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The effect of hydrofluoric acid concentration on the fatigue failure load of adhesively cemented feldspathic ceramic discs

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ABSTRACT

Objective. This study investigated the influence of hydrofluoric acid (HF) etching at different concentrations on the fatigue failure load of adhesively cemented feldspathic ceramic discs (Vita Mark II). Besides, their effect on the micromorphology of ceramic surface was investigated.
Methods. Eighty ceramic discs ($\phi = 10$ mm; thickness = 1.5 mm) were cemented to epoxy supporting discs ($\phi = 10$ mm; thickness = 2.0 mm) using different surface conditioning methods (n = 20): nonetched control (CTRL), or etched for 60 s with different HF concentrations: 1% (HF1), 5% (HF5), or 10% (HF10). All the ceramic discs received a silane application (Monobond Plus). The epoxy discs were etched with 10% HF for 60 s and received a primer coating (Multilink Primer A + B). Adhesively cementation was performed (Multilink Automix), and the assemblies (ceramic discs/epoxy discs) were subjected to cyclic loads in water by a staircase approach (500,000 cycles; 20 Hz; initial load = 290 N; step size = 30 N). Fatigue failure load data were analyzed using 1-way ANOVA and post-hoc Tukey's tests ($\alpha = .05$).
Results. Mean failure load of the HF5 group (255.0 ± 23.0 N) was significantly lower; HF1 group (301.7 ± 71.0 N) presented intermediate values, and the highest values were achieved in CTRL (351.7 ± 13.4 N) and HF10 (341.7 ± 20.6 N) groups. All the failures were radial cracks starting from the bonding surface.
Significance. In terms of fatigue failure load, etching with 1% and 5% HF had a deleterious effect on the fatigue behavior of an adhesively cemented feldspathic ceramic, while 10% HF had no negative influence.

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1. Introduction

Among a wide range of CAD/CAM blocks available to produce monolithic restorations, the feldspathic ceramic Vitablocs Mark II (Vita Zanhfabrik, Bad Säckingen, Germany) is fabricated using fine-structure ceramic powders under industrial sintering process, resulting in good polishing properties, decreased enamel wear, and increased strength due to a nearly pore free ceramic [1]. Also, the fracture resistance of the feldspathic ceramic restorations may be improved with appropriate bonding procedures [2]. To achieve successful bonding, the intaglio surface of these ceramics requires micro-mechanical interlocking by hydrofluoric acid etching, and chemical bonding by a silane coupling agent [3,4].

Even though that adhesive approach is a well-known and recommended method to increase bond strength to feldspathic ceramics, the high toxicity of hydrofluoric acid remains a concern among the clinicians and scientific literature [5] motivating the investigation of lower concentrations of hydrofluoric acid and alternative methods [6–9]. Besides, previous studies showed that mechanical strength of glass–ceramic materials may be negatively influenced by the hydrofluoric acid etching approaches [10–14]. The alterations of the surface flaws (defects' population) as consequence of the etching time and hydrofluoric acid concentration [11] could be the predictive factor of ceramic bulk fractures, mainly if the surface defects are not entirely filled by resin cement [15]. Thereby, only the flexural strength of the ceramic etched by hydrofluoric acid may not exactly reflect the actual strength of all-ceramic restoration [16] since it will be cemented to the tooth. The resin cement could improve the probability of survival of ceramic restorations to a certain extent by healing the cracks [17,18], which depends on the behavior of the resin penetrating the ceramic surface [19].

From the clinical perspective, ceramic restorations are constantly subjected to cyclic loading under wet conditions during chewing function, which should be considered during in vitro tests, since these conditions decrease the fracture load of restorative materials by slow-crack growth of intrinsic defects [20,21]. Studies about clinically failed glass–ceramic crowns reported that the majority of bulk fractures started from flaws and stresses at the cementation surface [22–24]. From that standpoint, it is important to investigate the influence of the etching protocols in a bonded ceramic surface in attempt to predict the clinical behavior using cyclic loading fatigue tests. Furthermore, limited information is available as to how the use of different hydrofluoric acid concentrations influences the fatigue failure load of adhesively cemented feldspathic ceramic.

Therefore, the current study aimed to evaluate the effect of different hydrofluoric acid concentrations on the fatigue failure loads of feldspathic ceramic discs adhesively bonded. Additionally, micro-morphological evaluations of the ceramic surface in response to the etching protocol and fractographic examinations of the failed specimens were executed. The hypotheses tested were: (1) acid etching would influence the fatigue failure loads compared with nonetched ceramic discs, and (2) different hydrofluoric acid concentrations would promote different fatigue failure loads.

2. Materials and methods

The materials used in this investigation are described in Table 1.

2.1. *Specimens preparation and cementation procedure*

Feldspathic ceramic blocks (VitaBlocks Mark II for CEREC/inLab, Vita Zahnfabrik, Bad Säckingen, Germany) were shaped into cylinders using a diamond drill ($\phi = 10$ mm; Diamant Boart, Brussels, Belgium) coupled to a bench drill (SBE 1010 Plus, Metabo; Nürtingen, Germany) under refrigeration. The cylinders were cut under water-cooling (Isomet 1000, Buehler, Lake Bluff, United States), resulting in 80 discs with an initial thickness of 1.6 mm. The 'occlusal' surface of the discs was polished with 600-grit SiC polish papers (Ecomet Polisher, CarbiMet SiC Abrasive Paper grit 600–P1200, Buehler)

Table 1 – Materials used in the study with manufacturer data and composition.

Material	Brand name; manufacturer (lot number)	Composition[a]
Fine-particle feldspar ceramic	VITABLOCS Mark II; Vita Zahnfabrik (LOT 28650)	Al_2O_3, SiO_2, K_2O, Na_2O, CaO, TiO_2, other oxides
Epoxy resin plate	Epoxy platte 150 × 350 × 2,0 mm; Carbotec GmbH & Co. KG, Germany	Epoxy resin
Dual-cured resin cement	Multilink Automix; Ivoclar Vivadent AG, Liechtenstein (LOT U19980)	Dimethacrylates, HEMA, barium glass filler, ytterbium trifluoride, highly dispersed silica, catalysts and stabilizer, pigments
Primer	Multilink Primer A and Primer B; Ivoclar Vivadent AG, Liechtenstein (LOT U22990)	Primer A: water, initiators; Primer B: phosphonic acid acrylate, hydroxyethyl methacrylate, methacrylate mod. polyacrylic acid, stabiliser
Silane	Monobond Plus; Ivoclar Vivadent AG, Liechtenstein (LOT U25466)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate
Hydrofluoric acid	Experimentally formulated 1% Condac Porcelana 5% Condac Porcelana 10% FGM, Brazil (LOT 030216)	Hydrofluoric acid at 1%, 5%, and 10%, water, thickener, surfactant, and coloring

[a] The chemical compositions are described according to the manufacturers' information.

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