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Original Article

Effect of hydrofluoric acid concentration and etching time on microtensile bond strength of suprinity and enamic CAD/CAM ceramics to resin cement

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Abstract

Introduction: The use of dental ceramics has increased due to their beauty and biocompatibility. The aim of this study was to evaluate the effect of hydrofluoric acid concentration and etching time on microtensile bond strength (μTBS) of the Enamic and Suprinity ceramics.

Material & Methods: In this in vitro study, two hydrofluoric acid (HFA) concentrations of 5% (A) and 10% (B) were used at the time of 20, 60 and 120 seconds (s) on the Suprinity and Enamic ceramics of CAD/CAM. The etched surfaces were impregnated with silane coupling agent as well as priming and Clearfil SE bond. Then, the Panavia F 2.0 resin cement was applied on the ceramic surfaces and light-cured. There were groups of EA20, EA60, EA120, EB20, EB60, EB120 for Vita Enamic and SA20, SA60, SA120, SB20, SB60, SB120 for Vita Suprinity. The μTBS between resin cement and porcelains was measured with universal testing machine. Mode of failure was observed under the stereo microscope at 40x magnification. Data were analyzed using ANOVA and Chi-square.

Results: The μTBS was significantly different between EB20 and EB60 (p=0.008), EB120 and EB20 (p=0.005), SA120 and SB120 (p=0.013), EA120 and EB120 (p=0.002) as well as EA60 and EB60 (p=0.44). In both ceramics, different concentrations and etching times had significant effect on the mean of μTBS (p=0.016). In both ceramics, the time had no effect on the failure mode. For Suprinity ceramic, the HFA concentration had effect on the failure mode (P=0.028).

Conclusion: The best surface treatment for Suprinity ceramic is 120 s with 5% HFA and for Enamic is 20 s with 10% HFA, which create the highest bond strength.

Keywords: Resin cement, Hydrofluoric acid, Ceramics, Adhesives

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بررسی اثرغلظت ها و زمان های مختلف اچ با اسید هیدروفلوئوریک براستحکام باند ریزکششی سرامیک های سوپرینیتی و انامیک CAD/CAM با سمان رزینی

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چکیده

مقدمه: میزان استفاده از سرامیک‌های دندانی به دلیل زیبایی و زیست سازگاری افزایش یافته است. هدف این مطالعه بررسی اثر غلظت-ها و زمان‌های مختلف اچینگ با اسید هیدروفلئوریک بر روی استحکام باند ریزکششی سرامیک‌های سوپرینیتی و انامیک می‌باشد.

مواد و روش ها: در این مطالعه آزمایشگاهی از دو غلظت 5٪ (A) و 10٪ (B) اسید هیدروفلئوریک در سه زمان ۲۰ و۶۰ و ۱۲۰ بر روی سرامیک¬های انامیک و سوپرینیتی CAD/CAM استفاده شد. سطح نمونه های اچ شده، به عامل اتصال سیلان، پرایمر و کلیر فیل اس ای باند آغشته شد. سپس سمان رزینی (Panavia F.2) بر روی سطوح سرامیکی بکار گرفته شد و با نور کیور گردید. گروه های EA20,EA60,EA120,EB20,EB60,EB120 برای سرامیک انامیک و SA20,SA60,SA120,SB20.SB60.SB120 برای سوپرینیتی را داشتیم. استحکام باند ریزکششی بین پرسلن و سمان رزینی با دستگاه universal testing machine اندازه گیری شد. نوع شکست نیز با بزرگنمایی ۴۰ با استریومیکروسکوپ بررسی شد. اطلاعات بدست آمده باتست¬های ANOVA وChi-square آنالیز گردید.

یافته ها: میزان استحکام باند ریزکششی در گروه‌هایEB20 وEB60 (P=0.008) و EB120 با EB20 (P=0.005) به طورمعنادار با هم متفاوت بود. در گروه‌های SA120 با (P=0.012) SB120 و EA120 با (P=0.002) EB120 وEA60 با EA120 (P=0.016) به طور معناداری متفاوت بود. در هر دو نوع سرامیک غلظت‌های مختلف و زمان‌های مختلف تاثیر معناداری بر میزان میانگین استحکام باند ریز کششی داشتند. در هر دو نوع سرامیک، زمان تاثیری بر نوع شکست نداشت. در سرامیک سوپرینیتی غلظت اسید بر نوع شکست موثر بود.(P=0. 028).

نتیجه گیری: بهترین آماده سازی در سرامیک سوپرینیتی غلظت ۵٪ اسید و زمان ۱۲۰ ثانیه و برای انامیک غلظت ۱۰٪ و زمان ۲۰ ثانیه می باشد که بالاترین استحکام باند را ایجاد می کنند.

واژگان کلیدی: سمان رزینی ، اسید هیدروفلئوریک، سرامیک ها، ادهزیو

Introduction

Dental ceramics are widely used in dentistry due to their natural appearance and biocompatibility .The high quality and improved mechanical properties of CAD/CAM restorations have led to their increasing use. [1]Many durable and advanced materials are available for CAD/CAM technology. All changes made to this system are designed to enhance the strength and simplify the use of it. CAD/CAM technology undoubtedly changes many aspects of dentistry in the future. [2] Due to the increasing prevalence of CAD/CAM restorations use, new materials have been produced to develop these restorations. These materials include matrix resin materials produced from the combination of non-organic and organic materials (polymer).Vita Enamic ceramic (Vita Zahnfabrik Germany) is made up of resin matrix ceramics so that 71±3 vol% of feldspathic ceramic is formed in the polymer network. In this ceramic, the ceramic particles

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are relatively synthesized and then entered into the polymer through capillary property with low viscosity. These ceramics include two networks: polymer and ceramic. Many in vitro studies have been carried out to determine the mechanical properties of these ceramics.
Enamic attrition against the front tooth is like the enamel-on-enamel wear. Although Enamic has a higher resistance to diamond bur, its flexural strength is lower compared to IPS e.max ceramic (146Mpa). Fracture toughness of Enamic is between composite and ceramic. It is glossy and has a lower translucency than glass ceramic like IPS e.max. Enamic has a higher strain resistance than Lava Ultimate and lower than IPS e.max. [3] VITA Suprinity (Vita Zahanfabrik Germany) recently introduced is the zirconia-reinforced lithium silicate glass ceramic. VITA Suprinity is composed of a glass matrix with zirconia crystals (46-56% silicone dioxide, 21-25% lithium dioxide, 8-12% zirconia and other components such as pigments). This ceramic has improved optical and mechanical properties compared to conventional lithium disilicate ceramics and its flexural strength is 440 Mpa. The presence of zirconia particles in a glass matrix can reinforce the ceramics by preventing crack propagation. [4] In addition, the lithium disilicate glass-matrix ceramic has better translucency than conventional lithium disilicate ceramic because of the crystalline particles. [5]

The most important aspect needed for the success of ceramic restorations is to establish an appropriate bond between substrate and adhesive. [4] Many studies have been used different methods to prepare the restoration's surface. Creating surface roughness with diamond bur, air abrasion with AL2O3 particles, and etch with different acids are introduced for micromechanical retention improvement. [6]

Hydrofluoric acid (HFA) is commonly used for ceramic indirect restorations. The advantage of HFA is the formation of a micromechanical pit and retention via dissolving the glass matrix. [7] After the etching operation, the surface is impregnated with the active silane to improve the chemical bond and create a precise and reliable chemical bond with resin cement. Silane is an inorganic-organo-functional trialkoxysilane monomer and is able to unify the organic and inorganic materials. Generally, silane has non-hydrolysable group (like methacrylate) and hydrolysable group (like ethoxy), and because of this, it is chemically bifunctional. When reactive silane is used on the etched ceramic surface, the hydrolysable alkoxy groups polymerize with exposed hydroxyl groups, and non-hydrolysable organic group react with unset resin cement. [1]

It has been shown that the difference in time and concentration of acid can cause a difference in surface morphology, and as a result, cause a difference in the bond. Bellan et al. evaluated the effect of different etching times on microtensile bond strength (μTBS) of ceramics and concluded that there was a significant difference between various times.[8] The increase of acid concentration and etching time rises the bond by enhancing the available surface for adhesion and reducing the contact angle. Of course, overetching in the flexural strength and fatigue behavior of glass ceramics is harmful. [9] In the study of Zogheib et al., the acid etching duration on the roughness and flexural strength of ceramic was examined and it was found that the roughness values were increased and the flexural strength was reduced with the increase of etching time.[10] Still, no study has been found to indicate the time and appropriate concentration of HFA to achieve the fine μTBS of these ceramics; therefore, the aim of this study was to evaluate and compare the effect of different times and concentrations of HFA on the microtensile strength of the Enamic and Suprinity ceramics.
The null hypotheses of this study were:

1) Mean μTBS of Enamic and Suprinity ceramics to resin cements enhances with the increase of etching time, 2) Mean μTBS of Enamic and Suprinity ceramics to resin cements elevates with the increase of HFA concentration.

Materials & Methods

Specimen preparation: This in vitro study was performed at the Dental Materials Research Center of Babol University of Medical Sciences in 2017. After obtaining the ethics approval (MUBABOL.REC.1396.59), two types of CAD/CAM chairside ceramic (Vita Enamic and Vita Suprinity) were used in the current study (table1). Four blocks (NO.14) of Suprinity (12× 14×18 mm) and four blocks (NO. 14) of Enamic (12 ×14× 18 mm) were used. Each block was horizontally sectioned into three slices using a low speed saw with a water-cooled diamond disk (Delta precision sectioning machine, Mashhad, Iran); thus, there were 12 specimens for each ceramic. The ceramic surface was ground using Blue diamond bur.

Specimens were polished with 800-grit silicon carbide paper to stimulate the CAD/CAM-milled surface. Suprinity specimens were heated in an oven (Vita Smart.Fire, Vita Zahanfabrik, Germany) according to the manufacture's instruction to complete their crystallization. After that, 4 cc and 6 cc of distilled water were added to 1cc 40% HFA for preparation of 10% and 5% acid concentrations, respectively. The HFA concentrations of 5% (A) and 10% (B) (Table1) were used to etch the ceramic, and three different etching times were assessed in this study; therefore, each subgroup had 2 specimens. EA20, EA60, EA120, EB20, EB60, EB120 (Vita Enamic subgroups) and SA20, SA60, SA120, SB20, SB60, SB120 (Vita Suprinity subgroups) after etching were rinsed with air water spray for 30 seconds (s). Before washing and placing in 99% alcohol, they were cleaned using ultrasonic machine for 5 minutes. Next, they were dried with compressed hot air.

Bonding method: Each ceramic had 2 subgroups and 12 specimens etched according to their subgroup. According to Afrasiabi et al. who used bonding agent with Panavia F 2.0 resin cement, the bonding agent increases the bond strength.[11] El Zohairy et al. suggested that bonding with resin cement enhances the bond strength[12]; hence, one layer of mixed silane coupling agent and Clearfil SE bond primer were used on all ceramic pieces, and after 60 s, one layer of Clearfil SE bond was applied onto ceramics. After that, an even amount of pastes A and B of Panavia F2.0 (Table1) was mixed for more than 20 s, Toffle mire strip (Arnel dental products, Washington,USA) was used around the specimens, the cement was applied onto the prepared ceramics and finally, the cement was cured for 20 s from each side using Valo LED (Ulteradent, USA) with 1000 mw/cm^2 intensity.

Table1. Materials used in this study

Material(manufacturer)	Description	Composition and batch number
Panavia F2.0: Kuraray Medical Inc., Osaka, Japan	Dual-cure single-step self-etch resin cement	ED Primer II; Liquid A: HEMA (30%-50%), MDP, Nmethacryloyl-5-aminosalicylic acid, water, accelerator (61185); ED primer II liquid B: N-methacryloyl-5-aminosalicylic acid, accelerator, water, sodium benzenesulfinate (61185); Paste A: hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, sodium aromatic sulfinate (TPBSS), N,N-diethanol-p-toluidine, surface-treated (functionalized) sodium fluoride ,10%, silanated barium glass (61185); Paste B: MDP, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica, photo initiator, dibenzoylperoxide (61185)
Clearfil SE Bond : Kuraray Medical Inc., Osaka, Japan	Light-cure self-etch adhesive	*Primer:* MDP,HEMA, Hydrophilic dimethacrylate, N,N-Diethanol , p-toluidine, water(00109A) Bonding: MDP, Bis-GMA,HEMA hydrophobic dimethacrylate, dl-Campherquinone, N,N-Diethanolp-toluidine, silanated silicate(00043A)
Clearfil Porcelain Bond Activator: Kuraray Medical Inc., Osaka, Japan	one bottle priactivated silane	Bisphenol A polyethoxy dimethacrylate, 3-methacryloyloxypropyltrimethoxy silane.(00241A)
Vita Suprinity :VITA Zahnfabrik, Bad Säckingen, Germany	Zirconia-reinforced glass-ceramic	56-64% SiO_2, 1-4%Al_2O_3, 15-21% Li_{20}, 8-12% ZrO_2 1-4% K_2O (37456)
Vita Enamic: Vita Zahnfabrik, Bad Säckingen, German	Dual-network ceramic	86% ceramic(58-63% SiO_2 , 20-23% Al_20_3, 9-11% Na_2O , 4-6% K_2O, 0-1% ZrO_2) 14% polymer (UDMA, TEGDMA) (37996)
Merk Hydrofluoric acid 40%: Merk, Darmstadt. Germany	Liquid 40% hydrofluoric 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)

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Microtensile bond strength test: The specimens were sectioned to prepare the beams with a bonding area of about 1mm^2 using a water-cooled diamond disk in a sectioning machine (Delta precision sectioning machine, Mashhad, Iran) so 10 beams of each experimental group were tested in terms of µTBS (n=10) (totally, 60 beams for µTBS test for each ceramic). The section area of each beam was measured using digital caliper (Shinwa Rules Co., Nigata, Japan).

The µTBS was measured with KOOPA universal testing machine (Koopa, Sari, Iran) at the crosshead speed of 0.5 mm/min until failure. The resultant forces (N) were divided by cross-sectional area and the µTBS values (Mpa) were calculated.

Failure mode: The failure mode of each specimen was evaluated using stereo microscope at 40x magnification and divided into three groups of failure in ceramic or cement (cohesive), failure in the interface of ceramic and cement (adhesive) as well as failure in ceramic, resin cement and interface (mixed).

Statistical analysis: For comparison between different etching times, SPSS 23 was used. One-way, two-way and three–way ANOVA was employed to assess the interactions among factors. Chi-square test was applied for mode of failure. Post hoc Tukey's test was used for µTBS comparison between different HFA concentrations and types of ceramic.

Results

Microtensile bond strength: Mean µTBS and standard deviation of the Enamic and Suprinity are shown in table2. The µTBS between EB20 and EB60 (p=0.008) as well as EB20 and EB120 (p=0.005) was significantly different. The µTBS between SA120 and SB120 (p=0.013), EA120 and EB120 as well as EA60 and EB60 (P=0.002, 0.44) was significantly different (one-way ANOVA). The highest µTBS was reported in EB20 and SA120. In both ceramics, different concentrations and etching times had significant effect on the mean of µTBS (p=0.016) (two-way ANOVA). Three-way ANOVA indicated that µTBS values were significantly different based on various concentrations, times and ceramics.

Mode of failure: Mode of failure is represented in table 3. Chi-square test for Suprinity groups demonstrated that different concentrations of HFA had significant effect on failure mode. For SB groups, the predominant failure was cohesive and for SA was adhesive. In Enamic groups, the HFA concentration had no significant effect on failure mode. In both ceramics, different etching times had no significant effect on failure mode (p=0.301).

Table 2. µTBS mean of Enamic and Suprinity ceramics

Vita Enamic		
Acid time	Acid concentration	
	5%	10%
20 s	24.67±4.25^{aA}	28.45±6.80^{Aa}
60s	25.94±6.96^{aA}	19.59±6.11^{bB}
120s	24.97±0.90^{aA}	18.99±5.11^{bB}
Vita Suprinity		
Acid time	Acid concentration	
	5%	10%
20 s	25.12±7.14^{aA}	22.17±5.56^{aA}
60s	18.89±7.30^{aA}	19.89±3.27^{aA}
120 s	27.50±8.82^{aA}	18.84±4.56^{Ab}
The different lowercase letters indicate a significant difference (p=0.05) between the etching times maintaining the same acid concentration different capital letters indicate a significant different (p=0.05) between acid concentration maintaining the same time.		

Table 3. Mode of failure after force in enamic and suprinity ceramics

Ceramic	HF concentration	Etching time	Adhesive failure	Cohesive failure	Mixed failure
Vita Enamic	5%	20 s	8(80%)	2(20%)	-
		60 s	8(80%)	2(20%)	-
		120 s	9(90%)	1(10%)	-
	10%	20s	0(0%)	10(100%)	-
		60s	7(70%)	3(30%)	-
		120 s	0(0%)	10(100%)	-
Vita Suprinity	5%	20 s	8(80%)	2(20%)	-
		60 s	9(90%)	1(10%)	-
		120 s	8(80%)	2(20%)	-
	10%	20 s	5(50%)	5(50%)	-
		60 s	9(90%)	1(10%)	-
		120 s	6(60%)	4(40%)	-

Discussion

In recent years, the adhesion of esthetic restorative materials to resin cements has improved. Successful adhesion of the indirect restorative materials between the luting agent and internal surface of the restoration can be reached with a reliable bond. In the present study, the bond strength of enamic and suprinity

ceramics to the dual-curing resin cement was evaluated by μTBS test after various preparations of ceramic surfaces. [13] The results of microtensile test represented that for both types of ceramics, there were significant differences between various concentrations of acid and etching times in some groups, so the null hypotheses of the study were accepted. According to the current study, the highest bond strength was obtained at 10% concentration and 20 s for Enamic ceramic, and at 5% concentration and 120 s for suprinity ceramic. The proper bond between the ceramic and luting agent requires surface preparation. A strong bond depends on micromechanical interlocking and chemical bonding. To achieve this, a rough and clean surface is needed.

Surface preparation increases the microprosities of the surface and thus makes the stronger bond. Conventional preparations include air abrasion, acid etching, sandblasting, or a combination of them .[14] Impregnation of the prepared surface by HFA with silane improves the wettability and covalent bond between ceramic and resin. [15]

Hence, the ceramic surface should be etched for strong chemical bonding. Since the theory of ceramic etching with HFA was introduced, it has shown that a certain acid concentration and time should be used for each specific type of ceramics to achieve an ideal bonding. [16]

The present study was conducted to find the appropriate time and concentration of HFA without weakening the enamic hybrid ceramic and suprinity ceramic. [1] Several studies have been carried out on the time and concentration of porcelain etching.[1,17]

Ramakrishnaiah et al. used various concentrations of HFA in etching time of 20, 40, 80 and 160 s for IPS e.max, Vita Mark II, Suprinity, Suprinity FC, Densply Celtra ceramics and with the increased etching time, the depth and number of pores, surface roughness and wettability were enhanced .[1] In contrast, Leite et al. used a variety of etching times and found that the increase of time had no effect on μTBS between resin cement and feldspathic ceramic although the ceramic type of their study was different from that of ours.[18]

However, the creation of sufficient porosity for the proper bond is controlled by the ceramic composition; thus, the present study based on the results proposes that the maximum of bond strength should be at 5% concentration for 120 s and 10% for 20 s in suprinity ceramic as well as at 10% concentration for 20 s and 5 % for 60 s in enamic ceramic. [1] In both types of ceramics, the μTBS elevated at 5% concentration of the acid with the increase of etching time, except for 60 s in suprinity, which could be due to the deeper and more microporosities, and subsequently, the bond strength increased, too. Histrova et al. used 4.5% HFA on IPS empress CAD, Vita mark II, KLEMA CAD CAM, Vita Enamic and IPS e.max CAD ceramics for 20, 30, 40, 50, 60, 75 90, 120 and 150 s, and it was observed that the surface energy and roughness enhanced with increasing etching time, but each of the ceramics had a various effect at different times of etching and general recommendations on the etching time cannot be made.[19]

Elsaka et al. used sandblast and 9% HFA for one minute on the lava ultimate and enamic ceramics, and no difference was observed in bond strength of different groups. Enamic ceramic indicated the highest bond strength in the use of acid and silane. The higher bond strength of enamic ceramic observed in their study and our study is related to its structure.[13] Enamic is a hybrid ceramic, whose feldspathic actually is changed and polymer is reduced (14% weight). This microstructure has an effect on mechanical properties such as increased chemical stability, increased strength and elasticity, biocompatibility and its resistance to high fracture. [13]

The analyzed scanning electron microscopy (SEM) images illustrated a mainly leucite and secondary zirconia crystalline structure surrounded by a polymer. This ceramic had a higher flexural strength than the fully sintered ceramics.[20] The elastic modulus of enamic is close to the dentin and resin cement and is lower than that of suprinity. [21]The elastic modulus plays an important role in the results of bond. Brittle materials such as suprinity compared to the elastic materials like enamic tend to break at the adhesive interfaces with lower bond strength values.

Analysis of SEM and energy-dispersive x-ray spectroscopy (EDX) indicated the leucite and zirconia particles surrounded by a polymer. The increase of porosity in these hybrid ceramics causes the decrease of their elasticity and hardness. [20] Since μTBS is more accurate than microscope, many studies apply the μTBS. On the other hand, homogenous and uniform stress creates during loading in μTBS and failures are mainly adhesive in the small bonded interface (1 mm^2).[13]

If the strength of the adherend is greater than that of adhesive, the adhesive failure occurs. Cohesive failure means that the strength of the adherend is lower than

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that of the adhesive. [22]In the present study, the type of failure was affected by the acid concentration in the suprinity ceramic so that cohesive failure was higher in the concentration of 10% than 5%. This might indicate a lower strength of adherend in this concentration although the difference in bond strength was not statistically significant except for 120 s. In enamic ceramic, concentration and time had no impact on the type of failure, and most of the failures were adhesive type, which can be due to more its elasticity and homogeneous stress distribution in micro tensile test.

Sundfeld et al. studied on the effect of etching time and different resin cement formulations on μTBS and they observed that the bond strength decreased after 6-month aging.[22] On the other hand, in Guess et al.'s study, thermocycling did not affect micro shear bond strength of ceramics,[23] concerning to these studies, aging can have effect on bond strength; therefore it is recommended to consider aging in the future studies.

Conclusion

The highest bond strength was observed in Suprinity when 10% acid was used for 20 s. According to the results, 10% concentration with 20-second etching time created a good bond for Suprinity and Enamic ceramics. If the concentration is 5%, then 120 s for these two ceramics are suggested until have a strong bond. Further studies are required to evaluate the properties of these new ceramics as well as the surface roughness in the future.

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Authors' Contributions

Homayoun Alaghehmand developed the original concept and design as well as supervised the in vitro procedure and preparation of the manuscript. Fatemeh Yazarloo carried out the in vitro procedures, collected the data and wrote the manuscript. Faraneh mokhtarpour developed the interpretation of data and statistical analysis.

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