Surface Treatment of Lithium Disilicate with Different Concentrations of Hydrofluoric Acid and Orthophosphoric Acid

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Abstract

Introduction: Ips Empress e.max Press is a vitreous ceramic reinforced with highly aesthetic lithium disilicate and with favorable mechanical properties, which is mainly used for anterior esthetic rehabilitations. Knowing the ideal treatment of ceramic surfaces is essential to increase and improve the long-term clinical behavior of the restorations.

Objectives: To evaluate if the different concentrations of HF used in this study affects the substrate provoking different surfaces and to know if the PA 36% application provoke any difference in the internal structure due to the elimination of sales precipitation.

Material and Method: 9 IPS e.max Press copings were selected and distributed in 2 groups according to the application of only hydrofluoric acid (HF) levels (n = 4): 5% 20 s, and 9.6% 10s, 20% 5s and monobond etch-and-prime or using an ethyl phosphoric acid etching (PA) at 36% 30s in an active application once the hydrofluoric acid has been applied at the different named concentrations (n = 4). A cap of e.max Press was evaluated and selected as a control group to compare with the different groups. The characterization of the surface was evaluated by scanning electron microscopy (SEM).

Results: A large difference was observed between group 1 (HF) and group 2 (HF + PA) with different etching patterns for different times. The surface treated with 5% HF and 36% PA shows a contrast, although it is not significant, compared to the group treated with HF at 9.6% and 36% PA, resulting in smoother and uniform retentive surfaces. The treatment with HF at 20% and PA at 36%, presents a great topographical difference with the two previous groups. This presents a disorganized area similar to the group treated with monobond etch-and-prime.

Conclusions: A 5% hydrofluoric acid concentration for 20s in lithium disilicate e.max Press is considered optimal. Moreover, an active application of phosphoric acid for 30s is necessary to discard the precipitation of sales. This provides a favorable microstructure and good bonding quality.

Keywords: Lithium Disilicate; Hydrofluoric Acid; Orthophosphoric Acid

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Introduction

According to the current classification system, glass matrix or vitreous ceramics, polycrystalline ceramics, and resin matrix ceramics are found. The first ones are composed of non-metallic inorganic ceramic materials that contain a glass phase and therefore can be engraved. These are subdivided into three subgroups, such as natural feldspathic ceramics, synthetic ceramics (lithium disilicate) and glass infiltrated ceramics [1-3].

It highlights the disilicate ceramics of crime within synthetic ceramics, which is a reinforced vitreous ceramic, which has a high aesthetic as well as a high mechanical resistance. Due to its high feldspar content, it has good optical behavior (translucency), does not suffer chipping and has good fluorescence characteristics. Regarding its clinical use, it is indicated for single-unit restorations (such as veneers or single crowns) and bridges in premolars of up to 3 units [3].

The form of presentation of these ceramics can be in two forms: injected lithium disilicate or IPS e.- max Press and lithium disilicate block or IPS e.max CAD.

The IPS e.max Press is an improvement of the Empress II system with a higher percentage of lithium disilicate crystals, which improves its resistance to bending up to 470 MPa. His technique of handling is the injection under pressure and presents with two degrees of translucency/opacity, increasing his indications not only to teeth with clear substrates but also to teeth with dark substrates [4,5,8].

With respect to IPS e.max CAD, these are blocks processed by CAD/CAM of lithium disilicate with the same composition as the previous one based on lithium disilicate. These blocks can be processed in a CAD/CAM unit in its pre-sintered or pre-crystallized state (whitish blue and bluish gray) and can be milled in the Cerec3 and Kavo Everest systems among many others [1,4,5,8,12].

Being a vitreous porcelain, the lithium disilicate cemented is made from the adhesive technique. The conditioning of the ceramic surface to prepare the cementation is decisive to generate a bond between the cementing material and the restoration of total ceramic. Thus, etching with hydrofluoric acid produces retentive bonding surfaces, which increases the bond between the resinous cement and the totally ceramic restoration [1].

Therefore, it is important to determine which is the treaty protocol to obtain an adequate surface and thereby establish an optimal adhesion between the restoration and dental tissue.

It was proposed as a hypothesis in the present investigation that: (1) the different concentrations of AF (5%, 9.6%, 20% and MBEP) provoke different surface treatments in the internal adhesive zone of the restoration through the use of the scanning electron microscope (SEM); (2) if when we increase the concentration of AF we also increase the precipitation of salts of lithium disilicate by the MEB; (3) if by removing the precipitation of salts of lithium disilicate with orthophosphoric acid (AO) to 36% we obtain a more retentive and salt-free internal adhesive surface and with structural differences in the samples evaluated.

The objective of the study was:

- Evaluate if the different concentrations of hydrofluoric acid used in the study cause different surfaces in the internal substrate of the lithium disilicate, by means of the evaluation in the scanning electron microscope at 100X and 500X.
- Evaluate whether the application of orthophosphoric acid to 36% after the different concentrations of hydrofluoric acid used causes differences in the internal structure of lithium disilicate due to the elimination of salt precipitation.

Materials and Methods

Study design

In the department of the European University of Madrid, between November 2017 and June 2018, an in vitro study was carried out to assess the effect that orthophosphoric acid has on the elimination of precipitation of salts that are created once the acid has been applied. hydrofluoric and evaluate what type of affection it has in adhesion.
Two study groups were established in which hydrofluoric acid was applied at different concentrations in the first (GE1) without making a subsequent waste removal and only in the second (GE2) was a subsequent washing with orthophosphoric acid. In addition, a control group (GC) in which no surface treatment was carried out. The samples were randomly assigned and the surface treatment performed by the same operator. Later they were evaluated by means of a scanning electron microscope (SEM).

**Materials used**

<table>
<thead>
<tr>
<th>Material</th>
<th>Trade Name</th>
<th>Commercial Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ceramic lithium disilicate</td>
<td>IPS e.max Press</td>
<td>Ivoclar Vivadent-Schaan, Liechtenstein.</td>
</tr>
<tr>
<td>Hydrofluoric acid 4.5%</td>
<td>IPS Ceramic Gel</td>
<td>Ivoclar Vivadent-Schaan, Liechtenstein.</td>
</tr>
<tr>
<td>Hydrofluoric acid 9%</td>
<td>Ultradent Porcelain Etch</td>
<td>Ultradent Products Inc, South Jordan, UT, USA.</td>
</tr>
<tr>
<td>Hydrofluoric acid 20%</td>
<td>Hydrofluoric acid Gel</td>
<td>Dentaflux</td>
</tr>
<tr>
<td>Monobond etch and prime (MBEP)</td>
<td>Monobond etch and prime</td>
<td>Ivoclar Vivadent-Schaan, Liechtenstein.</td>
</tr>
<tr>
<td>Orthophosphoric acid 36%</td>
<td>Conditioner 36 etching Gel</td>
<td>Dentsply Detrey- Germany.</td>
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</tbody>
</table>

**Method**

**Procedure**

To carry out the objective of the study, 9 lithium disilicate ceramic copings (IPS e.max Press) were selected and distributed in 2 groups according to the application of only hydrofluoric acid concentrations (n = 4): 5% (Ivoclar Vivadent), and 9.6% (Ultradent), 20% (Dentaflux) and monobond etch-and-prime (Ivoclar vivadent) or using an etching with 36% orthophosphoric acid (Ivoclar Vivadent) once the hydrofluoric acid is applied to the different named concentrations (n = 4) [15-19].

In addition, an additional distribution of the subgroups was performed according to the engraving times (n = 4) 20s, 10s, 5s and 40s respectively at each acid concentration, and was evaluated by scanning electron microscopy (SEM) for the evaluation of the Characterization of the surface. The time is adapted according to the applied concentration, selecting smaller times for higher concentrations. This is because the AF in contact with the surface for long periods of time, has more time to react with the silicon present in the vitreous matrix, eliminating more vitreous phase and consequently creating greater superficial irregularities that can sometimes appear sharp [20].

**Selection of samples**

Nine lithium disilicate ceramic copings (IPS e.max Press) were selected for our study. The samples collected were injected lithium disilicate copings of an unspecified size, which once selected were each observed in groups of 7 and 2 in the microscope to rule out the presence of cracks or defects.

The samples were numbered from 1 to 9 to facilitate the preparation process of the copings.

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Preparation of the samples according to study groups

In each of the copings the following surface preparation protocol was made: (1) Application of hydrofluoric acid at concentrations of 5% for 20s in the first cap, 9.6% for 10s in the second, 20% for 5s in the third, and placement of monobond etch-and-prime actively 20s leaving acting for 40s in the fourth, in the two groups [12,20,21]; (2) Surface washing and drying; (3) Subsequent active application of orthophosphoric acid at 36% in the second group. 4. Washing and drying of the surfaces.

In addition to these two groups there is a control group to which no surface treatment was applied.

MEB analysis

The samples were prepared to be observed under the scanning electron microscope XL30 ESEM at room temperature. All samples were glued with cyanoacrylate in a sample holder. After this they were introduced into the microscope under controlled pressure or environmental mode to get them an electron beam and thus be visible in the MEB. Samples were examined with 100X and 500X magnification.

Results

Morphological changes

Comparison between study groups

The images acquired through the scanning electron microscopy (SEM) at 500x show morphological changes according to the different treatment methods. With this, different etching patterns are observed for different times, finding an adequate vitreous matrix elimination and superficial features not very acute in 5% concentrations.

With the treatment only with HF (Figure 2) saturated areas of salts are observed that once eliminated with H₃PO₄ surfaces appear free of residues causing the formation of microretentive grooves (Figure 2). In comparison with the untreated control group, a large morphological difference can be observed at 500x once the surface is treated.

**Figure 2:** Comparative image of the disilicate surfaces treated at different concentrations.
Regarding the surface treated with 5% HF and 37% H$_3$PO$_4$, it shows a contrast, although not significant, compared to the group treated with HF at 9.6% and 37% H$_3$PO$_4$ since both present microretentive sulci of shallow inside a broken surface. Both surfaces treated with HF at 5% and 9.6% result in smoother and more uniform surfaces, and deep and distinguishable channels compared to the other results, since at lower concentration, less depth but higher surface quality.

The group treated with HF at 20% and H$_3$PO$_4$ at 37%, presents a great remarkable topographical difference compared with the two groups indicated above. This presents a mostly disorganized area more similar to the group treated with monobond etch-and-prime and H$_3$PO$_4$. In this treaty there is a major alteration of the morphology of the surface, with a disorganized pattern, a little uniform area and little distinguishable microplastics. This is due to the fact that the higher the concentration applied, the greater the change in the surface, also causing a greater presence of waste to be subsequently removed with the washing with phosphoric acid.

**Figure 3**

*Photo preparation of COFIAS group 1*

HF, Hydrofluoric acid 5%  HF, Hydrofluoric acid 9.6%  HF, Hydrofluoric acid 20%  Monobond etch-and-prime

*Photo preparation of COFIAS group 2*

HF, Hydrofluoric acid 5%  HF, Hydrofluoric acid 9.6%  HF, Hydrofluoric acid 20%  Monobond etch-and-prime

AO, Orthophosphoric acid

Lithium disilicate COFIA without treatment
Surface Treatment of Lithium Disilicate with Different Concentrations of Hydrofluoric Acid and Orthophosphoric Acid

Discussion

In order to improve the bond between the resin and the ceramic, methods have been developed to create an optimum roughness with the purpose of propitiating a longer time in the mouth of our restorations [15,22].

However, despite the fact that the conditioning of the ceramic surfaces produces a result in the roughness of these areas, the application protocol is essential, since there may be a disparity of results that can be obtained from an insufficient preparation, to a degradation causing microcracks that commit to restoration [12,15,18,20,23,24].

One of the characteristics that represents hydrofluoric acid is its aggressiveness on ceramic surfaces based on silica. As for its form of affection in ceramics, it acts selectively attacking the glass phase causing an exposure of the disilicate crystals (silicon dioxide or SiO₂) that at not very high concentrations can result in an optimum surface for adhesion. This produces an irregular porous surface that allows a penetration of the resinous materials by micromechanical retention and in turn an improvement in the chemical bond between the silane and the resin cements [15,17,18,20,23,25].

Puppin-Rontani, et al. in their study they explain the reason for the selectivity of fluorohydrin acid etching in the vitreous matrix. Said elimination occurs because the affinity of fluorine for silicon is greater than for oxygen, which makes it possible for an attack on the glass matrix by the ionized HF. Therefore, the higher the concentration of HF, the greater the elimination of the vitreous matrix due to a greater presence of ionized HF available to react with silicon dioxide (SiO₂) [20].

Also, Murillo-Gómez, et al. 2018 highlight in their study the resistance of the disilicate crystal to fluorohydrin acid. In its publication it is observed that the stronger the engraving, the lower the content of silicon (Si) registered in the ceramic surface. In addition, the etching pattern of hydrofluoric acid is irregular in each case, since the dissolution of the internal structure depends on the composition of the substrate being recorded, and on the chemical affinity that this acid gel has with the substrate. The mechanism mainly consists of an ion exchange that is responsible for the removal of the silicon content [20].

As already mentioned above, the time and concentration of hydrofluoric acid used influence both the creation of surface irregularities as well as the bond strength. For this reason, it is vitally important to adjust the time to concentration and thereby optimize the binding force, according to Canay, et al. 2001 in his study carried out [17,18,20,23].

Thus, the engraving recommendation for e.max Press ceramic is 5% for 20 seconds according to the commercial house, however there are many in vitro studies that use different concentrations and times for this type of reinforced vitreous ceramic. This study also shows that the higher concentration used there are more failures in terms of adhesion and ceramic strength and may damage the integrity of the material due to increased defects of the internal structure, in cases where the concentration of acid is high (> 10%) [17,20].

It has also been found that concentrations lower than 5% are not efficient in terms of the creation of a retentive surface, and in addition they must be applied for a longer time. However, concentrations of more than 10% decrease the bond strength due to the highly irregular surface that is produced [12]. Thus, it is recommended that the lithium disilicate be etched at 5% concentrations for 20 seconds because it contains a smaller amount of glassy phase. This concentration and time is considered the least harmful compared to the others since it is able to create a favorable surface for adhesion [12].

Furthermore, in order to improve the porous surface formed by hydrofluoric acid, it is necessary to clean said extension to eliminate the salts formed. Likewise, the crystalline precipitates formed on the surfaces etched with this acid are the result of the reaction of sodium, potassium, calcium and aluminum, which, if not discarded, will remain on the ceramic surface, preventing and weakening the bonds between the resin and porcelain. These must be suppressed by means of a specific mechanism, their expulsion not being effective only with water under pressure since they are not water-soluble [14,18,23]. Thus, after washing the ceramic once the hydrofluoric acid has been applied, the residues and salts must be eliminated, the traditional method being the placement of the restoration in distilled

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water, 95% alcohol or acetone in an ultrasonic bath for 4 or 5 minutes [14,20,23,26]. However, the ease of handling, the time gain, and the elimination quality of the salts are advantages that we do not find in the conventional ultrasonic bath washing process, considered as a not efficient technique [2,3].

Puppin-Rontani., et al. 2017 highlights that the replacement of the traditional method with distilled water in an ultrasonic bath by phosphoric acid can cause a time gain and ease of handling, as well as quality in the elimination of salts [20].

The majority of results of studies with scanning electron microscopy (SEM) reveal the different form of action of different acids such as hydrofluoric acid and orthophosphoric in porcelain restorations [15]. Although the application of hydrofluoric acid reacts and dissolves the crystals causing holes in the surface, the use of only this acid without adequate subsequent cleaning, is not entirely effective. Likewise, the application only of phosphoric acid does not bring changes in the surface causing a minimum roughness.

Although it is true that treatment with silane after the application of hydrofluoric acid decreases the chances of failure, Maruo., et al. [15] in their study, they found that etching with hydrofluoric acid produces insoluble salts of silica and fluoride that precipitate on the ceramic surface and hamper resin-ceramic bonding [15,17,18,23]. This results in a difficulty for the accommodation of the silane and the resinous cement on the surface of the ceramic. On the other hand, the orthophosphoric acid does not originate any compound that is deposited on the surface of the ceramic, so it could be an effective potential product to wash said surface once the hydrofluoric acid is applied, and with this to achieve an increase in the bonding strength between the resin and the lithium disilicate ceramics. This method of applying 37% orthophosphoric acid is interesting as a substitute for the traditional ceramic cleaning system. Likewise, Canay., et al. 2001 in his study observed a lower amount of precipitation and crystalline residues (such as silicon tetrafluoride) in disilicate ceramics compared to conventional feldspathic, which may be due to the greater presence of vitreous matrix in the latter with the consequent appearance of greater precipitation of salts [20,23].

So important is the application of orthophosphoric acid for the elimination of salts, as well as its active application form [16]. Studies such as Guiraldo., et al. 2016 have investigated that the active application of orthophosphoric acid against passive application, can truly act as a cleansing agent for vitreous waste being an alternative to the traditional method [16,27].

As for the analysis of the images obtained after the study with the scanning electron microscope, there is a great visual inequality between them. The results of this study showed that the variations in the acid etch protocols affect differently in the surface microstructure, as well as the engraving depth that also depends on the concentration used. Each of the images represents a lithium disilicate coping that is engraved at a certain concentration and adapting the time to each concentration, that is, shorter times are applied for higher concentrations. With this, different engraving patterns are observed for different periods.

In addition, it must be considered that in order to obtain the highest possible surface quality, protocols with not very high times and concentrations must be applied, since they produce sharp irregularities and with it a decrease in the tensile strength, causing fractures of the restoration in the long term [2,3].

Thus a clearer and cleaner surface is observed when concentrations of 5% have been applied and, on the contrary, in solutions of 20%, the disilicate area appears deteriorated. This highlights that time is not a condition in this study, since this parameter is adjusted according to the concentration used. This is the reason why the result of the surface engraved at 20% is not enough. On the other hand, similar results can be seen in the surfaces recorded at 5% and 9.6%, however in the latter an area with less clarity is observed, where the micro-retentions created by this acid concentration simulate less uniform. Murillo-Gómez., et al. 2018 in his study, reports that concentrations of 10% are already considered aggressive for the surface, since they dissolve a large amount of vitreous phase and produce more irregularities that can become unfavorable. However, this author uses a time of 20s for a concentration of 10%, this factor may be the one that alters the result, since it would not hurt the use of a shorter time, as has been done in this study (at higher concentration less time) [12].
The difference between concentrations of 5% and 9% observed in photographs compared with the concentration of 20% is disturbing (Figure 3), which is why it is practically obsolete by dental professionals and is also difficult to find in the market. Puppin-Rontani, et al. 2018 in his study highlights the fact that at concentrations greater than 10% a decreased bond strength is observed. There are no articles in the literature that deal with concentrations of 20%, which may be due to the fact that concentrations greater than 10% are becoming unfavorable for micromechanical adhesion and retention, in addition to being considered a dangerous concentration in relation to soft tissues [12,17].

In addition, the similarity of the surfaces etched with 20% hydrofluoric acid and the surface in which monobond etch-and-prime has been applied is of great curiosity, since they present similar features despite the fact that the hydrofluoric acid concentration of both it does not coincide, the latter being a concentration of% hydrofluoric acid together with the presence of silane.

It should be noted that in the group to which monobond etch-and-prime was applied and subsequently an etching with active orthophosphoric acid for the elimination of salts, the subsequent application of a pure silane is questionable, since this washing with orthophosphoric acid would eliminate these precipitations of salts as well as the silane that takes presents. For this reason, it is proposed not to apply this protocol for this type of monocomponent acid, since if this is the case, we will not take advantage of its main advantage. Murillo-Gómez, et al. 2018 in his study, he observes that the surfaces that were treated with monobond-etch-and-prime, resulted in very soft areas and of lower roughness, being the least harmful option with respect to the others. However, in the literature and after the results obtained in this study, its performance continues to be questioned, since contrary effects are observed. This product is composed of acid, silane, fluorides and methacrylates that can be harmful in adhesion after a long period of time [12].

The instructions for use for this first monocomponent conditioner is based on a simplification of steps, combining the step of recorder and silane reducing the conditioning process. According to the manufacturer, a profuse wash with water is indicated for the elimination of residues, however, it is clear from numerous studies [14-16] that it is not enough to finish with the precipitations formed by the hydrofluoric acid. Therefore, a subsequent active application of a phosphoric acid that would assume this task would be necessary, despite the uncertainty that results from the need for the application or not of a pure silane once the phosphoric acid is placed [12].

On the other hand, the silane provides a chemical bond with the silica, which can also be altered if it is not used in its full purity. The use of a pure silane is recommended since monobond plus is considered to contain components that interfere with adhesion, such as fluorides and functional groups such as methacrylates (silane methacrylate, phosphoric acid methacrylate and sulfur methacrylate), and these are not profitable in terms of membership and may interfere in this in the long term. On the other hand, pure silane is composed of ethanol in more than 85% and contains more than 10% of water in addition to methacrylxypropyltrimethoxysilane in a smaller proportion of 5%, so applied to the ceramic surface produces a clean and interference-free union. Thus, the application of hydrofluoric acid as well as silane is very important to obtain a good adhesion [18,20,21,28].

Although it is a simple bonding process, since it only requires a uniform reaction time of 60 seconds after the application of 5% hydrofluoric acid for 60 seconds on any type of surface, this universal primer is particularly indicated when they go to use cements from the Ivoclar Vivadent house, such as Variolink Esthetic and Multilink Automix, so their use is limited to some extent.

The null hypothesis of the present study is therefore approved, since: (1) the different concentrations of AF (5%, 9.6%, and 20%) cause different surface treatments in the internal adhesive zone of the restoration using the use of the scanning electron microscope (SEM); (2) if we increase the AF concentration we also increase the precipitation of lithium disilicate salts by the SEM; (3) when removing the precipitation of lithium disilicate salts with 37% orthophosphoric acid, we obtain a more retentive and salt-free internal adhesive surface and with structural differences in the samples evaluated.

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Conclusion

The lack of studies and adequate standardization of the preparation of ceramic surfaces and definition of the process, has led to obtain the following conclusions:

1. A joining quality is required carried out by deep grooves, abundant and not too extensive to create a good retention with the treated surface with hydrofluoric acid.
2. To achieve this quality of union, it is necessary to wash the surface to eliminate the precipitate of salts that appears after the application of AF.
3. Pressurized water does not eliminate the precipitation of salts, so a more specific treatment is required for its elimination, such as the application of 37% phosphoric acid, or ultrasonic bath with distilled water; 95% alcohol or acetone. for 4 or 5 minutes.
4. An active application of phosphoric acid is required for 30s to dispose of residual salts.
5. The ease of handling, the gain of time, and the quality of elimination of the salts are some of the advantages of the washing with phosphoric acid with respect to the bath with conventional ultrasounds. The conventional technique for washing the precipitation of salts is not efficient in daily work.
6. Higher concentrations or high acid exposure times produce acute irregularities that are unfavorable in adhesion as well as increasing the risk of fracture (> 10%).
7. The depth of engraving depends on the engraving protocol used, producing different effects according to the concentration of hydrofluoric acid used in each cap.
8. A concentration of 5% for 20 seconds in lithium disilicate e.max.Press produces a favorable microstructure, with regular and retentive grooves.

Bibliography


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