



# The Effect of Different Surface Treatment Protocols on the Shear Bond Strength to Repressed Lithium Disilicate Glass Ceramics

Efeito de diferentes protocolos de tratamento de superfície na resistência ao cisalhamento de vitrocerâmica de dissilicato de lítio prensado

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## ABSTRACT

**Objective:** The present study aimed to evaluate the effect of repressing and different surface treatment protocols on the shear bond strength of lithium disilicate glass-ceramics. **Material and Methods:** A total of 52 lithium disilicate glass-ceramic discs (IPS emax Press, Ivoclar Vivadent) were fabricated using the heat-press technique. The discs were divided into two groups; group (P): discs fabricated from new e.max ingots (n=26), group (R): discs fabricated from reused e.max buttons (n=26). Each group was subdivided into subgroup (E): discs were etched with hydrofluoric acid (9.5%) (n=13), subgroup (S): discs were air-abraded with 110  $\mu\text{m}$  alumina particles. All specimens were subjected to X-ray Diffraction analysis, Scanning Electron Microscope, Energy Dispersive X-Ray, Thermo-Cycling, and Shear Bond Strength Testing. **Results:** Repressed Etched subgroup (RE) recorded the statistically highest shear bond strength value, followed by the Pressed Etched subgroup (PE), while the statistically lowest shear bond strength value was recorded for the Pressed Air-Abraded subgroup (PS) and Repressed Air-Abraded subgroup (RS). **Conclusion:** Repressing the leftover buttons for the construction of new lithium disilicate glass-ceramic restorations has no adverse effect on the bond strength of the resin cement to the ceramic. Hydrofluoric acid surface treatment improves the shear bond strength and durability of resin cement bond to both pressed and repressed lithium disilicate glass-ceramic. Air-abrasion cannot be considered as a reliable surface treatment when bonding to lithium disilicate glass-ceramics.

## KEYWORDS

Heat pressed; Lithium disilicate glass-ceramics; Repressing; Shear bond strength; Surface treatment.

## RESUMO

**Objetivo:** O presente estudo teve como objetivo avaliar o efeito da repressagem e de diferentes protocolos de tratamento de superfície na resistência ao cisalhamento de vitrocerâmica de dissilicato de lítio. **Materiais e Métodos:** Um total de 52 discos de vitrocerâmica de dissilicato de lítio (IPS emax Press, Ivoclar Vivadent) foram fabricadas usando a técnica de prensagem quente. Os discos foram divididos em dois grupos: grupo (P): discos fabricados a partir de novo lingotes de e-max (n=26), grupo (R): discos fabricados a partir de botões de emax reutilizados (n=26). Cada grupo foi subdividido em subgrupo (E): discos condicionados com ácido fluorídrico (9,5%) (n=13), subgrupo (S) discos foram abrasivos com partícula 110  $\mu\text{m}$  de alumínio. Todos os espécimes foram submetidos a análise de difração de raio-x, microscópio eletrônico de varredura, raio-x de energia dispersiva, termociclagem e teste de resistência de cisalhamento. **Resultados:** O subgrupo Repressado-condicionado (RE) registrou o valor estatisticamente mais alta em relação a resistência ao cisalhamento, seguido pelo subgrupo Prensado-condicionado (PE), enquanto o valor estatisticamente mais baixo de resistência ao cisalhamento foi o subgrupo Prensado-Jateado (OS) e o subgrupo Repressado-Jateado (RS). **Conclusão:** A repressagem dos botões restantes para a construção de novas restaurações vitrocerâmicas de dissilicato de lítio não apresentou efeitos adversos na resistência de unicãp do cimento resinoso à cerâmica. O tratamento de superfície com ácido fluorídrico melhora a resistência ao cisalhamento e a durabilidade do cimento resinoso para vitrocerâmica de dissilicato de lítio prensada e reprimida. A abrasão por ar não pode ser considerada um tratamento de superfície confiável quanto a colagem da vitrocerâmica de dissilicato de lítio.

## PALAVRAS-CHAVE

Prensagem quente; Vitrocerâmica de dissilicato de lítio; Repressagem; Resistência ao cisalhamento; Tratamento de superfície.

## INTRODUCTION

Dental practitioners have adopted glass-ceramic restorations due to their ability to mimic natural tooth structure esthetics. Lithium disilicate glass-ceramics contains a 70% crystalline phase that increases its flexural strength than that of leucite reinforced glass-ceramics. [1,2] Lithium disilicate glass-ceramics have superior clinical properties; they bond adhesively to dental tissues, have high esthetics and biocompatible. These optimum optical and mechanical properties lead to high survival rates and hence their wide use in dentistry to restore lost and decayed teeth [3-11].

Lithium disilicate glass-ceramic restorations are cemented traditionally with glass ionomer cements or bonded adhesively with resin cements. [12,13] Adhesive bonding is recommended as its strength is superior to traditional cementation, [14-16] and also it increases the fracture resistance of lithium disilicate restorations. [17] Hydrofluoric acid (HF) etching has been recommended before the resin cement application, [18] as it partially dissolute the glassy phase [14,15] exposing the crystalline phase and increasing the surface area for bonding, [16-18] thus improving the bond strength of resin cement to lithium disilicate glass-ceramics. [19-23] The application of silane coupling agent will follow this, then a thin layer of unfilled resin is applied to promote a better infiltration to the surface irregularities of the etched lithium disilicate ceramic surfaces. [24,25]

Several studies compared etching with hydrofluoric acid to other surface treatments as air-abrasion and reported a decrease in shear bond strength when the heat-pressed glass-ceramic surface was air-abraded with alumina oxide particles. [26-30]

Heat-pressing of lithium disilicate glass-ceramics utilizes the lost wax technique. It has been reported that some dental laboratories reuse the residual glass-ceramic materials remaining from the heat-pressing procedures to press new restorations. [31,32] Studies showed

that repressing did not affect biaxial flexural strength (BFS), [31,32]. Hardness and flexural toughness [32] of heat-pressed glass-ceramics. In contrast to an another study, which reported a significant increase in BFS of repressed glass-ceramic material. [33] X-ray diffraction (XRD) and scanning electron microscopy (SEM) showed no difference in the crystalline composition of repressed glass-ceramics. Finally, it was concluded that lithium disilicate glass-ceramic could be repressed while maintaining good mechanical properties and without significantly altering the crystalline composition. [31-33]

The recycling of heat pressed lithium disilicate glass-ceramic leftover material has been reported to be done by dental laboratories. The effect of this procedure on the shear bond strength of repressed lithium disilicate glass-ceramic material is unknown, and has not yet been investigated especially with subsequent exposure to temperature changes. The present study aimed to evaluate the effect of repressing and different surface treatment protocols on the shear bond strength of lithium disilicate glass-ceramics. The null hypothesis is that the processing technique, as well as different surface treatment protocols, will have no significant effect on the shear bond strength of lithium disilicate glass-ceramics.

## MATERIAL AND METHODS

A total of 52 lithium disilicate glass-ceramic discs (IPS emax Press, Ivoclar Vivadent) were fabricated by using the heat-press technique. The discs were divided into two groups; group (P): discs fabricated from new e.max ingots (n=26), group (R): discs fabricated from reused e.max buttons (n=26). Each group was subdivided into subgroup (E): discs were etched with hydrofluoric acid (9.5%) (n=13), subgroup (S): discs were air-abraded with 110 um alumina particles. To fabricate specimens of group (P), wax discs (Geo Classic, Renfert) of 10 mm diameter and 2 mm thickness, were fabricated with the aid of a specially designed Teflon mold. The discs were sprued and invested (IPS

PressVest Premium, Ivoclar Vivadent) according to the manufacturer instructions. After the investment set (30 minutes), the investment ring was placed inside a burnout furnace (Ney, US Dental) for wax elimination according to manufacturer instructions. The ring was then transferred to a porcelain furnace (EP 3000, Ivoclar Vivadent), where it was heat-pressed using lithium disilicate glass-ceramic (A3, LT, IPS emax Press, Ivoclar Vivadent). After cooling (1 hour), rough divesting was done under 4 bar pressure followed by soft divesting under 2 bar pressure using 110  $\mu\text{m}$  alumina particles. The pressed object was placed in a 1% hydrofluoric acid (Invex liquid, Ivoclar Vivadent) for 10 minutes, then washed with water, air-dried, and air-abraded with 110  $\mu\text{m}$  alumina particles under 2 bar pressure to remove the remaining reaction layer. The discs were then cut from the sprues, and the remaining button was finished to resemble a new ingot using diamond stones. The leftover buttons were heat-pressed to fabricate the discs of group (R).

#### **X-ray Diffraction (XRD)**

X-ray Diffraction analysis was carried out to study the produced phases by using the PANalytical X-Ray Diffraction equipment model X'Pert PRO with Secondary Monochromator, Cu-radiation ( $\lambda=1.542\text{\AA}$ ) at 45 K.V., 35 M.A. and scanning speed 0.04o/seconds. The diffraction peaks between  $2\Theta = 20$  and  $60$ o, corresponding spacing ( $d$ ,  $\text{\AA}$ ) and relative intensities ( $I/I_0$ ) were obtained and compared with ICDD files.

#### **Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX)**

The SEM was carried out (at 6000x and 10000x) to study the surface topography of specimens using SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit to study the elemental structure of specimens, with accelerating voltage 30 K.V., magnification 14x up to 1000000 and resolution for Gun.1n.

#### **Surface Etching protocol**

Group (E) specimens were etched for 20 seconds with 9.5% buffered hydrofluoric acid

(Porcelain Etchant, Bisco), then rinsed with water, dried with oil-free moisture-free air, followed by silane coupling agent application (Porcelain Primer, Bisco) for 60 seconds then air-dried for 5 seconds. [3,22,23]

#### **Air-Abrasion protocol**

Group (S) specimens were air-abraded with 110  $\mu\text{m}$  aluminum oxide particles (Renfert) under pressure 2.8 atm, from a 1 mm distance for 15 seconds, then cleaned with alcohol, dried with oil-free moisture-free air, followed by silane coupling agent (Porcelain Primer, Bisco) application for 60 seconds then air-dried for 5 seconds. [26-30]

To ensure a standardized resin cement dimensions and to allow the mono-beveled chisel of the universal testing machine to introduce a compressive force at the ceramic resin interface, a pediatric catheter was cut into a height of 2 mm piece and attached using a single bond universal to the center of each disc. Care was taken not to contaminate the center of the disc with the adhesive bond. The catheter was secured in place by using light-cure for 20 seconds from all aspects, then the catheter was filled with dual-cure self-adhesive resin cement (Breeze, Pentron) and cured for 20 seconds from all aspects. After curing, the catheter was cut using a sharp lancet leaving a 2 mm height cylinder of resin cement attached to the disc and ready to be tested. [34,35]

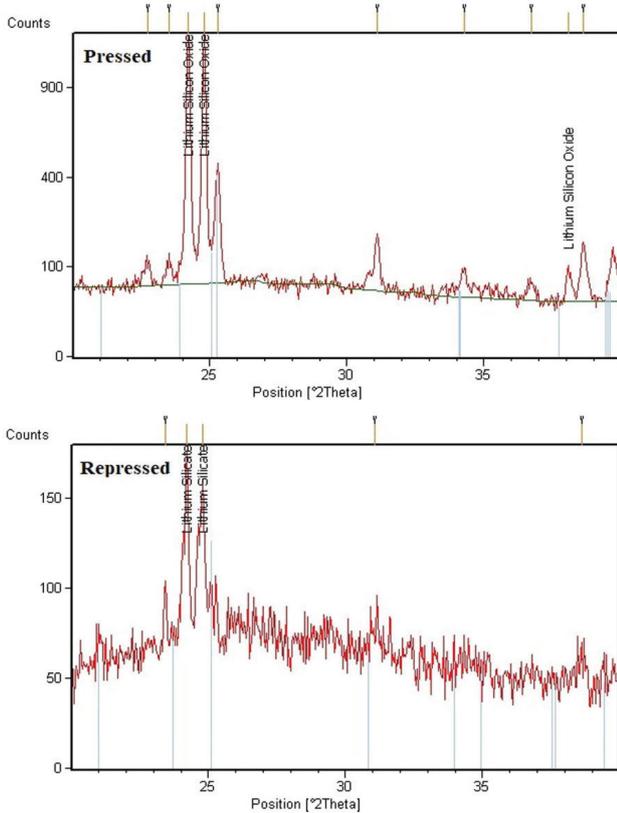
#### **Thermo-Cycling**

Thermal aging was performed for all specimens after adhesive bonding using Robota automated thermal cycle (BILGE, Turkey). The number of cycles was 5000 cycles (equivalent to 6 months inside oral condition). Dwell times were 25 in each water bath with a lag time of 10 seconds. The low-temperature point was 5  $^{\circ}\text{C}$ . The high-temperature point was 55  $^{\circ}\text{C}$ . [36,37]

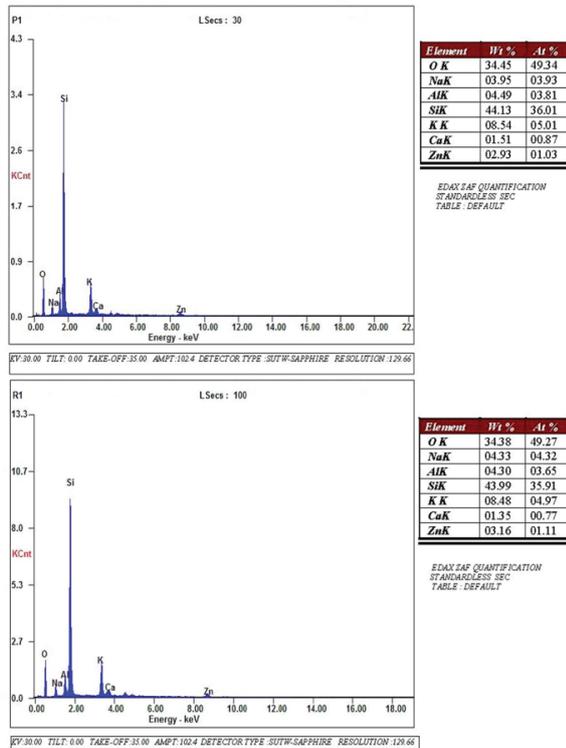
#### **Shear Bond Strength Testing**

A circular interface shear test was performed using a computer-controlled materials testing machine (Model 3345) with a loadcell of 5 KN. The data were recorded

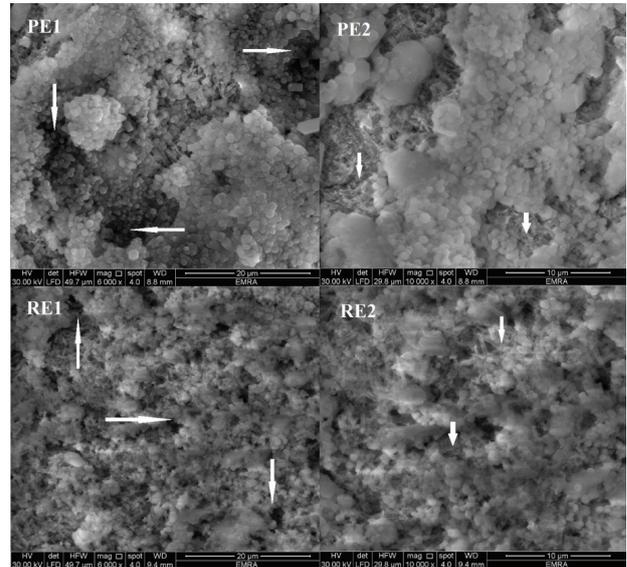




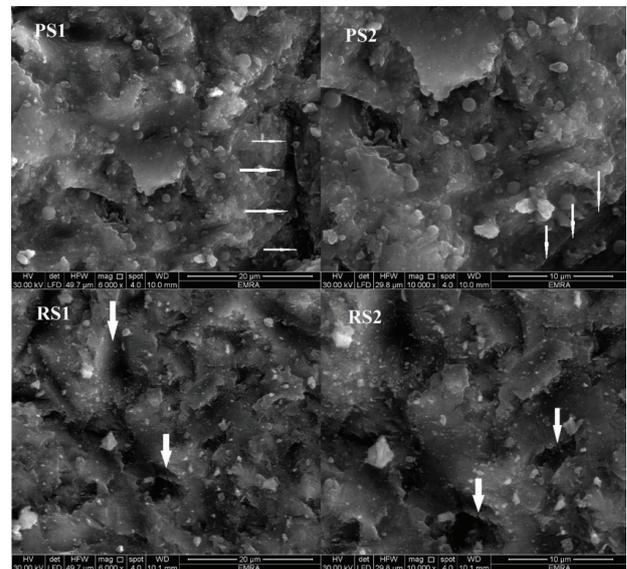
**Figure 1** - X-ray diffraction (XRD) patterns of IPS e.max Press (P) and (R) showing peak positions in agreement with those of standard Lithium disilicate.



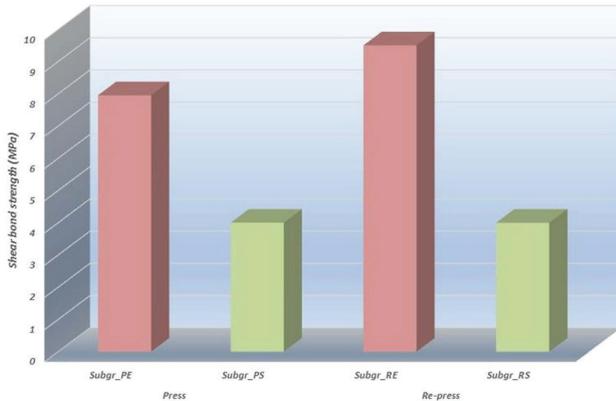
**Figure 2** - Microanalysis by EDAX of IPS e.max Press (P) and (R) groups.



**Figure 3** - SEM images for (E) group (6000X and 10000X) showing a deeper etching pattern with exposition of crystals in the (PE) specimens, while the (RE) specimens showed a more homogenized surface with the persistence of crystals.



**Figure 4** - SEM images for (S) group (6000X and 10000X) the surface of the (PS) specimens is characterized by various deep grooves, valleys and irregularly shaped defects, while that of the (RS) specimens showed crater-shaped cavities that are larger in size and greater in number.



**Figure 5** - Column chart of shear bond strength mean values for both groups with different surface treatment.

**Effect of Etching on the Bond Strength**

It was found that etching significantly increased the shear bond strength mean values of the tested specimens.

**Effect of Air-Abrasion on the Bond Strength**

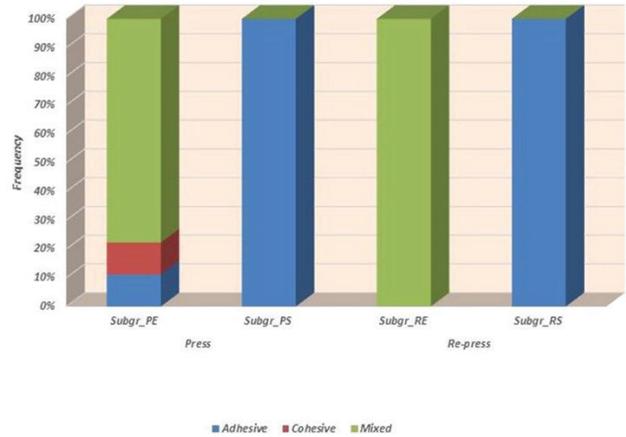
It was found that air-abrasion had no significant effect on the shear bond strength mean values of the tested specimens.

**Mode of Failure**

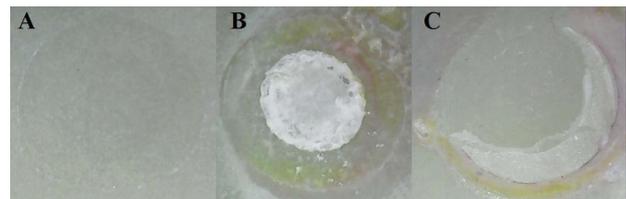
Frequent distribution of failure modes scores (%) for both groups as a function of two surface treatments (Table II, Figure 6). The difference in the frequent distribution of failure modes scores between groups was statistically significant, as indicated by the chi-square test ( $p < 0.0001 < 0.05$ ). The air-abrasion surface treatment showed a 100% Adhesive failure with both Pressed and Repressed specimens while the Acid Etching surface treatment showed a 100% Mixed failure with Repressed specimens and a lower mixed failure 77.8% with the Pressed specimens.

**Table II** - Frequent distribution of failure modes scores (%)

Variables		Failure modes		
		Adhesive	Cohesive	Mixed
Pressed (P)	Subgroup PE	11.1	11.1	77.8
	Subgroup PS	100	0	0
Repressed (R)	Subgroup RE	0	0	100
	Subgroup RS	100	0	0



**Figure 6** - Stacked column chart comparing the frequent distribution of failure modes scores for both groups with both adhesive approaches.



**Figure 7** - Representative microscopic image showing (A) Adhesive, (B) Cohesive and (C) Mixed failure mode at 25x magnification.

**DISCUSSION**

The null hypothesis of the present study was rejected. A significant difference was found between the shear bond strength of acid-etched pressed and repressed groups. A significant difference was found between the shear bond strength of etched and air-abraded subgroups.

The bonding effectiveness of resin cement to an all-ceramic restoration has a significant impact on its strength. Thus, the cementation process plays a vital role in the clinical success of an all-ceramic restoration. It has been reported that some dental laboratories reuse the leftover material of previous heat pressing procedures to fabricate new restorations. [31,32] The effect of this procedure on the bond strength of resin cement to lithium disilicate glass-ceramics is unknown.

Bonded specimens were subjected to thermocycling in an attempt to stimulate the

aging process occurring in the oral cavity. There is a considerable variation in the number of thermal cycles and the temperature extremes between studies. In the present study, the number of cycles used was 5000 cycles simulating thermal change for approximately 6 months in the oral cavity. The low-temperature point was 5 °C, and the high-temperature point was 55 °C. [34,35]

Although many studies reported that there was a decrease in shear bond strength when the heat-pressed glass-ceramic surface was air-abraded with alumina oxide particles when compared with a heat pressed glass-ceramic surface etched with hydrofluoric acid. [26-30] Yet, air-abrasion was used in this study as its effect on repressed lithium disilicate glass-ceramics was unknown, and whether air-abrasion will affect the shear bond strength of the repressed ceramics was still to be investigated. Air-abrasion protocol used was the recommended by the manufacturer for conventional cementation of lithium disilicate glass-ceramics. Specimens with alumina oxide air-abrasion demonstrated the significantly lowest mean bond strength compared to those etched with hydrofluoric acid. This may be attributed to the inability of the air-abrasion to provide a mechanically retentive surface that is as efficient as etching with hydrofluoric acid.

Results of the present study showed that etching surface treatment recorded statistically higher shear bond strength than air-abrasion surface treatment with both tested groups (pressed and repressed). This goes in agreement with Guarda et al. [20], whose results showed that the etching procedure with hydrofluoric acid resulted in the highest bond strength with a statistically significant difference when compared with air-abrasion using alumina oxide particles.

The bond strength difference can be explained based on morphology created on both specimens, as confirmed by SEM pictures. Hydrofluoric acid etching caused the dissolution of the specimens glassy matrix. This dissolution

extended to the depth of a few microns that were enough to enable the protrusion of the lithium disilicate crystals from the glass matrix. Elongated crystals and shallow irregularities were observed. The change in the surface morphology of the specimens treated with hydrofluoric acid increased the surface area required for the penetration and retention of resin cement into the micro-retentions of the treated surface. [26-39] (RE) showed higher shear bond strength than (PE) specimens which highlights the effect of repressing and this result may be attributed to the more homogenized surface with the persistence of crystals in the (RE) than that of the (PE) specimens as seen in the SEM pictures.

Failure mode showed that all specimens treated with air-abrasion had a 100% adhesive failure, which is concurrent with our results that showed that air-abraded samples had much lower shear bond strength when compared with acid-etched specimens. All specimens treated with acid etching showed mixed adhesive and cohesive failures, which elaborate much stronger shear bond strength. This may be attributed to surface topography of air-abraded specimens seen in SEM images. The surface of the (PS) specimens was characterized by various deep grooves, valleys and irregularly shaped defects, while that of the (RS) specimens showed crater-shaped cavities that are larger in size and greater in number than that of PS specimens. These defects affect the wettability of the adhesive to the specimens surface and may entrap air that negatively affects the bond strength.

#### Limitations of the study

1- Discs samples were used to investigate the bond strength thus the effect of the significant increase in bond strength to repressed specimens on the final strength of lithium disilicate restorations is still unknown.

2- Only Shear bond strength was used and not microShear or microtensile test.

## CONCLUSION

1- Repressing the leftover buttons for the construction of new lithium disilicate glass-ceramic restorations has no adverse effect on the bond strength of the resin cement to the ceramic.

2- Hydrofluoric acid surface treatment improves the shear bond strength and durability of resin cement bond to both pressed and repressed lithium disilicate glass-ceramic.

3- Air-abrasion cannot be considered as a reliable surface treatment when bonding to lithium disilicate glass-ceramics.

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## Conflict of interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

## Regulatory Statement

This study was exempted from the ethical committee as it is an invitro study.

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