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# Heat treatment-improved bond strength of resin cement to lithium disilicate dental glass-ceramic

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ABSTRACT

This study investigated the influence of different hydrofluoric acid (HF) concentrations and heat treatments applied to a lithium disilicate dental glass-ceramic (EMX) on surface morphology and micro-shear bond strength (μSBS) to resin cement. Five HF concentrations (1%, 2.5%, 5%, 7.5% and 10%) and four different heat treatments applied before etching were assessed: 1. etching at room temperature with no previous heat treatment (control group); 2. HF stored at 70 °C for 1 min applied to the ceramic surface at room temperature; 3. HF at room temperature applied after a hot air stream is applied perpendicularly to the ceramic surface for 1 min; 4. the combination of previously heated HF and heated EMX surface. The etching time was fixed for 20 s for all groups. Etched EMX specimens were analyzed on field-emission scanning electron microscope (FE-SEM) and the μSBS was carried out on a universal testing machine at a crosshead speed of 1 mm/min until fracture. For the control groups, FE-SEM images showed greater glassy matrix dissolution and higher μSBS for 7.5% and 10% HF concentrations. The previous heat treatments enhanced the glassy matrix dissolution more evidently for 1%, 2.5% and 5% and yielded increased μSBS values, which were not statistically different for 7.5% and 10% HF concentrations (control group). HF concentrations and previous heat treatments did show to have an influence on the etching/bonding characteristics to lithium disilicate dental glass-ceramic.



## 1. Introduction

Dental ceramics have gained great notoriety in the last decade due to the remarkable improvement of their mechanical strength and ability to mimic dental tissues. Clinical indications comprise restoring tooth tissues that were decayed/fractured, replacement of unsatisfactory/failed clinical dental restorations or to reconfigure the anatomical shape of mal-positioned teeth with an improvement on the design and the esthetic appearance of the smile. Nowadays, glass-ceramics are among the most commonly used indirect restorative materials in Dentistry.

Among glass-ceramics, some materials are reinforced by lithium disilicate crystals, and have recently become a popular restorative material for esthetic and functional rehabilitations [1]. The first lithium disilicate glass-ceramic introduced in the dental market was the IPS Empress II (Ivoclar Vivadent, Schaan, Liechtenstein) in 1998. This glass-ceramic contained around 60 vol% of lithium disilicate crystals dispersed in an amorphous vitreous phase and commercially available in different shades and opacities. The IPS e.max Press (Ivoclar Vivadent, Schaan, Liechtenstein) (EMX), a later version of IPS Empress II, was released in 2005 with some changes in the microstructure. EMX is a bioceramic composed by refined lithium disilicate crystals (± 70 vol%-$Li_2Si_2O_5$. Crystals: 3–6 μm in length) embedded in a glassy matrix (information provided by the manufacturer) and it is indicated either as a full-contour restoration (monolithic) or as a core for further porcelain veneering [2]. It presents statistically similar occlusal wear as natural enamel [3] as well as translucency and high strength as a monolithic ceramic [4]. These factors, combined with the emerging demand for metal free restorations, explain the widespread use of lithium disilicate glass-ceramics [4].

The lithium disilicate glass-ceramics can be chemically bonded to tooth tissues by methacrylate-based materials, such as resin

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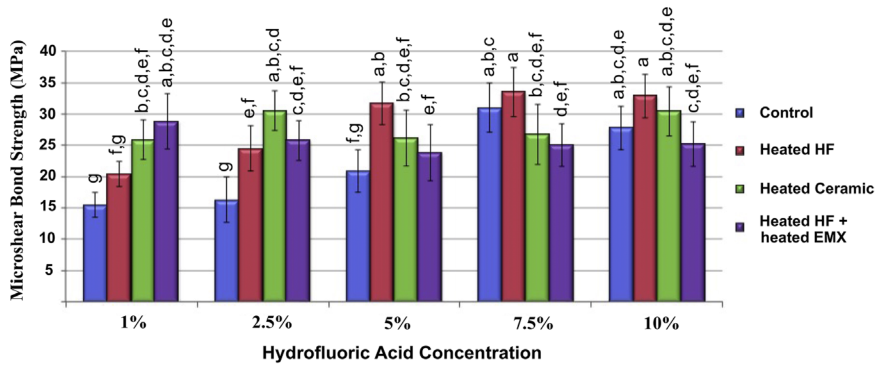


**Fig. 1.** Average values of micro-shear bond strength with error bars ( ± Standard Deviation). Columns with different lowercase letters in vertical position indicate significant statistical difference (two-way ANOVA—Tukey's multiple comparisons test, $p < 0.05$).

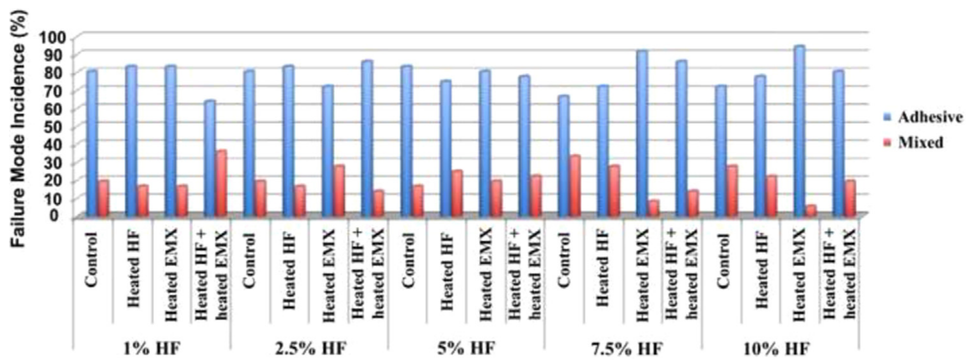


**Fig. 2.** Failure mode results of debonded resin cement specimens. Hydrofluoric acid (HF).

cements, and their interaction is one of the key factors to long-term clinical success [5]. Ideal bond to dental glass-ceramics is achieved by the sum of two techniques: (1) surface modification in order to increase surface area; (2) chemical bonding via silane coupling agent, which makes possible to bond an inorganic material (glassy matrix) to the resin cement (organic compound). Despite its inherent brittleness [6], resin cement bonding is able to strengthen the dental ceramic [7].

Regarding physical surface modifications of EMX, no other method has proved to be as efficient as etching with hydrofluoric acid (HF) [8–13]. HF acid etching increases the roughness [7], therefore the surface energy and wettability [14,15], and selectively dissolves the glassy matrix, exposing lithium disilicate crystals, which is essential to increase the micromechanical retention between restoration and resin cement [9,10,16]. Based on scientific and clinical evidence, HF etching followed by silane application are necessary and have become the most widely accepted surface treatments for glass-ceramics [5].

The manufacturer recommended that IPS Empress II should be etched with 10% HF for 60 s at the time it was released on the market. However, the etching time was later modified to 20 s according to the findings obtained by Spohr et al. [17]. Today, the manufacturer recommends that EMX should be etched with 4.8% HF for 20 s. However, clinically, the optimal HF acid etching time and concentration to treat glass-ceramic is not clear [7]. Since HF is hazardous to soft tissue, lower HF concentrations have been evaluated to reduce the risk of tissue damage [18]. Unfortunately, previous study has reported that HF concentrations ranging from 1% to 2.5% were not able to provide adequate bond strength to ceramic substrate [16].

In an attempt to improve the bonding potential, some researchers [19–21] have reported increased roughness/surface area/bond strength when hot etched solutions were applied onto zirconia dental ceramic. Moreover, Liu et al. [22] reported increased bond strength to zirconia dental ceramic when 48% hydrofluoric acid was previously heated to 100 °C. To date, there are not investigations concerning the assessment of previous heat treatments on the etching morphology/interfacial bond strength of lithium disilicate glass-ceramic.

Thus, the aim of this study is to assess the effects of previous heat treatments applied to five different hydrofluoric acid concentrations and to ceramic surface on the etched surface morphology and micro-shear bond strength (μSBS) between lithium disilicate dental glass-ceramic (EMX - IPS e.max Press) and one commercial resin cement.

## 2. Materials and methods

### *2.1. IPS e.max Press blocks*

Two hundred and sixty ceramic blocks of IPS e.max Press (8 mm × 8 mm × 3 mm), shade LT A2, were fabricated according to the manufacturer's instructions. The entire detailed laboratorial steps used to fabricate the specimens are reported in a previous study [16].

### *2.2. IPS e.max Press surface treatments*

After being divested, the ceramic blocks were embedded in polyester resin (Resapol T208, Difibra/Fiberglass Ltda, Mogi das Cruzes, SP, Brazil) in rigid polyvinyl chloride (PVC) tubes and subjected to wet polishing with 1000-, 2500-and 4000-grit silicon carbide abrasive papers (Buehler, Lake Buff, IL, USA) in order to obtain a flat, polished and standardized surface. Then, all specimens were ultrasonically cleaned in distilled water for 20 min.

The ceramic specimens were randomly assigned to 5 groups ($n$=52) according to the hydrofluoric acid concentrations: 1%,