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Effect of acid etching on tridimensional microstructure of etchable CAD/CAM materials

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ABSTRACT

Objective. Evaluate if etching protocols affect superficial/internal microstructural integrity of CAD/CAM ceramic materials.

Methods. Sixty blocks ($3 \times 3 \times 3$ mm) of IPS/Empress-LEU, IPS/e.max-LDC (Ivoclar-Vivadent) and Enamic-PIC (VITA) were used. Lateral surfaces from each block were isolated with Teflon strip and petroleum jelly to keep them untouched. Specimens were distributed into 6 groups ($n = 10$): 1. no treatment (C); 2. hydrofluoric acid (HF) 5%, 20 s (HF5%20 s); 3. HF5%60 s; 4. HF10%20 s; 5. HF10%60 s; 6. Monobond Etch&Prime (MBEP). Surface roughness (Sa) and 3D profile were obtained using a confocal-laser-optical-microscope (LEXT OLS 4000, Olympus), while element ratios (Si/K for LEU and LDC; Si/C for PIC) were recorded using energy dispersive spectroscopy (EDS). Superior (treated) and lateral (non-treated) surfaces were analyzed using scanning electron microscopy (SEM) (JSM 5600 LV, JEOL). Etching depth was measured on lateral surfaces. Data were submitted to ANOVA-One-Way and Tukey test ($\alpha = 0.05$).

Results. For LEU, only HF10% treatments produced statistically different roughness values and Si/K ratios compared to C group. Regarding LDC and PIC, groups HF5%60 s and HF10% showed higher roughness values than C group. In the case of PIC, all treatments (except MBEP) produced lower Si/C ratios than C group. All treatments (except MBEP) produced higher etching depth values than C group for all materials, being HF10%60 s the highest (LEU:403.2 ± 11.4 μm; LDC:617.4 ± 75.7; PIC:291.6 ± 6.5 μm). HF10% produced more aggressive etching morphology patterns on superior and lateral surfaces (SEM). Treatments MBEP and HF5%20s, produced the least aggressive structural alterations. Acid etching produces superficial and internal alterations on ceramics' structural configuration.

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Significance. Aggressive etching protocols of glass-ceramics may cause internal material loss, consequently, milder etching is recommended to treat those materials before adhesion procedures.

1. Introduction

Full-ceramic restorations have been highly utilized in recent years because of their biocompatibility, improved mechanical properties, and optimal aesthetics [1]. Nowadays, a pre-sintered-ready-to-use format associated to computerized-aided technology (CAD/CAM blocks), has reached a strong position on dental ceramics market due to its convenience in reducing some laboratory steps besides maintaining materials positive characteristics [2]. In this regard, most materials available in pre-processed CAD/CAM block presentation are composites and glass-ceramics [2].

Glass-ceramics are characterized for containing a high percentage of glassy phase on its composition [3]. Inside this group of materials, leucite-based and lithium disilicate reinforced glass-ceramics combine optimal aesthetics and optical properties, along with improved mechanical properties due to reinforcement given by a crystalline phase [4]. On the other hand, pre-polymerized composite resin blocks show more flexibility and machining facility when compared to glass-ceramics [2,5]. With the intention of combining advantageous characteristics of ceramics and composites, a polymer infiltrated ceramic network material has been developed [6]. This material is composed of a feldspar ceramic network (86%wt) infiltrated by a polymer matrix (14%wt) [7]. Some advantages previously attributed to these novel materials are: improved damage tolerance, higher flexibility, less susceptibility to suffer slow crack growth [6,8], similar physical properties of those from natural teeth and comparable mechanical behavior to other ceramic materials [9].

In a recent classification of ceramic materials proposed by Gracis et al. [3], lithium disilicate and leucite-reinforced ceramics are coded as “Glass-matrix ceramics”. Polymer infiltrated ceramics (PIC) are classified as “Resin-matrix ceramics” as they are composed of 86%wt of feldspathic ceramic [3]. This implies that PICs should be treated in the same manner as glass-ceramics when preparing them to be bonded to tooth structure. Some previous works have stated that PIC respond well in terms of bond strength, to surface treatments already used to treat glass-ceramics [10–13], but other side effects of those treatments still have to be evaluated [3,12]. Two main approaches are the most accepted in literature to fulfill this task: mechanical and chemical [14]. The former consists in producing a selective glass-content removal through etching the ceramic surface using hydrofluoric acid (HF), exposing crystalline structure (when present), raise surface energy and facilitate mechanical interlocking of the resin cement [15–17]. While the later provides chemical adhesion between resin cements and silica-containing ceramic substrates [14,15]. Regarding hydrofluoric acid (HF) etching, concern is still patent as it may weaken ceramic material [18,19], or even influence its bonding performance to resin cement depending on its concentration and application time [20,21]. Recently, a simplified acid ceramic primer has been introduced, claiming to perform a mild acid etching (very smooth etching pattern) and silanize using a single solution [22], however literature regarding its performance is still quite scant.

A vast number of previous works have investigated the effect of variations on HF etching protocols on ceramic materials/resin cement bonding [10–13,19–24], ignoring the current effect of those variations on internal microstructure, amount of lost glassy phase or the power of HF to etch deep inside materials’ body. These parameters seem to be of great importance nowadays, as minimally invasive dentistry has led to a reduction in ceramic restorations’ thickness (sometimes less than 0,5 mm), leaving a very narrow margin in terms of dissolving part of materials’ structure, as small changes may produce great effects on its mechanical properties. This gains even more relevance at the time that a wide variety of materials with different compositions are available, being imperative to analyze structural effects of HF on each material according to each specific composition.

Thus, the aim of this study is to evaluate if different acid etching protocols affect the superficial microstructural integrity and glassy-phase dissolution depth on different types of CAD/CAM ceramic materials. The null hypothesis set is that variations on etching protocols have no effect on ceramics’ microstructural integrity and etching depth.

2. Materials and methods

2.1. Specimen preparation and group division

Three CAD/CAM materials were evaluated: a leucite-based glass ceramic (IPS Empress CAD-Ivoclar Vivadent- Schaan, Lichtenstein) (LEU), one lithium disilicate reinforced glass ceramic (IPS e.max CAD-Ivoclar Vivadent) (LDC) and a polymer infiltrated glass ceramic (VITA Enamic-VITA Zahnfabrik-Bad Säckingen, Germany) (PIC). Materials’ compositions are described in Table 1. Sixty blocks from each material were cut ($3 \pm 0.3 \times 3 \pm 0.3 \times 3 \pm 0.3$ (thick) mm) using a diamond saw. LDC blocks were crystallized according to manufacturers’ instructions. All blocks were polished using SiC 600# grit sandpaper (to standardize all specimens’ roughness baseline and simulate the effect produced by CAD/CAM machining process) at all faces and ultrasonically cleaned in distilled water for 5 min.

Sample size was determined using a power/sample size calculation test (Minitab v17.2.1, Minitab Inc.; State College, PA, USA) under a confidence degree of 95%. Parameters used to obtain sample size were extracted from our pilot study