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Evaluation of the Effect of Various Cementation Protocols Used for 10% Zirconia-Reinforced Lithium Glass Ceramic Veneer on Shear Bond Strength to Resin Cement (An In Vitro Study)

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Abstract: Bonding failure between ceramic restoration and cement dramatically influences the success of resin-bonded ceramic restoration. This study evaluates the influence of various fitting surface treatments of 10% zirconia-reinforced lithium glass ceramic (ZLS) on its shear bonding strength to resin cement. Sixty blocks sized 8 × 8 × 2 mm^3 were cut from a ZLS. All specimens were fired for 10 min and separated into six groups according to surface treatment: GI (Without treatment), GII (10% HF acid, Ultra Sound water bath US, and silane coupling agent S), GIII (HF, US, S, and bonding without light cure B1), GIV (HF, US, 37% phosphoric acid PA, US, S, B1), GV (HF, US, S, and bonding with light curing B2), VI (HF, US, PA, US, S, and B2). The treated specimens were cemented to a resin cement cylinder of 3.5 mm in diameter and 2 mm in height (Variolink, Esthetic. Neutral). A universal test machine was used to test the shear bond strength (SBS) and SEM for failure mode. The result indicated that applying a bonding agent on the prepared surface of ZLS without curing before cementation significantly increases the shear bond strength and affects the failure mode. In contrast, the application of PA does not influence bond strength.

Keywords: shear bond strength; Celtra Duo; hydrofluoric acid; surface treatment; adhesive failure

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1. Introduction

Patients increasingly desire improvement of esthetic teeth with minimally invasive treatment and a decrease in the time between treatment steps to help decrease sensitivity and exclude the need for temporary restorations [1,2]. These factors have encouraged dentists and researchers to introduce more retentive esthetic materials and processing methods. CAD/CAM tools have allowed dentists to simplify laboratory steps and shorten their duration from a few weeks to a single day. Meanwhile, glass-ceramic restorations have demonstrated high strength and bonding ability to tooth structures [1,3].

Various factors act on the bond strength of the glass-ceramic to the tooth structure, such as the luting cement, adhesive, fitting surface preparation, and cutting depth in the tooth structure [4–6]. Bonding of ceramic to the tooth substance is based on the adhesion of luting resin to the ceramic substrate on one side and enamel and dentin on the other, which requires a sequence of procedures. The procedure starts with the preparation of the fitting surface of the glass-ceramic with hydrofluoric acid (HF) and the application of a silane (S) coupling agent, followed by the tooth surface preparation with 37% phosphoric acid, bonding application. Finally, resin cement is applied, and the glass-ceramic restoration is fitted under controlled pressure. The first step of this process is selectively dissolving the glassy matrix of ceramic by etching with hydrofluoric acid, followed by colonization [4]. Because HF is a weak acid, the hydrogen fluoride in the water is not entirely ionized. Its conjugate base, the fluoride ion, can re-associate to form HF in solutions with low pH [7] Energy Dispersive X-ray Spectroscopy (EDS) analyses of etched porcelain surfaces with HF acid show that precipitates such as Na, K, Ca, and Al remain on the surface after acid application [8]. Eliminating excess acid and acid precipitates from prepared porcelain surfaces enhances their bonding strength to resin cement [5] In addition to HF etching to glass-ceramic to improve the bond strength to resin cement, different available surface treatments have been investigated, such as roughening with burs, air-particle abrasion with aluminum oxide, laser, and etching with acids [9,10]. Cleaning the HF-etched ceramic surface ultrasonically (US) in distilled water has increased the shear bond strength compared with rinsing it with air-water spray only or using alcohol or acetone [5]. A study that attempted to neutralize the HF precipitate found that this seemed to reduce the bond strength between the dentin and glass-ceramic [11].

Meanwhile, a 1070 nm fiber laser can be considered a suitable device to increase the adhesion of lithium disilicate ceramics when optimum parameters are considered [9]. A study of the influence of the active and passive application of phosphoric acid (PA) on the SBS of lithium disilicate to resin cement indicated that the active application of 37% PA after 9.6% HA increases the micro shear bond strength [12]. In addition, silane application followed by laser treatment as a combination surface treatment was feasible and efficacious for chairside porcelain repair [10]. ZLS, especially the fine-grained form, has been found to have some properties and indications comparable to lithium disilicate-based ceramics. ZLS ceramics have been recently used as materials for hot press and CAD/CAM techniques. Fully crystallized glass ceramic Celtra Duo, DeguDent GmbH/Germany (ZLS), according to the manufacturing company, is mainly composed of 58% silica, lithium metasilicate, disilicate, and phosphate crystals, and 10% zirconia crystals in addition to other minor oxides and ingredients that can be cemented directly after milling with simple polishing and with or without heating for 10 min help to decrease treatment time [13,14]. Due to its composition, ZLS offers a combination of excellent optical properties and high fracture strength above 350 MPa [10,15,16]. The surface treatment methods of glass ceramics are paramount for proper adhesion [17,18]. Preceding studies investigated the bonding strength with full-ceramic restorations by changing the surface properties of ceramic materials. Although comparative studies showed the advantages of various types of surface-conditioning methods on different ceramics surface treatments, there is no consensus in the literature regarding the best surface-conditioning method to produce optimal bond strengths [8,9,12,19–22]. Therefore the objectives of this study were to evaluate the effects of the adhesive application with light curing and without light curing on the etched and slanted (ZLS) glass-ceramic on SBS and the mode of failure of the resin cement. The following hypotheses were tested: (a) application of bonding agent to the slanted surface (ZLS) and light curing increases bond strength; (b) 37% phosphoric acid application and ultrasonic cleaning of the (ZLS) glass-ceramic after HF etching have more effect than cleaning with air-water spray on enhancing bond strength.

2. Materials and Methods

Materials used in this study and samples grouped according to ceramic fitting surface treatment were listed in Tables 1 and 2.

Table 1. Materials used in this study.

Material	Exp. Date	Manufacturer
Celtra Duo block HT A1 C14	1 November 2033	DeguDent GmbH/Germany
N- Etch. Etching Gel 37% Phosphoric acid	24 March 2023	Ivoclar Vivadent/Liechenstein
Tetric N-Bond Universal	10 July 2022	Ivoclar Vivadent/Liechenstein
Monobond N	15 October 2022	Ivoclar Vivadent/Liechenstein
Variolink Esthetic neutral light-curing cement	9 January 2023	Ivoclar Vivadent/Liechenstein
Condac porcelain, Hydrofluoric acid 10%	12 May 2023	FGM, Joinville, SC, Brazil

Table 2. Treatments of ZLS were applied to study groups.

Group Name	Type of Ceramic Fitting Surface Treatment	Bonding Application Method
G1	Without treatment (control group)	-
GII	HF acid, US, and coupling agent only	-
GIII	HF acid, US, silane coupling agent, and bonding	Light curing the bonding on the ceramic fitting surface after application of resin cement cylinder
GIV	HF acid, US, 37% phosphoric acid, US, coupling agent, and bonding agent	
GV	HF acid, US, coupling agent, and bonding	Light curing the bonding of ceramic fitting surface for 15 s before the application of resin cement cylinder
GVI	HF acid, US, 37% phosphoric acid, (US), coupling agent, and bonding agent.	

2.1. Preparation of the Specimens

A pilot study was made to determine the number of specimens that would be required for the test group, a power analysis was calculated using GPower software version 3. Considering six groups, one-way analysis of variance (ANOVA), an effect size of 0.604 for, a sample of 10 specimens per each group was indicated to detect significant differences [23].

Sixty rectangular blocks, 8 × 8 × 2 mm^3 in size, were cut from Celtra Duo block HT A1 C14 (lithium silicate reinforced by 10% Zirconia CAD/CAM blocks, DeguDent, Hanau, Germany) using a diamond disk (Isomet 1000, Buehler; Lake Bluff, IL, USA) at low-speed rotation under water cooling, then fired without adding any glaze materials by CEREC SpeedFire Dental Furnace Dentsply for 10 min. After the cut was made, all the blocks' surfaces were investigated with a stereoscopic magnifying X40 (OMAX 20×–40× Binocular Stereo Microscope) to check for probable cracks and fractures to exclude them from the specimens. Next, the selected sound blocks were embedded in acrylic resin using a plastic ring mold 10 mm in diameter and 20 mm high, leaving one surface uncovered. The exposed surface of the ZLS block was polished using standard silicon carbide sandpapers, and the disk was polished with decreasing grit (#600, #800, and #1200) under water cooling as shown in Figure 1A. Subsequently, the block surfaces were submitted to an ultrasound bath (Ultrasonic Cleaner Easy Home) in distilled water for 60 s. According to the manufacturer's recommendation, all ZLS fitting surfaces of the specimens were etched with hydrofluoric acid 10% for 20 s as shown in Figure 1B, and rinsed with distilled water for 60 s under ventilation and protective measures next submitted to an ultrasound distilled water bath for 60 s.

2.2. Grouping the Sample

The specimens were divided randomly into six groups (n = 10) according to the fitting surface preparation sequences shown in Table 2. After that, 37% phosphoric acid N-Etch Etching Gel was applied, without rubbing the surface (passive mode), to GIV and GVI for 30 s only as shown in Figure 1C [12]. All samples were submitted to an ultrasound bath in distilled water for 60 s and dried before the application of the coupling agent. According to the manufacturer's instruction, the coupling agent (Monobond N silane, Ivoclar Vivadent; Schaan, Liechtenstein) was applied to the etched surface for all specimens and was left for the 60 s, again according to the manufacturer's instruction, then dried with oil-free air pressure as shown in Figure 1D. Tetric N-Bond Universal agent was applied to G (III, IV, V, and VI) specimens and GV and GVI only were light cured for 15 s before the application of resin cement cylinder whereas the GII and GIV light curing the bonding on the ceramic fitting surface after application of resin cement cylinder. Tetric N-Bond Universal contains methacrylates, ethanol, water, highly dispersed silicon dioxide, initiators, and stabilizers.

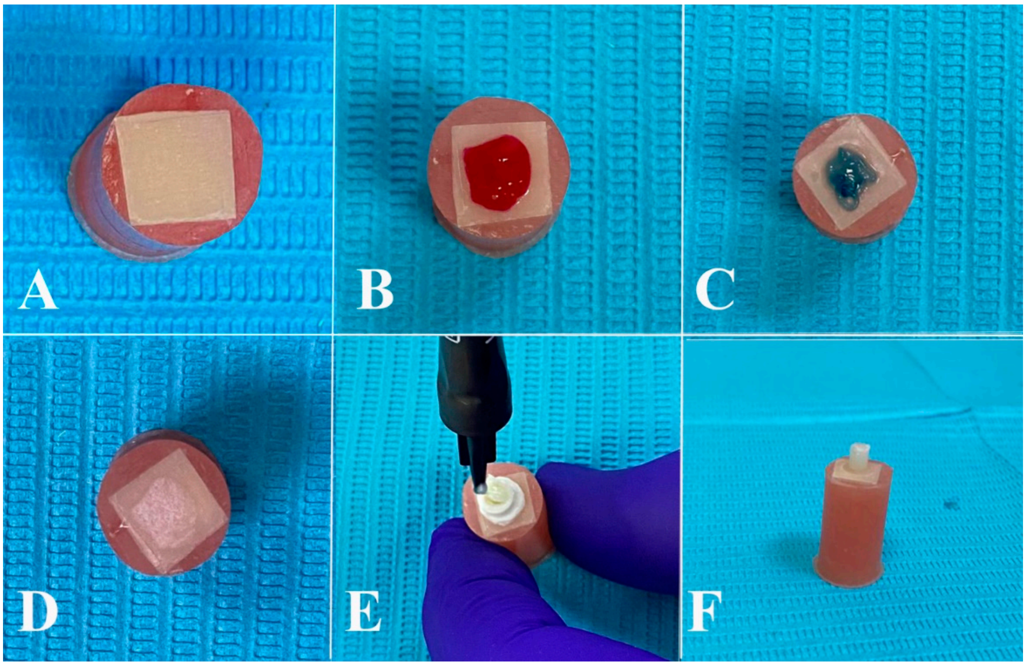

Figure 1. Steps of sample preparation: (**A**)—ZLS block was polished, (**B**)—HF acid application, (**C**)—37% phosphoric acid application, (**D**)—adhesive applied after washing in US and dryness, (**E**)—resin cement poured in the mold, (**F**)—final sample.

2.3. Shear Bond Strength Test (SBS)

For the SBS test, 3.5 mm in diameter and 2.5 mm in height cylinders were fabricated from luting resin (Variolink Esthetic neutral light-curing cement Ivoclar Vivadent; Schaan, Liechtenstein) using a transparent plastic mold with a perforation in the center. The luting resin was injected into the mold in 2 mm increments and light cured (Hy-G31 Light cure) for 40 s on four sides of the resin cement; the steps and final sample shape are shown in Figure 1E,F. After carefully removing the excess resin cement with disposable applicators (KG Brush), all specimens were tested after thermocycling (conditions: 5000 times, 5–55 C, 30 s). The SBS of luting cement to ceramics was measured using a universal testing machine (TERCO MT 3037) at 0.5 mm/min speed on the surface at the resin/ceramic interface, as shown in Figure 2. The load value until failure, determined in MPa, was recorded, and statistical analysis was performed.

2.4. Failure Mode Investigation

The failure modes were evaluated through a stereo microscope X40 (OMAX 20×–40× Binocular) and divided into

1. Adhesive failure: fracture between ceramic and resin.
2. Cohesive failure: include internal fracture of ceramic or resin cement.
3. Mixed failure: include adhesive with cohesive in the ceramic or resin cement.

Two representative specimens from each group were sent to the scanning electron microscope (SEM) to analyze the failure mode.

2.5. Statistical Analysis

The data were tested for normality and homogeneity of variance using Shapiro-Wilk Statistic and Levene tests before further statistical analysis (SPSS v 26.0 software for windows, SPSS; Chicago, IL, USA). Bond strength results (MPa) were analyzed using one-way ANOVA. Tukey's HSD post hoc test was used for multiple comparisons.

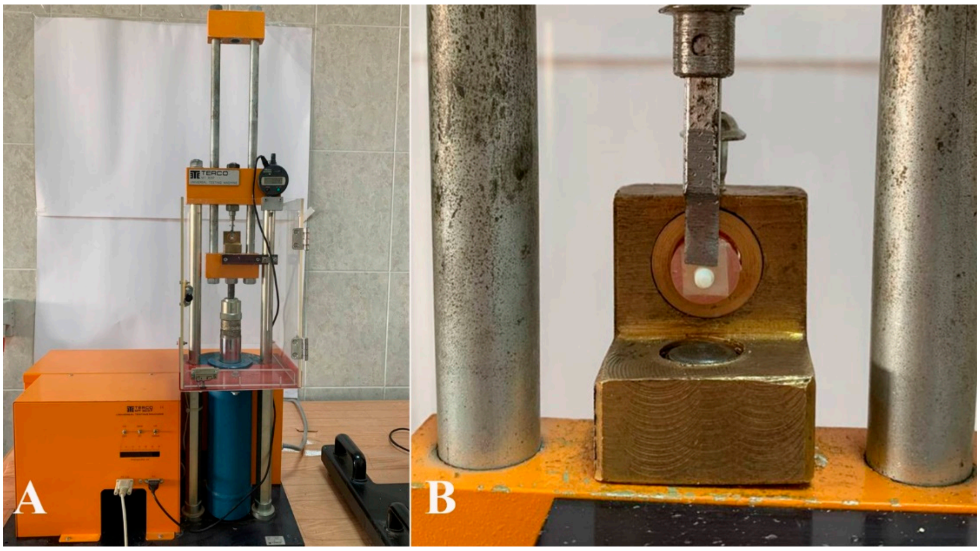

Figure 2. (**A**). Testing the SBS of the sample with the universal testing machine, (**B**). The position of the load application for SBS test.

3. Results

The Shapiro–Wilk test verified the normality of data, and Levene's test of equality indicated homogenous distribution of bond strength values among all tested groups. The result of one-way ANOVA indicated a statistically significant difference between the tested groups, as shown in Table 3. SBS results in Table 4 and Figure 3 show a significant difference between all groups and GI, and between GII and GIII. The results also indicated no significant difference between GII and GIV, GV, and GVI, or between GIII and GIV.

Table 3. One-way ANOVA testing results across the studied groups.

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	577.978	5	115.596	27.438	0.000
Within Groups	227.502	54	4.213		
Total	805.480	59			

Table 4. The mean values with standard deviations for each group's shear bond strength.

Groups	Mean ± St. Deviation
GI [a]	2.62 ± 1.07
Gil [b]	9.14 ± 2.108
GIll [c]	12.64 ± 2.67
GlV [bc]	10.89 ± 2.45
GV [b]	9.35 ± 1.905
GVl [b]	9.36 ± 1.69

Identical superscript letters indicate no significant difference (Tukey's test, $\alpha \leq 0.05$).

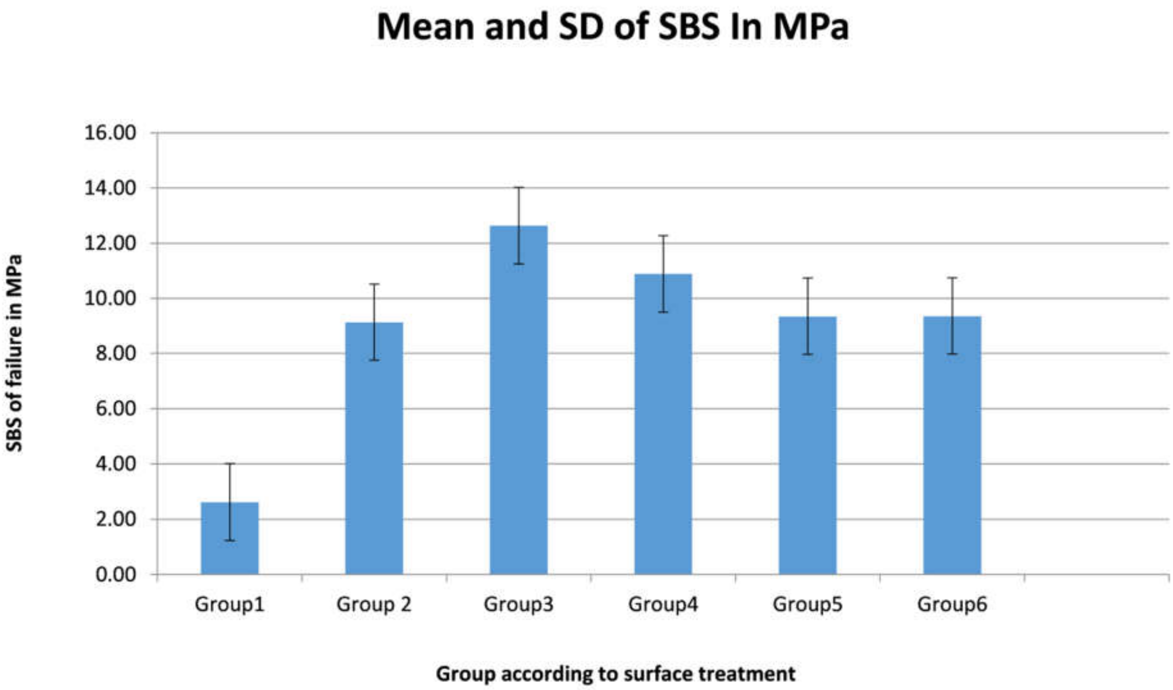

Figure 3. The mean values and SD of the shear bond strength for each group.

Figure 4 shows that in respect of the mode of failure between the ceramic and luting resin, the adhesive failure was more frequent in G (I, II, V, and VI) compared with G (III and IV), which showed more cohesive failure and mixed failure. The mode of failure results showed one dislodgment of the ceramic from the acrylic mold in G (I, IV, V, and VI), while there were two in GIV. Figure 5 shows a sample of adhesive, cohesive, and mixed failure modes for representative specimens investigated with SEM.

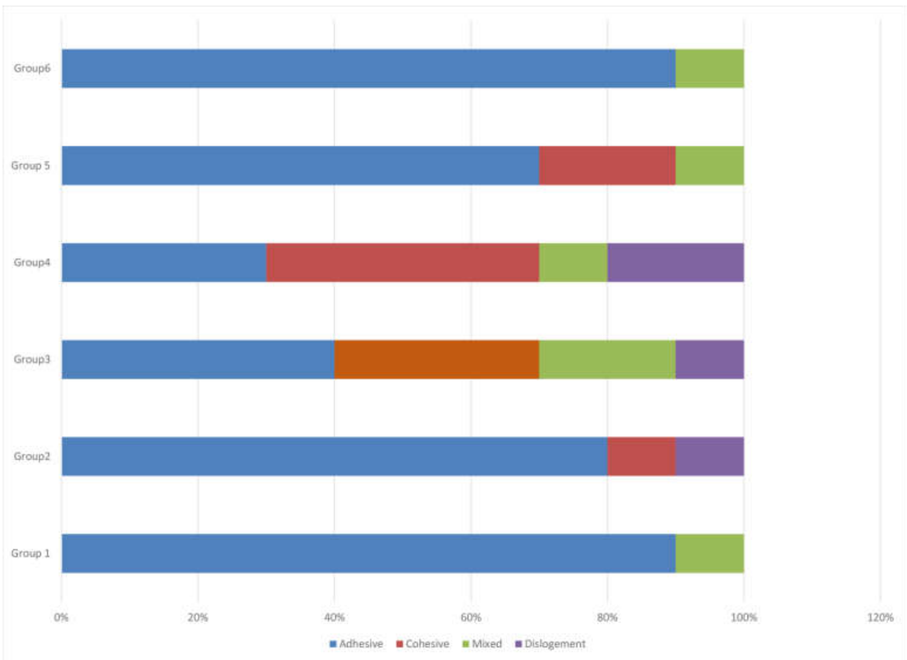

Figure 4. Types of failure between luting resin cement and the Celtra Duo glass ceramic.

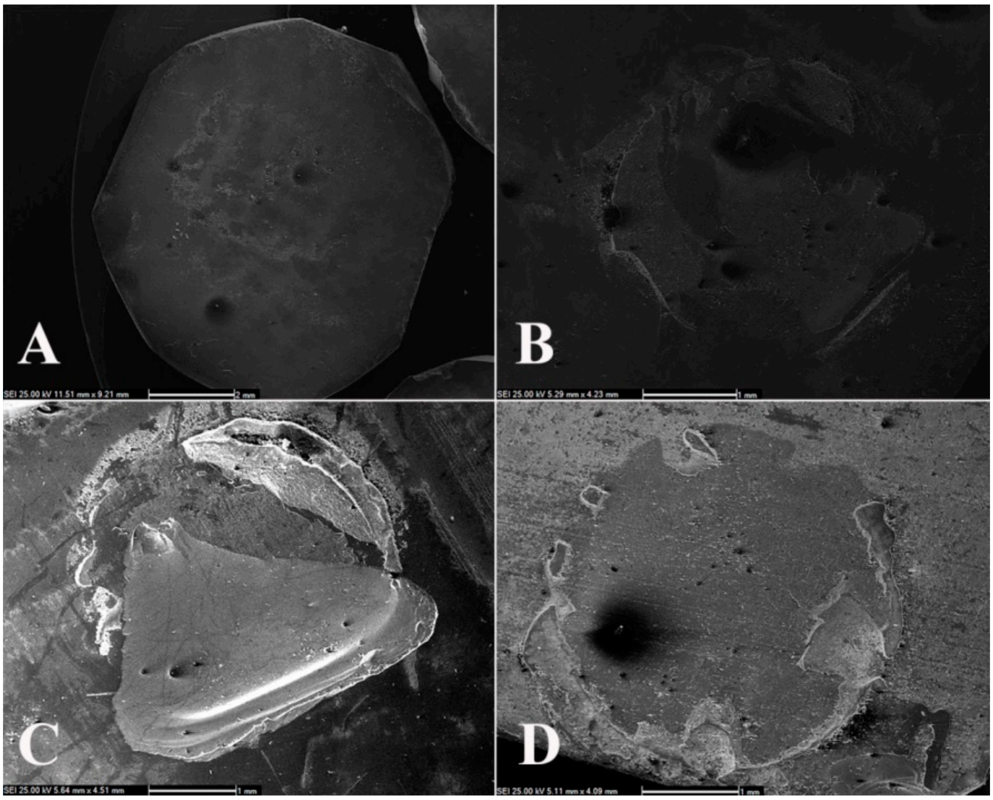

Figure 5. Scanning electron microscope image shows (**A**) adhesive between ceramic and resin cement, (**B**) cohesive in ceramic, (**C**) cohesive in resin cement, and (**D**) mixed failure.

4. Discussion

Treating the ceramic surface to increase surface roughness is a key to the success and prognosis of the bond strength between ceramic and resin luting cement, which may increase the bond strength between the ceramic surface and the resin cement [24–26]. The recently introduced fully crystallized glass ceramic, ZLS, which can be cemented directly after milling with simple polishing or heating for 10 min, has also helped to decrease treatment time. ZLS, after glazing and firing, approximates enamel's mechanical properties in terms of wear resistance [27]. In this study, the Celtra Duo was selected as a type of ZLS glass-ceramic to test its ability to bond to the luting resin cement using different methods to treat the fitting surface. Treatment with 10% HF acid for 20 s and washing with distilled water and ultrasound was applied to all groups except the control group. Higher SBS was recorded with the ultrasound cleaning the etched glass ceramic surface with hydrofluoric acid, resulting in a surface without fluorosilicate precipitate [22]. The Tetric N-Bond Universal was used with the application of saline separately, according to the manufacturer's recommendation used in this study. Silane coupling agents are widely used for unifying dissimilar materials [27]. In in vitro studies, thermal cycling has been performed to mimic intraoral temperature changes. All ceramic samples prepared were subjected to 5000 thermal cycles before the SBS test [28].

Failure of the shear bond is considered one of the common causes of failures of veneer restorations. Bonding failure of a veneer to the tooth structure can be related to many reasons, including cutting depth in the enamel, tooth surface preparation, and ceramic fitting surface preparation, in addition to the light cure type and resin luting cement used [2,4–6]. In this study, shear bond failure, rather than tensile failure, which is more common with other types of restoration, such as Inlay and Onlay, was therefore selected for testing the bond strength [29].

The results indicated that the application of adhesive after silane application on the etched ceramic surface without light curing increases the bond strength significantly compared with curing the adhesive before cementation; these results agree with the study by Murillo-Gomez F et al., who found that the application of silane followed by an adhesive system application can improve ceramic/resin cement bond strength for both short- and long-term water aging. However, it was applied for a different reason [30,31]. This finding may be due to more time being allowed for integration between resin cement and flowable bonding before light curing [32]. The results also indicated that the application of 37% phosphoric acid to clean off the HF acid residue does not increase the SBS strength. The use of phosphoric acid did not completely remove the residues of fluorine deposited on the specimen's surface, verified by the SEM images and the EDS test, which detected the presence of F on the surface. Despite the remaining, a small percentage of F could have influenced the SBS [21,22].

Moreover, low SBS may be due to the action of phosphoric acid application that degraded the saline layer that was not completely eliminated with the US. This finding agrees with Giraldo TC et al. (2016), who investigated the active and passive application of 37% phosphoric acid to the etched lithium disilicate glass-ceramic with HF acid on shear bond strength. They found that the active application of phosphoric acid increased the shear bond strength; however, the passive application had no effect [12]. The results of failure mode between Celtra Duo ZLS and resin cement showed a high percentage of adhesive failure in G (I, II, V, VI), whereas adhesive failure was less frequent in G (III and IV). In addition, there was a higher incidence of the cohesive and mixed types in the G (III and IV), which can be related to the high SBS in these two groups without curing the adhesive before cementation. This study rejects the first hypothesis, curing the adhesive before cementation increases the SBS, and partially rejects the second hypothesis. While applying US with distilled water increased the SBS, washing the Celtra Duo etched surface with HF followed by 37% phosphoric acid in a passive mode did not increase the SBS. Within the limitations of this study, the clinical significance is that the application of adhesive to the treated ceramic surface with HF and silane without curing can increase the SBS. This cementation protocol needs to be supported by studying the influence of curing the adhesive or without curing before the cementation of the veneer on the cement gap and microleakage at the margin.

5. Conclusions

The results indicate that applying a bonding agent on the prepared surface of Celtra Duo glass ceramic (ZLS) with HF and S without curing before cementation increases the shear bond strength and affects the failure mode.

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