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Original Research Article

Influence of the conditioning technique of a lithium disilicate vitroceramic

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Abstract

Introduction: Lithium disilicate reinforced glass ceramics are materials that require good adhesion to ensure clinical success. **Objective:** To evaluate the bond strength of resin cements to lithium-disilicate-enhanced vitroceramics using different cleaning techniques of recently conditioned ceramics. **Material and methods:** Twelve ceramic discs (IPS Empress II) were made and inserted into PVC pipes using acrylic resin. The ceramic surface was designed and submitted to a 10% hydrofluoric acid conditioning process for 20s. Then, the specimens were divided into 3 groups (n = 16) according to the cleaning techniques of recently conditioned ceramics: 1) control - conventional technique (no cleaning after the conditioning process); 2) application of 37% phosphoric acid for 20s; 3) 90% ethyl alcohol bath in a ultrasound tub for 4 min. After cleaning, the silane agent was applied for 1 minute and silicon matrices (1 mm in diameter x 1 mm in height) were made for further application of the resin cement (Vitique, DMG), which was handled according to the manufacturer's instructions. Four cylinders were prepared on each ceramic surface. The specimens were stored in distilled water for 48 hours and subjected to the micro-shear test in a universal testing machine. After the micro-shear test, a failure analysis of the specimens was performed. Data were submitted to ANOVA (analysis of variance for a criterion) with a significance level of 5%. **Results:** There was no significant difference between groups ($p > 0.05$). **Conclusion:** the cleaning technique of the recently conditioned ceramics does not interfere with the resin/cement bond strength values.

Introduction

Dental ceramics have become very popular for presenting favorable aesthetic characteristics, such as the ability to mimic dental structure [14]. Among the various materials, vitroceramics enhanced with lithium disilicate have very favorable optical and mechanical properties and high translucency, with great versatility to be used for indirect restorations, such as inlays, onlays, veneers and crowns [10, 15, 29]. They are highly favorable materials for adhesive cementation because they undergo acid conditioning, besides allowing union through the silane [19]. However, ceramics do not only require the good characteristics of the material to ensure longevity. They also depend on the adhesive relationship between the different substrates and/or materials used to ensure clinical success [3, 20, 25, 35].

Resin-based cements have become popular because of their potential to overcome the disadvantages of solubility, aesthetics, support and lack of adhesion [14] when compared to zinc phosphate cement [24]. However, the cementing procedure of ceramic restorations by their vitreous composition is a complex procedure that requires correct treatment of the internal surface of the piece and the correct application of the bonding agent, which will depend on the micromechanical retention in the porosities coming from the hydrofluoric acid conditioning, associated to a silane bonding agent that will make the chemical bond between the inorganic phase of the ceramic and the organic phase of the resin-based adhesive material, which will ensure better interaction between the part and the cement used [1]. Although it is theoretically possible to apply the cement directly on the silane, the application of an adhesive is recommended so that there is better adhesive resistance between the different materials used that will be used in the cementation process [18, 22].

Vitroceramics enhanced with lithium disilicate are materials that are sensitive to chemical conditioning (hydrofluoric acid and silane) for [1]. The use of hydrofluoric acid exposes the crystals on the surface of the ceramic structure, creating areas of micro retention [34]. The result of the chemical reaction between this acid and the silica present in the ceramic is a salt called hexafluorosilic, which must be removed by water jet as it forms corrosive precipitates caused by hydrofluoric acid [13, 30, 32].

Some authors suggest that this layer should be removed by bathing in an alcohol solution in an ultrasonic vat or by further conditioning with phosphoric acid, removing the by-products on the surface of the ceramic, causing obliteration of the microporosities previously formed by acid conditioning, impairing the bond strength [9, 17, 19, 23, 31]. However, there are different conclusions in the literature on the effect of surface treatments related to bond strength assessments between ceramic and resin cement [4, 16, 10, 27].

Thus, the objective of this study was to evaluate the bond strength of a photoactivated resin cement to a vitroceramic enhanced with lithium disilicate after conventional hydrofluoric acid conditioning technique and alternative techniques in which performing cleaning with phosphoric acid or ultrasound bath of newly conditioned ceramic. The null hypothesis to be studied is that there would be no significant difference between the different ceramic surface cleaning techniques after the hydrofluoric acid conditioning.

Material and methods

Preparation of specimens

Twelve discs were made of an injectable vitroceramic (IPS e.max Press HT ingot - Ivoclar Vivadent, Liechtenstein) with 1mm x 15mm thickness. To obtain the discs, the wax patterns were prepared with the approximate dimensions for later inclusion in the coating (e.max Ivoclar Vivadent), before put in the furnace (EDG 3000 – São Carlos, SP, Brazil) for a period of one hour at a temperature of 850°C for complete removal of the wax and gasses.

The coating was then placed in the furnace (Duramat EP 3000 – Ivoclar Vivadent – Liechtenstein) for injection of the tablets and boiling of the discs. After the cooling period of approximately one hour, the disks were blasted with aluminum oxide for divesting and then received a bath of 1% hydrofluoric acid (HF/H_2SO_4) (Invex liquid, e.max press – Ivoclar Vivadent – Liechtenstein) for five minutes to remove surface roughness and cleanliness. Afterwards, a manual finishing with diamond discs was performed to remove sprues. The thicknesses of all ceramic discs were measured with a digital caliper (Starrett, Itu, Brazil) with an accuracy of 0.01mm.

The disks were embedded in ¾" PVC tubes with self-curing acrylic resin (Jet, Artigos Odontológicos Clássico Ltda, São Paulo, Brazil) and had the surfaces polished in a semi-automatic polishing automática (Ecomet 3, Buehler, Lake Bluff, IL, EUA) using sequential grain size grinding disks (#300, 400 and 600).

The disks were randomly distributed into three groups (n=4), represented by three surface treatments: 1) control – conditioning with 10% hydrofluoric acid for 20s, washing for 20s, silane application; 2) conditioning with hydrofluoric acid for 20s, washing for 20s, cleaning with phosphoric acid for 20s, washing for 20s, silane application; 3) conditioning with hydrofluoric acid for 20s, washing for 20s, cleaning with bath with ethyl alcohol at 90° in ultrasonic vat (Cristofoli, Campo Mourão, PR, Brazil) for 4 min, washing for 20s, silane application.

Then, 4 silicone mats, made from surgical catheters with a diameter of 1mm diameter and 1mm hight, were placed on each ceramic substrate and their internal volume was filled with the photoactivated resin cement (Vitique, DMG, Germany), totaling 4 specimens per ceramic surface (n=16). The cement was handled according to the manufacturer's recommendations and photoactivated for 40 seconds with a LED-curing light (Poly Wireless, Kavo, Joinville, SC, Brazil) with an irradiance of 1100mW/cm2.

After ten minutes, the silicon mold was carefully removed with the aid of a scalpel to expose the cement cylinders with a bond area of 0,79 mm^2 attached to the surface of the ceramic substrate. The specimens were then stored in distilled water at 37°C for 48 hours.

Micro-shear bond strength

After 48 hours, the specimens were adapted to the test device and tested in a universal testing machine (EMIC, DL2000 model, São José dos Pinhais, PR, Brazil). The shear loading was applied at the base of the rolls with a steel wire (0,2 mm diameter) at velocity 0,5 mm/min until breaking. The bond strength was calculated by dividing the maximum force recorded during the test (in N) by the bond area (in mm^2) and expressed in MSBS.

Fault analysis and determination of fracture mode

After performing the micro-shear strength tests, the surfaces of the specimens were examined with a stereoscopic magnifying glass with 57x magnification (SZX9, Olympus, Tokyo, Japan) to determine the failure mode, classifying them as adhesive failures (at the bond interface), cohesive (in ceramic or cement) or mixed.

Analysis of results

Data were tabulated and submitted to statistical analysis (ANOVA to a criterion) with significance level of 5%, using the Statistica program.

Results

The values of the micro-shear bond strength average (MSBS) and standard deviation of each group studied are shown in table I and figure 1.

Table I – Average (MSBS) and standard deviation obtained through the micro-shear bond strength test in the different groups

Groups	Bond Strength (MPa)
G1 – control	11,49 (5,58) A
G2 – phosforic acid	12,51 (4,03) A
G3 – ultrasonic alcohol bath	10,11 (5,33) A

* averages followed by equal letters in the column did not present significant statistical difference (p>0,05)

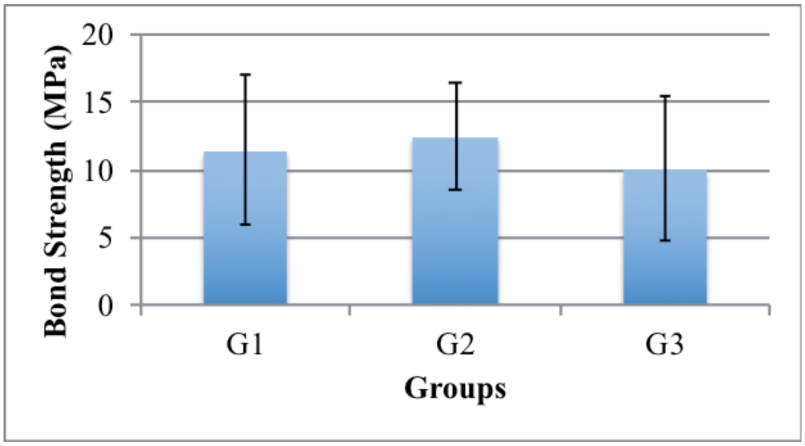

Figure 1 – Micro-shear bond strength average values of the different groups

According to the analysis of variance to a criterion, there was no statistically significant difference between the groups ($p>0,05$). With regard to the failure analysis after mechanical test of micro-shear, it can be observed that most of the groups showed a higher frequency of adhesive failures (between cement and ceramics), as shown in figure 2.

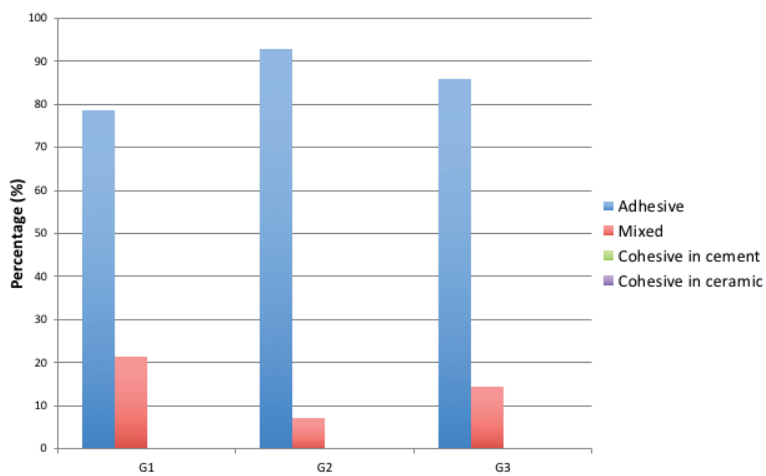

Figure 2 – Percentage of type of failure after mechanical test of the different groups

Discussion

The internal conditioning of silica-based ceramics with hydrofluoric acid at concentrations ranging from 4 to 10% renders the surface rougher through the selective dissolution of the vitreous and crystalline phases [3, 12, 27] and promotes increased cement adhesion [6]. Therefore, the removal of by-products resulting from the ceramic surface conditioning process is considered an important step to ensure a good adhesion performance to the ceramic substrate [5, 23, 30]. These by-products are the result of corrosion caused by the action of hydrofluoric acid, which may impair the infiltration of the adhesive material into the microporosities created [2, 32]. The composition of these by-products depends on both the conditioning agent and the type of [12].

In order to reduce the number of residues deposited on the ceramic surface after conditioning with hydrofluoric acid, several cleaning protocols have been suggested, such as the application of phosphoric acid, alcohol and ultrasonic vats. The hypothesis of cleaning the internal surface of a vitroceramic reinforced with lithium disilicate conditioned with hydrofluoric acid would influence the adhesive strength of a photopolymerizable resin cement. This hypothesis, however, was rejected since the surface cleaning techniques studied did not influence the adhesive strength of the photoactivated resin cement. These results are in agreement with the results of Belli et al. [5], in which a vitroceramic enhanced with lithium disilicate was not influenced by the ceramic cleaning technique after conditioning with 10% hydrofluoric acid. According to these authors, the residue layer formed after the hydrofluoric acid conditioning is thinner and does not negatively affect the adhesive strength of the resin-based cement to the ceramic, once the silanization is done [5].

A difference in the conditioning pattern between vitroceramic enhanced with lithium disilicate and those enhanced with leucite has been reported. While in a leucite-enhanced ceramic the dissolution occurs both in the glassy phase and in the leucite crystals, in a ceramic enhanced with lithium disilicate the dissolution occurs only in the glass phase [5, 12, 21]. In addition to the different conditioning pattern, leucite-reinforced vitroceramics present a bigger and thicker layer of residues after application of hydrofluoric acid [12].

A cement based on photoactivated resin was used in the present work. This type of material has been indicated for the cementation of indirect restorations, guaranteeing high bond strength and esthetics [14], guaranteeing clinical longevity. It is important to point out that, in this work, the cement was exposed directly to the light of the photopolymerizer. The possible attenuating effect of the irradiance caused by the interposition of ceramics could generate different results. It is also worth mentioning that the type, opacity and thickness of the ceramic could also generate different results, considering its effect on the degree of conversion in this type of cement activated exclusively by the light action [8, 33].

In general, adhesive failures were observed between ceramic and resin cement. These results may be related to the stress distribution at the adhesive interface caused by micro-shear strength tests, in which the area corresponding to the adhesive interface is smaller [7]. In conventional shear strength tests, in which the samples have relatively large areas (usually 3-6mm in diameter), this stress distribution occurs unevenly [11]. In the present work, the tension applied on the adhesive interface was generated through an orthodontic wire instead of a chisel, which has also been associated with the better voltage distribution at the adhesive interface [7, 11].

An alternative to micro-shear tests is the use of microtensile. This method presents as advantages the small area of cross-section in the region of the adhesive interface and the homogeneous distribution of stresses during load application [28]. This method, however, was not used in the present work because, besides technically it is more difficult obtaining specimens, the cutting procedure with diamond disk to obtain of ceramic/cement sticks could induce defects and defects in the adhesive interface.

In this work, the adhesive strength was evaluated after storage of the specimens in distilled water at 37°C for 48 hours. Different results could be observed if the specimens were stored for longer times or even subjected to mechanical fatigue tests to be performed prior to micro-shear strength tests.

It is important to note that the internal treatment of a fully ceramic prosthetic part is not limited to the cleaning of any salts formed after the application of hydrofluoric acid. Silanization and the application of adhesive are also important steps that cannot be neglected, as they are as important as the internal conditioning of the ceramic. The silane, a bifunctional molecule that will act as the intermediary of the adhesion between the inorganic phase of the ceramic and the organic phase of the bonding agent, cement [3, 26], which will guarantee a good adhesion between the ceramic and the bonding agent, by increasing the ceramic surface and promoting better wettability of the cement [26]. However, studies show that the application of an adhesive system after silane application can significantly increase the bond strength between the different materials. This may be due to the fact that the linkage between the silane and the MDP present in the adhesive systems [18, 22].

Conclusion

Based on the results of this study, it can be concluded that the cleaning technique of the newly conditioned ceramics does not interfere in the resin/cement bond strength values.

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