Effect of different surface treatments on bond strength of different resin cements to lithium disilicate glass ceramic: an in vitro study

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ABSTRACT
The aim of this study was to evaluate the bond strength of different resin cements in lithium disilicate glass ceramics on which different surface treatments are used. For this study, 120 unit IPS e.max Press samples were prepared for the bond strength test. Samples were randomly divided into six groups. The following treatments were applied on ceramic surfaces: (1) control (no surface treatment), (2) hydrofluoric acid (HF), (3) sandblasting (Al₂O₃ powder), (4) Co-Jet (silica-coated Al₂O₃ powder), (5) Er:YAG (erbium-doped yttrium aluminium garnet) laser and (6) Nd:YAG laser (neodymium-doped yttrium aluminium garnet). SEM (scanning electron microscopy) was performed on one sample from each group. Dual-cure resin cement was stuck on half of the samples, and self-cure resin cement was stuck on the other half of the samples, and shear strength was applied until breakage occurred. The data was analyzed with variance analysis (ANOVA). According to the results from the bond strength tests, dual-cure and self-cure resin cements showed statistically significantly higher bond strength in the samples on which Co-Jet was applied, while the least bond strength was observed in the samples in the control group. Additionally, self-cure resin cement showed significantly higher bond strength values than dual-cure resin cement. Different surface treatments affect the bonding of different resin cement to IPS e.max Press.

Introduction
Dental ceramics have superior features such as chemical stability, biocompatibility, low thermal conductivity, high pressure resistance, translucency and fluorescence [1]. Lithium disilicate glass ceramics (LDC) are a new generation of heat-pressure ceramics that show higher fracture strength and bending resistance than the first generation of ceramics reinforced by leucite [2]. When compared with feldspar-containing ceramics, the presence of LD crystals increases the durability and longevity of the ceramic material [3]. These features enable the inlays, onlays, laminate and implementation of three-tooth bridges extending to the second premolars [4]. In general, the LDC microstructure is composed of two components: silica, which serves as a glass matrix, and lithium oxide (Li₂O), which serves as a flux to decrease the increasing temperature of the glass matrix that is raised approximately to 2000 °C from 1100 °C [5].

Advances in adhesive dentistry have resulted in the investigation of different surface treatments to achieve high bond strengths (BS). As a ceramic surface treatment, methods such as sandblasting, acid etching and silica coating are suggested [6]. The application of a silane coupling agent after ceramics are etched with acids at different concentrations is one of the known and recommended methods to increase BS [7].

Etching with hydrofluoric acid (HF) selectively dissolves surfaces that contain a glass matrix and crystals [8]. It also helps to increase the surface energy for the implementation of acidified surface silane agent [8,9]. As a result, the chemical bond between the inorganic matrix and organic matrix of resin cement is improved [10].

Modification of the LDC surfaces after different surface treatment affects the shear BS to resin cement [11]. In addition to existing ceramic surface treatment applications, laser irradiation of ceramic surfaces is also investigated. However, there is limited literature regarding the laser application on dental ceramics [12]. During the laser treatment, the exaggerated temperature changes in the heating and cooling phase damage the material by creating internal tensions in the ceramic; therefore, appropriate laser parameters must be applied [13].

The clinical success of ceramic restorations depends on many different factors, including cement survival time [1]. In vitro studies performed on the cementation of the restorations made from LDC have reported that bonding with resin cement shows stronger BS and increases its fracture resistance [14–16]. Depending on

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the indication, dentists need to choose resin-based cements with different bonding strategies. Resin cements are recommended for several reasons, including low solubility in the oral environment, an ability to reduce micro leakage between the tooth and the restoration, favourable optical properties, marginal discoloration and a low recurrent decay rate [17].

There are limited studies regarding laser application in the evaluation of bonding of LDC with resin cement. The difference between traditional surface treatment and laser application has not been fully evaluated. The null hypothesis of this study is that laser irradiation is an alternative method to other methods for resin cement bonding of LDC. The aim of this study was to evaluate the effect of different surface treatments on the BS between LDC and different resin cements.

**Materials and methods**

The materials used in this study are listed in Table 1. For this study, 120 units of IPS e.max Press with a 10-mm diameter and 2-mm thickness were prepared in the laboratory and in accordance with the manufacturer’s instructions. All of the specimens prepared were embedded in autopolymerized acrylic resin (in silicone molds with a 15-mm diameter and 20-mm height) to fit the test device in which the test was performed.

Polishing and finishing discs (Sof-Lex; 3M ESPE, Neuss, Germany) were used, respectively, to ensure that the specimens would be completely flat and smooth. Ceramic specimens were cleaned of residuals by keeping them in an ultrasonic cleaner for 15 min and then made ready to be applied as surface treatments. The specimens were then randomly divided into 6 groups, with 20 specimens per each group. Different surface treatments were used for each group. The surface treatments applied on the specimens were as follows:

1. **Control group (C):** No surface treatment was applied on the ceramic specimens in this group.
2. **Acid etching (HF):** 9.5% HF (Bisco, Schaumburg, USA) was applied on 20 specimen pieces of ceramic for 120 s. The specimens were washed with pressurized water for 20 s to remove the acid from the specimens and were then dried.
3. **Sandblasting (SB):** 50-μm Al₂O₃ sand (Aluminum oxide; Dentsply, Bohemia, USA) was applied with a fine-tipped sandblasting device on 20 ceramic specimens under atmospheric pressure of 2.8 atm, from a 1-mm distance, and this was done for about 15 s. Sandblasting was performed by a single person, on the whole surface and from different angles. Following sandblasting, the specimens were washed for 30 s and then dried.
4. **Sandblasting process with silica-coated aluminum oxide powder (SC):** 30-μm silanized Al₂O₃ sand (CoJet™ Sand; 3M ESPE, Neuss, Germany) was applied with a fine-tipped Co-Jet sandblasting device on 20 specimen pieces of ceramic, under atmospheric pressure of 2.8 atm, from a 10-mm distance, for about 15 s. Sandblasting was performed by a single person, on the whole surface and from different angles. Following the surface treatment, no cleaning was performed on the surface of the specimen in order not to destroy the silanization that was obtained.
5. **Er:YAG laser application (ER):** A water-cooled laser was applied on the surfaces of 20 ceramic specimens at 400 mJ, 20 Hz, 8 W power output, in the short pulse mode, using an Er:YAG laser unit (Smart 2940D PLUS Deka, Serial No. FA9A41041AQ, Italy). The application was performed with a focal distance of 1 mm for 40 s on each specimen.
6. **Nd:YAG laser application (ND):** A water-cooled laser was applied on the surfaces of 20 ceramic specimens at 100 mJ, 20 Hz, 2 W power output through an Nd:YAG laser unit (Smartlife, DEKA, Serial No. FA9A41041AQ, Italy). The application was performed with a focal distance of 1 mm for 40 s on each specimen.

A specimen from each group was selected after surface treatment for the purpose of SEM analysis (Inspect S50, FI Company Inspect™, USA). The specimens in each group were divided into two equal groups: dual-cure

### Table 1. Materials used in this study.

| Brand name          | Type                              | Manufacturer                      | Batch No.  |
|---------------------|-----------------------------------|-----------------------------------|------------|
| IPS e.max Press     | Lithium disilicate glass ceramic  | Ivoclar Vivadent, Liechtenstein   | U28244     |
| S.C. Self cure      | Autopolymerizing acrylic resin    | Imcryl, Turkey                    | 14123      |
| Sof-Lex             | Extra thin contouring and polishing discs | 3M ESPE, USA                  | N357198    |
| Bisco, Porcelain Etchant (9.5%) | 9.5% Buffered hydrofluoric acid gel | Bisco, USA                      | 1100008120 |
| Aluminium oxide     | 50-micron Al₂O₃ sand              | Dentsply International Inc., USA  | 16194      |
| CoJet™ Sand         | Blast-coating agent 30 μm         | 3M ESPE, Germany                  | 477110     |
| Panavia F 2.0       | Dual-cured resin cement           | Kuraray Med. Inc., Japan         | 041332     |
| Multilink® N        | Self-curing resin cement          | Ivoclar Vivadent, Liechtenstein  | 500630     |
| Elite-HD Putty Soft | Silicone impression material       | Zhermack, Italy                   | 73518      |
| Panavia F 2.0 Oxyguard II | Liquid oxyguard                  | Kuraray Noritake Dental Inc., Japan | 00665A    |
Results and discussion

In the present study, the difference between the BS values of self-cure and dual-cure resin cements was found to be statistically significant ($p < 0.01$), while the total BS value of self-cure resin cement ($21.62 \pm 7.72$ MPa) was observed to be much higher than that of dual-cure resin cement ($16.23 \pm 5.49$ MPa) (Tables 2 and 3). It was observed that different surface treatments applied on LDC significantly affected its bonding with resin cements. The differences between the average BS values with different surface treatments were found to be statistically significant ($p < 0.01$). Among the six different surface treatment processes, group SC had the highest BS value ($28.73 \pm 5.07$ MPa), and group C had the lowest BS value ($10.63 \pm 2.05$ MPa) (Table 3). The most effective surface treatments for LDC were SC and HF, whereas, contrary to the hypothesis, laser irradiation was not an effective alternative method. However, laser irradiation still gave significantly high BS values in the control group, without demonstrating a negative effect on BS. LDC does not interact with the laser beam because of its high reflectivity. Therefore, the application of graphite powder increases the effect of the laser beam [18]. It is possible that if graphite powder is used, the results could be different.

Besides the differences between the studied groups, the interaction between either dual-cure or self-cure resin cements and surface treatments was found to be statistically significant ($p < 0.01$). The highest BS values were obtained in group SC, followed by group HF, group SB, group ER and group ND ($p < 0.01$). When the overall BS strength values of the IPS e.max Press specimens were considered, the same trend was observed in the effects of the tested surface treatments. The clinical success of ceramic restorations depends on the adhesive cements and the cementation process [19]. Therefore, the determination of BS of the ceramic material is of great importance when evaluating the success of the restoration [1]. The shear bond strength test was used to evaluate the BS between LDC and resin cements because this testing method is advantageous in terms of involving an easy-to-apply procedure and giving reliable results [20]. The durability of ceramic restorations is affected by the pre-treatments applied on the crown and the bridge, and the bonding

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Table 2. Variance analysis results of bond strength values.

| Variation                  | Df | MS     | F      | p     |
|----------------------------|----|--------|--------|-------|
| Cement                     | 1  | 870.65 | 237.52 | 0.00  |
| Surface treatment          | 5  | 949.05 | 258.91 | 0.00  |
| Cement × surface treatment | 5  | 29.90  | 8.16   | 0.00  |
| Error                      | 108| 3.67   |        |       |

Df, degrees of freedom; MS, mean square ($p < 0.01$).

Table 3. Bond strength values (MPa) within the groups.

| Group                        | C $\pm$ SD | HF $\pm$ SD | SC $\pm$ SD | SB $\pm$ SD | ER $\pm$ SD | ND $\pm$ SD | Total $\pm$ SD |
|------------------------------|------------|-------------|-------------|-------------|-------------|-------------|----------------|
| Dual-cure resin cement       | 9.01 (1.34)| 21.07 (1.73)| 24.20 (1.97)| 17.17 (1.61)| 13.59 (1.79)| 12.34 (1.74)| 16.23 (5.49)   |
| Self-cure resin cement       | 12.44 (1.12)| 28.57 (1.85)| 33.25 (2.23)| 22.61 (3.56)| 17.72 (1.33)| 15.32 (1.55)| 21.62 (7.72)   |
| Total                        | 10.63 (2.05)| 24.82 (4.22)| 26.73 (5.07)| 19.89 (3.87)| 15.65 (2.62)| 13.83 (2.22)| 18.93 (6.70)   |

*Mean values with standard deviation ($\pm$SD). Means shown with different letters are statistically different from each other ($p < 0.01$).
method [21]. In order to provide adequate stability, to increase the fracture resistance of the restoration and the bridge, and to prevent post-operative sensitivity, full ceramic restorations should be bonded to the tooth structure with resin cements because of their superior mechanical, physical, and aesthetic properties [22–24]. The fracture resistance of full ceramic restorations increases when they are bonded with resin cements, because resin cements block the progression of cracks by filling the cracks and irregularities in the interior surface of the restoration [25,26].

In the study performed by Spohr et al. [27], in which the effects of alternative ceramic surface treatments on the BS in the bonding of RelyX ARC resin cement to IPS Empress 2 ceramic discs were investigated, it was reported that the application of 10% HF acid and silane created the highest BS compared to sandblasting only, sandblasting and silanization and HF application only. These findings are supported the results from the present study.

An evaluation of the bonding of feldspathic ceramic with resin cements has demonstrated that the BS of each specimen’s surface after sandblasting with 50-μm Al2O3 powder is similar to the BSs of the specimens treated with Er:YAG and Nd:YAG lasers [28]. In the present study, in contrast to feldspathic ceramic, the SB group had higher BS values than those achieved following Er:YAG and Nd:YAG laser irradiation for the bonding of IPS e.max Press. The results from the present study regarding the bonding between IPS e.max Press and resin cement based on different surface treatments are in agreement with Guarda et al. [1], who reported that etching with 10% HF shows higher BS values than the sandblasting with 50-μm Al2O3. Other authors, Salvio et al. [29], Spohr et al. [27], Ayad et al. [30] and Attia [31], have also reported that sandblasting with 50-μm Al2O3 causes a decrease in BS as compared to etching with 10% HF acid. Maruo et al. [32] demonstrated that, when evaluating the surface morphology and adhesiveness of LDC, etching with 5% HF acid or phosphoric acid yielded higher shear BS than sandblasting between LDC and self-cure adhesive resin cement. In line with these reports, in the present study, higher resin cement BS values were determined in the IPS e.max Press specimens etched with HF acid compared to the specimens sandblasted with 50-μm Al2O3.

Giraldo et al. [33] observed that the active application of phosphoric acid after 9.6% HF acid increases the microshear BS values between the resin cement and LDC. In the study of Lise et al. [34], HF acid etching showed higher microshear BS values than those observed in the control group.

In addition to the traditional surface treatments that were used to increase the BS between the ceramic surface and resin cement, in the present study, we aimed at evaluating the effect of laser irradiation on BS; however, there have been few studies on laser irradiation [7,32]. The results obtained in the present study are in agreement with Kursuoglu et al. [11], who reported that laser irradiation led to higher BS in the bonding between IPS Empress II and resin cement compared to a control group but to lower BS compared to that achieved through acid etching. Additionally, the high laser power output appears to weaken the bonding between full ceramic restoration and resin cement [31].

To further explore the effect of surface treatments on the surface of IPS e.max Press, we performed SEM analysis. The SEM images at ×2000 magnification revealed a very smooth surface of the specimen in control group.

![SEM images: control (a), acid etching (b), sandblasting (c), Co-Jet (d), Er:YAG laser (e), and Nd:YAG laser (f).](image-url)
Although the SEM images of the surfaces of the specimens in group ER and group ND provided similar images to the specimen in the control group, too few linear retentive areas were observed. Uniform and regular retentive areas in the SEM image of the specimen surface were observed in group HF. Various irregular retentive areas that were similar to each other were observed in the SEM images of groups SC and SB (Figure 1). These observations support the results from the SB analysis.

The present study, however, has a limitation in that graphite powder was not used, which would probably have increased the effect of the laser beam. Furthermore, different laser power outputs need to be evaluated in future, which, together with application of graphite powder, could potentially identify more optimal treatment conditions.

Conclusions

Within the limitations of the present investigation, Co-Jet application on IPS e.max Press was shown to be important for increased BS to the tested resin cements. HF acid etching showed higher BS values than sandblasting. Laser application had no negative effect on the BS of IPS e.max Press between resin cements, but it also increased the BS, though at too low a level to be considered significant. On the other hand, self-cure resin cement had higher BS values than dual-cure resin cement in the bonding of IPS e.max Press. Based on these results, in dental practice, Co-Jet application or HF acid etching could be considered preferable in cementation of LDC restoration to increase the bonding of resin cement. Furthermore, self-cure resin cement could be recommended in cementation of LDC restorations.

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Disclosure statement

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