

Effect of Hydrofluoric Acid Etching Duration on Fracture Load and Surface Properties of Three CAD/CAM Glass-Ceramics

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Abstract

Background: The purpose of this study was to investigate the effect of etching time using 9% hydrofluoric acid on Fracture Load (FL) and surface properties, such as Surface Free Energy (SFE) and Surface Roughness (SR) of three CAD/CAM glass-ceramics.

Methods: Following glass-ceramics were investigated: EMP (IPS Empress CAD), KLE (KLEMA CAD/CAM glass-ceramic) and EMC (IPS e.max CAD). For FL, standardized crowns for maxillary canines (N=270, n=90) were milled. Each material group was divided into six subgroups (n=15) and etched for 0, 30, 60, 90, 120, and 150s. Crowns were adhesively cemented to metal abutments and subsequently loaded until fracture. For surface property investigations, 55 specimens per material were fabricated, divided into 11 groups (n=5) and etched for: 0, 20, 30, 40, 50, 60, 75, 90, 105, 120, and 150s. SFE using contact angle measurements, SR using profilometer and surface topography with scanning electron microscopy were determined. Data were analyzed using linear covariance analyses/regressions, and two/one-way ANOVA with post-hoc Scheffé test ($p < 0.05$).

Results: Unetched EMP crowns showed lower FL than etched for 150s. Unetched and etched for 150s KLE crowns presented lower FL than those etched for 90s. Within EMC, no effect of etching time was observed. The highest SFE was detected for EMC, the lowest for KLE. The highest SR showed KLE, the lowest EMC. KLE and EMP showed no impact of etching time on SFE. EMC increased in SFE with increase of etching time. EMC showed higher SR than KLE and EMP. EMC showed lower SR than KLE. All materials showed an increase of SR dependent on the etching time.

Conclusion: General recommendations on etching time cannot be made as etching time showed different effects on fracture load and surface properties.

Key Words: Glass-ceramic, Etching time, Hydrofluoric acid, Fracture load, Roughness, Surface free energy

Introduction

Recent trends in aesthetic dentistry include the increased substitution of prosthetic restorations manufactured on metal basis. Against this backdrop, dental ceramics are extensively used because of their similar optical properties to the natural tooth structure [1], physical and mechanical characteristics [2-4], and biocompatibility [5]. Different classes of glass-ceramic materials are available for use in Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) restorative dentistry: feldspar/leucite ceramics based on silica and alumina [6], ceramics containing crystalline lithium disilicate, or recent developments such as hybrid ceramics with a dual-network structure [7]. In summary, all these glass-ceramics show differences in optical, mechanical and chemical properties dependent on their composition.

Lithium disilicate ceramics show higher flexural strength and fracture toughness compared to leucite-based ceramics, based on the higher volume fraction of crystals and therefore a tighter interlocking matrix of the disilicate-based materials [8-10]. Adhesive cementation with resin-based composite cements enhances additionally the clinical efficiency [11] and increases the stability and fracture resistance of those restorations [12-17]. Therefore, glass-ceramics should be cemented adhesively [18]. An interaction between the resin composite cement and the micro porosities of the ceramic is determined by the capability of the resin cement to wet the

ceramic surface [19], dependent on the surface chemistry and roughness of the ceramic [20] as well as the viscosity and composition of the resin cements [21]. Before cementation, all glass-ceramic surfaces must be etched using hydrofluoric acid (HF) for increase of Surface Roughness (SR) and Surface Free Energy (SFE). A clinical study showed the importance of bonding where acid etching of glass-ceramic crowns decreased the annual failure risk by about 50% [22]. However, previous studies have also reported about a weakening effect of HF etching on glass-ceramics [23].

In general, any ceramic surface is inert and does not adhere readily to other materials. Achieving defined SFE and SR is necessary for proper bonding. The SFE is defined, as the work required for increasing the area of a substance of 1 cm². It can be determined by measuring the contact angle formed by a range of liquids on a defined surface (e.g. water and diiodomethane) [24-26]. When adhesion is required, high SFE is favored and, on the contrary, undesirable when plaque accumulation should be avoided [25,27]. The wettability of a solid surface by a liquid is estimated by the dimensions of the contact angle; the lower the contact angle, the more the wettability of the surface [20,24,28-30]. Etching is a reliable procedure to have a dissolving effect on the superficial layer of silicate ceramics [31,32]. This roughly etched surface offers more SFE [19,28]. Using HF is the predominantly used surface treatment process prior to resin bonding [33]. In vitro studies observed a positive

effect of HF etching on the surface topography by increasing its roughness [34-36], leading to micromechanical retention of the luting cement [37,38]. Applied on glass-ceramics, this method removes the glass matrix selectively and exposes the crystalline structure beneath [31,32]. However, the authors identified limited information to date about the influence of etching time of different glass-ceramics on SR, wettability, SFE, and surface topography.

Given that both the crystalline content and the etching time may affect the surface properties and the stability of the ceramic restoration, this study aimed:

(a) To evaluate the influence of etching time on fracture loads of CAD/CAM glass-ceramic crowns and

(b) To investigate the effect of HF etching duration on surface properties of glass-ceramics. The null hypothesis tested that the etching time has no effect on fracture load and on surface properties.

Materials and Methods

Three types of CAD/CAM glass-ceramics were selected for the Fracture Load (FL) and surface property measurements: IPS Empress CAD (EMP), KLEMA CAD/CAM glass-ceramic (KLE), and IPS E.MaxCAD (EMC). *Table 1* shows all tested CAD/CAM glass-ceramics, their chemical composition, manufacturers, lot numbers, and the abbreviations used in this study.

Determination of fracture load

A metal tooth analog with the shape of a prepared maxillary canine with a chamfer preparation of 1 mm was cast from a Cobalt-Chrome (CoCr) alloy (Zenotec NP; Wieland Dental) and was then scanned using the Cerec AC intraoral scanning device with Bluecam (Sirona Dental Systems). An anatomical master upper jaw canine crown was digitalized and a master Surface Tessellation Language (STL) dataset was generated (Software Cerec 4.1; Sirona Dental Systems) for milling of the standardized crowns (*Figure 1*). Ninety identical crowns of each glass-ceramic-based material were milled (SironaCerec

MC XL (D3439); Sirona Dental Systems). This resulted in a total number of 270 specimens. The crowns showed a layer thickness of 4.95 mm at their incisal peak portion and an axial wall thickness diminishing to 1.0 mm at the margin, with a maximum height of 11.96 mm and a maximum width of 7.74 mm. The crowns were then fitted to the metal abutments. The adjustment was fundamental so that every crown could reach the maximum seating at the margin. The glazing of the crowns was performed according to the manufacturers' instructions. *Table 2* describes the manufacturers, furnaces, of the used glazing pastes.

Before cementation, the crowns were ultrasonically cleaned in distilled water for 5 min (Sonorex RK102H; Bandelin electronic), air-dried with care and then randomly divided into 6 subgroups for different etching times: 0 s (control group), 30, 60, 90, 120 and 150 s. Each subgroup included 15 specimens. The internal surfaces of the crowns were etched with 9% HF (LOT B863L; Ultradent Products Inc.), rinsed with flowing water for 15 s, ultrasonically cleaned (isopropyl alcohol; Merk) and adhesively cemented (Multilink Automix, LOT S0382; Ivoclar Vivadent) on the metal abutment. Excess material was carefully removed and crowns were light cured for 60 s each from the mesial, distal, palatal and vestibular sides (Elipar S10; 3M ESPE).

After 24 h, the cemented crowns were measured in a universal testing machine (1 mm/min, Zwick 1445; Zwick). The load was induced with a flat jig on the oral surface of the incisal edge at an angle of 45 degrees to the long axis of the tooth (*Figure 2*). A tin foil (LOT 432819; Dentaurum) with a thickness of 0.5 mm was placed between the crown and the loading jig to avoid load peaks in the contact area.

Determination of surface properties

Fifty-five specimens with a height of 1.5 mm, length of 10 mm, and width of 10 mm were fabricated from each glass-ceramic. This resulted in a total number of 165 specimens. For this, the CAD/CAM blocks were sectioned under water cooling (Accutom-50; Struers) with a diamond cut-off wheel

Table 1. Summary of all tested glass-ceramic-based materials, their abbreviations, Lot numbers, manufacturers, and chemical composition in weight percentage (%).

Ceramic	Abbreviation	Lot No.	Manufacturer	Composition
IPS Empress CAD	EMP	J17565	Ivoclar Vivadent, Schaan, Liechtenstein	SiO ₂ 60%-65%, Al ₂ O ₃ 16%-20%, K ₂ O 10%-14%, Na ₂ O 3,5%-6.5%, other oxides 0.5%-7%, pigments 0.2%-1%
KLEMA CAD/CAM glass-ceramic	KLE	2008/ K-4849	Klema Dentalprodukte, Meiningen, Austria	SiO ₂ 55%-65%, Al ₂ O ₃ 20%-25%, K ₂ O 5%-10%, Na ₂ O 8%-12%, MgO < 0.1%, CaO 1%-2%, BaO 0.5%, TiO ₂ , ZrO ₂ , P ₂ O ₅ , CeO ₂ , CeF ₃ , SnO ₂ < 0.1%, pigments 1%-5%
IPS e.max CAD	EMC	P81551	Ivoclar Vivadent, Schaan, Liechtenstein	SiO ₂ 57%-80%, Li ₂ O 11%-19%, K ₂ O 0%-13%, P ₂ O ₅ 0%-11%, ZrO ₂ 0%-8%, ZnO 0%-8%, Al ₂ O ₃ 0%-5%, MgO 0%-5%, pigments 0%-8%

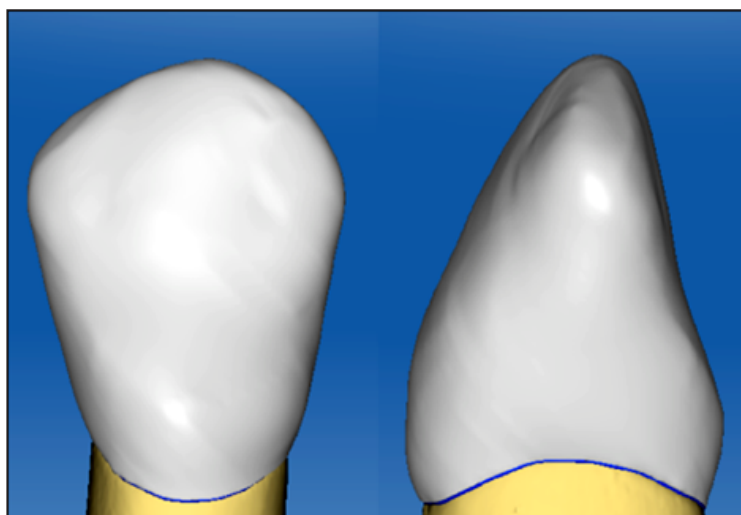


Figure 1. Screenshots of the CAD/CAM canine crowns (Cerec software).

Table 2. Summary of the firing processes used for glazing the ceramic crowns previous to fracture load measurement. Labelling, Manufacturer and Lot numbers of the glazing pastes.

Ceramic	Glazing paste	Furnace
Empress CAD	IPS Empress Universal Glaze Paste (D64847) IPS Empress Universal Glaze Liquid (E15607) Ivoclar Vivadent, Schaan, Liechtenstein	Vita Vacumat 40 VITA Zahnfabrik, Bad Säckingen, Germany
KLEMA CAD/CAM	Glasur NT Paste (B196) Glasur NT Liquid (K4142) Klema Dentalprodukte, Meiningen, Austria	Vita Vacumat 40 VITA Zahnfabrik, Bad Säckingen, Germany
IPS e.max CAD	IPS e.max CAD Crystall./Glaze Paste (N01905) IPS e.max CAD Crystall./Glaze Liquid (L49949) IPS Object Fix Putty (S06646) Ivoclar Vivadent, Schaan, Liechtenstein	Programat EP 5000 Ivoclar Vivadent, Schaan, Liechtenstein

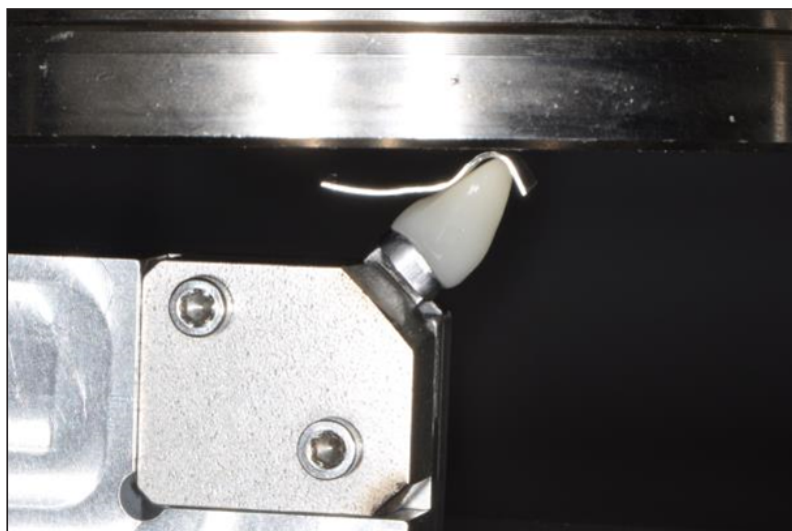


Figure 2. Test design of the FL experiment.

with a cutting speed of 1,000 rpm and a medium force of 40 N. Subsequently, all specimen surfaces were polished (in the following order: 40 μm diamond pad, 20 μm diamond pad, MD-Largo + DiaPro Allegro/Largo, MD-Largo + DiaPro Largo, MD-Chem + OP-S) with a microprocessor-controlled tabletop machine (Abramin; Struers). EMC specimens were additionally crystallized in a press furnace (Programat EP 5000; IvoclarVivadent) with the following crystallization parameters: closing time: 6 min; stand-by temperature: 403°C; heating rate: 90°C/min; holding time: 10 min; heating rate: 30°C/min; firing temperature: 840°C; holding time: 7 min; and long-term cooling: 700°C/min. Each glass-ceramic-based material was then randomly divided into 11 subgroups and

etched (9% HF, LOT B6X7B; Ultradent Products Inc.) with the following etching times: 0 (control group), 20, 30, 40, 50, 60, 75, 90, 105, 120, and 150 s. The etched specimens were adhered to aluminum SEM carriers for better fixation in the following experiments and manually cleaned with distilled water prior to performing the measurements of SFE and SR.

Surface free energy

To measure the contact angle between a liquid (water/diiodomethane) and a solid (glass-ceramic-based specimens), a special device (Krüss Easy Drop; Krüss GmbH) was used. The contact angle device with a manual double dosing system with two glass syringes, one filled with distilled water and the other with diiodomethane (99%, CAS: 15.842-9, LOT:

S65447-448; Sigma-Aldrich), was used. Each test drop of water contained 10 μl , and each test drop of diiodomethane contained 5 μl of the respective fluid. Measurement was executed 5 s after the drop made contact with the specimen surface. The contact angle was determined for six independent drops of liquid (three drops of water and three drops of diiodomethane). The tangent-1 method was used for angles above 20 degrees and the circle method for angles under 20 degrees. The SFE was calculated from the results of the Owens-Wendt-Rabel-Kaelble method [39,40].

Surface roughness

The SR measurements were provided using a profilometer (Mar Surf M400; Mahr). To achieve accurate and reproducible results, the specimens were fixed in a special holding device to retain the surface parallel to the platform of the machine. The 90° measuring sensor has a diamond probe tip (diameter 2 μm). The contact force was approximately 0.7 mN. Six measurements with a measuring track of 6 mm per specimen were performed. The means of these six measurements were calculated for each specimen.

SEM surface topography

For Scanning Electron Microscopy (SEM) analyses, two specimens per subgroup were selected. The specimens were ultrasonically cleaned in distilled water and then gold sputtered (layer thickness: 6 nm). Surface topography was evaluated under a SEM (Carl Zeiss Supra 50 VP FESEM; Carl Zeiss) operating at 10 kV with a working distance of 7.0

mm to 12.4 mm.

Statistical analysis

Descriptive statistics were calculated. The normality of data distribution was tested using the Kolmogorov-Smirnov and Shapiro-Wilk tests. Two- and one-way ANOVA followed by a post-hoc Scheffé test were used to determine the significant differences between groups. In the next step, the data were plotted in scatter diagrams. Linear covariance analysis was computed to investigate the differing associations provided by the glass-ceramics between etching time and outcomes (such as SFE and SR). In addition, given significant interactions ($p < 0.001$), linear regressions for each outcome with respect to each etching time for all tested materials were computed separately. P values smaller than 5% were considered to be statistically significant in all tests. The data were analyzed using SPSS Version 20 (SPSS Inc., Chicago, Illinois).

Results

Fracture load

The descriptive statistics for the FL results are summarized in Table 3. Figure 3 presents the boxplots for all three tested glass-ceramics dependent on the etching time. Kolmogorov-Smirnov and Shapiro-Wilk test indicated no violation of the assumption of normality of all tested groups. Therefore, for the statistical analysis a normal distribution assumption was employed. Within EMP, crowns without etching showed significantly lower FL than crowns etched for 150 s. For

Table 3. Mean and standard deviation for fracture load values (N). Differing letters within a row correspond to differing FL means between treatment groups according to the post hoc Scheffé test.

Etching time	EMP	KLE	EMC
	Mean \pm SD	Mean \pm SD	Mean \pm SD
0 s	646,57 \pm 143,26 ^a	533,88 \pm 101,91 ^b	1380,51 \pm 397,34 ^a
30 s	810,32 \pm 204,60 ^{ab}	445,15 \pm 101,74 ^{ab}	1390,40 \pm 237,49 ^a
60 s	815,44 \pm 183,2 ^{ab}	505,20 \pm 92,36 ^{ab}	1227,86 \pm 277,37 ^a
90 s	766,91 \pm 170,09 ^{ab}	408,35 \pm 121,28 ^a	1318,70 \pm 221,16 ^a
120 s	679,77 \pm 125,4 ^{ab}	471,48 \pm 76,36 ^{ab}	1241,37 \pm 207,49 ^a
150 s	852,71 \pm 139,4 ^b	544,20 \pm 67,95 ^b	1107,52 \pm 180,54 ^a

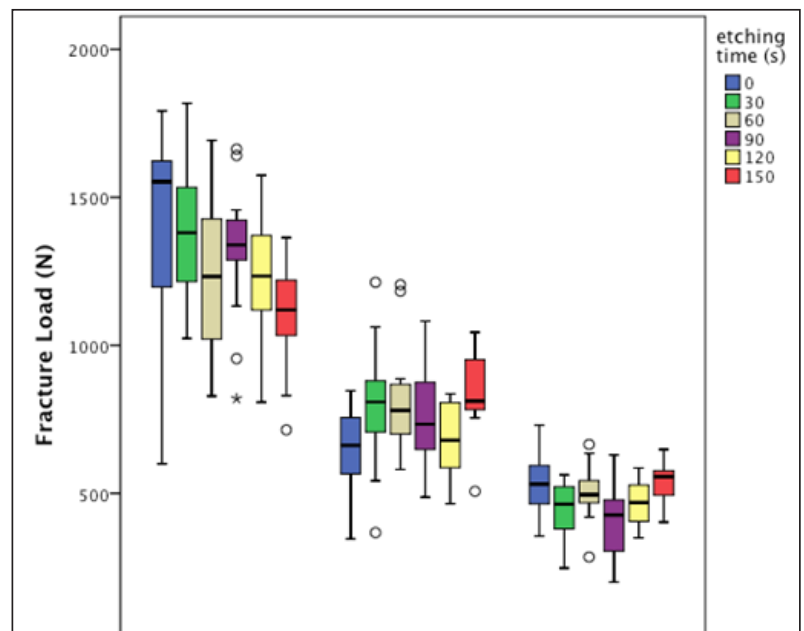


Figure 3. Boxplot with FL-values (N) for each tested glass-ceramic-based material at each etching time level.

KLE, crowns without etching and crowns etched for 150 s, presented significantly lower FL than crowns etched for 90 s. Within EMC, etching time showed no significant effect on FL.

Surface properties

Surface free energy: According Kolmogorov-Smirnov and Shapiro-Wilk tests 6% of the tested groups (2 of 33 groups) showed a violation of the assumption of normal distribution. Therefore, the statistical differences between the tested groups were analyzed using linear covariance analyses and linear regressions. The descriptive statistics for the SFE results are summarized in *Table 4*. *Figure 4* presents the scatter diagram for all three tested glass-ceramic-based materials dependent on the etching time. In general, KLE and EMP showed no impact of etching time on SFE. By contrast, EMC presented an increase in SFE with an increase of etching time. The measured SFE for EMC was higher than those of KLE and EMP.

Surface roughness: According Kolmogorov-Smirnov and Shapiro-Wilk test indicated no violation of the assumption of

normality for 97% of the tested groups. Only 3% were not normally distributed (this 1 not normally distributed group out of 33 contained no outliers) which is the type I error for a statistical test. Therefore, the statistical differences between the tested groups were analyzed using linear covariance analyses and linear regressions. *Table 4* shows the mean SR with standard deviations of all tested glass-ceramics. EMC showed significantly lower mean SR than KLE.

No differences between KLE and EMP were observed. All materials showed an increase of SR dependent on the etching time (*Figure 5*). No associations between SFE and SR were observed.

SEM surface topography: The SEM pictures are presented in *Figure 6*. EMP and KLE showed a clear change in surface topography with an increase of etching time. The etching time of EMC caused only minimal changes of the surface. EMP showed a “swiss hole cheese pattern” after etching while KLE

Table 4. Mean and standard deviation for surface free energy (SFE) values (mN) and surface roughness (SR) values (μm). Different letters within a row correspond to differing SFE or SR means between treatment groups according to the post hoc Scheffé test.

Etching time	SFE			SR		
	EMP	KLE	EMC	EMP	KLE	EMC
	Mean \pm SD	Mean \pm SD	Mean \pm SD	Mean \pm SD	Mean \pm SD	Mean \pm SD
0 s	56.7 \pm 12.7 ^b	41.2 \pm 13.5 ^b	55.1 \pm 8.8 ^a	0.67 \pm 0.16 ^a	0.63 \pm 0.58 ^a	0.02 \pm 0.01 ^a
20 s	26.7 \pm 2.3 ^a	16.9 \pm 3.5 ^a	70.9 \pm 1.8 ^b	0.72 \pm 0.37 ^a	1.19 \pm 0.67 ^{abc}	0.07 \pm 0.01 ^{ab}
30 s	29.9 \pm 6.4 ^a	15.3 \pm 1.9 ^a	67.9 \pm 3.1 ^b	0.82 \pm 0.67 ^{ab}	1.02 \pm 0.22 ^{ab}	0.07 \pm 0.01 ^{ab}
40 s	21.1 \pm 2.8 ^a	15.4 \pm 1.8 ^a	64.0 \pm 6.1 ^{ab}	0.93 \pm 0.16 ^{ab}	1.41 \pm 0.15 ^{bc}	0.08 \pm 0.01 ^{ab}
50 s	24.1 \pm 3.9 ^a	13.6 \pm 4.3 ^a	69.2 \pm 2.6 ^b	0.81 \pm 0.58 ^{ab}	1.51 \pm 0.14 ^{bcd}	0.09 \pm 0.01 ^{ab}
60 s	20.8 \pm 3.9 ^a	19.8 \pm 6.8 ^{a*}	70.4 \pm 0.6 ^b	1.23 \pm 0.92 ^{bc}	1.68 \pm 0.10 ^{cd}	0.10 \pm 0.02 ^{ab}
75 s	18.2 \pm 6.2 ^a	13.3 \pm 1.2 ^a	70.0 \pm 2.4 ^b	1.23 \pm 0.3 ^{bc}	1.52 \pm 1.00 ^{bcd}	0.10 \pm 0.01 ^{ab}
90 s	40.1 \pm 13.5 ^{ab}	12.0 \pm 1.8 ^a	69.1 \pm 1.5 ^b	0.98 \pm 0.15 ^{ab}	1.58 \pm 0.43 ^{bcd}	0.10 \pm 0.01 ^{ab}
105 s	41.3 \pm 7.9 ^{ab}	12.1 \pm 1.6 ^a	68.4 \pm 3.1 ^{b*}	0.91 \pm 0.12 ^{ab}	1.16 \pm 0.09 ^{abc}	0.13 \pm 0.02 ^b
120 s	33.3 \pm 14.2 ^{ab}	13.9 \pm 2.3 ^a	69.6 \pm 2.4 ^b	0.91 \pm 0.32 ^{ab}	1.53 \pm 0.17 ^{bcd}	0.15 \pm 0.06 ^b
150 s	17.9 \pm 5.7 ^a	10.9 \pm 2.2 ^a	73.1 \pm 1.7 ^b	1.50 \pm 0.15 ^c	2.06 \pm 0.31 ^d	0.15 \pm 0.07 ^{b*}
Lin. reg. intercept	34.81	24.72	63.58	0.713	0.973	0.043
Slope	-0.071	-0.119	0.065	0.004	0.006	0.001

Figure 4. Scatter diagram of Surface Free Energy (SFE) for each tested glass-ceramic-based material at each etching time level.

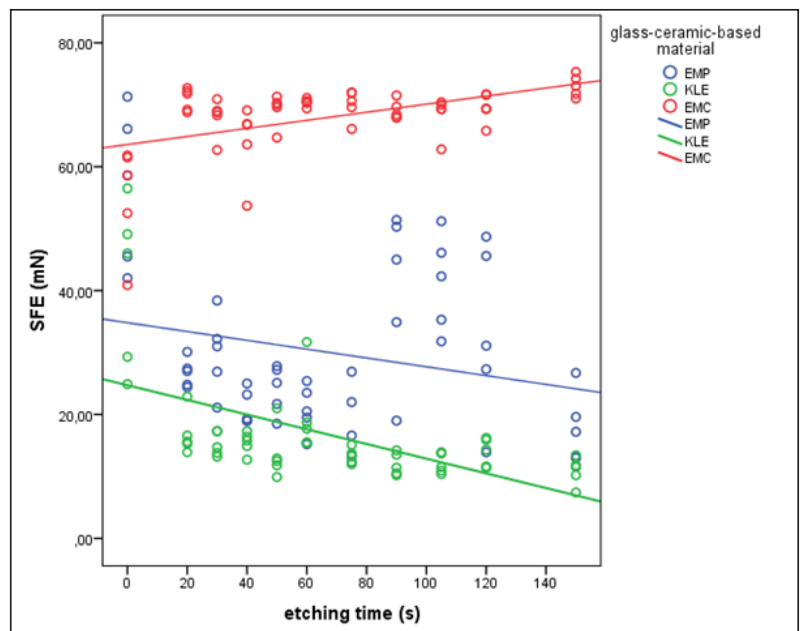


Figure 5. Scatter diagram of surface roughness (SR) for each tested glass-ceramic-based material at each etching time level.

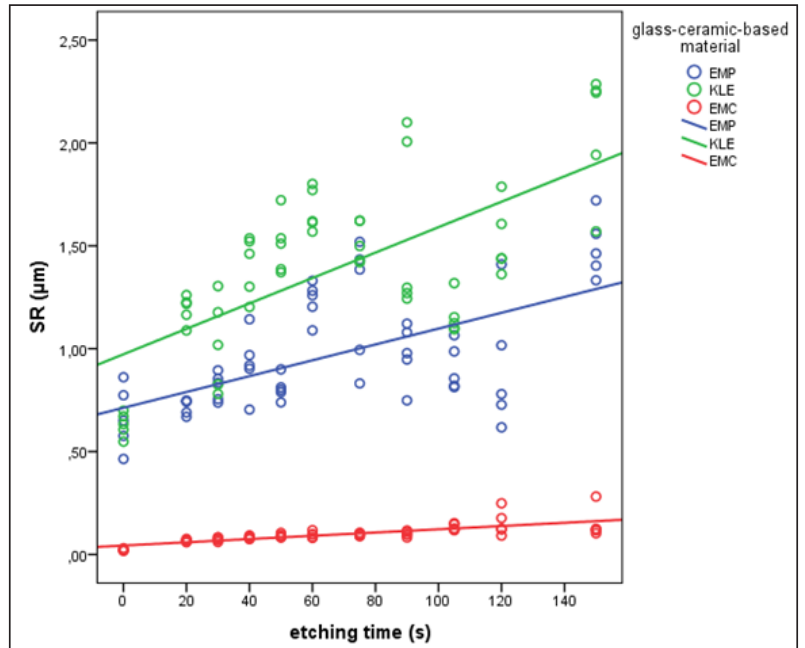
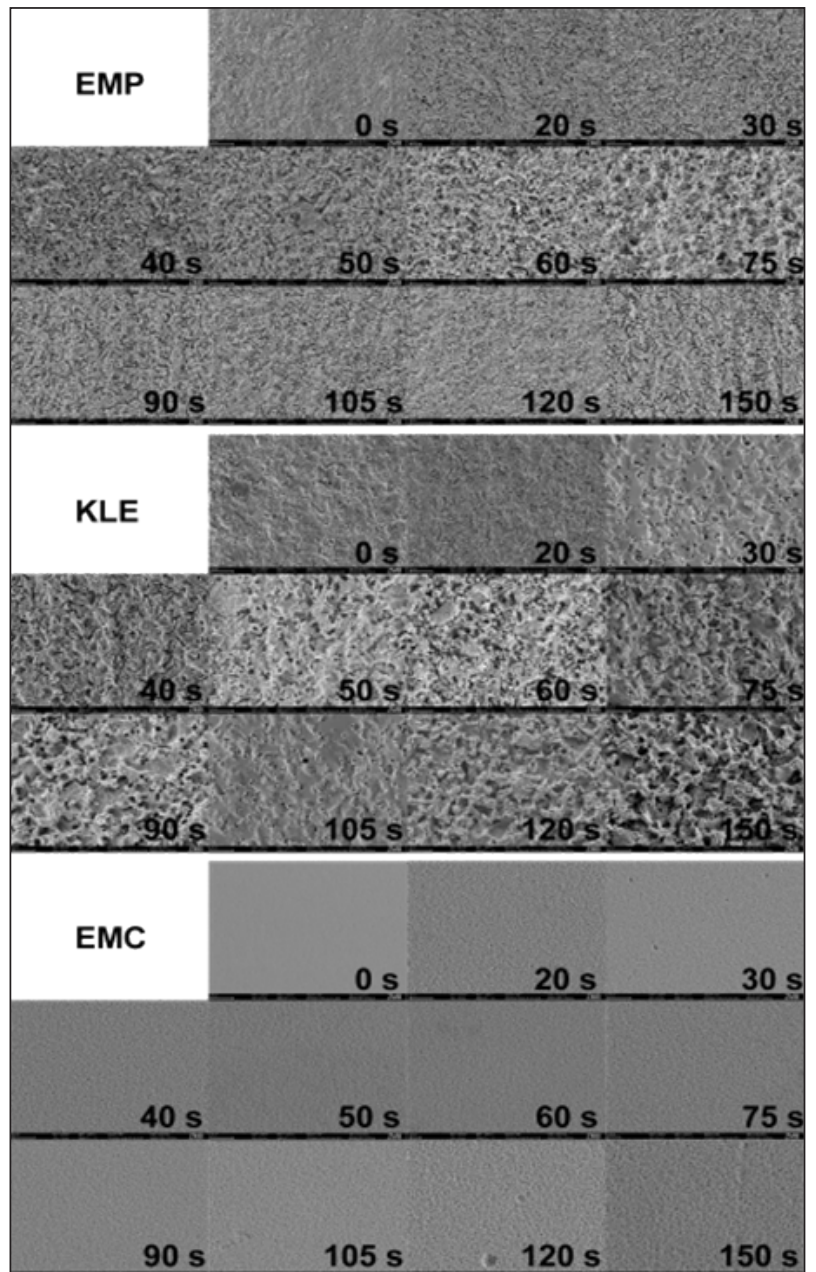


Figure 6. SEM pictures of etched ceramic surfaces in order of glass-ceramic-based materials and etching time.



exhibited very irregular features, such as big holes, fissures, scratch-like gaps, and areas with grain pullout.

Discussion

This study investigated the influence of different HF etching durations on the FL values for lithium disilicate-based (EMC) and two leucite-based ceramics (EMP and KLE). EMP is commonly used and established in CAD/CAM restorative dentistry, whereas KLE is a new product that has yet to be tested clinically and experimentally. The results show that similar surface treatments were associated with significantly different dependencies of etching time for the leucite-based ceramics on the FL values. Therefore, the null hypothesis that the etching duration has no effect on the FL has to be rejected.

The microstructural difference between the two leucite ceramics is a major controlling factor on the quality of adhesive bonding after etching. Thorough observation of SEM pictures shows that the degree of crystallinity of EMP is higher than that of KLE. The etched surface of KLE exhibits very irregular features, probably caused by the preferential attack of HF on the grain boundaries at the interface of leucite crystals and the glass phase. Deep fissures, scratch-like gaps, and areas with grain pullout may affect the material flow and interpenetration of the etched ceramic surface by the resin cement, leaving deeper defects unreached by the cement and resulting in higher instability. During the visual examination of the fracture types, the resin cement often stayed on the metal abutment for KLE, illustrating a failure at the ceramic-cement interface. In contrast, the resin composite cement was still adhering to the ceramic for groups EMP. Further studies should investigate the influence of different silane coupling agents prior to cementation and whether they may improve the quality for bonding of KLE.

For EMC, the FL values measured in this study correspond to the observed surface properties (SFE, SR, and SEM). Given that the different etching durations did not induce significant morphologic changes, and surface roughening was least efficient for EMC, no significant dependence between FL values and etching time was expected. Although this lithium disilicate ceramic is a brittle material, its strength is so high that cementing with adhesive resin cements after thorough etching and bonding cannot further increase the FL. Among recent studies, one study showed that cementation type has no significant effect on fracture load results for lithium disilicate-based ceramic [18].

Additionally to the determination of the FL, the surface properties of the glass-ceramics were investigated. It could be shown that the etching time had a significant effect on the SFE of the tested materials. In general, different effects of etching times on the tested materials were observed. For KLE and EMP the SFE decreased with an increase of etching time while, on the contrary, the SFE increased for EMC. Roughening by HF etching was the least efficient for EMC, and the highest SR values were measured for KLE. Therefore, the tested null hypothesis that all tested materials present similar surface properties after HF treatment dependent on the etching time is rejected. All tested materials are based on silica oxide combined with different reinforcement particles. Therefore, it can be stated that the percentage of silica oxide as

well as the different particles such as alumina oxide, leucite, and lithium silicate have a significant effect on the etching duration and surface properties.

Knowledge of the SFE of dental materials and especially the interaction with different liquids is very important. Numerous approaches for measuring the SFE have been described in the literature. According to Owens et al., using the geometric mean approach or the harmonic mean method, SFE can be estimated by measuring contact angles with two liquids [39]. In this study, a two-liquid method was used, making a distinction between dispersive and polar components. Still, very controversial opinions exist regarding the most accurate method in defining SFE. Combe et al. suggested at least five test liquids for precise results [26], while Carlen et al. favored three liquids [25]. Furthermore, according to the literature available, discrepancies exist in view of the surface tension of the test liquids what may affect the experimental results. Unfortunately, a comparison with other glass-ceramics cannot be made because no other studies dealing with SFE values were identified.

Acid etching of ceramic has been widely used in dentistry to increase retention between the resin composite cement and the ceramic restoration. Etching is a dynamic process, and the effect is dependent on the type of etchant, etching time, ceramic microstructure, plus composition [35]. It causes a preferential dissolution of the weaker glass phase and the introduction of new surface defects or the extension of preexisting ones [33,35]. HF etching provides the necessary roughness for mechanical interlocking; nevertheless, over-etching was described to weaken the porcelain [35]. These considerations have encouraged numerous studies to attempt the adequate HF etching duration for micromechanical retention of all types of different ceramic products [28,37]. Jardel et al. concluded that HF gel combined with a silane coupling agent is the most effective treatment for ceramic surfaces [28]. Zogheib et al. etched a lithium-disilicate ceramic using 4.9% HF for different etching periods and measured significantly higher roughness values for all etching periods than for the unetched group [37]. Similar results were achieved by Wolf et al., who studied the surface properties of a feldspathic ceramic etched with 9.5% HF for different etching times to find a positive correlation between roughness and etching period [36], agreeing with further literature [35] and with the present results. In the present study 9% HF etching resulted in rougher. This result was highly anticipated because all tested ceramics contain a glass matrix with a high silicate weight percentage (*Table 1*). The efficiency of surface treatment is highly dependent on the composition of the ceramics. In contrast, ceramics with high alumina content and no glass phase remained unetched [33,34].

In the present study, the SEM images correspond with the measured SR - the longer the etching time, the more pronounced the microscopic irregularities in the images. The SEM micrographs clearly revealed the effect of the different etching periods on the microstructure of the ceramic. HF etching significantly modified the morphological surface of EMP and KLE while the morphological characteristics of EMC with its typical elongated crystal structure remained

constant.

In contrast to the situation in vivo, the specimens in this laboratory test design of FL were not stressed under cyclic loading. This can be considered as a limitation of the study, because materials tested in the laboratory should produce failures that are comparable with those in clinical situations [38]. In addition, the crowns were not exposed to a humid environment, which is the case in clinical scenarios. Further studies should examine the effect of dentin bonding on the FL because a CoCr abutment does not represent the important qualities of the natural tooth substance and supports conceivably the crowns by its high modulus of elasticity during the loading test. One further limitation of this study is that the effect of only a single concentration of HF (9%) was evaluated. There are other acid etchants, such as phosphoric acid, that do not etch ceramics but still have to be considered because they may improve the SFE by cleaning the ceramic surface. Additionally, further studies should investigate the

effect of etching duration on flexural strength and the dynamic fatigue of the ceramic materials.

Conclusions

Within the limitations of this study, the following can be concluded:

- Each tested glass-ceramic presented a different effect of etching time on the surface properties and fracture loads.
- General recommendations on the etching time cannot be made.

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Conflict of Interest

The authors disclose that no conflict of interest exists.

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