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Hydrofluoric acid concentrations: Effect on the cyclic load-to-failure of machined lithium disilicate restorations

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ABSTRACT

Objectives. To evaluate the effects of the etching with different hydrofluoric acid (HF) concentrations on the cyclic load-to-failure (C_{Lf}) of machined lithium disilicate crowns cemented to dentin analogue material.

Methods. Pairs of dentin analogue prosthetic preparations and lithium disilicate ceramic crowns with simplified and standardized designs were machined (n = 18). The preparations were etched with 10% HF (60 s), followed by primer application. The intaglio surface of the ceramic crowns was treated as follows: non-etched (control, CTRL); or etched for 20 s with different HF concentrations — 3% (HF3), or 5% (HF5), or 10% (HF10). A silane coating was then applied onto the treated ceramic surfaces, and they were adhesively cemented to the preparations. To perform the fatigue tests (staircase approach), a hemispheric stainless-steel piston (Ø = 40 mm) applied cyclic loads in the center of the crowns under water (initial load: 720 N; step-size: 70 N; cycles: 500,000; frequency: 20 Hz). Additionally, topographic, fractographic, and fractal analyses were carried out. The fatigue data were analyzed using the Dixon and Mood method.

Results. Although the topographic and fractal analyses depicted the action of HF etching altering the superficial complexity and topography, the preponderant topography

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pattern was established by machining on CAD/CAM. All groups showed similar C_{Lf} (in N) (CTRL = 805.00 ± 91.23; HF3 = 781.25 ± 29.87; HF5 = 755.00 ± 154.49; HF10 = 833.75 ± 100.74).
Significance. Etching with different HF acid concentrations did not promote a deleterious effect on the cyclic load-to-failure of machined lithium disilicate crowns.

1. Introduction

With an increasing demand for aesthetics and mechanical reliability, lithium disilicate ceramic has been meeting an outstanding role among the glass-ceramics available for monolithic restorations. This ceramic material, feasible to machining by CAD/CAM (computer-aided design/computer-aided manufacturing) presents a good success rate (~95%) for single crowns for up to 10 years of follow-up [1–3].

As a relevant issue, a strong bond between the ceramic restoration and tooth structure provides good support for the restoration and actively transmits functional loads through the bonded interface, thus improving clinical success [4,5]. Regarding vitreous ceramic restorations, to achieve successful bonding, the glass matrix of the intaglio ceramic surface may be chemically modified by hydrofluoric (HF) acid etching in order to promote an irregular surface to provide a mechanical interlocking with resin cement [6–9]. A silane coupling agent is applied on the etched surface to promote a chemical bond between the inorganic phase of the ceramic (i.e., silica), and organic phase (i.e., polymer) of resin cement [10–12]. Studies have shown that HF acid etching or silane coating, when applied separately, do not promote stable bond strength [13,14].

The manufacturer's recommendation to etch lithium disilicate is with 5% HF acid for 20 s [15]. Despite the increase in ceramic surface roughness to promote micromechanical interlocking, HF acid is capable of decreasing the ceramic material's strength, as a consequence of the modification of the resident flaw population [16–21]. Since there is a direct association between defects on the bulk structure of ceramic materials and their resistance [22,23], over-etching (i.e., higher exposure time and acid concentration) could negatively influence the long-term success of ceramic restorations. Moreover, HF acid is also a hazardous substance; exposure to it can lead to damage to the eyes, soft tissues, necrosis, and bone decalcification, also related to its time and concentration [24–26].

Some authors have researched about the exact concentration and time applicable to lithium disilicate ceramic, but these studies only applied static loads (monotonic tests), using specimens as bars or discs, and did not consider the geometry of an all-ceramic restoration [17,18,27,28]. Cyclic fatigue tests under intermittent loads are crucial to assessing the ceramic material's mechanical performance, thus providing information about their fatigue strength and also influence of the different factors related to it. These tests have been employed to more closely reproduce the clinical conditions under which restorative materials are subjected during function [29,30].

It is important to elucidate the increase of a glass-ceramic's strength when adhesively cemented [31], but also its degradation when the surface is roughened [28,32]. According to Seydler and Schmitter [33], CAD/CAM systems produce ceramic restorations with few defects due to industrially-prepared ceramics blocks, which have reduced internal defects and flaws [34]. However, some studies have shown that the machining process may introduce new defects on ceramic surfaces and sub-surface, leading to a decrease in mechanical strength [35,36].

Therefore, adequate HF acid etching protocol for micromechanical retention and resin cement bonding without weakening the ceramic should be investigated. Moreover, the final strength of etched ceramics evaluated by monotonic tests and the use of disc- or bar-shaped specimens may not exactly reflect the real strength of all-ceramic restorations, since the defects produced by conditioning could be filled-up by resin cement [21,37]. Limited information is available regarding the influence of different HF acid concentrations under cyclic fatigue of adhesively cemented lithium disilicate crowns, as well as about the combinatorial effect of hard machining.

Besides, fractal analysis has been applied in dentistry to assess the complexity of oral mucosa, the roughness of implants, the surface characterization of zirconia (Y-TZP) ceramics, and also to analyze failures of clinically-failed all-ceramic restorations [38–41]. This mathematical tool can provide a new way to account for complexity of the topographical pattern of the treated ceramic surface. Likewise, the fractal dimension, which is a measurement of how complex the topography is, when increased by 10%, causes the surface stiffness to decrease more than one order of magnitude [42].

Herewith, the present study aims to evaluate the cyclic load-to-failure (C_{Lf}) of simplified lithium disilicate crowns machined by the CAD/CAM system etched with different HF acid concentrations. Additionally, topographic, fractographic, and fractal analyses have been carried out. The assumed null hypotheses were: (1) different HF acid concentrations would not promote different cyclic loads-to-failure, and (2) HF acid etching would not reduce the cyclic loads-to-failure when compared with non-etched crowns.

2. Materials and methods

The general description of the materials used in the present study, their manufacturers, composition, and batch numbers are listed in Table 1.

2.1. Specimen preparation

Initially, a model was machined from a dentin analogue material (epoxy resin, G10, FR4 Laminate Round Rods Epoxyglass, NEMA grade FR4, Accurate Plastics, Inc., Yonkers, USA) in a