Effect of different etching time and concentration on microshear bond strength of CAD/CAM glass-ceramic blocks to composite resin

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Effect of different etching time and concentration on microshear bond strength of CAD/CAM glass-ceramic blocks to composite resin

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This certifies that the Masters Thesis of

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감사의 글

2010년 설레임과 두려움을 안고 보존과에 첫 발을 내디뎠을 때가 엊그제 같은 데 어느새 이렇게 논문을 작성하는 시간까지 오게 되었습니다. 부족하지만 이렇게 하나의 결실을 맺게 되어 도움과 의지가 되어 주신 많은 분들께 감사의 말씀을 전하고자 합니다.

먼저 논문은 물론 수련 생활의 시작부터 끝까지 든든한 버팀목이 되어 주신 지도 교수 노병덕 선생님께 진심으로 감사 드립니다. 항상 애정 어린 가르침과 격려를 주신 박성호 선생님과 꼼꼼한 지적으로 완성도 높은 논문을 작성하도록 도와 주신 박정원 선생님께도 깊은 감사의 말씀을 드립니다. 또한 오늘의 제가 있기까지 힘이 되고 방향을 잡아주셨던 이찬영 선생님, 이승종 선생님, 김의성 선생님, 정일영 선생님, 신수정 선생님, 신유석 선생님께도 감사 드립니다. 가르침 받들어 자만하지 않고 항상 겸손한 자세로 꾸준히 노력하는 좋은 모습을 보이도록 하겠습니다.

그 동안 같이 생활하면서 정들었던 2년차 선생님들과, 앞으로 든든하게 과를 꾸려나갈 1년차 선생님들에게도 고마운 마음을 전합니다. 특히 3년 동안 같이 수련을 받으며 기쁘고 힘든 일들을 함께한 수련동기들이 있었기에 이 논문이 있을 수 있었다고 생각합니다.

마지막으로 늘 한결 같은 마음으로 딸을 곁에서 지켜보고 지지하고 지원해주시는 부모님과 힘든 와중에도 열렬히 응원해주는 든든한 동생 유현에게 그 동안 못다한 감사와 사랑을 전합니다.

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김 유 경

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Abstract

Effect of different etching time and concentration on microshear bond strength of CAD/CAM glass-ceramic blocks to composite resin

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1. Objective

Optimal surface preparation techniques for chemical and/or mechanical bonding to ceramic substrates are crucial in order to ensure clinical success when placing indirect ceramic restorations. The purpose of this article is to evaluate the effect of different etching time and concentration on microshear bond strength of two different CAD/CAM glass ceramic blocks to composite resin.

2. Materials and methods

140 ceramic plates were prepared, 70 from leucite based IPS Empress® CAD and another 70 from lithium disilicate based e.max® CAD. The ceramic surfaces were assigned into 7 groups of different surface treatments. The variables were the hydrofluoric acid etching time (0, 20, 60, 120 seconds and 10 minutes) and the concentration of the gel (5% and

9.5%). After composite resin bonding, microshear bond test was carried out using INSTRON universal testing machine and all debonded specimens were observed under X40 stereoscope. Additionally specimens in each group with different hydrofluoric acid surface treatment were observed under scanning electron microscope for detailed evaluation of surface morphology.

3. Result

The mean microshear bond strength of IPS Empress CAD and e.max CAD was 43.38 and 36.43 MPa, respectively. In Empress blocks, all groups were homogenous with no statistical differences. Altered hydrofluoric acid etching time and concentration did not influence the results. However in e.max blocks, higher bond strength was associated with longer etching time and higher hydrofluoric acid concentration. In Empress blocks, cohesive fractures within ceramic occurred most frequently. On the other hand in e.max blocks, the failures were predominantly adhesive.

4. Conclusion

(1) In IPS Empress CAD, hydrofluoric acid conditioning time and concentration did not influence the microshear bond strength decisively.

(2) In IPS e.max CAD, changing etching time and concentration had stronger effect on the surface microstructure, therefore resulted in positive relationship with microshear bond strength.

Key words : microshear bond test, cerec3, CAD/CAM, glass ceramic, hydrofluoric acid, etching time, concentration

Effect of different etching time and concentration on microshear bond strength of CAD/CAM glass-ceramic blocks to composite resin

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Department of Dental Science

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I. Introduction

Advances in computer-aided design (CAD) and computer-aided manufacturing (CAM) systems are providing new options for dentistry and creating an alternative to the conventional impression and casting technique for

producing dental restorations. (Kamada et al., 1998) The CEREC (CEramic REConstruction) system represents a unique CAD/CAM system that is used by dentists for chairside fabrication and delivery of ceramic restorations. It offers a considerable time savings over conventional, laboratory-generated restorations that require multiple appointments.(Mehl and Hickel, 1999; Mormann, 1992)

A number of ceramic materials are available for use in CEREC restorations. (Fasbinder, 2002) From the early feldspathic Vita Mark II (Vita, Bad Säckingen, Germany) blocks, leucite reinforced glass-ceramic IPS Empress CAD (Ivoclar vivadent AG, Schaan, Liechtenstein), and most recent lithium disilicate glass-ceramic IPS e.max CAD (Ivoclar vivadent AG, Schaan, Liechtenstein) blocks are provided and widely used by clinicians nowadays.

Optimal surface preparation techniques for chemical and/or mechanical bonding to ceramic substrates are crucial in order to ensure clinical success when placing indirect ceramic restorations and, when required, repairing them intraorally (Alex, 2008). The modern generation of bonded porcelain restorations was first described in 1980s, from Calamia's works (Calamia, 1983, 1985; Calamia and Simonsen, 1984). He successfully utilized hydrofluoric acid etching on porcelain surfaces in order to increase micromechanical retention, and then applied silane to achieve stable chemical bonds between luting agent and silicon dioxide on the ceramic surface. From then, hydrofluoric acid and silane surface treatment was accepted as standard procedure in adhesive cementation of ceramic restorations.

About usefulness of silane, common consensus has been reached (Hayakawa et al., 1992; Matinlinna et al., 2004; Shimada et al., 2002). Not only providing

chemical interaction, which is attributed to its bifunctional characteristic, but it also increases wettability of ceramic surfaces and creates better adhesive environment.

But regarding hydrofluoric acid etching so far, the question is often raised if it is generally required. Some studies clearly demonstrate that etching with HF has the potential to significantly increase its bond strength to composite (Chen et al., 1998; Nagayassu et al., 2006; Pisani-Proenca et al., 2006), while others lead to the conclusion that acid etching can be eliminated, but not on the silanization, relying more on the chemical adhesion (Aida et al., 1995; Hayakawa et al., 1992; Shimada et al., 2002).

Moreover, when it comes to the specific time and concentration, the matter becomes even more complex. In the initial stages, Calamia applied hydrofluoric acid as long as 20 minutes, since he treated more acid-resistant feldspar ceramics (Calamia, 1983). In recent years, numerous studies have tested ceramic to resin bond, but the authors were inconsistent with their usage in hydrofluoric acid, varying from 2.5 to 52% in concentration, 20 seconds to several minutes with application time. In clinical situations, this inconsistency may confuse dentists and laboratory technicians.

The purpose of this article is to evaluate the effect of different etching time and concentration on microshear bond strength of two different CAD/CAM glass ceramic blocks to composite resin. Leucite based IPS empress CAD and Lithium disilicate based IPS e.max CAD were tested.

II. Materials and Methods

1. Materials

Two recent CAD/CAM glass-ceramic blocks and hydrofluoric acid with two different concentrations were prepared. The materials employed in this study are listed in Table 1 and 2.

Table 1. Ceramic block

Product	Composition	Shade	Size	Lot	Manufacturer
IPS Empress® CAD	Leucite-reinforced glass ceramic	LT A3	C14	L39492	Ivoclar vivadent AG, Schaan, Liechtenstein
IPS e.max® CAD	Lithium disilicate glass-ceramic	LT A3	C14	N03151	Ivoclar vivadent AG, Schaan, Liechtenstein

Table 2. Hydrofluoric acid

Product	Composition	Lot	Manufacturer
Porcelain Etchant	9.5% Hydrofluoric acid	1100000095	Bisco, Inc. Schaumburg, IL

IPS® Ceramic etching gel	<5% Hydrofluoric acid	P02565	Ivoclar vivadent AG, Schaan, Liechtenstein

2. Ceramic specimen preparation

140 rectangular ceramic plates were made using low-speed diamond wheel (Struers Minitom, DK-2610 Rodovre, Denmark), 70 from IPS Empress®CAD and another 70 from IPS e.max®CAD blocks. The dimensions were 12mm in width, 14mm in length and 2mm in thickness. E.max blocks were milled first in bluish grey pre-crystallized metasilicate phase, and then followed by subsequent crystallization process in Programat P300 (Ivoclar vivadent AG, Schaan, Liechtenstein) furnace under crystallization temperature of 820~840℃ (Program no.81).

3. Experimental groups

Prepared specimens were randomly assigned to 7 groups of different surface treatments. The variables were the hydrofluoric acid etching time (0, 20, 60, 120 seconds and 10 minutes) and the concentration of the gel (5% and 9.5%). The descriptions of the tested groups are given in Table 3. In each groups, 10 samples were assigned.

Table 3. Experimental groups

HF Surface treatment		Materials	
Time	Concentration	Empress® CAD	e.max® CAD
0 sec	–	P0	M0
20 sec	5%	P2	M2
60 sec	5%	P6	M6
120 sec	5%	P12	M12
10 min	5%	PM	MM
20 sec	9.5%	P2H	M2H
60 sec	9.5%	P6H	M6H

4. Bonding procedure

The specimens were treated with hydrofluoric acid according to their groups, except the P0 and M0 group with no acid etching. Critical variable of this study was the application time in seconds, so a stopwatch was used to control it exactly. After etching, all the samples were thoroughly rinsed with water and air-dried. A universal primer containing silane coupling agent, Monobond Plus (Ivoclar vivadent AG, Schaan, Liechtenstein) was applied on the etched surface for 1 min and air-dried to remove excess solvent.

In order to control the bonding area, acid/solvent-resistant adhesive tape (Scotch Tape, 3M) with 4 holes (1.2mm diameter) was first attached to the ceramic plates, completely covering the surface except for the hole area. Resin bonding agent, Heliobond (Ivoclar vivadent AG, Schaan, Liechtenstein) was applied over the tape and light cured for 30 seconds (600 mW/cm^2, Smart LED

plus, Sungbotech, Seoul, Korea).

Translucent elastomer Bioplast® (Scheu, Am Burgberg, Iserlohn, Germany) mold was made to control resin placement onto the ceramic plate. The mold was positioned over the tape, ensuring that their lumen coincided with the circular bonded areas (Figure 1). Metafil Flo α (Sun Medical co., Japan) was inserted into the hole, and light cured for 40 seconds. The mold and tape was carefully removed, exposing the resin composite cylinders bonded to the ceramic surface. Bonded area was meticulously checked and specimens with gap, defect or void were excluded.

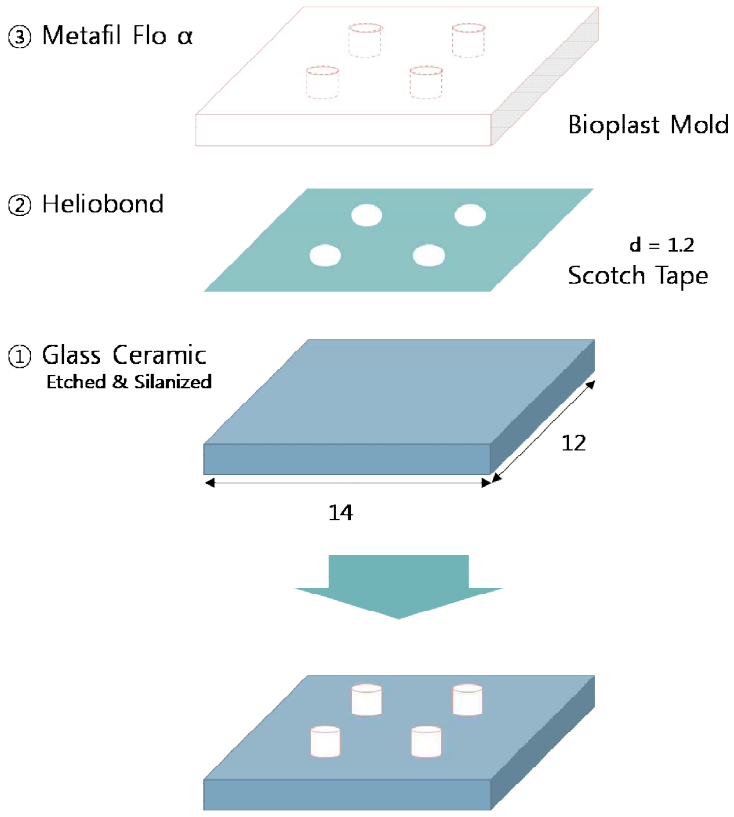


Fig 1. Specimen Preparation

5. Microshear bond test

Following storage in distilled water at 37℃ for 24 hours, microshear bond test was carried out using INSTRON universal testing machine (Model 3366, Instron Co., Massachusetts, USA) with cross-head speed of 1mm/min (Figure 2). Shear load was applied until bond failure of the specimen occurred. The load at failure was recorded in Neutons(N) and converted to shear bond strength in MPa ($\tau=4P/\pi d^2$).

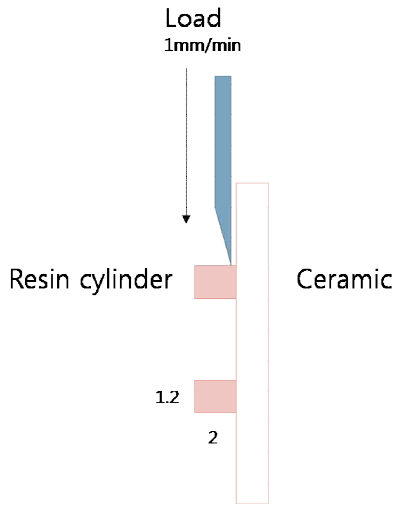


Fig 2. Microshear bond test

6. Surface evaluation

All debonded specimens after shear bond test were observed under X40 stereoscope (Leica, Microsystems Inc., Depew, New York, USA) to determine the mode of failure. Failure modes were classified as follows:

- Adhesive failure at resin-ceramic interface
- Cohesive failure within ceramic.
- Mixed failure, involving bonding agent, resin and ceramic interfaces.

Furthermore, specimens in each group with different hydrofluoric acid surface treatment were observed under scanning electron microscope (Hitachi, Tokyo, Japan) for detailed evaluation of surface morphology.

7. Statistical analysis

Statistical analyses were performed using SPSS 11.5 software for Windows (SPSS Inc., Chicago, IL, USA). 3-way analysis of variance (ANOVA) was applied using bond strength (MPa) as the dependent variable and material, etching time and concentration as factors. Tukey test was used in the post hoc comparisons. When an interaction between the 3 factors was identified, the differences were assessed statistically using two-way ANOVA, and Tukey tests. In all analyses, the level of significance was set at $\alpha = 0.05$.

III. Result

1. Microshear Bond Strength

Results for the bond test are summarized in Table 4 and 5. The mean microshear bond strength of IPS Empress CAD and e.max CAD was 43.38 and 36.43 MPa, respectively. 3-way ANOVA analysis (Table 6) showed interactions between all three variables, therefore the effects of time and concentration were separately analyzed by 2-way ANOVA in IPS Empress CAD and e.max CAD blocks. In Empress blocks, all groups were homogenous with no statistical differences. Altered hydrofluoric acid etching time and concentration did not influence the results. However, e.max samples showed contrasting results. 2-way ANOVA analysis showed significant effects of both time ($p<0.0001$) and concentration ($p<0.0001$) on the bond strength of e.max blocks. Interaction terms were also significant ($p<0.05$). In e.max blocks, higher bond strength was associated with longer etching time and higher hydrofluoric acid concentration. Figure 3 and 4 displays the clear differences between two blocks.

2. Failure Analysis

The distribution of failure modes is shown in Figure 5 and 6. In Empress blocks, cohesive fractures within ceramic occurred most frequently. On the other hand in e.max blocks, the failures were predominantly adhesive. The representive micrographs are presented in figure 7.

Table 4. Microshear bond strength of IPS Empress® CAD

Group	HF		Mean bond strength (Mpa)	SD	Tukey Grouping
	Time	Con			
P0	0	0	40.07	6.45	A
P2	20	5	44.82	7.35	A
P6	60	5	42.30	5.82	A
P12	120	5	45.74	6.90	A
PM	600	5	46.65	3.71	A
P2H	20	9.5	38.97	6.72	A
P6H	60	9.5	48.28	6.35	A

Table 5. Microshear bond strength of IPS e.max® CAD

Group	HF		Mean bond strength (Mpa)	SD	Tukey Grouping
	Time	Con			
M0	0	0	19.62	4.00	A
M2	20	5	26.66	4.10	B
M6	60	5	32.39	4.89	BC
M12	120	5	34.66	5.17	C
MM	600	5	42.50	5.91	D
M2H	20	9.5	43.39	5.87	ABCD
M6H	60	9.5	55.77	4.78	ABCDE

Table 6. Result of 3-way ANOVA analysis

Source	Sum of squares	Mean square	F	Sig.
Material	1322.458	1322.458	36.243	.000
Time	1823.483	607.828	16.658	.000
Con	2052.534	2052.534	56.251	.000
Material*Time	405.851	135.284	3.708	.013
Material*Con	1970.211	1970.211	53.994	.000
Time*Con	398.687	398.687	10.926	.001
Material*Time*Con	26.006	26.006	.713	.400

Table 7. Result of 2-way ANOVA in IPS Empress® CAD

Source	Sum of squares	Mean square	F	Sig.
Time	281.251	93.750	1.958	.129
Concentration	.421	.421	.009	.926
Time * Con	314.171	314.171	6.563	.013

Table 8. Result of 2-way ANOVA in IPS e.max® CAD

Source	Sum of squares	Mean square	F	Sig.
Time	1948.083	649.361	25.865	.000
Concentration	4022.324	4022.324	160.217	.000
Time * Con	110.522	110.522	4.402	.040

Fig 3. Mean bond strength values (MPa) of IPS Empress® CAD.

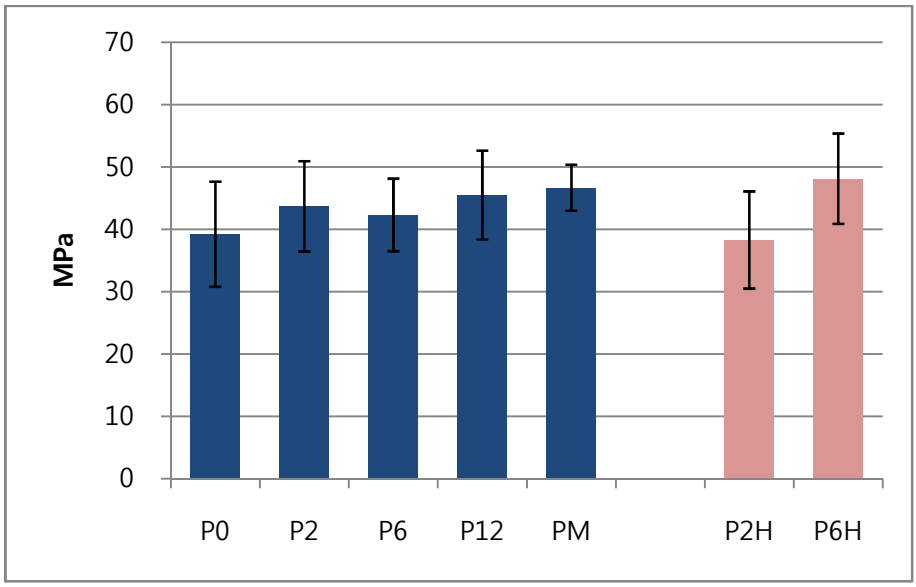


Fig 4. Mean bond strength values (MPa) of IPS e.max® CAD

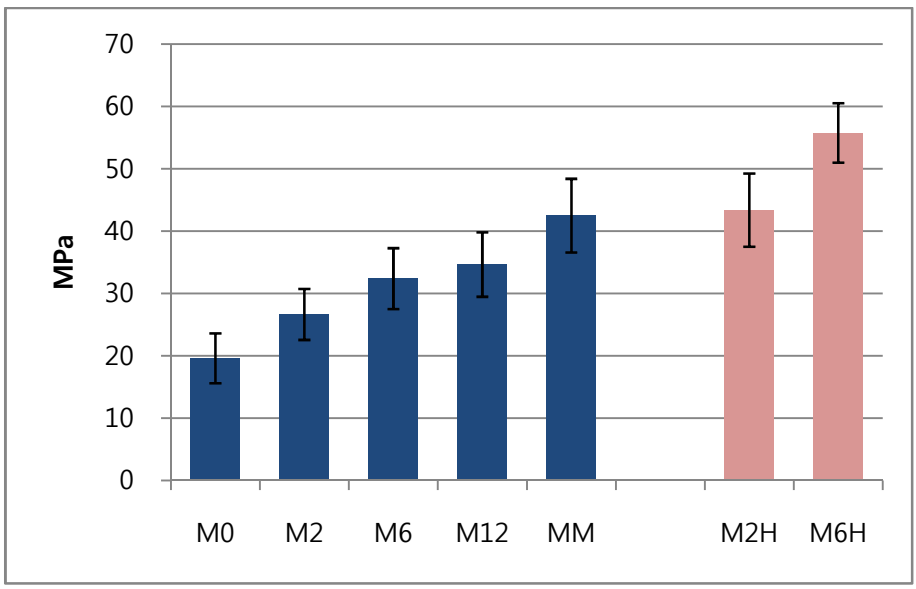
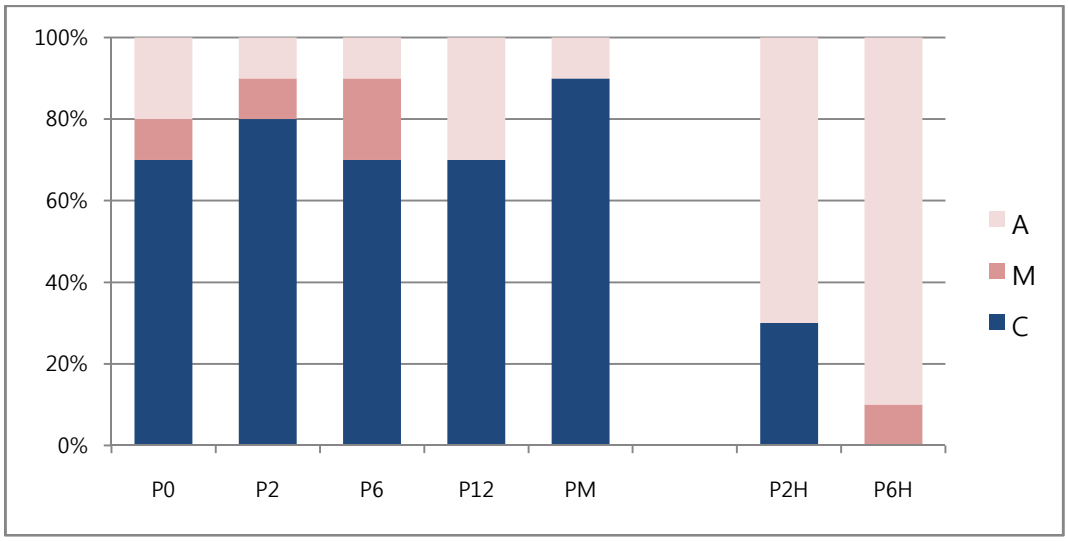
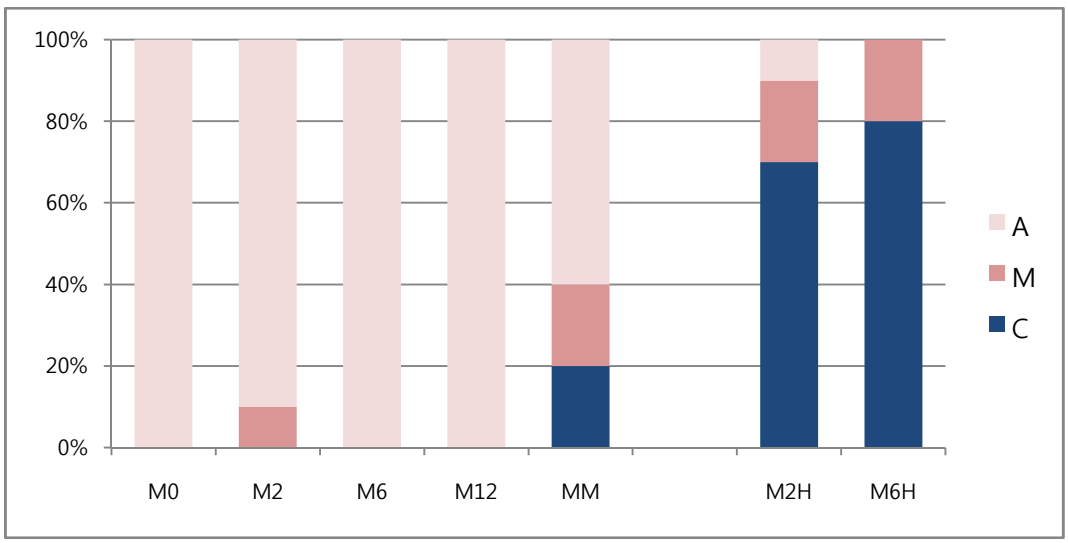


Fig 5. Distribution of failure modes (%) among groups in IPS Empress® CAD



(C) Cohesive (M) Mixed (A) Adhesive

Fig 6. Distribution of failure modes (%) among groups in IPS e.max® CAD



(C) Cohesive (M) Mixed (A) Adhesive

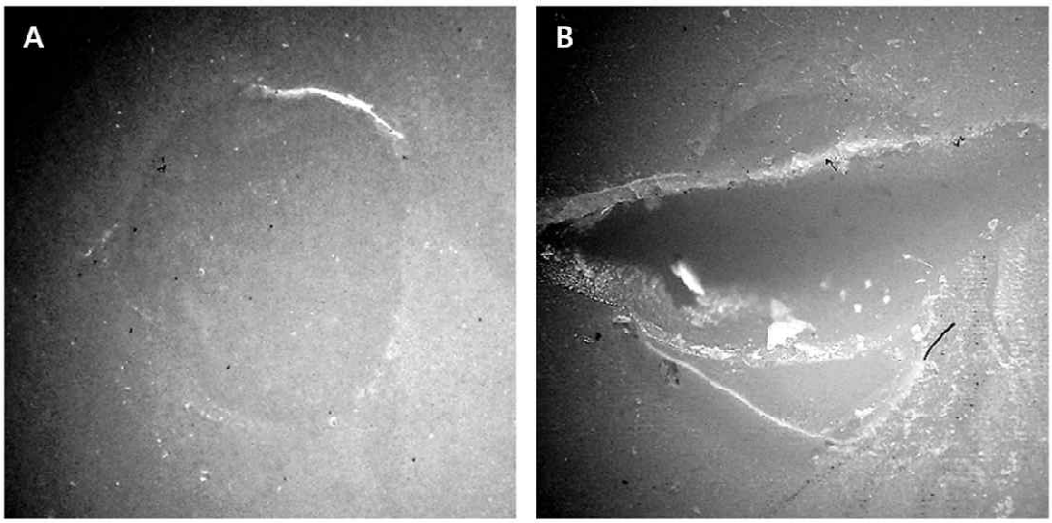


Fig 7. Representive micrograph of two failure modes

(A) Adhesive failure, (B) Cohesive failure within ceramic

3. SEM Evaluation

SEM images of etched surfaces are shown in Figure 8 and 9. There was a distinct change in surface morphology as the etching time and concentration was modified, but the appearance was different in two materials.

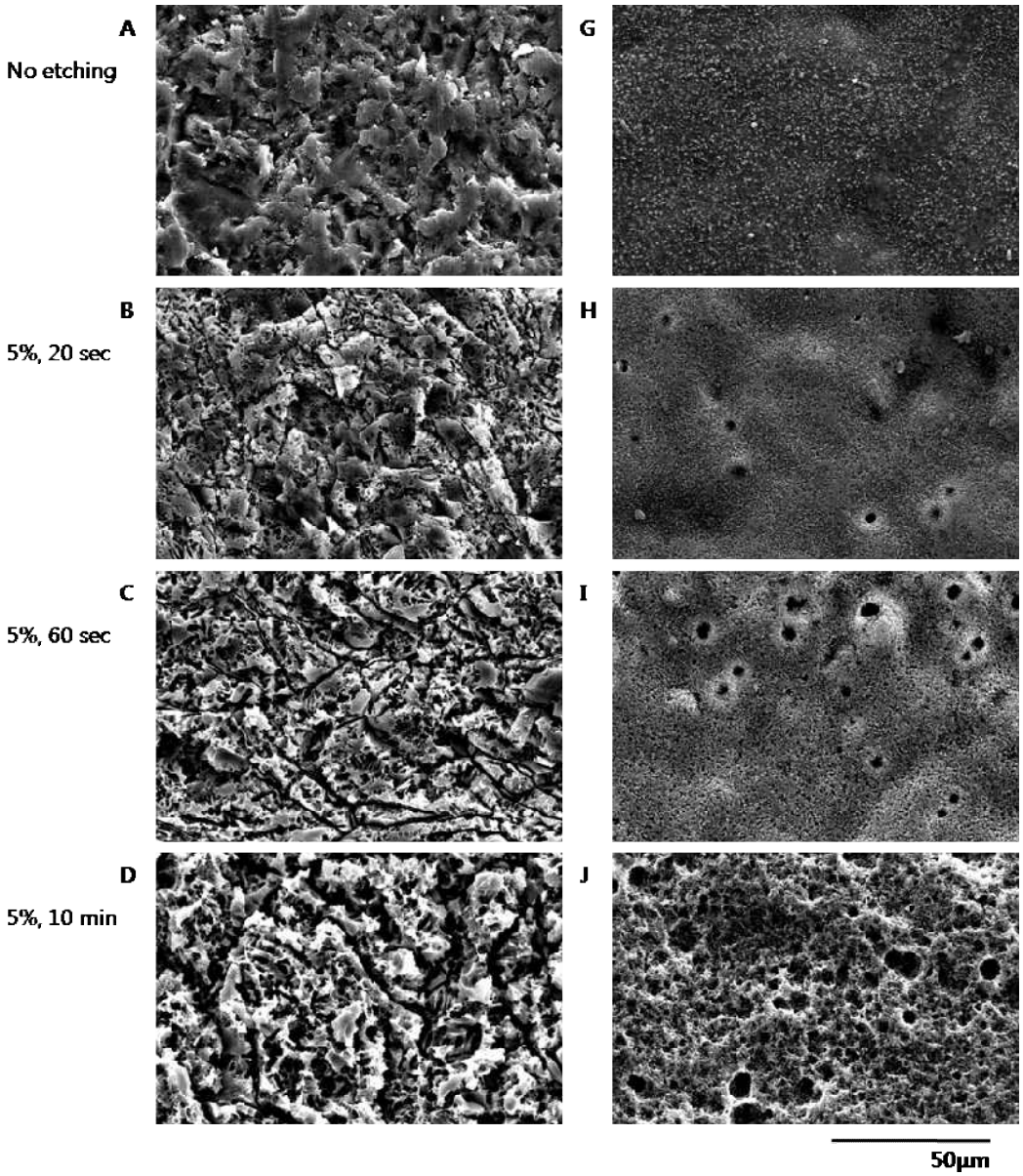


Fig 8. SEM images of acid-etched surfaces (X1000)

(A-F) IPS Empress CAD, (G-L) IPS e.max CAD.

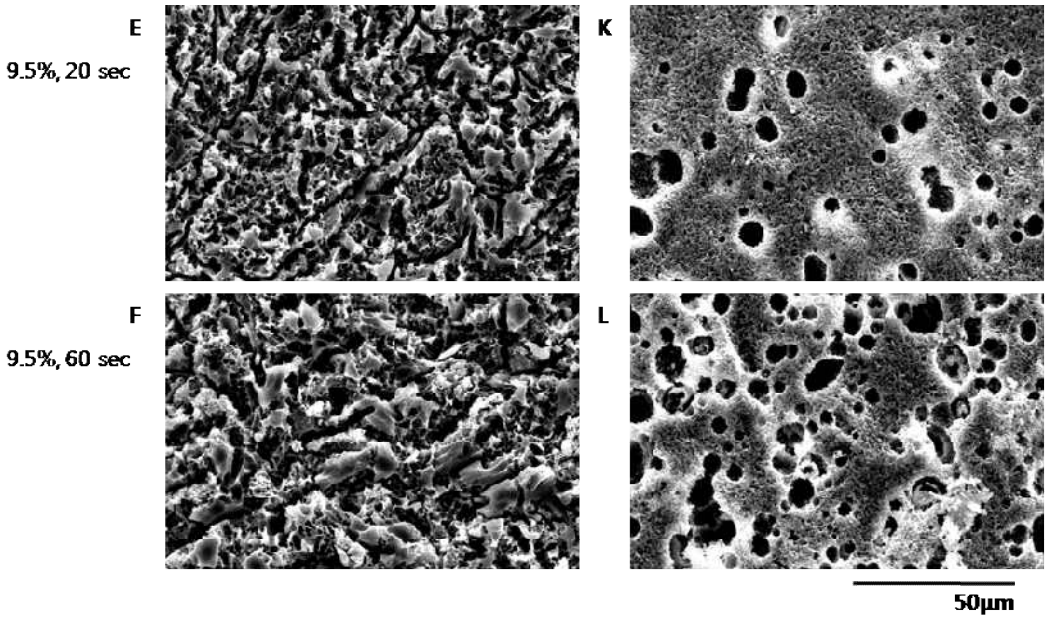


Fig 8. (cont.)

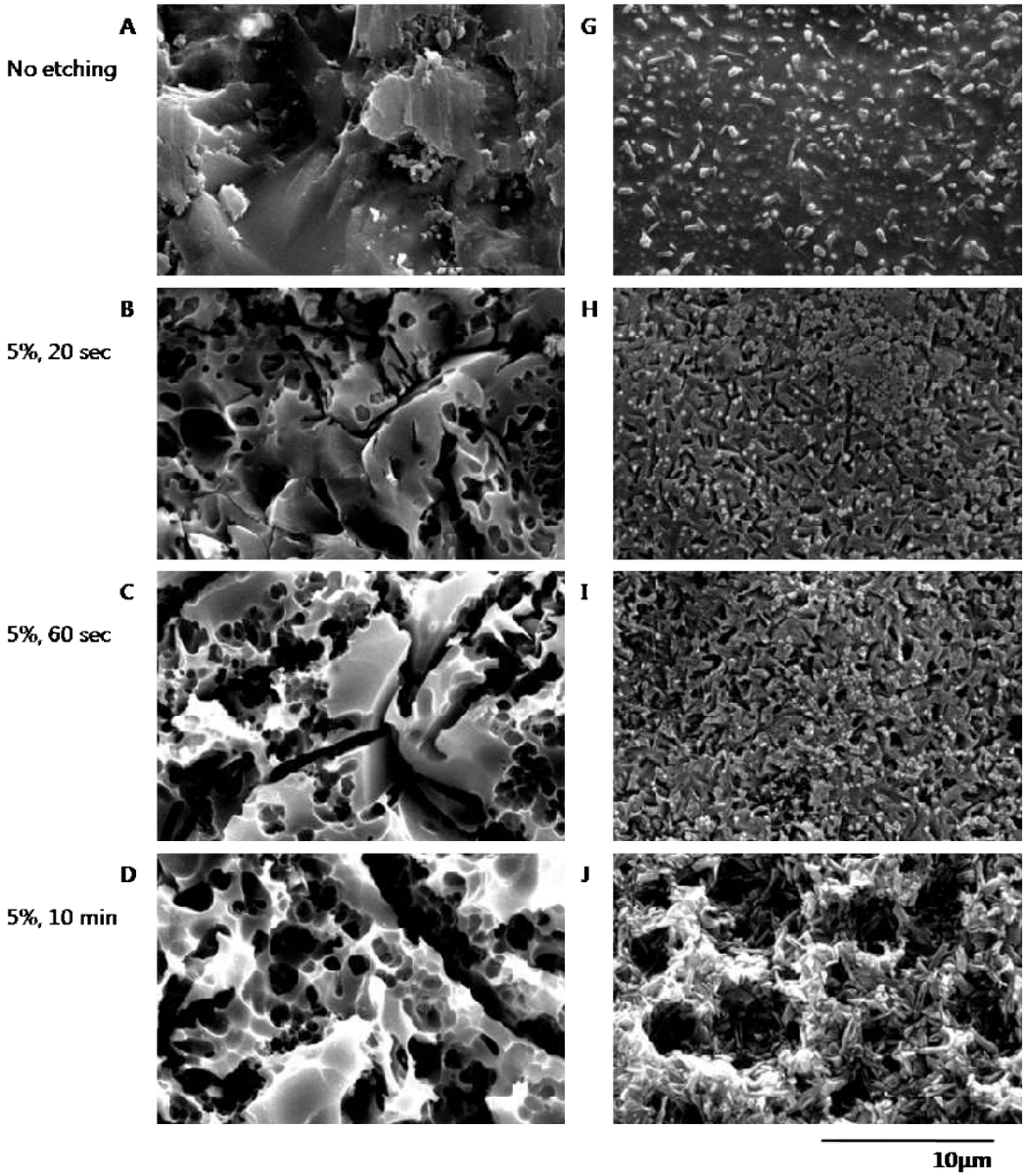


Fig 9. SEM images of acid-etched surfaces (X4000)

(A-F) IPS Empress CAD, (G-L) IPS e.max CAD.

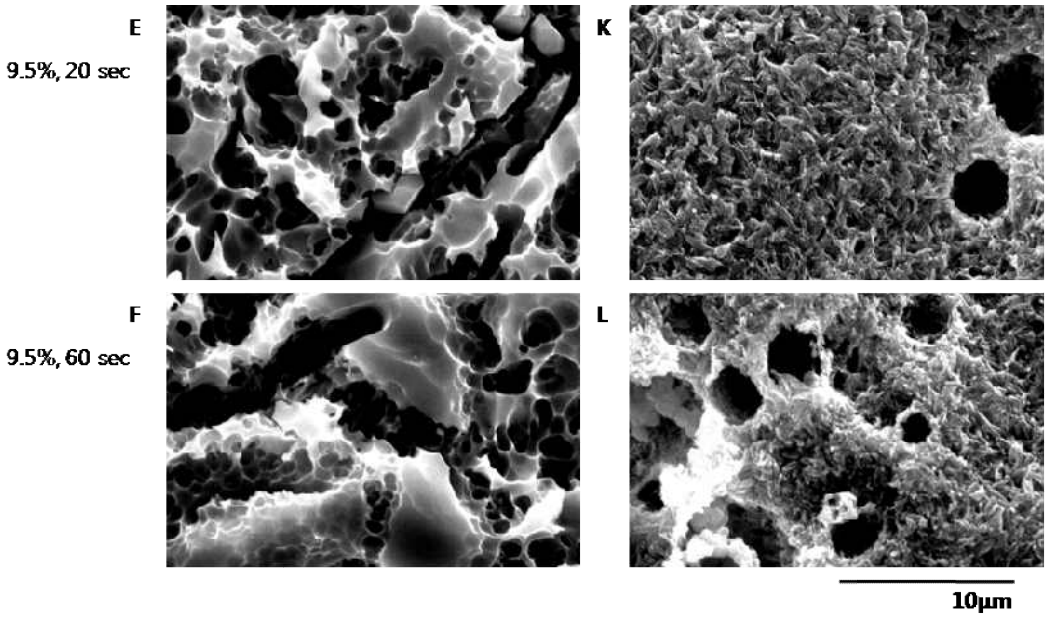


Fig 9. (cont.)

IV. Discussion

IPS Empress CAD and e.max CAD are recommended to be conditioned for 60 seconds and 20 seconds with 5% hydrofluoric acid, respectively. Surprisingly, the results of this study were quite inconsistent with the manufacturer's instructions.

First of all, there was a noticeable difference between the two CAD/CAM blocks used in this study. From the beginning stages of adhesive cementation, it was recommended to adjust etching time and concentration of hydrofluoric acid depending on the specific porcelain being treated (Calamia, 1985). Since then, several researchers have shown that the effects of different surface treatments on bonding are strongly dependent on the type of ceramics (Aida et al., 1995; Chen et al., 1998; Lacy et al., 1988).

Table 9. Standard composition of IPS Empress® CAD and IPS e.max® CAD (wt%)

IPS Empress® CAD		IPS e.max® CAD	
SiO2	57-80	SiO2	60-65
Li2O	11-19	Al2O3	16-20
K2O	0-13	K2O	10-14
P2O5	0-11	Na2O	3.5-6.5
ZrO2	0-8	Other oxides	0.5-7
ZnO	0-8	Pigments	0.2-1
Others	0-12		

The different responses to hydrofluoric acid appear to come from the differences in ceramic microstructure and composition. IPS Empress CAD is a leucite-based glass ceramic of the SiO_2-Al_2O_3-K_2O materials system, but e.max CAD is a lithium disilicate glass-ceramic, and the chemical basis for the material is the SiO_2-Li_2O system ("IPS e.max CAD: Scientific documentation; IPS Empress CAD: Scientific documentation,"). From a materials perspective, therefore, these materials are entirely different (Holand, 1998). Apart from the chemical composition, there are also considerable differences in the microstructures which is apparent in SEM images of non-etched samples (Figure 8 and 9, A and G). The surface of e.max CAD shows dense microstructure of lithium disilicate crystals measuring 0.5 to 5μm. The content of these phyllosilicate crystals approximates 70% in volume of the glass-ceramic, and is considerably higher than that of Empress CAD (35~45% vol%). Stresses initiated by acid etching begin to form in the glass-crystal interface and longer etching periods lead to the dissolution of crystals on the surface of the glass-ceramic (Dorsch and Holand, 1994). Accordingly the microstructures created by the glass and ceramic phases can greatly affect the surface morphology created by acid etching. Therefore in this perspective, Empress and e.max blocks will be discussed separately from now on.

In the Empress specimens, all groups showed no statistical differences. Recently, Naves conducted a similar microshear bond test using 10% hydrofluoric acid, changing etching periods from 10 to 120 seconds (Naves et al., 2010). When unfilled resin was used, 60-second samples presented significantly higher bond strength, but all the other groups showed similar

results without statistical differences. He suggested that combination of silane and unfilled resin is responsible to this similarity, demonstrating complete infiltration of resin into the irregularities created even by prolonged etching periods. In an experiment run by the manufacturer itself (Dorsch and Holand, 1994) also revealed no differences in shear bond strength between etched and non-etched samples.

These results can be somewhat confusing and difficult to interpret, because etching with hydrofluoric acid clearly produced microporous and dendritic retentive appearance on the ceramic surface in consistent with earlier studies utilizing Leucite glass ceramics (Nagayassu et al., 2006; Naves et al., 2010). But these mechanical interlocking appear to have little effect on the bond strength of IPS Empress CAD. Advances in dental silane agent have brought us several studies proposing possibilities to skip HF etching procedures (Aida et al., 1995; Hayakawa et al., 1992; Shimada et al., 2002). Perhaps, relative high proportion of glass matrix can explain this result. The dependence on chemical bonding by silane coupling agent may override the influence of micro-mechanical retention in Empress CAD.

Furthermore, there was no negative effect in bond strength from so-called 'over-etching' described in previous articles (Chen et al., 1998; Shimada et al., 2002; Yen et al., 1993), despite the control group was applied as long as 10 minutes. The SEM images of current study could afford some explanation to this result (Figure 8 and 9). After 60 seconds of 5% HF etching, the change over time was relatively mild in Empress CAD samples. Samples treated with 9.5% HF also showed similar surfaces with 5% samples. Maybe this is because the

crystalline content on the surface of Empress CAD is limited to certain degree (35-45%). After exceeding this limit, perhaps which is after 60 seconds, there may be no more crystals left to be dissolved by acid attacks. Therefore, no more surface changes can be achieved, resulting in similar bond strengths.

Failure mode was predominantly cohesive within glass ceramics. This result is in accordance with those of several studies that evaluated the bond strength at ceramic-composite interface after silanization (Aida et al., 1995; Lu et al., 1992; Nagayassu et al., 2006; Stangel et al., 1987). Some criticizes the cohesive fractures associated with shear bond tests as a result of experimental design and stress concentration (Armstrong et al., 2010; Braga et al., 2010; Della Bona and van Noort, 1995), but in dealing with ceramic-composite interfaces this mode to some extent may be inevitable because of brittleness of ceramic materials. Thus, cautious interpretation is needed.

In e.max specimens, the results were totally different. 2-way ANOVA showed positive correlation between shear bond strength and both of the variations, etching time and concentration. Up to date, there are no published articles dealing with bonding of IPS e.max, but there was one study with IPS Empress 2, precursor of e.max, composed of same lithium disilicate crystals (Kim et al., 2004). The result of their study showed similar results with this study concluding that Empress 2 all-ceramic restorations require etching with 10% hydrofluoric acid for 180~300 seconds to enhance the bond strength. This is also lot more than the recommended of 5% and 20 seconds.

In the SEM images, in comparison with Empress samples, consequential changes in the surface after increasing time and concentration were more

apparent on the e.max samples (Figure 6, G~L). These distinct increases in microporosity can contribute to higher bond strength by providing more bond area and creating undercuts for resin cements (al Edris et al., 1990). Calamia evaluated the bond strengths of feldspatic porcelain etched for 2.5, 5, 10, and 20 minutes and found increased bond strength with increased etch times (Calamia, 1983). His SEM analysis of the porcelain surface was in accordance with ours, indicating a definite potential for increased retention at each etch time. As discussed above, the dense crystalline microstructures of e.max CAD surface possibly have attributed to this high reactivity to hydrofluoric acid. In theory, the reaction will continue until the available surface crystals are all used up.

Despite the apparent correlation with bond strength shown in this study, there may be disadvantages utilizing higher HF concentrations and longer etching periods. The manufacturer advice to etch for a very short period of 20 seconds and they are unclear about what this recommendation is based on. Maybe the answer to this question can be found in the material properties.

Lithium disilicate crystals are known be relatively susceptive to chemical attacks (Holand, 1998). A recent study has been designed to evaluate the durability of all-ceramic materials in oral environment. Substance lost following wear testing with storage in artificial saliva was measured, and the result showed significantly higher element release in lithium disilicate group than leucite based groups (Dundar et al., 2003). According to the ISO standard regarding dental ceramics ("ISO Dental Norm 6872,"), chemical resistance of a material is tested by loss of mass with 4% acetic acid treatment for 16 hours at 80℃. Chemical solubility of IPS e.max CAD shows 40μg/cm^2 and it is almost

twice compared with 25μg/cm^2 of IPS Empress CAD ("IPS e.max CAD: Scientific documentation; IPS Empress CAD: Scientific documentation,"). This value is lower than maximum level permitted by the relevant standard (<100μg/cm2), but it may affect the material in the acidic environments caused by etching procedures. The biaxial strength of IPS e.max CAD after 20 seconds of hydrofluoric acid etching was not reduced significantly (Holand, 1998), but no data is provided with longer etching period or higher concentration.

All e.max specimens with only one exception etched with 5% hydrofluoric acid showed adhesive failures till 120 seconds, but in the 10−minute etching group, mixed and cohesive failure was increased. Particularly, specimens etched with 9.5% HF showed predominantly cohesive failures regardless of etching periods. The SEM images uphold these findings (Figure 8, K and L). 9.5% HF created highly destructive pitting on ceramic surfaces, even in the 20 second samples. The involved area was not limited to the surface crystals measuring 0.5 to 1μm; it contained much larger mass along with the glass matrix (~10μm).

Nevertheless, according to study of Yen, alteration of surface topography by increased acid etching period from 30 seconds to 5 minutes did not have a deleterious effect on the flexural strength of feldspathic porcelain and glass ceramics (Yen et al., 1993). He concluded that flexural strength of ceramic material is more dependent on internal bulk texture than on surface characteristics. The clinical significance of these surface defects created by acid etching is currently unknown.

The results of the current study have clinical implications. In Empress CAD, prolonged etching periods or high HF concentration may not be necessary,

because it does not bring about higher bond strength. But in e.max CAD, the bond strength increased significantly following longer etching time and higher HF concentration. Therefore in this material, application of 9.5% HF in laboratory use or 5% HF for a longer etching period in chairside may be encouraged in clinical practice.

In this study, microshear test was performed to compare the bond strength. Until the mid-nineties, bond strength tests were performed in specimens with relatively large bonded areas, usually 3~6mm in diameter. However, problem in stress distribution at the bonded interface was pointed out, and the need for new methods to overcome these limitations led to the use of specimens with small bonding areas, in the so-called micro-tensile and micro-shear tests(Braga et al., 2010). Though, the general finding based upon finite element stress analysis (FEA) shows that the shear force has its inherent limitations, which tends to concentrate its force on to the base material rather than the strength of the adhesive interface, resulting in cohesive fracture of the base material (Armstrong et al., 2010; Della Bona and van Noort, 1995). But unfortunately, the microtensile bond tests, although an effective method, also does not totally eliminate the possibility of cohesive failures (Scherrer et al., 2010; Souza et al., 2011), and above all, it is very technique sensitive to specimen preparation procedures (Shimada et al., 2002). The high percentage of pre-test failures reported and lack of standard interpreting these failures (Fabianelli et al., 2010; Souza et al., 2011) can be one of the reasons not favoring this method especially in the case of glass-ceramic samples. Compared to microtensile bond test, trimming of the sample after the bonding procedure is not necessary for the

microshear test. Accordingly, the majority of studies on resin to ceramic bond were designed and conducted as shear tests. (Hayakawa et al., 1992; Kukiattrakoon and Thammasitboon, 2007; Lu et al., 1992; Nagayassu et al., 2006; Naves et al., 2010; Shimada et al., 2002)

Lastly, it should be added that only the early bonding ability was investigated in this study. It plays a fairly important role in clinical situations but of course, the effects of aging on bonding should also be taken into consideration. There are evidences that chemical adhesion of silane is unstable in aging situations. (Meng et al., 2010; Pisani-Proenca et al., 2006) The oral environment continuously stresses the bond interface by thermal changes, masticatory forces and acidic challenges. Thus, further long-term investigations and clinical trials are desired.

V. Conclusion

The aim of this article was to evaluate the effect of different etching time and concentration on microshear bond strength of IPS Empress CAD and IPS e.max CAD, two CAD/CAM blocks to composite resin.

Within the limitation of this study, the following conclusions were drawn:

(1) In IPS Empress CAD, hydrofluoric acid conditioning time and concentration did not influence the microshear bond strength decisively.

(2) In IPS e.max CAD, changing etching time and concentration had stronger effect on the surface microstructure, therefore resulted in positive relationship with microshear bond strength.

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국문 요약

불산 농도 및 적용 시간에 따른 CAD/CAM 세라믹의 복합 레진에 대한 미세 전단 접착 강도 비교

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김유경

1. 서론

기계적, 화학적 접착을 위한 표면 처리는 성공적인 세라믹 수복물의 합착을 위해서 필수적인 부분이다. 하지만 임상가들은 물론 연구자들 간에도 적절한 표면 처리에 대한 합의가 이루어지지 않아 혼동을 주고 있다.

이에 본 연구에서는 불산의 농도 및 적용 시간에 따른 CAD/CAM 세라믹의 복합 레진에 대한 전단 접착 강도를 비교하고자 하였다.

2. 본론

Leucite 기반의 IPS Empress®CAD와 lithium disilicate 기반의 e.max®CAD, 두 종류의 세라믹 블록과 5%, 9.5% 두 가지 농도의 불산을 사용하였다. 세라믹 시편

제작 후 불산을 0초, 20초, 60초, 120초 및 10분간 각각 적용하였고, silane(Monobond Plus) 및 bonding agent(Heliobond)를 도포하였다. 복합 레진 접착 후 인스트론 만능 시험기를 사용하여 미세 전단 접착 강도(MPa)를 측정하였고, stereoscope으로 파절면을 관찰하여 파절 양상을 기록하였다. 추가적으로 주사 전자 현미경으로 불산 처리 시간 및 농도에 따른 세라믹 표면 결정 구조의 변화를 관찰하였다. Two-way ANOVA test로 군간 접착 강도 사이의 유의성을 평가하였다.

실험 결과, IPS Empress CAD 는 모든 군에서 유의한 차이를 나타내지 않았으나, IPS e.max CAD 의 경우, two-way ANOVA test 에서 불산 처리 시간 및 농도 모두 미세 전단 접착 강도와 유의한 관련성을 보였으며, 긴 처리 시간 및 높은 농도에서 보다 높은 미세 전단 접착 강도를 나타내었다.

파절 양상 관찰 결과 Empress CAD 는 세라믹 수복물의 cohesive failure 가 주를 이루었고, e.max CAD 의 경우 대부분 접착 계면에서 adhesive failure 를 나타내었다.

3. 결론

1) IPS Empress CAD 의 표면 처리시, 불산 적용 시간 및 농도를 변화시켜도 미세 전단 접착 강도에 유의한 변화가 나타나지 않았다.

2) IPS e.max CAD 의 경우 불산 적용 시간을 증가시키면 미세 전단 접착 강도도 유의하게 증가되는 양의 상관관계를 보였다.

3) IPS e.max CAD 의 경우 불산 농도를 5%에서 9.5%로 대체하여 사용 시 유의하게 높은 미세 전단 접착 강도를 보였으며, 세라믹에서의 cohesive failure 비중이 증가하였다.

Key words : microshear bond test, cerec3, CAD/CAM, glass ceramic, hydrofluoric acid, etching time, concentration