MATERIAL | CERAMIC SURFACE |
|---|---|
| Condac porcelana | Apply for 20s, wash and dry. |
| Monobond N | Apply a thin layer with a microbrush and leave to act with 60 sec. Remove any excess with a strong jeto fair. |
| Single Bond Universal | Apply a layer to the pretreated surface with hydrofluoric acid, remove excess and apply a strong jet of air. |
| Monobond Etch&Prime | Apply with a microbrush for 20 s, leave to act for 40 s. Rinse the surface and dry. |

### Pressed ceramic discs

Acrylic resin cylinders (Duralay, Reliance Dental MFG Company, Illinois, USA) with 10 mm in diameter were made in a putty consistency of Poly Dimetil Siloxane (Zetaplus, Zermack, Italy) and following cut in discs in the thickness of 1.0 mm using a 0.5 mm-diamond saw (Buehler, Lake Bluff, IL, USA) coupled to IsoMet precision machine (Isomet 1000-Buehler, Lake Bluff, IL, USA). After, they were sprued in silicone cylinders, attached to a flask base, invested with phosphate-based material (IPS Press Vest Speed, Ivoclar Vivadent, Schaan, Liechtenstein) and eliminated in an automatic furnace (EDG 3000, São Carlos, SP, Brazil) at temperature of 850°C for 60 min using the lost wax technique. The IPS e.max Press ceramic ingots were pressed into the investment molds in an automatic press furnace (EP 3000, Ivoclar Vivadent, Schaan, Liechtenstein). After removing the disc from the investment material by sandblasting(4 bar to remove the coarse part and 2 bar for removal of coatings near the samples), all samples were ultrasonically cleaned in deionized water (Ultrasonic Cleaner 1440 D, Odontobrás, Ribeirão Preto, SP, Brazil) for 10 min, dried with compressed air. The final disc thicknesses (1.0 mm) were confirmed with a digital caliper (Mitutoyo Corporation, Tokyo, Japan), with accuracy of 0.01 mm.

### CAD/CAM ceramic blocks

The CAD/CAM samples were made by cutting the ceramic block with a cutting apparatus (Isomet 1000-Buehler, Lake Bluff, IL, USA) and 0.5 mm diamond disk (Buehler, Lake Bluff, IL, USA).The final thickness was checked the same way as described to the pressed discs. Following, the blocks were crystalized in an automatic press furnace (EP 3000, Ivoclar Vivadent, Schaan, Liechtenstein). The crystallization process takes between 20 to 31 minutes, and the blocks do not shrinkage significantly. The process happens between 840 to 850 °C and it produces microstructure modification, which is a controlled growing of the disilicate crystals.

## Surface treatment for cementation

After fabrication, the samples were divided into eight experimental groups, according to the type of surface treatment and processing, as shown in figure 1, where:

- ✓ NT = no treatment;
- ✓ HFS = hydrofluoric acid and silane
- ✓ HFU = hydrofluoric acid and universal adhesive
- ✓ EP = monobond Etch & Prime

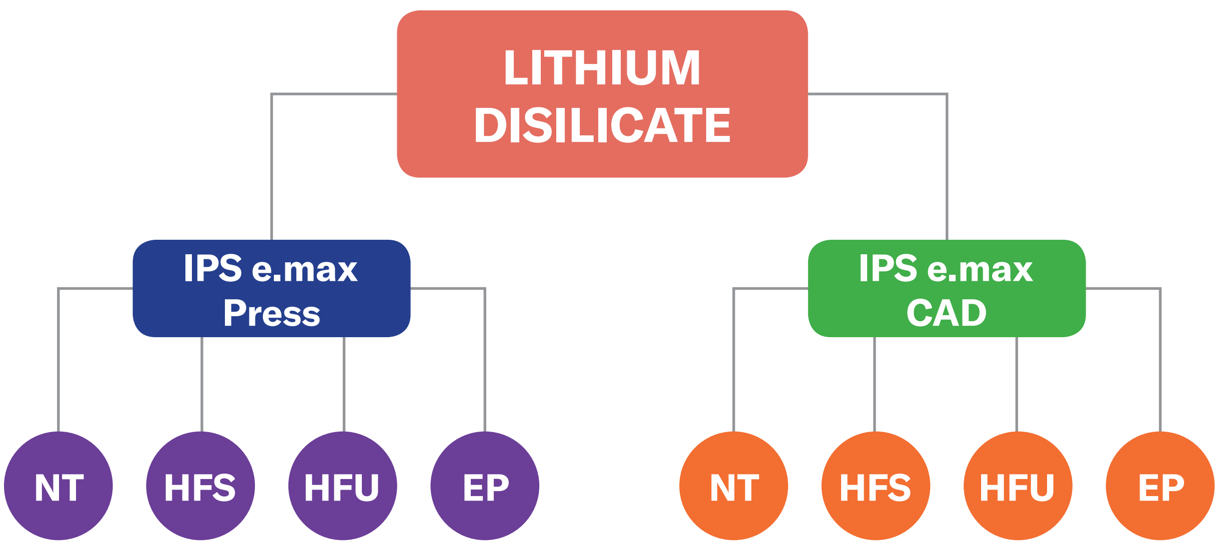


**Figure 1:** Organization chart of experimental groups.

### Cementation procedure

Polyvinyl siloxane molds (Virtual, Ivoclar Vivadent, Schaan, Liechtenstein), 0.5 mm thick, were fabricated using five cylinder-shaped orifices (0.8 mm in diameter) and were placed on the ceramic disc surface to determine the adhesion area. Before positioning the mold, each surface treatment was applied to the surface of each experimental group.

Surface treatment and cementing procedure were performed by the same operator under controlled temperature (23 ± 2ºC). The resin cement (Variolink Esthetic LC, Ivoclar Vivadent, Schaan, Liechtenstein) was prepared according to the manufacturer's instructions and inserted into the orifice of the mold, with a spoon excavator (Duflex, Juiz de Fora, MG, Brazil). Excess cement was removed using a resin spatula #01 (Duflex, Juiz de Fora, MG, Brazil). The resin cement was photo activated for 40 s, using a continuous mode with a LED Radii Cal (SDI, Victoria, Australia) and an irradiance of 500 mW/cm$^2$, as verified with radiometer (Kerr, Joinvile, SC, Brazil). After 10 min the silicone matrix was removed and cement cylinders were carefully evaluated with a optical microscope to observe the bonding area. Following, they were stored for 24h at 37ºC, 100% relative humidity until the bond strength test.

### Micro shear bond strength test

Microshear bond strength (μSBS) testing was performed in a testing machine (EMIC DL 3000 ¯ EMIC - Equipamentos e Sistemas de Ensaios Ltda. São José dos Pinhais, Brazil). A stainless steel chisel was attached to the load cell and the test was carried out at 0.5 mm/min crosshead speed until failure. The average of each resin cement cylinder on the ceramic specimens was calculated to obtain the mean value of the bond strength of each sample. The testing machine software was set to give the results in MPa.

### Statistical analysis

The mean of the total samples of each group was submitted to the t student test to normal and homogenous distribution variable among the groups. Following, Kruskal-Wallis test and Student-Newman-Keuls post hoc test were carried out. Differences were considered significant at $p< 0.05$.

### Failure mode analysis and cement/ceramic interface

After the rupture of the resin cement cylinders were observed in scanning electron microscopy (SEM) (JSM-5600LV, Jeol Ltd., Tokyo, Japan) at 15Kv. The specimens were mounted on coded brass stubs coated with sputter coating (SCD

050, BAL-TEC, Liechtenstein) for 180 s at 40 mA. And the images were classified as cohesive (COH) (failure within the cement layer), adhesive (ADH) (failure between ceramic and cement), or mixed (MIX) (involving cement and ceramic substrates).

Additional specimens of ceramics-cement-ceramics were obtained for each group; two ceramic samples conditioned with different surface treatment were bonded together using resin cement. The specimens were embedded cross sectionally in epoxy resin in order for the ceramic-cement interfaces to be viewed. After 24 h, the specimens were wet-polished with 600-, 1200- and 2000-grit SiC paper followed by polishing with 3 µm, 1 µm and 0.5 µm diamond compounds. The cross-section profiles were examined by SEM, focusing on the integrity, homogeneity and continuity along the bonding interface.

## RESULTS

### Statistical analysis

The data are presented in Table 3 and the highest bond strength values in MPa were presented by HFU (16.8 ± 6.26) and PE (12.9 ± 3.05) pressed ceramics groups, followed by HFS groups (5.92 ± 3.51) and NT (2.31 ± 1.66). Among the surface treatments of CAD/CAM ceramics, the highest statistical values of union strength were for the HFS group (8.17 ± 4.81), but were not statistically different in comparison to HFU (7.83 ± 5, 30) and EP (4.34 ± 2.78). Statistically, the lowest bond strength values among the CAD/CAM ceramics were demonstrated by NT 1.24 ± 1.23. Overall, all types of surface treatment of CAD/CAM ceramics were statistically lower than pressed ceramics, with the exception of HFS, which did not present statistically different values among ceramic manufacturing methods.

**Table 3:** Means and standard deviations of the micro shear bond strength (MPa) values of different experimental groups.

| GROUP | PRESS MEAN (SD) | CAD MEAN (SD) |
|---|---|---|
| NT | 2,31 (1,66)$^{Ac}$ | 1,24 (1,23)$^{Bc}$ |
| HFS | 5,92 (3,51)$^{Ab}$ | 8,17 (4,81)$^{Aa}$ |
| HFU | 16,8 (6,26)$^{Aa}$ | 7,83 (5,30)$^{Bab}$ |
| EP | 12,9 (3,05)$^{Aa}$ | 4,34 (2,78)$^{Bb}$ |

Different superscript uppercase letters in the same row indicate significant difference (p>0.05). Different superscript lowercase letters in the same column indicate significant difference (p>0.05).

## Failure mode analysis and cement/ceramic interface

The SEM images showed that, regarding the failure mode, only NT groups, both CAD and Press, presented adhesive failures and the other groups presented mixed failures. The interface between the resin cement and the glass ceramic was continuous without voids or failures for all groups, except NT that showed discontinuity for both CAD and Press, being the CAD group with a larger gap (Fig 2 -9).

[ A ] [ B ]

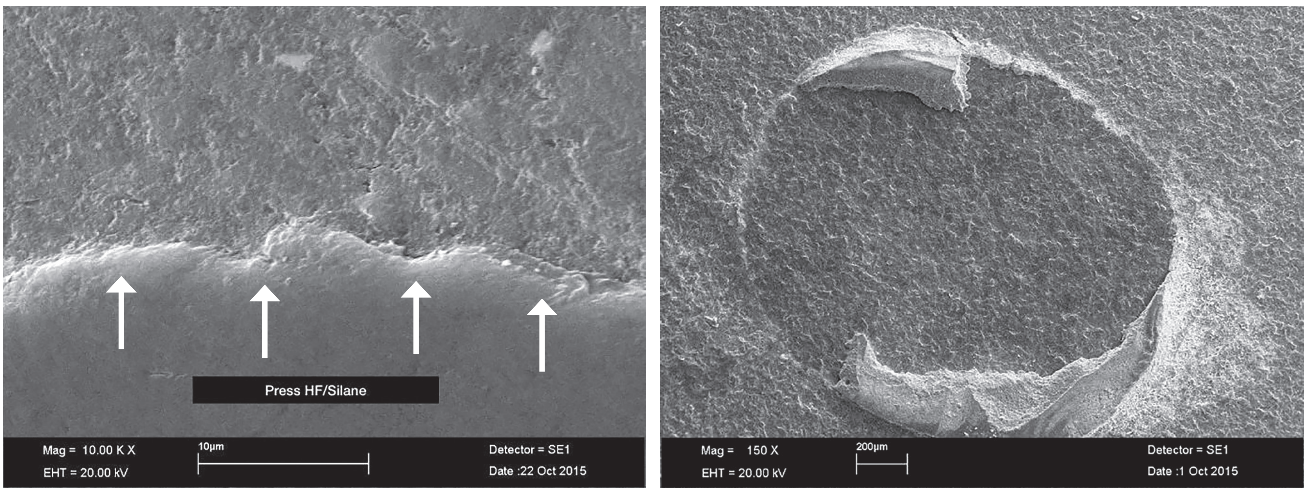


**Figure 2:** SEM images. **A** HF/Silane interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X10000) and **B** mixed failure for Press fabrication (Original magnification X150)

[ A ] [ B ]

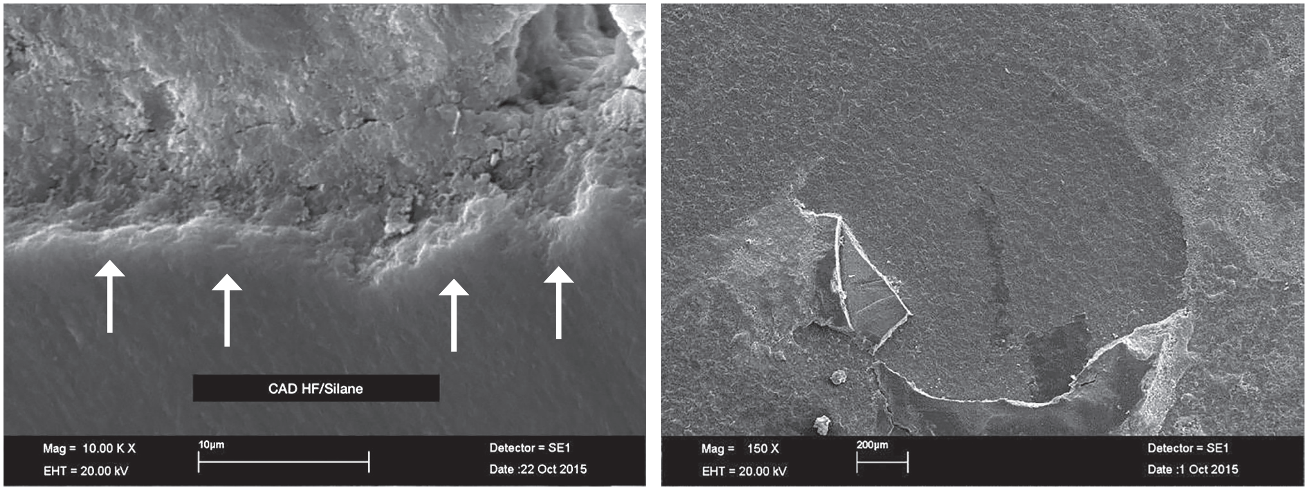


**Figure 3:** SEM images. **A** HF/Silane interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X10000) and **B** mixed failure for CAD fabrication (Original magnification X150)

[ A ] [ B ]

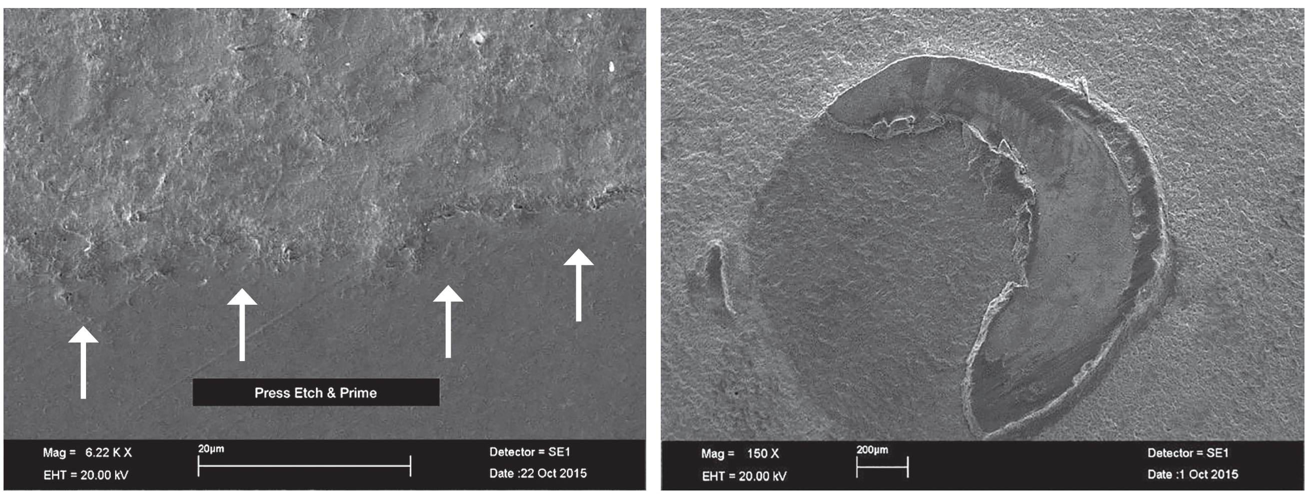


**Figure 4:** SEM images. **A** Etch and Prime interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X6220) and **B** mixed failure for Press fabrication (Original magnification X150)

[A] [B]

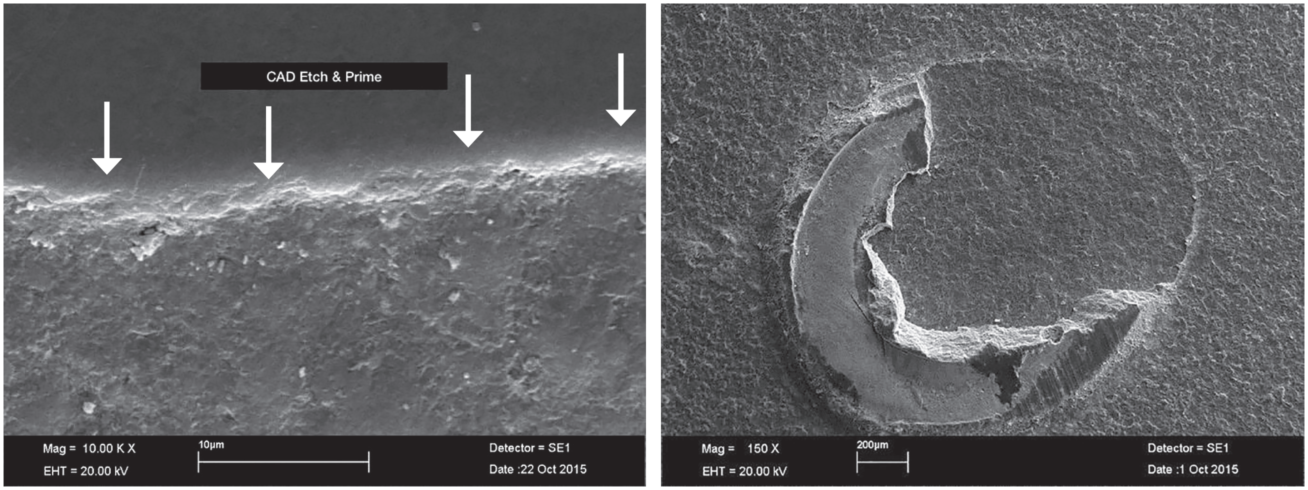


**Figure 5:** SEM images. **A** Etch and Prime interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X10000) and **B** mixed failure for CAD fabrication (Original magnification X150)

[A] [B]

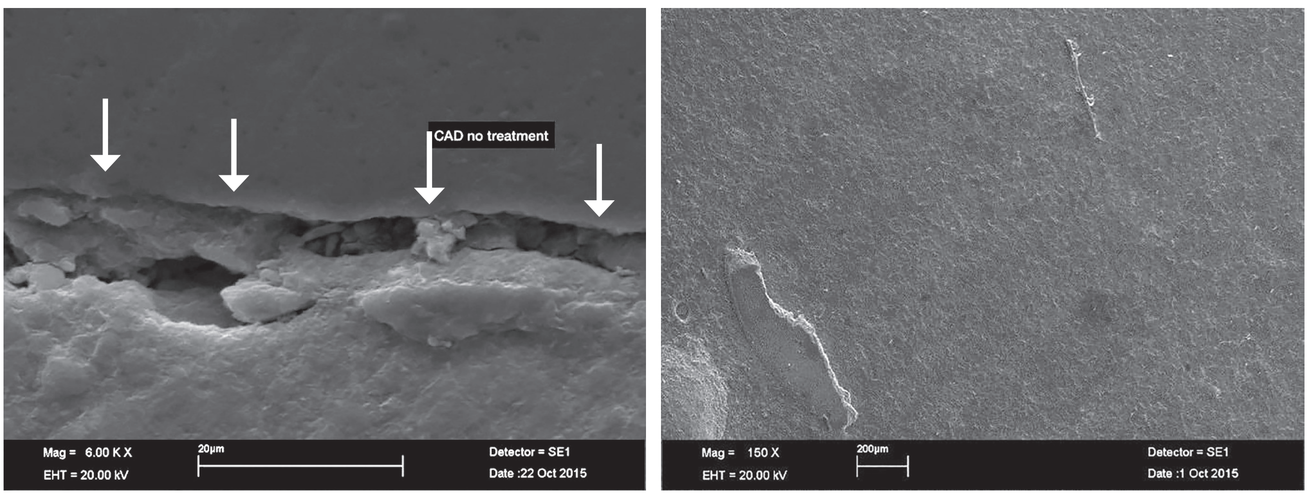


**Figure 6:** SEM images. **A** No treatment interface (indicated by the white arrows) with discontinuity and failure/gaps (Original magnification X6000) and **B** adhesive failure for CAD fabrication (Original magnification X150)

[ A ] [ B ]

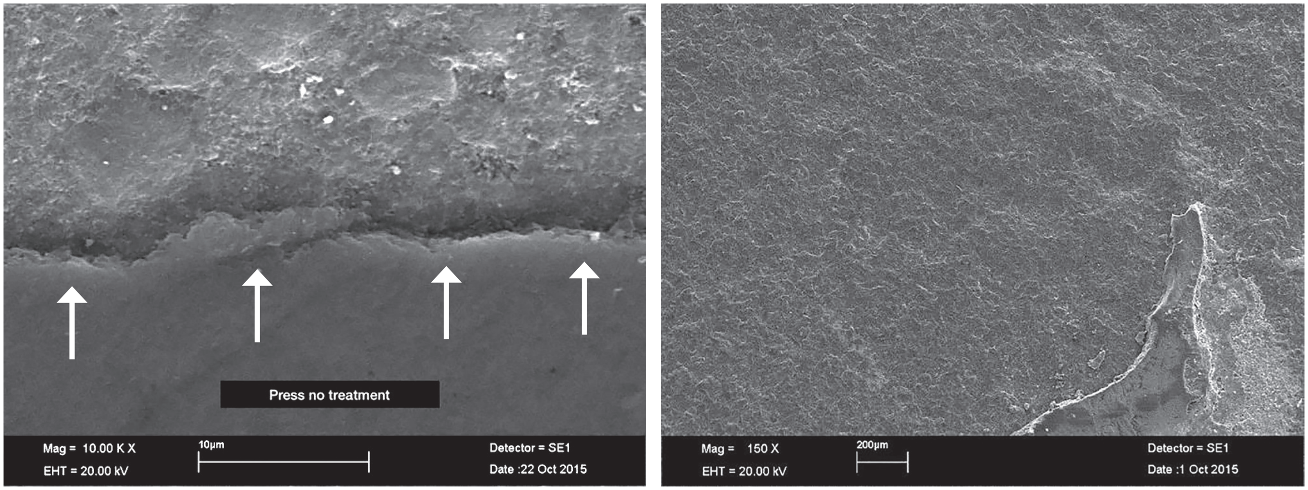


**Figure 7:** SEM images. **A** No treatment interface (indicated by the white arrows) with discontinuity and failure/gaps (Original magnification X10000) and **B** adhesive failure for Press fabrication (Original magnification X150)

[ A ] [ B ]

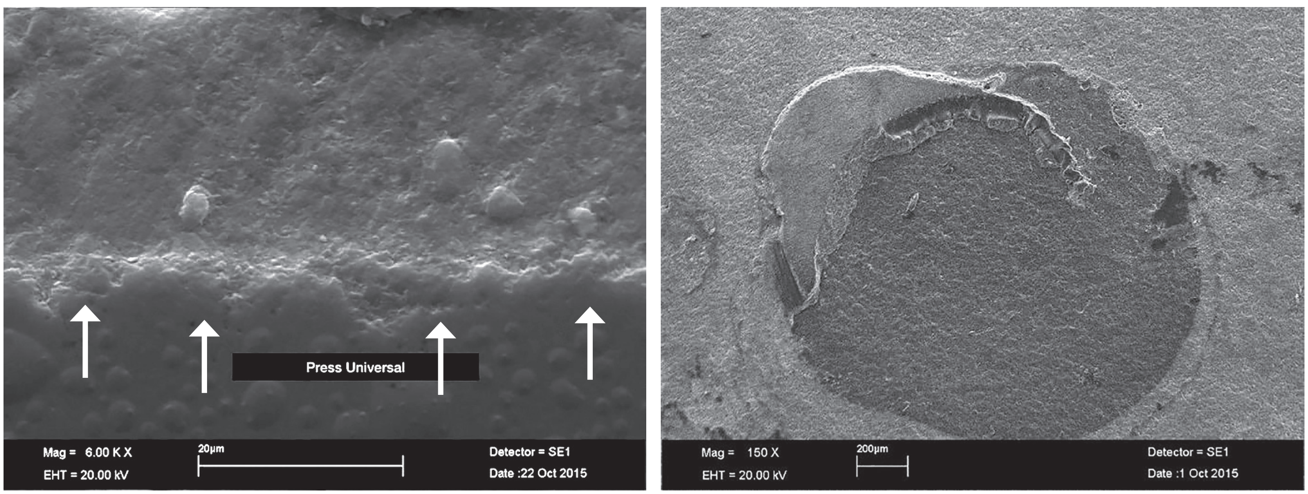


**Figure 8:** SEM images. **A** Universal adhesive interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X6000) and **B** mixed failure for Press fabrication (Original magnification X150)

[A] [B]

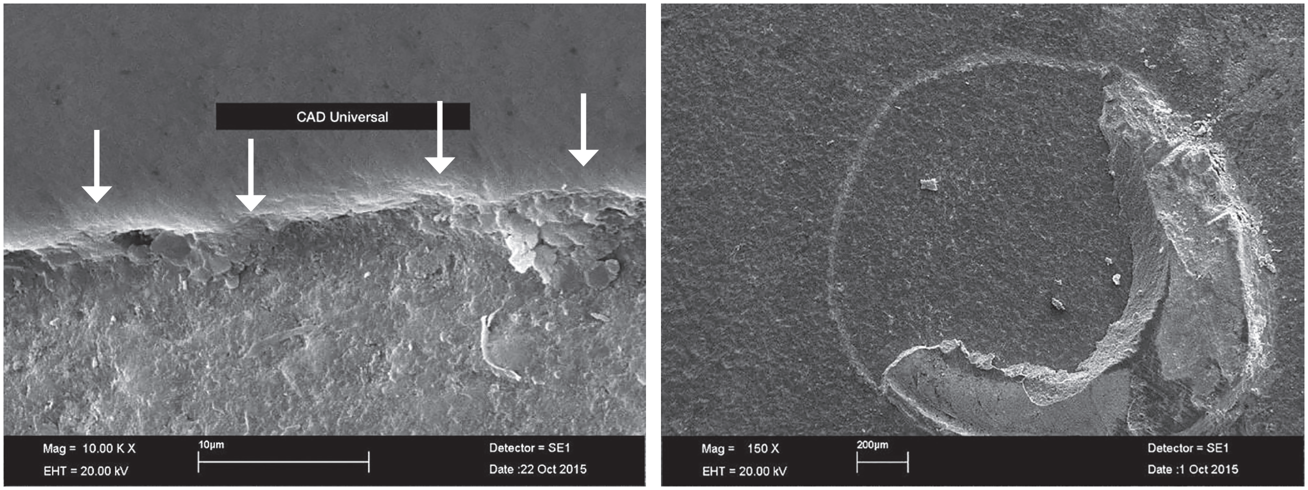


**Figure 9:** SEM images. **A** Universal adhesive interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X10000) and **B** mixed failure for CAD fabrication (Original magnification X150)

## DISCUSSION

The results showed that there was a statistical difference of bond strength between the types of surface treatment and the types of ceramic manufacture. Therefore, the hypotheses of the study were rejected. In the consulted literature, no study was found comparing the bond strength between the pressed and CAD/CAM systems of the lithium disilicate ceramic, using the same surface treatments in a single research. Both universal adhesives and ceramic primers are materials where the manufacturer brings the proposal to simplify clinical steps, when in only one bottle there are the etching and silane of the ceramic structure. Nevertheless, studies still show that the individual use of the silane coating agent has an important role in bond strength between ceramics, pressed and CAD/CAM, and resin cement.[22-26]

Universal adhesives, as used in the present study, Single Bond Universal (3M ESPE), contains silane and an acidic monomer called 10-methacryloxydecyl phosphate (MDP) in its composition. The silane is an important substance in the bond between lithium disilicate ceramics and resin cement. It is a bifunctional molecule that binds both the organic part of the ceramic and the inorganic one of the resin cement, but it is sensitive to the pH value of the solution. Usually the material presents a pH value between 4 and 5, on the other hand, the universal adhesives the pH is about 2.7.[27] The reaction between silane and MDP promotes the adhesion mechanism, improving surface wettability,[28] butthe pH value of an MDP molecule is between 2 and 2.7, which contributes to the low pH value of the universal adhesive, compromising the ideal chemical interaction of the silane with the ceramic.[27] This may have happened in the experiments of this study that led to the lower of results of the samples, treated with universal adhesive in the group of CAD/CAM ceramics, in comparison to the group treated with HF and silane. Furthermore, some studies also show worst results when using universal adhesive, such as Kalavacharla et al. in 2015[29] and Murillo-Gómez et al. in 2017[30] which demonstrated bond strength data when used silane plus a statistically better universal adhesive compared to the same application of the adhesive alone. Advising that, the realization of silane application is necessary for the surface treatment of CAD/CAM ceramics based on lithium disilicate.

The Etch & Prime monobond contains ammonium polyfluoride, which is an acid salt that corrodes glasses and silicates, reaching a porous aspect and resulting in micro-mechanical retentions, but has a softer acidity compared to hydrofluoric acid, leading to a pattern of weak conditioning.[31] Both El-Damanhoury in 2017[32] and Lyan et al. in 2018 [31]showed that, in comparison to the HF conditioning, the use of EP results in inferior bond strength values between ceramic and resin cement. In the same way, in the present study, better results can also be observed with the use of HFS and HFU in CAD/CAM ceramics than the use of EP.

The results of Press group treated with HFS were different than expected, considering the pertinent literature. [21-25]This factor is related to the concentration of HF used in the study. HF is responsible for removing part of the silica matrix of a glass ceramic, promoting a porous surface, allowing the micromechanical retention, besides providing greater area available for adhesion. [33]This material can be found in concentrations between 1 and 10%. It has already been demonstrated that, HF 10%, resulted in increase of bond strength between ceramic and resin cement because it results in more micro retentions than the other concentrations,[34] however, this high concentration can lead to an extensive removal of the vitreous matrix and the removal of the crystals of lithium disilicate, generating failures as gaps in the bond, acting as initiators of cracks.[33]

With the exception of the HFS CAD group, the remaining within CAD showed statistically lower bond strength values than all groups of pressed ceramics. This can be explained due to the grounding procedure used in CAD/CAM. Grounding by machining of a material is characterized by the process of removal of fragments by a tool (diamond tips and stainless steel burs). In the present study, diamond tips were not used as in clinical reality, but cutting with a diamond blade may have resulted in surface damage associated with the removal of the material, affecting the bond strength between this type procedure and the resin cement. These would induce cracks on the surface of the ceramic, that would propagate and resulting in catastrophic failure.[35] Another relevant explanation for the lower performances of CAD/CAM ceramics was reported in 2016.[11] In this study it was reported that CAD/CAM ceramics have lower fracture toughness values ($K_{IC}$) than pressed ceramics and SEM images of ceramic surface characterization, demonstrated that CAD/CAM ceramics present a surface smooth, indicating a crack propagation through the glass matrix, while pressed ceramics present a more rough and irregular surface, with several visible crystals embedded in the glass matrix. The difference between the $K_{IC}$ values between IPS e.max Press and CAD/CAM seems to be related to the higher amount of glass matrix, reduced crystalline phase and the smaller crystal size of the IPS e.max CAD, leading to larger failures of CAD/CAM ceramics.

Within the limitations of this study, in vitro, pressed ceramics resulted in values of bond strength statistically superior to CAD/CAM, when using universal adhesive and ceramic primer. High HF concentration did not show efficacy in pressed ceramics as shown in CAD/CAM ceramics. This leads us to reflect on the choice of the best surface treatment of CAD/CAM ceramics, if there would be a need for a specific treatment for this type of manufacturing method, even the ceramics are of the same composition. More studies are necessary to make clearer if there is a difference between the methods and treatments regarding not only bond strength, but also longevity of the restorations clinically.

## CONCLUSION

1) The use of hydrofluoric acid and universal adhesive proved to be the best surface treatment for pressed lithium disilicate ceramics. In contrast, the surface treatment of CAD/CAM ceramics was shown to be more effective when using hydrofluoric acid and silane.
2) SEM images showed significant discontinuity and presence of faults/gaps in ceramics no surface treatment, but the same findings were not found among the other treatments.
3 The fracture pattern between ceramic and resin cement showed both areas of resin cement failure and bonding agents, except in no treatment samples that showed almost total absence of resinous cement residue.

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