Comparative Evaluation of Lithium Disilicate Ceramic Surface and Bond Strength to Dentin Surface after Treatment with Hydrofluoric Acid and Acidulated Phosphate Fluoride Gel: An In Vitro Study

Abstract

Context: Long-term retention of the restoration depends on strength and durability of the bond of the luting composite resin to the tooth and the porcelain substrates. Aims: This in vitro study was conducted to compare and evaluate the influence of hydrofluoric acid (HF) and acidulated phosphate fluoride (APF) gel-etched treatments on surface roughness and bond strength to dentin of a commercially available lithium disilicate ceramic (E-Max). Subjects and Methods: Sixty lithium disilicate ceramic disks measuring 5 mm in diameter and 2 mm thick were fabricated and divided into three groups: Group A (n = 24), Group B (n = 24), and Group C (n = 12) and were subdivided to Group A₁, A₂, and A₃ which were surface treated with 1.23% APF gel (pH = 3–4) at different time intervals 4, 7, and 10 min, respectively. Group B₁, B₂, and B₃ were surface treated with 1% APF gel (pH = 1–2) at different time intervals 4, 7, and 10 min, respectively. Group C were surface treated with 9.6% HF (pH = 1–2) for 1 min. Morphological changes obtained with the surface treatments were analyzed using a surface profilometer. Statistical Analysis: All specimens were subjected to a tensile bond strength test using a tensometer, and the obtained data were statistically analyzed using Kruskal–Wallis test. Results: The surface roughness (µm) and bond strength (MPa) of lithium disilicate discs (samples) etched with 1.23% APF gel and 1% APF gel for 10 min and etched with 9.6% HF for 1 min showed no statistical significant difference among them. Conclusions: In this study, the lithium disilicate discs etched with 1.23% APF gel and 1% APF gel for 10 min showed similar surface roughness and bond strength to those etched with 9.6% HF for a minute.

Keywords: Acidulate phosphate fluoride gel, hydrofluoric acid, lithium disilicate ceramic, profilometer, tensometer

Introduction

All-ceramic restorations have gained acceptance among clinicians and patients because of their superior esthetics and the possibility of conservative tooth preparations.[1] Long-term retention of the restoration depends primarily on strength and durability of the bond of the luting composite resin to the tooth and the porcelain substrates to prevent fracture, marginal discoloration, and secondary caries. To achieve this bond, the porcelain surface may be modified chemically or mechanically to promote surface roughness to the luting agent.[2] Surface treatment of porcelain increases the surface area and creates micro-porosities, which enhance the potential for mechanical retention of the luting composite resin. This physical bond combines with the chemical bond obtained from the use of a silane coupling agent to provide a high strength bond between the components of treated porcelain-bonded restorations. Chemical surface treatments of dental ceramics include the use of 50% or 60% orthophosphoric acid, 10% sulfuric acid, 0.5 N nitric acid, 9.6% hydrofluoric (HF) acid, 1.23% acidulated phosphate fluoride (APF) gel, ammonium hydrogen difluoride, and airborne particle abrasion with 50 µm or 250 µm alumina.[2] Among all, 9.6% HF acid is considered as the gold standard to etch ceramic before final cementation.[2] Although it is widely used, it has many disadvantages causing tissue rash, burns, and contact poison as it is a highly corrosive liquid. The results of in vitro studies have indicated that various topical fluorides such as APF gel which is safe for oral tissues may etch or react with porcelain, glass ionomer, fissure sealant, and composite restorative materials.[3] HF acid which is component of APF preparations has a destructive effect on composite restorations.

How to cite this article: Mallikarjuna DM, Surendra GB Kumar¹, Shilpa Shetty¹, Mallika Shetty, Bharath Raj¹

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Access this article online
Website: www.ijdr.in
DOI: 10.4103/ijdr.IJDR_230_17

Quick Response Code:
and causes wear and loss of integrity. Hence, the aim of the study was to compare and evaluate the influence of HF acid and APF gel surface treatments on surface roughness and bond strength to dentin of a commercially available Lithium disilicate ceramic.

**Subjects and Methods**

Sixty sound, freshly extracted, noncarious maxillary first premolars were used for the study.

Maxillary first premolars with caries, hypoplasia, attrition, abrasion, erosion, fluorosis, and restorations were excluded from the study.

**Collection of extracted teeth**

The collected specimens were scaled to remove any surface deposits and stored in 0.02% thymol solution.

**Mounting of specimens**

The roots of the teeth were embedded in acrylic resin blocks and grooves were placed on the lingual surface of the tooth to aid in retention of the cylindrical extension attached to the occlusolingual surface of teeth, which was used to facilitate mounting of specimen on tensometer [Figure 1a and b].

**Tooth preparation**

A silicone mold (Aquasil Soft Putty/Regular Set) of each specimen was made before preparation and was used to standardize the preparation procedure. All the specimens were prepared only on the buccal surface to receive a 5-mm ceramic disc. Tooth preparation was done using a high-speed, high torque Airotor Handpiece and diamond burs according the biomechanical principles of tooth preparation for all-ceramic crowns. The prepared samples were stored in distilled water.

**Fabrication of lithium disilicate ceramic (IPS e. max®) discs**

Wax patterns were fabricated using mold with uniform thickness of 2 and 5 mm diameter with inlay wax (Kronenwachs Bego inlay wax) and sprued. Casting ring was invested using Cergo® fit speed investment material immediately to prevent distortion. It was then vacuum mixed for 90 s. The mix was used to invest the patterns in the rings. The invested rings were set aside for 60 min. The investment cylinders were then preheated in a conventional preheating furnace from room temperature up to 850°C (1 h). The investment cylinders along with the lithium disilicate ingots were placed at the center of the hot press furnace and pressed. Once the entire process was completed, investment cylinders were removed and allowed to cool to room temperature. After divestment, the pressed ceramic discs were subjected to sandblasting with 50 μ particles for 20 s at 2 bar pressure so that all the investment material was removed completely from the ceramic disc surface. To remove any residue, the samples were cleaned in an ultrasonic cleaner for 60 s. Then, the pressed ceramic discs were cut from the sprues (with 3 mm of sprue intact) with a diamond-coated disc and cleaned with a jet steam machine [Figure 2]. Acrylic cylindrical extensions were attached to each sprue which was used to facilitate mounting of specimen on a tensometer. The ceramic discs were then finished and polished [Figures 1b and 3].

**Surface treatment**

The specimens were divided into three main groups − Group A (n = 24), Group B (n = 24), and Group C (n = 12). Group A and Group B were further divided into three subgroups each − Group A₁ (n = 8), Group A₂ (n = 8), Group A₃ (n = 8), Group B₁ (n = 8), Group B₂ (n = 8), and Group B₃ (n = 8). Group A₁, A₂, and A₃ were surface treated with 1.23% APF gel (pH = 3–4) at different time intervals 4, 7, and 10 min, respectively. Group B₁, B₂, and B₃ were surface treated with 1.0% APF gel (1% APF gel [pH = 1–2]) was prepared by adding 0.2 ml of 48% HF acid to 1 ml of 1.23% APF gel to make it more acidic) at different time intervals 4, 7, and 10 min, respectively. Group C was surface treated with 9.6% HF acid (pH = 1–2) for 1 min. Treatments were applied to each
ceramic disc from cotton pliers and washed with water spray for 10 s. The surface roughness of all sixty specimens was measured with a surface profilometer [Figure 4]; they were later used for the tensile bond test.

**Surface roughness measurement**

Each traverse of the profilometer stylus was made across each disc approximately parallel to the specimen base and the surface roughness was measured in Rq (root mean square).

**Bond strength measurement**

Resin cement (RelyX™U100) was mixed according to the manufacturer’s recommendations. The mixed cement was applied to both the prepared dentin and the ceramic. The prepared tooth did not require etching as the cement used was self-adhesive. The ceramic was placed upon the flattened dentin surface with finger pressure. The excess cement was removed with an explorer after the initial polymerization; the resin cement was light polymerized with a light intensity of 480 nm and a power of 1100 mw/cm² (±10%) for 40 s. The light source was held at distance of 10 mm from the specimens. The bonded specimens were stored at room temperature with 100% relative humidity for 48 h before the tensile bond strength test. Each specimen was then mounted on a metal holder in the tensometer [Figure 5]. A tensile load was applied until failure occurred. The ultimate load to failure was recorded in Newton (N). The average bond strength (MPa) was calculated by dividing the maximum ultimate load to failure (N) by the bonded cross-sectional area (mm²). The data obtained were then subjected to statistical analysis using Mann–Whitney U-test and Kruskal–Wallis test.

**Results**

In this study, we evaluated the surface topography of lithium disilicate ceramic after surface treatment with 9.6% HF acid at 1 min and 1.23% APF gel and 1% APF gel at 4, 7, and 10 min interval, respectively, and bond strength of lithium disilicate ceramic to dentin.

The results of this study revealed that there was significant difference in mean surface roughness (µm) among the three groups (P = 0.0003). There was significant difference between the Group A (1.23% APF gel) and Group B (1% APF gel) and also between the Group A (1.23% APF gel) and Group C (9.6% HF) with higher mean value for 9.6% HF followed by 1% APF gel and 1.23% APF gel [Table 1].

There was significant difference in mean bond strength (MPa) among the Group A (1.23% APF gel), Group B (1% APF gel) and Group C (9.6% APF gel). There was significant difference between the mean bond strength (MPa) of Group A (1.23% APF gel) and Group B (1% APF gel), between the Group A (1.23% APF gel) and Group C (9.6% APF gel), and also between the Group B (1% APF gel) and Group C (9.6% HF) with higher mean value for Group C (9.6% HF) followed by Group B (1% APF gel) and Group A (1.23% APF gel) [Table 2].

There was no significance difference in mean surface roughness (µm) and mean bond strength (MPa) between
In the present study, the mean surface roughness (µm) and mean bond strength (MPa) of Group A3 (1.23% APF gel-10 min), Group B3 (1% APF gel-10 min), and Group C (9.6% HF-1 min) were almost similar with no statistically significant difference among them.

Discussion

Bonding ceramic restorations to tooth structure relies on treatment of the ceramic intaglio surface, selection of a suitable resin luting agent, and appropriate treatment of prepared tooth structure. Long-term retention of the restoration depends primarily on strength and durability of the bond of the luting composite resin to the tooth and the porcelain substrates to prevent fracture, marginal discoloration, and secondary caries. The strong interlocking of the luting composite into the retentive etch pits produced by surface treatment contributes to strong adhesion of the porcelain restoration, with good retention. Various ceramic surface treatments have been advocated which produce different topographies and bond strengths. Ayad et al.[2] found that HF acid treatment resulted in the generation of pores and grooves that produced the greatest bond strength between the ceramic and tooth dentin among 50%, or 60% orthophosphoric acid and airborne-particle abrasion with 50-µm, or 250-µm alumina for 10 s, and orthophosphoric acid treatment was the least effective surface treatment method evaluated.[2] Kukiattrakoon and Thammasitboon[4] found that shear bond strength values between composite resin and high leucite ceramics after etching with 1.23% APF gel for 7-10 min were not significantly different than after etching with 9.6% HF acid for 4 min.[4]

The strength of the enamel/composite resin/ceramic bond depends primarily on the silanization of the etched ceramic surfaces. Jardel et al.[5] reported that etching with HF acid alone is not sufficient to produce a strong bond with dental ceramics, but when a silane coupling agent is also used, the adhesion bond increases.[5] 9.6% HF acid is considered as the gold standard to etch ceramic before final cementation.[5] Although it is widely used, it has many disadvantages such as it is a highly corrosive liquid, it causes contact poison, it penetrates tissues more quickly than typical acids, and it interferes with nerve function. The results of in vitro studies have indicated that various topical fluorides such as APF gel, which is safe for oral tissues may etch or react with porcelain, glass ionomer, fissure sealant, and composite restorative materials.[6]

Although a number of studies have been undertaken to determine ceramic surface and bond strength to dentin surface after treatment with HF acid, the review of literature revealed very few studies to determine the comparative evaluation of the influence of HF acid and APF gel-etched treatments on surface roughness and bond strength.
strength to dentin of a commercially available lithium disilicate ceramic at different time intervals.

Every effort was made to simulate in vivo conditions in this in vitro study.

According to Nattress et al.,[7] free-hand preparation can result in variable depth of preparation. To minimize variation, tooth preparations were evaluated using respective silicone molds of the specimens made before the preparation.

APF gel, used widely for in-office fluoride application, consists of sodium fluoride, phosphoric acid, and HF acid.[9] It is safe for oral tissue, unlike HF acid, which can produce tissue rash and burn.

APF gel has been proposed as an alternative for ceramic surface etching before bonding with composite resin. Lacy et al. reported no significant difference in the shear bond strength between composite resin and feldspathic porcelain treated with 9.6% HF acid for 4 min or 1.23% APF gel for 10 min. These results were similar to the results of Tylka et al.,[9] in which no significant difference in bond strength was found between composite resin and two types of ceramics after treatment with either 9.6% HF acid for 5 min or 1.23% APF gel for 10 min. Interestingly, surface analyses of HF acid-etched and APF gel-etched ceramics have shown different etching patterns.[9] A previous study showed that HF acid interacts with silica in a glass matrix in feldspathic porcelain, forming hexa-fluorosilicate at the rate of 0.44 μm/min.[10] This creates small pits around the leucite crystals in the ceramics. However, APF gel produced minimal surface topography change. The difference in the etching patterns were produced in relation to the etching time and concentration of the etching agents. HF acid etching time has been reported to range from 60 s to 20 min, while 10 min ceramic etching is recommended when 1.23% APF gel is used.[11]

There are certain limitations to this in vitro study. The methodology used does not accurately reproduce clinical factors such as oral temperature changes, oclusal forces, saliva of varying pH, buffer capacity and flow rate, and abrasion resistance of the cement and the acid environment produced by bacteria such as Streptococcus mutans and Lactobacilli, which may affect the degradation of the cement. Therefore, further in vivo investigations related to 1.23% APF gel and 9.6% HF required.

Conclusions

From the results and statistical analysis, the following conclusions were drawn:

1. The lithium disilicate discs etched with 1.23% APF gel and 1% APF gel for 10 min, respectively, showed similar surface roughness and bond strength to 9.6% HF acid etched for a minute

2. Duration of etching 1.23% APF gel and 1% APF gel for 10 min was longer compared with 9.6% HF acid for a minute. However, both 1.23% APF gel and 1% APF gel negated all the disadvantages of 9.6% HF acid.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

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