Does Post-etching Cleaning Influence Bond Strength of Lithium Disilicate Laminate Veneers?

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Abstract

This paper aims to present a clinical case of laminate veneers in lithium disilicate (LD) reinforced ceramic, complemented with scanning electron microscopy and energy dispersive x-ray spectroscopy analysis of this ceramic’s surfaces submitted to some of the different post-etching cleaning protocols proposed by literature. Many dentists are applying all-ceramic restorations, especially veneers, with the most applied material being LD, due to its good mechanical properties, esthetics, and ability to adhere to tooth substrate. The cementation process is critical to clinical success of indirect restorations. Combination of hydrofluoric acid and silane application on the inner surface of ceramic has been the most recommended method to increase bond strength between ceramic and tooth substrate. As a result of surface corrosion, residual subproducts are formed and precipitate on the ceramic surface, affecting the bonding process to the resin cement. Elimination of residue is recommended in the literature and can be done via 3 common techniques: thorough washing with air/water spray for 30 seconds, 37% phosphoric acid for 60 seconds, or ultrasonic bath for 4 to 10 minutes. Little information on LD ceramics post-etching cleaning methods is available in the literature. For this ceramic, the thorough washing with air/water spray for 30 seconds appeared to remove most of the residue without damaging the material. Being also the most practical and easiest of the reviewed procedures, it was, therefore, the protocol of choice for the clinical cases presented in this paper with success after 18 months.

A significant aspect of clinical routine today involves using all-ceramic indirect restoration, especially lithium disilicate (LD) laminate veneers. Adhesive cementation is considered one of the most critical and meticulous steps, due to its numerous variables and long working time. Everything that is performed previous to it—treatment planning, evaluation of tooth wear, impressions with accurate details reproduction, and adequate materials selection for adhesion—could be lost if the cementation is done with technical indifference. Scientific evidence guides the careful execution of this process to achieve predictable outcomes.

Ceramic and tooth-surface treatments are essential to obtaining an efficient adhesion. The combination of hydrofluoric acid (HF) and silane application on the inner surface of the ceramic material has been the most recommended method to increase bond strength between ceramic and tooth substrate.

HF etching is the first clinical step on surface treatment of etchable ceramics and should be applied to selectively dissolve their crystalline, or glassy, phase and create superficial
microporosity, resulting in a highly retentive surface, receptive to silane and cement application. As a result of surface corrosion caused by acid etching with HF, residual subproducts are formed and precipitate on the ceramic surface, giving it a white and opaque appearance, affecting the bonding process to cement. These precipitated salts of silica and fluorine are insoluble and remain attached to the ceramic surface even after acid rinsing. This layer over the etched ceramic proved to change the dynamics of bonding, leading to decreased bond strength between resin cement and the ceramic.

Although some authors did not report differences among groups that were submitted to various post-etching cleaning procedures, residue elimination is recommended in common techniques: thorough washing with air/water spray for 30 seconds, 37% phosphoric acid for 60 seconds followed by washing, or ultrasonic bath for 4 to 10 minutes. However, the literature findings are not conclusive regarding the best technique. Too little information is available concerning the use of LD ceramics. This paper aims to present a clinical case of LD-reinforced ceramic, with the use of scanning electron microscopy (SEM) images and energy dispersive x-ray (EDX) spectroscopy analysis of ceramic surfaces submitted to some various postetching cleaning protocols proposed by the literature.

**SEM and EDX Analysis**

To evaluate the effect of different post-etching cleaning protocols for the ceramic surface, 6 disc specimens (11.0-mm-diameter x 0.5-mm-thickness) of LD-reinforced (e.max CAD, Ivoclar Vivadent) were prepared in the computer-aided design/computer-aided manufacturing (CAD/CAM) system (Weiland, Ivoclar Vivadent). These discs did not undergo any finishing and polishing to simulate the inner surface of laminate veneers. One ceramic disc was not submitted to HF etching to evaluate surface characteristics before any treatment. Other discs were submitted to etching with 5% HF (Power CEtching, BM4) for 20 seconds. Postetching cleaning procedures used are described in Table 1.

After these surface treatments, all discs were prepared for analysis in SEM. First, specimens were placed in metal stubs and positioned in a sputtering system to be covered with a thin layer of gold. Then, they were examined in a high-vacuum SEM (JEOL JSM-IT300, equipped with EDX, Thermo Scientific NSS Spectral Imaging, thermals, thermofisher.com). Only one examiner performed the analysis to standardize the image analysis and obtention and also to guarantee that all images would be evaluated according to the same parameters. Subsequently, other investigators were consulted to confirm the findings and opinions of the original examiner. Images were collected at x3000 magnification and voltage of 15 kV.

The same areas of the samples used for SEM observation (at x5000 magnification) were additionally x-ray scanned using the Oxford Instruments X-Max NPXL-30 SEM x-ray detector system for qualitative analysis to determine elemental distribution profile. After identification of all peaks on the EDX spectrum, data on O, F, Na, Al, Si, K, Ca, and Ti elements were registered.

**Discussion**

The era of conservative dentistry had brought to light the concern with obtaining the best adhesion, as most of dental wear nowadays is carried out without mechanical retention, leading to adhesion in enamel. In the case reported, LD laminated veneers were indicated as a conservative and well-established treatment for minor smile
Dental veneers, like the case reported, make it even more important to have a good adhesion and to minimize as much as possible the factors that may jeopardize the clinical longevity of this restorative procedure.

Researches have shown the presence of residue on ceramic inner surface after etching can significantly influence bond strength values in microtensile bond strength tests. Figures 1 (a-f), show LD-reinforced ceramic and result of acid etching and different cleaning protocols. Bond strength results presented in literature along with images obtained confirm the importance of establishing correct clinical protocols for post-etching cleaning of different ceramics available.

Formation and composition of this residue is directly associated with the composition of acid used and of ceramic conditioned.

In an EDX analysis of ceramic samples that were submitted to post-etching cleaning protocols, Belli et al. observed the presence of residue in specimen surface was associated to the observation of fluorine picks in samples (around 15%), while specimens submitted to post-etching cleaning did not present any fluorine. In the studies of Canay et al. and Sposetti et al., residues of fluorosilicate products of Al, K, Na, and Ca were also found. Therefore, residues observed in the ceramic's surface are formed by the reaction of fluorine present in HF with elements Si, Ca, K, and Ti present in ceramic.

Contrary to that, for the LD ceramic no fluorine was observed in any EDX analysis and all specimens presented similar graphics. This is probably due to the short time of acid etching (20 seconds), resulting in a small fluorine release and deposition in surface.

Ceramics have different types of crystals and percentages of silica and the preferential dissolution of the phases is dependent of etching time. Literature indicates etching of ceramics according to the amount of silica they contain: feldspathic porcelains for 120-seconds, due to few silica content, leucite reinforced ceramics for 60 seconds, and of LD reinforced ceramics for 20 seconds, due to great amount of silica. The longer etching time, the more residues produced, therefore feldspathic porcelain is supposed to be more responsive to different post-etching cleaning procedures, in comparison to LD based ones.

Most studies showed that the best method to remove residues formed by acid etching in feldpathic porcelain is the ultrasonic bath. Magne and Cascione found the best results for the same material, in SEM and bond strength evaluation, when cleaning with 37% phosphoric acid etching followed by ultrasonic bath. Belli et al. concluded that air/water spray, phosphoric acid etching, phosphoric acid and ultrasonic bath cleaning, and just ultrasonic bath were all similarly good for removing the residue layer and produced good bond strength for leucite and LD-reinforced ceramic.

In Brazil, it is a common practice for ceramic etching to be performed only in the office, after try-in, as described in the Case Report section. In the United States, however, most laboratory technicians condition the restoration, which is then sent to the dental office for try-in. The try-in procedure can contaminate the ceramic surface, which will then need to be etched again and cleaned. Ivoclean (Ivoclar Vivadent) is a new universal cleaning paste, created to clean an already etched surface. This product is based on sodium hydroxide and appears to effectively remove various contaminants from the ceramic surface, and provide a clean surface for resin bonding when used before and after etching.

Images obtained for LD-based ceramic showed similar surface pattern in most cleaning methods; however, use of ultrasonic bath resulted in a surface covered with smaller crystals that appeared to be broken crystals, and phosphoric acid also seemed to have caused a new etching, resulting in smaller crystals as well. Accordingly, thorough washing with air/water spray for 30 seconds seemed to be the post-etching cleaning method that presented less residue and broken crystals, is easier to perform, and does not need new apparatus to be performed. In the case reported, the LD veneers’ surface preparation was carried out according
to the findings observed in SEM analysis, in order to obtain the best quality of the adhesive interface. In a follow-up of 18 months, no effects on adhesion were observed.

**Case Report**

A 36-year-old woman presented at the restorative dentistry specialization at the Brazilian Association of Dentistry in Goiânia, Brazil, with concerns about her smile esthetics. Full-facial photographs confirmed the complaint and the esthetic problem created due to absence of the left upper central incisor (Figure 3 and Figure 4). After performing a detailed analysis of the intraoral photographs (Figure 5), taking the patient’s medical and dental histories, and completing the dental examination, the esthetic-functional rehabilitation was planned, presented to the patient, and approved by her.

First, an impression was taken with silicone (Virtual, Ivoclar Vivadent, ivoclarvivadent.us) to obtain a wax-up of the intended final result and perform a mockup with bis-acrylic resin (Protemp™ 4, 3M ESPE, 3m.com). The mock-up guided the periodontal surgery, enabling the clinician to obtain a surgical crown lengthening of the left maxillary lateral incisor, to transform it into a central incisor. After 120 days, a new impression was taken to make a new wax-up and mockup to analyze the size, form, and anatomic details (Figure 6).

After the patient approved the mockup, guides made with putty silicone material (Zetalabor, Zhermack, http://en.zhermack.com) were obtained to help the evaluation of areas to be prepared. This wear was minimal, and a fine-grit diamond bur was used (Figure 7).

Final impressions were taken with silicone (Virtual) using a 2-step technique and 2 knitted cords of different diameters (Ultrapak 00 and 000, Ultradent Products, ultradent.com). Impressions and registers obtained were sent to the dental laboratory technician. Laminated veneers were made on glass ceramic reinforced with LD (color HTBL3, IPS e.max®, Ivoclar Vivadent) through injection molding followed by extrinsic staining techniques. Figure 8 shows veneers positioned on plaster casts.

Veneers were tried-in in the patient’s mouth with translucid try-in paste (Variolink Veneer Try-In, Ivoclar Vivadent). After the dentist and patient provided approval, the ceramics were submitted to surface treatments necessary before cementation.

The first step was to use 5% HF (Power CEtching, BM4) for 20 seconds on the ceramic surface (Figure 9a). After the correct conditioning time, acid was removed from the ceramic surface with thorough washing with air/water spray for 30 seconds (Figure 9b).

Then, ceramic surface was air dried and silane (Monobond Plus, Ivoclar Vivadent) was applied (Figure 9c) to create a chemical durable bond between the ceramic and resin cement. To help solvents with evaporation, after 60 seconds, an air spray was used on the ceramic inner surface, before cement application (Figure 9d).

Cementation was performed tooth by tooth, so the first to be cemented was separated from the others with polytetrafluoroethylene strips (Figure 10), then 37% phosphoric acid (Power Etching, BM4, Florianópolis, SC, Brazil) was applied, respecting the 30 seconds of conditioning time for enamel and 15 seconds for dentin (Figure 10). After the sufficient time, the substrate was thoroughly washed and dried, then adhesive (ExciTE F, Ivoclar Vivadent) was applied (Figure 10), followed by the use of an air spray to remove excesses and obtain a thin adhesive film. Then, resin luting cement, which was selected in the try-in phase, was applied on the laminated veneer’s inner surface, which adhered to the substrate (Figure 10). The restoration was positioned with continuous and light finger pressure, cement excesses were removed with a brush, and then, photoactivation (Bluephase, Ivoclar Vivadent) was carried out for 60 seconds on the buccal surface. For all teeth, the same sequence was repeated.

Buccal margins were finished with a scalpel blade No. 12 and then polished with a sequence of abrasive silicone (porcelain veneer kit, Shofu Dental, shofu.com). The last step was to use a felt disc with polishing diamond pastes. Figure 11 to Figure 13 show the excellent esthetic-functional result obtained. Figure 14 and Figure 15 depict the 18 months follow-up, preserving the same healthy and harmonious condition of the immediate result.
Conclusion

Thorough washing with air/water spray for 30 seconds can be used to obtain good post-etching cleaning of LD-based ceramics. Further investigation of bond strength must be done comparing all protocols proposed in the literature; however, considering clinical routine, this technique seems to be the best option and, therefore, was used in the case reported with success after 18 months.

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