Caspian J of Dent Res

Original Article

Effect of hydrofluoric acid concentration and etching time on the surface roughness of CAD/CAM ceramics

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Received: 15 Apr 2018 Accepted: 28 Aug 2019

Abstract

Introduction: Dental ceramics are considered as materials that can restore the appearance of natural teeth. Etching the inner surface of a ceramic restoration with hydrofluoric acid (HF) followed by using a silane coupling agent is a well-known and recommended method to increase the bond strength. The aim of etching on ceramic structure is to enhance the surface roughness (Ra) and energy and to cleanse the bonding area. The aim of this study was to evaluate the effect of different HF concentrations and etching times on the Ra of IPS e.max CAD™ and Vita mark II™.

Material & Methods: Two HF concentrations (5% and 10%) and three etching times (20, 60 and 120 seconds) were evaluated. Etched patterns were observed by scanning electron microscopy (SEM) and Ra was measured using atomic force microscopy (AFM). Surface element analysis was performed using energy dispersive X-ray spectroscopy (EDAX). Data were analyzed on SPSS 20 using ANOVA and T-test.

Results: The Ra had no significant difference among various Vita mark II™ specimens (P=0.973). Among IPS e.max™ specimens etched with 5% HF, the AFM results showed that 20-s etching time had the lowest Ra and among those etched with 10% HF and 120-s etching time had the most Ra. In IPS e.max™ specimens etched with acid for 20 s, a significant difference was observed in Ra of 5% and 10% acid concentrations (5% HF lower than 10% HF) (p=0.012).

Conclusion: Among IPS e.max™ specimens etched with 5% and 10% HF, increasing the etching time lead to higher Ra. For both IPS e.max™ and Vita mark II™, 20-s etching with 5% HF provides acceptable Ra for the bond.

Keywords: Ceramics, Hydrofluoric acid, Scanning electron microscopy

Citation for article: Mokhtarpour F, Alaghehmand H, Khafri S, Mahdian M. Effect of hydrofluoric acid concentration and etching time on the surface roughness of CAD/CAM ceramics. Caspian J Dent Res 2019; 8: 8-15.

[Downloaded from cjdr.ir on 2026-11-03]

[DOR: 20.1001.1.22519890.2019.8.2.1.8]

[DOI: 10.22088/cjdr.8.2.8]

تأثیر غلظت اسید هیدروفلوئوریک و زمان اچ کردن بر خشونت سطحی سرامیک های CAD/CAM

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چکیده

مقدمه: سرامیک های دندانی به عنوان موادی ارزیابی می شوند که می توانند نمای دندان های طبیعی را بازسازی کنند. اچ کردن سطح داخلی ترمیم های سرامیکی با هیدروفلوئوریک اسید و به دنبال آن کاربرد عامل جفت‌کننده سیلانی روشی شناخته و توصیه شده برای افزایش استحکام باند می باشد. هدف اچ کردن ساختار سرامیکی، افزایش خشونت سطحی و انرژی سطحی و تمیز کردن ناحیه باند می باشد. هدف این مطالعه ارزیابی تأثیر غلظت های مختلف اسید هیدروفلوئوریک و زمان های اچ با آن بر خشونت سطحی سرامیک های IPS e.max^{TM} CAD و Vita mark II^{TM} می باشد.

مواد و روش ها: دو غلظت مختلف اسید هیدروفلوئوریک (۵٪ و ۱۰٪) و سه زمان مختلف اچ (۲۰و ۶۰ و ۱۲۰ ثانیه) مورد ارزیابی قرار گرفت. الگوی اچ توسط میکروسکوپ الکترونی SEM بررسی شد و میزان خشونت سطحی توسط AFM اندازه گیری گردید. آنالیز عناصر سطحی هم با استفاده از طیف‌سنجی پراش انرژی پرتو ایکس انجام شد. داده‌ها در $^{+}$ نسخه ۲۰ با استفاده از ANOVA و T-test انجام شد.

یافته ها: خشونت سطحی بین گروه های مختلف در سرامیک Vita mark II^{TM} تفاوت معنی داری را نشان نداد (P=0.973). نتایج AFM نشان داد بین گروه های سرامیک IPS e.max^{TM} که با اسید ۵٪ اچ شدند کمترین میزان خشونت سطحی در ۲۰ ثانیه اچ دیده شد و بین گروه های اچ شده با اسید ۱۰٪، ۱۲۰ ثانیه اچ بیشترین خشونت سطحی را ایجاد کرد. در گروه سرامیک های IPS e.max^{TM} در زمان ۲۰ ثانیه اچ با اسید، تفاوت معنی‌داری در خشونت سطحی غلظت های اسید ۵٪ و ۱۰٪ مشاهده شد (در غلظت ۵٪ کمتر از ۱۰٪) (p=0.012).

نتیجه گیری: بین گروه های سرامیک IPS e.max^{TM} با اسید ۵٪ و ۱۰٪ اچ شدند ، افزایش زمان اچ کردن باعث افزایش خشونت سطحی می شود. برای هر دو سرامیک IPS e.max^{TM} و Vita mark II^{TM} ۲۰ ثانیه اچ با اسید ۵٪ خشونت سطحی قابل قبولی را برای باند فراهم می کند.

واژگان کلیدی: سرامیک ، اسید هیدروفلوئوریک ، میکروسکوپ الکترونی روبشی

Introduction

Dental ceramics compared to other materials are a group of restorative dental materials with high esthetic properties and great ability to simulate the appearance of natural tooth. [1] The use of all-ceramic prostheses many of which can be fabricated through traditional laboratory methods or CAD/CAM technology has become increasingly popular in restorative dentistry. The traditional method is unpredictable, technique sensitive and time-consuming owing to many variables; therefore, the CAD/CAM technology may be an appropriate alternative for dental practitioner and laboratory. [2] CAD/CAM may also decline the fabrication time of high-strength ceramics by 90%. Moreover, the industrial fabricated blocks with minimal flaws are more homogenous. [3] Silica-based ceramics include lithium disilicate and feldspathic ceramic. Feldspathic Glass and leucite (potassium aluminosilicate) constitute the feldspathic porcelain, and lithium disilicate ceramics are composed of about 65%

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[DOR: 20.1001.1.22519890.2019.8.2.1.8]
[DOI: 10.22088/cjdr.8.2.8]

volume of highly interlocking lithium disilicate crystals dispersed in a glassy matrix. [4]

In 1991, Vita mark II (Vita Zahn-fabrik, Bad Saöckingen, Germany) as a CAD/CAM feldspathic ceramic was introduced for Cerec systems [5] and IPS™ e.max CAD (Ivoclar-Vivadent) as a lithium disilicate ceramic was presented for chairside use in 2006. [3, 6]

The porcelain laminate veneers should rely on the synergistic bond between porcelain and resin cement to survive in the oral environment. [7, 8] Internal porcelain surface modification for enhanced bond strength may be achieved via air abrasion with alumina particles or exposing the porcelain surface to acid. [9, 10] Etching the inner surface of some kind of ceramic restorations such as feldspathic and lithium disilicate with hydrofluoric acid (HF) followed by using a silane coupling agent is a well-known and recommended method to increase the bond strength. [11- 13] This process is not applicable on the zirconia-based ceramics. [6]

This surface pre-treatment method is adopted to boost the surface energy and roughness as well as cleanse the bonding area. [14] The etched porcelain surface dissolves various porcelain phases preferably depending on the porcelain composition and makes more conducive surface for bonding. [15, 16] In ceramic surface treatment, the acid reacts with silica glass matrix and glass matrix is selectively removed. As a result, the ceramic surface becomes rough, leading to micromechanical retention on the ceramic surface. [17- 19] In addition, this etched surface helps to provide more surface energy before mixing with silane solution. [18, 20] Ever since the introduction of HF acid etching as a ceramic surface pre-treatment for resin bonding, various etching protocols have been proposed. [1] The increase of etching time from 0 to 120 s using HF acid is associated with higher shear bond strength (SBS) between resin adhesive and dental CAD/CAM porcelain. [19] The recommended etching time, on behalf of manufacturer, for cementation of the IPS e.max Press glass ceramic restorations with a luting resin is 20 s. In 1998, Chen et al. have suggested that the maximum bond strength is gained by using the 120-s etching time with 5% HF acid for Vita mark II. [19] Nevertheless, clinically, the optimal concentration and etching time of HF acid for the treatment of glass ceramic restoration are unclear and there is lack of sufficient evidence on appropriate etching time for CAD/CAM ceramics. Hence, knowing the optimal and proper HF etching time for resin cement bonding without weakening the ceramic is very important. [1]

The aim of this study was to evaluate the effect of different etching times and HF concentrations on the roughness of feldspathic and lithium disilicate CAD/CAM ceramics as well as the analysis of surface elements in each surface treatment protocols. Null hypotheses of this study were a) Increasing etching time will enhance the surface roughness (Ra), and b) Increasing HF concentration will increase the Ra.

Materials & Methods

Specimen preparation: This study was approved by Ethical Committee of Babol University of Medical Sciences (mubabol.rec.1393.148). In this experimental study, two types of CAD/CAM chairside ceramics-feldspathic and lithium disilicate (table 1) were subjected to Ra analysis and surface element analysis, following different surface treatment protocols using HF acid etching technique. Totally, 40 ceramic blocks (size 14) with dimensions of 12 x 14 x 18 mm (twenty of each ceramic) were horizontally sectioned to render 5 pieces (100 specimens from each ceramic) using a water-cooled diamond disk with a low-speed saw machine. Thirty specimens from each ceramic were subjected to micro shear bond strength analysis and 70 specimens for Ra evaluation.

In order to achieve a standard surface for all ceramic blocks, ceramic surfaces were grinded using a blue diamond bur. E.max specimens were heated in the furnace (Programat P3 10, Ivoclar Vivadent, Lichtenstein) in vacuum conditions to complete crystallization. For Ra test and EDAX, 6 surface treatment protocols with two different HF acid concentrations i.e. 5% and 10% (table 1) applied at three different etching times (20 s, 60s and 120 s) were tested for each ceramic. Among them, 10 specimens from each ceramic group did not receive any surface treatment, served as control group rendering a total of 7 subgroups for each ceramic (n=10 in each subgroup, total number of specimens were 140).

Subsequently, the specimens were rinsed with air-water spray for 30s and ultrasonically cleaned in distilled water for 5 minutes. To eliminate any remaining surface contamination from the specimens, phosphoric etchant gel was applied (table 1) for 5s, rinsed, air dried and placed in 99% alcohol, and ultimately dried with compressed hot air.

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Table 1. Material descriptions, manufacturers, compositions and batch number

Material(manufacturer)	description	Composition and batch number
VITA BLOCS mark II: VITA Zahnfabrik, Bad Säckingen, Germany	CEREC/inLab (2M1C I12) block	Mixture of feldspathic crystalline particles embedded in a glassy matrix Vol % ≈30(15670)
IPS e.max CAD blocks: Ivoclar Vivadent, Liechtenstein	Lithium disilicate blocks	SiO_2 (57–80%), Li_2O (11–19%), K_2O (0–13%), P_2O_5 (0–11%), ZrO_2 (0–8%), Al_2O_3 (0–5%), MgO 90–5%) and coloring oxides (0–8%)(R64456)
Merk HF acid 40%: Merk, Darmstadt. Germany	Liquid 40% HF acid	Chloride:1ppm,Hexafluorosilicate :50 ppm,phosphate:0.5 ppm,Sulphate:2 ppm, Arsenic & Antimony:0.03 ppm,Silver:0.020 ppm, Aluminium:0.050 ppm,Barium:0.050 ppm, Beryllium: 0.020 ppm, Bismuth:0.020 ppm, Calcium:0.200 ppm (B0710538231)

Ra Seventy specimens from each ceramic were used for Ra evaluation. Ra was calculated as R_a (µm) for each specimen using atomic forced microscopy (AFM) (Nano surf easy scan 2 flex AFM, Swiss).

Scanning electron micrography: One specimen from each subgroup (total of 14 specimens) was subjected to surface elements analysis using scanning electron microscopy (SEM) (energy dispersive X-ray spectroscopy (EDAX)) (VEGA\\ TESCAN, Check Republic) (Fig1).

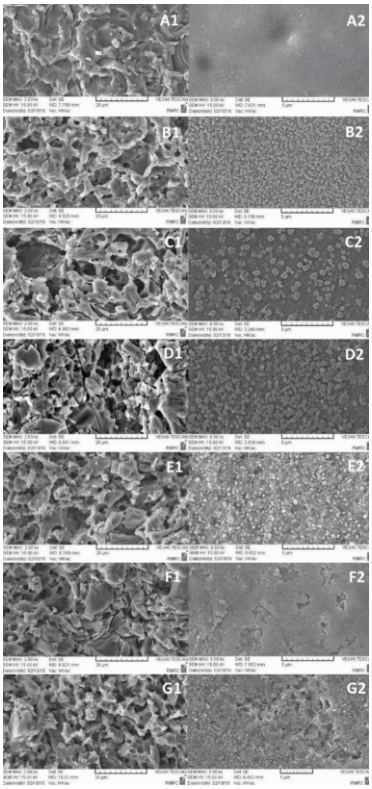


Figure 1. scanning electron micrographs of vita mark II (200 x magnification) (1) & Emax (50x magnification) (2): A. without etching. B. 20s etch with 5% HF. C. 20s etch with 10% HF. D. 60s etch with 5% HF. E. 60s etch with 10% HF. F. 120s etch with 5% HF. G. 120s etch with 10% HF.

Statistical analysis: T-test was used for Ra comparison between different HF concentrations and type of ceramic. One–way ANOVA was used for comparison between different etching times. Two-way and three-way ANOVA were applied to evaluate the interactions among factors.

Results

Surface Roughness: Mean R_a values and standard deviation of the IPS e.max™ and Vita mark II are shown in table 2. Mean R_a and standard deviation for untreated ceramics were 16.03±15.19 µm and 104.42±45.09 µm for IPS e.max™ and Vita mark II ™, respectively.

Table 2. Ra(R_a) of IPS e.max™ & Vita mark II™

IPS e.max™ Concentration	R_a (µm)	
Times	5%	10%
20 s	16.57±4.08^{aA}	32.92±8.16^{aB}
60s	47.22±19.22^{bA}	44.4±19.91^{abA}
120s	52.14±23.01^{bA}	60.3±24.1^{bA}
Vita mark II™ Concentration	R_a (µm)	
Times	5%	10%
20 s	202.29±145.09^{aA}	175.6±85.55^{aA}
60s	207.38±163.59^{aA}	264.51±174.26^{aA}
120s	251.25±131.94^{aA}	175.43±100.2^{aA}

* The different lowercase letters indicate a significant difference (p=0.05) between etching times maintaining the same acid concentration. Different capital letters indicate a significant difference (p=0.05) between acid concentrations maintaining the same time.

Vita markII than IPS e.max™ demonstrated significantly higher Ra (p value=0.00, one-way ANOVA). In the IPS e.max™ groups with 20-s etching

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[DOI: 10.22088/cjdr.8.2.8]

time, the Ra was significantly higher in 10% than 5% HF concentration (p=0.012). Ra of unetched IPS e.max™ ceramics was significantly lower than that of etched surfaces with 5% HF for 60 s (p=0.001) and 120 s (p=0.00). Ra of unetched Vita mark II™ ceramics was significantly lower than that of etched surfaces for all etching times and HF concentrations.

Scanning electron micrography: Figure 1 illustrates the porcelain surfaces before and after etching in different concentrations and etching times. As seen in fig. 1, increasing the etching time and HF concentration, escalates the surface rupture and makes cracks, leading to the weakening of ceramics. EDAX results are represented in table 3.

Table 3. EDAX analysis of IPS emax and Vita Mark II

	concentration	5%			10%			
	Time(s)	20	60	120	20	60	120	unetched
	Elements (at%)							
Vita mark II™	O_2	64.31	67.41	62.54	60.14	64.23	61.69	64.17
	Na	4.26	3.50	3.53	4.66	3.80	4.36	4.49
	Al	7.74	6.59	7.68	8.40	7.24	8.01	8.09
	Si	20.14	19.09	23.15	23.23	21.46	22.72	19.76
	K	2.77	3.13	5.68	3.43	3.13	3.22	3.21
IPS e.max™	O_2	69.11	68.14	79.81	70.04	70.77	72.25	74.02
	Al	0.64	1.45	0.00	-----	0.70	1.22	0.95
	Si	23.60	24.61	11.52	24.39	22.45	22.07	21.10
	P	2.48	2.83	-------	1.93	2.19	1.95	1.72
	K	3.23	2.62	7.56	3.05	3.58	2.29	2.10

Discussion

This study revealed that the Ra showed no significant difference in Vita mark II groups. Null hypothesis 1 was rejected for all groups except for comparing 20-and 120-s etching time with 10% HF. Null hypothesis 2 was rejected for IPS e.max™ groups except for comparing 20-s etching time using 5% and 10% HF. Among IPS e.max™ ceramics etched with 5% HF, the highest Ra was observed at 60- and 120-s etching time, and the highest Ra was observed at 120-s etching time for those groups etched with 10% HF. The Ra of IPS e.max™ ceramics etched with 5% HF for 20 s was significantly lower than that of IPS e.max™ specimens etched with 10% HF for 20 s.

A combination of chemical and mechanical retention should be happened for a reliable bonding between ceramic and resin cement. Porcelain surface treatments alter its texture, leading to the increase of the micromechanical retention of the resin cement. The use of silane agents creates the chemical retention reacted with the composite organic matrix and glassy compounds of the ceramic;[21, 22] thus, the HF acid was used for treating the ceramic surfaces in the current study. Following the introduction of the concept of etching porcelain surfaces and adhesive cementation of porcelain laminate veneers, many authors have demonstrated that the concentrations and etching periods must be adjusted to each specific type of ceramic in order to optimize the bond strength. [11, 15, 17, 19, 22-24] Knowing the optimal and proper HF etching time for micromechanical retention without weakening the ceramicis very important. [18] Therefore, the present study investigated the adequate etching protocol for a lithium disilicate-based and feldespatic glass ceramic. Numerous studies have evaluated different etching periods with various kinds of ceramics and HF etchants. [17-19, 25, 26] Mokhtarpour et al. assessed the μSBS of feldspathic and lithium disilicate CAD/CAM ceramics with resin cement using different HF acid concentrations (5% and 10%) and etching times (20, 60 and 120 s). Their result indicated no significant difference in μSBS between 5% and 10% HF as well as 20-, 60- and 120-s etching times in each ceramic and the μSBS of IPS e.max™ was significantly higher than that of Vita mark II. [27]

The results of the current study explained that the increase in etching time led to the enhancement in Ra that was significant among some experimental groups (table 2.), which inconsistent with those of Mokhtarpour et al. [27] who declared that the increase in Ra had no effect on μSBS. Thus, the best etching time for these ceramics is 20 s that makes enough μSBS without

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weakening ceramics. The surface treatment that creates more irregularities on the porcelain surface causes good adhesion of resin cement to it. [11] IPS e.max CAD has a high crystalline content (70 vol.%) in glassy matrix and is mainly constitutes 58% silica (SiO2), 10% zirconia crystals in addition to lithium-metasilicate, -phosphate and -disilicate crystals.

Vita mark II as a ceramic material with no zirconium reinforcement is made up of weak glass matrix phase and one/more irregularly-shaped crystalline phases which are more brittle than zirconia, resulting in its lower fracture strength compared to IPS e.max^{TM}. [28] Some studies , [17, 18, 25,29] evaluated the bond strength to resin and Ra .Their results manifested a positive correlation between ceramic Ra and increasing HF etching time, which are consistent with those of the present study.

In the current study, HF etching increased ceramic roughness in all experimental groups, even for periods as short as 20 s. For cementation of e.max CAD restorations, the manufacturer also recommends an etching time of 20 s with 4.9% HF gel. [30] In addition, the ceramics etched with 5%HF for 20 s (figures B1 and B2) compared to other etched surfaces display minor surface disruptions. Therefore, according to this study and considering the weakening effect of acid on porcelain surfaces [25, 29, 31], the best etching protocol is 20-s etching time with 5% HF.

Zogheib et al. [18] stated that the flexural strength of lithium disilicate-based glass ceramic was decreased after surface treatment using HF acid which could be due to the amounts of the glass phase involving in the lithium disilicate crystals. Increasing the etching time removes greater amount of glass phase. Numerous studies on various types of ceramics documented the weakening effect of HF etching. [26, 29, 32]

Surface disruption analysis evaluating failure modes revealed that in the IPS e.max^{TM} group, higher acid concentration (10% compared to 5%) was associated with a shift from adhesive failure to mixed failure. Adhesive failure illustrated that the strength of the adherent was greater than that of the adhesive whereas cohesive failure displayed that the strength of the adherent was less than that of the adhesive, and mixed failure represented that the strength of the adherent and adhesive was equal.

Findings of the present study exhibited higher HF acid concentration, and the extended etching time was associated with increased surface disruption resulting in cohesive or mixed failure and to a lesser extent adhesive failure (Fig 1).

The bond between porcelain and composite resin is achieved either by chemical or mechanical methods. Etching the porcelain surface with HF acid creates micro-mechanical retention. HF acid selectively dissolves the weaker glass phase and creates a retentive surface. The porous irregular surface facilitates the penetration of resin into the microretentions of the treated ceramic surface.

Silane-coupling agents can be used in combination with the surface alteration method such as etching with HF acid for chemical bonding. [33] Silane promotes a chemical bond between the silica phase of these ceramics and methacrylate groups of the silane coupling agent. [32, 34] The chemical bonding of silane and resin cement to the ceramic can be possible via the high percentage of silica in porcelain. [16, 18] In this study, the EDAX was applied to measure the surfaces of silica-containing ceramics. The results obtained from the EDAX group indicated a positive correlation between the surfaces of silica-containing ceramics and μSBS. In this study, etching with HF acid led to an increase in the percentage of atomic silica in the ceramic surfaces. In the IPS e.max^{TM} groups and Vita mark II groups, the highest silica content and μSBS were observed at 60- and 20-s surface treatment time using 10% H, respectively. Moreover, there was a relationship between step-down silica and μSBS in IPS e.max^{TM} ceramic etched with 5% HF for 120 s, Vita mark II ceramic etched with 5% HF for 60 s and Vita mark II etched with 10% HF for 20 s.

Energy dispersive spectrometers usually are usable to all elements down to atomic number 11(sodium) although they may be used down to atomic number 6 (carbon) with special provision. [35] Hence, the EDAX is not applicable for detection of hydrogen, lithium and beryllium with atomic number 1, 3 and 4, respectively. Thus, no lithium element of IPS e.max^{TM} (lithium disilicate ceramic) was reported in EDAX results of this study. Besides, no fluoride was detected in the EDAX, and it was shown that the fluoridate salts were produced from HF etching and rinsed off from ceramic surfaces, indicating that the methods used in the present study to clean the etched surfaces was successful. The comparison of the results of the running study with those of other studies is limited due to the newer CAD/CAM materials used in the present study. The further study should be done to assess the efficacy of

other concentrations, etching times and protocols on a wider variety of ceramics.

In conclusion Among IPS e.max^{TM} ceramics etched with 5% and 10% HF, increasing the etching time leads to higher Ra. According to SEM graphs, increasing the etching time and HF concentration causes surface disruption of ceramics and makes cracks. Therefore, we prefered to choose minimum etching time and HF concentration that create sufficient Ra for bonding.

Acknowledgments

The authors would like to thank the Dental Materials Research Center and Research Council of Babol University of Medical Sciences.

Funding: This study was a part of research project (Grant No: 9440722) supported and funded by Babol University of Medical Sciences.

Conflict of interest: There is no conflict of interest.

Author Contributions

Homayoon Alaghehmand developed the original idea and protocol as well as abstracted and wrote the manuscript. Faraneh Mokhtarpour contributed to the development of the protocol, abstracted data and prepared the manuscript. Soraya Khafri analyzed the data and Mina Mahdian edited the article.

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