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# Fatigue failure load of zirconia-reinforced lithium silicate glass ceramic cemented to a dentin analogue: Effect of etching time and hydrofluoric acid concentration

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A B S T R A C T

This study aimed to evaluate the effect of etching time and hydrofluoric acid (HF) concentration on the fatigue failure load and surface characteristics of zirconia-reinforced lithium silicate glass (ZLS) ceramic cemented to a dentin-like, fiber reinforced epoxy resin. Ceramic (Suprinity, VITA) (1.0 mm thick) and epoxy resin (2.5 mm thick) discs (10 mm diameter) were produced. The bonding surface of the ceramic samples was nonetched (control group), or etched for 30, 60 or 90 s by 5% or 10% HF. The epoxy resin discs were etched by 10% HF for 30 s followed by the application of an adhesive material (Single Bond Universal, 3M ESPE). Pairs of ceramic/epoxy resin discs were cemented with a dual cure resin cement. The fatigue failure load was determined by the staircase method (500,000 cycles at 20 Hz; initial load = 925 N; step size = 45 N). In 10% HF the etching time was shown to influence the fatigue failure load, which increased as the etching time increased (30 s < 60 s < 90 s), and in 5% HF the fatigue failure load was not shown to be affected by the etching time; the lowest fatigue failure loads were produced in the control group without ceramic etching followed by 10% HF acid etching for 30 s. Topography analysis showed variations based on the etching protocols. All fractures (radial cracks) were shown to originate from defects at the ceramic surface on the cementing interface. For fatigue loading improvements of ZLS ceramic, 10% HF acid etching for 90 s and silanization of the ceramic surface is recommended.

## 1. Introduction

Recently, zirconia-reinforced lithium silicate glass (ZLS) ceramics were introduced on the market for CAD/CAM restorations, such as Vita Suprinity (Vita Zahnfabrik, Bad Säckingen, Germany), composed of a synthetic glass matrix with zirconia crystals (56–64% silicon dioxide, 15–21% lithium oxide, 8–12% zirconia, and other components, e.g. pigments) (Vita Suprinity, 2013; Gracis et al., 2015). It combines excellent optical and mechanical properties, with flexural strength of approximately 440 MPa (Elsaka and Elnaghy, 2016), and has been considered for monolithic full-contour restorations (Rinke et al., 2016a, 2016b).

An important aspect required for the success of such restorations is the establishment of proper adhesion between substrate and adherent (Tsujimoto et al., 2017). In this sense, the gold-standard protocol for resin bonding to glass ceramics is the etching with hydrofluoric acid (HF) followed by the application of a silane coupling agent (chemical and micro-mechanical bond) (Sattabanasuk et al., 2016; Sato et al., 2016). Variations in HF acid etching (for instance, time and concentration) have been shown to change the surface micro-morphology of glass ceramics (surface defect population) (Traini et al., 2016) and resin adhesion (Leite et al., 2013; Venturini et al., 2015b), being the

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increase in HF acid concentration and etching time associated with an increase on surface area available to adhesion with resin cement and a decreasing on contact angle values (Ozcan and Valittu, 2003; Venturini et al., 2015b; Sato et al., 2016).

Even that rougher surfaces are related to better adhesive potential, HF over-etching (e.g., increased time and concentration) is reported as detrimental to flexural strength as well as the fatigue behavior of glass ceramics (Addison et al., 2007; Hooshmand et al., 2008; Zogheib et al., 2011; Venturini et al., 2015a; Venturini et al., 2017). However, some reports have shown that adhesively cementation is able to promote a strengthening of the assembly (May et al., 2012; Posritong et al., 2013; Venturini et al., 2017), filling up the defects and flaws produced by HF acid etching (Anusavice and Hojjatie, 1992).

Clinically, ceramic restorations are susceptible to fatigue failure in response to an environment in the presence of moisture and cyclic masticatory forces (Gonzaga et al., 2011; Morimoto et al., 2016). Hence, fatigue failure may be defined as the cumulative damage triggered by cyclic forces, resulting in slow-crack growth of defects that will lead to catastrophic failure of a restoration under loads below the normal characteristic strength of a specific material (Wiskott et al., 1995; May et al., 2015). Although, the fatigue behavior of ZLS ceramics, including the effect of different etching protocols on the fatigue load bearing capability of the material, has not been studied. Thus, the question is: do the surface topographic variations of ZLS glass ceramic affect the fatigue behavior of this material adhesively cemented?

Thus, this study aimed to elucidate and compare the effect of different HF acid concentrations and etching time on the surface characteristics and fatigue failure load of a ZLS ceramic cemented to a dentin analogue. The null hypotheses were: (1) mean fatigue failure loads will not be influenced by the etching time; (2) HF acid concentrations will not affect the mean fatigue failure loads.

## 2. Materials and methods

### 2.1. Specimen assembly description

A simplified tri-layer setup was designed, as presented by Chen et al. (2014), to simulate an occlusal restoration for a posterior tooth (molar). ZLS discs, reproducing the occlusal restoration, were cemented on a glass fiber reinforced epoxy resin disc, which simulated dentin. The discs were 10 mm in diameter, the average dimension of molars (Ferrario et al., 1999). The bonded tri-layer discs had a final thickness of 3.5 mm, equivalent to the average thickness from pulp wall to occlusal surface (Sulieman et al., 2005; Harris and Hicks, 1998).

### 2.2. Ceramic specimens manufacturing

Pre-fabricated ceramic blocks of Vita Suprinity (Vita Zahnfabrik H. Rauter GmbH & Co., Bad Säckingen, Germany; Lot No. 49142) were shaped into cylinders (10 mm in diameter) using silicon carbide (SiC) papers with 180-grit sandpaper in a polishing machine (Ecomet 250 Grinder Polisher, Buehler, Illinois, USA). The cylinders were cut with a diamond blade under constant water-cooling (Isomet 1000, Buehler, Illinois, USA), resulting in 150 discs with a standard thickness of 1.1 mm. The specimens were crystallized in a furnace according to the manufacturer's instructions (VACUMAT 6000 MP, VITA; 840 °C, 8 min vacuum). The 'occlusal' surface of the discs (opposite the cementation surface) was polished with SiC paper of increasing grit-size (600-, 800- and 1200-grit) until a final thickness of 1.0 mm was achieved.

### 2.3. Epoxy resin discs manufacturing

The used epoxy resin sheet (150 mm × 350 mm, 2.5 mm thick) was acquired from Carbotec GmbH & Co. KG. (Königs Wusterhausen; Lot No. 295795). The aforementioned ceramic specimens methodology was used for shaping the epoxy resin into cylinders (10 mm in diameter).

**Table 1**
Experimental design; parameters for fatigue tests: mean monotonic load-to-failure, initial load for fatigue tests (50% of mean monotonic load-to-failure), and step size (5% of initial fatigue load). Fatigue results: mean fatigue failure load (σf), standard deviation (SD), 95% confidence interval (CI) according to Dixon and Mood's method, and percentage of decreasing in load, comparing the mean value of monotonic load-to-failure Vs mean fatigue failure load.

| | Study' factors | | Parameters for fatigue tests | | | Fatigue failure load findings | | |
|---|---|---|---|---|---|---|---|---|
| **Groups' codes** | **HF acid concentration** | **Etching time** | **Mean monotonic load-to-failure (N)** | **Initial load for fatigue tests (N)** | **Step size increment (N)** | **$\sigma_f$ (SD) (N)*** | **95% CI** | **Load decrease (%) in relation to the mean value of monotonic load-to-failure** |
| **CTRL** | **Without HF** | | 1850 | 925 | 45 | 257.5 (23.9)$^{A}$ | 241.1–273.9 | 86% |
| **HF5-30s** | **5%** | **30 s** | | | | 857.5 (43.8)$^{C}$ | 825.1–889.9 | 54% |
| **HF5-60s** | | **60 s** | | | | 857.5 (99.3)$^{C}$ | 796.5–918.5 | 54% |
| **HF5-90s** | | **90 s** | | | | 795.6 (37.4)$^{C}$ | 764.5–826.8 | 57% |
| **HF10-30s** | **10%** | **30 s** | | | | 610 (23.9)$^{B}$ | 588.6–631.4 | 67% |
| **HF10-60s** | | **60 s** | | | | 837.5 (84.9)$^{C}$ | 784.8–890.2 | 55% |
| **HF10-90s** | | **90 s** | | | | 1052.5 (99.3)$^{D}$ | 992.2–1112.8 | 43% |

* Different superscript letters mean statistically significant differences.