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Influence of Different Etching Protocols on the Reliability of Resin Bonding to CAD/CAM Feldspathic Porcelain

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Abstract

Objectives: To evaluate the effects of different acid etching times on the mechanical strength of dental porcelain as well as the influence on the reliability of resin bonded CAD/CAM porcelain veneer.

Material and Methods: Rectangular CAM/CAM feldspathic porcelain (Mark II, Vita Zahnfabrik) specimens (12mm × 10mm × 4mm) were prepared and polished with silicon carbide abrasive paper under running water. All the samples were randomly divided into four groups according to the corresponding etching protocols: control group (without any treatment), group A (etched with an gel etchant containing 5% hydrofluoric acid for 30 s and rinsed with de-ionized water), group B (etched for 1 min and rinsed), group C (etched for 2 min and rinsed). After silanization, resin stubs were adhered on porcelain surface. There are 25 resin-porcelain samples prepared in each group and subjected to the shear bond strength testing. Weibull analysis was conducted to evaluate of the reliability of resin-porcelain bonding. For each of the etching method, eight additional porcelain samples (3mm×2mm×10mm) were prepared and etched. Then, surface roughness (R_a), microhardness (Vickers Hardness) and biaxial flexural strengths were measured on these porcelain specimens. Energy dispersive X-ray spectrometry technique was used to assess the changes in surface chemical composition after etching and the surface topography was recorded under atomic force microscope (AFM) and scanned electron microscopy (SEM).

Results: The reliability of resin to CAD/CAM porcelain bonding was decreased with the increase in HF etching time. The application of HF etching for 30 s decreased the Vickers hardness number (HV) significantly from 651.6 (control group) to 488.7 (group A). With the extension of etching time, the Vickers hardness number was further reduced to 430.1 (group B) and 305.7 (group C). However, the biaxial flexural strengths among these four groups were not statistically significant different ($p>0.05$). AFM revealed the porous structures on the porcelain surface at microscopic level.

Conclusions: The application of HF to etch the CAD/CAM feldspathic porcelain surface reduced the microhardness number. Etching with 5% hydrofluoric acid on dental porcelain for more than 1 min might impair the reliability of resin bonded porcelain veneer.

Keywords: Surface treatment, adhesion, etching, resin cement, dental veneer, Weibull analysis

1. Introduction

The application of porcelain laminate veneers in the restoration of oral anterior area has now been increasingly developed due to their highly esthetic appearance and the relatively conservative requirements in tooth preparation [1]. However, the failure rate of porcelain veneers was relatively high when compared with full-crown restorations. Its 5-year failure rate was found to be within the range between 2% to 14% [2-5], and the 10-year failure rate of porcelain veneer was 47% according to an investigation over 2,500 prostheses [6]. Thus, many efforts have been done for improving the clinical performance in the past few decades. Particularly, the introduction of computer-aided design/computer-aided manufacturing(CAD/CAM) technique into dental practice has not only increased the speed of production due to its ease in manipulation, but also the clinical performance of laminate veneer restorations [7]. The excellent marginal fitness of CAD/CAM produced veneers could help to reduce the occurrence of micro-leakage. Moreover, with the application of multicolored ceramic blocks, the esthetic appearance of such restorations will be further improved [8].

A failure of porcelain veneer restoration might be caused by some influencing factors, such as the breakdown in resin porcelain adhesion, marginal micro-leakage, cohesive failure of veneer or tooth structure, poor marginal adaption, and improper occlusion relation [9]. A strong adhesion between porcelain veneer and tooth tissue is considered to be one of the most important factors for achieving a successful clinical performance of laminate veneer restoration [10]. Therefore, the conduction of an appropriate etching process on porcelain surface combined with the application of silane coupling agent was suggested to be a routine surface pre-treatment for the restoration with dental porcelain veneers [11]. The aim of etching on porcelain structure is to increase the surface roughness, promote the surface energy, and cleanse the bonding area [12]. Etching with 5% hydrofluoric acid (HF), which is a common regime for dental application, was found to generate the improved resin-porcelain adhesion. It is also important to note that the etching mechanism of HF on the porcelain surface is not due to the acid (i.e. H^+) but the chemical reaction of SiO_2 in glass with HF. Therefore, “acid etching” might be erroneous and “HF etching” is preferred [13]. Indeed, some other mineral acid, such as orthophosphoric acid and sulfuric acid, could not modify silicate ceramic surface effectively under normal conditions in a short time [14, 15]. Furthermore, the extension of etching duration time (HF) was found to result in the higher shear bond strength between resin adhesive and dental CAD/CAM porcelain within the range from 0 to 120 s [16].

However, the negative influence of HF etching should not be neglected. Despite the HF etching was suggested to modify the porcelain surface for a long time, its influence on the reliability and stability of resin-porcelain bonding interface has not been thoroughly investigated. Some of the surface contents, such as silicates, are dissolved with the application of HF, and thus porosities were created on porcelain surface [17]. Therefore, the strength of porcelain surface could be adversely affected, especially when a higher concentration of acid material or a longer etching duration time is adopted. Subsequently, the mechanical strength of resin bonded porcelain restoration might be impaired and the risk of failure might also be increased.

This study thus aimed to investigate the effects of different etching protocols on the mechanical strength of dental porcelain and evaluate the most appropriate setting of HF etching for dental practice. The hypothesis of the study was that HF etching would not impair the reliability of resin bonded porcelain veneer restoration.

2. Materials and methods

2.1. Preparation and grouping of porcelain specimens

Porcelain specimens with a rectangle shape (12mm × 10mm × 4mm) were sectioned from commercial dental CAD/CAM feldspathic porcelain blocks (Mark II,Vita Zahnfabrik, Germany) using a diamond saw (Microslice, Metal Research limited company, England) under running water. Subsequently, the porcelain specimens were polished with SiC abrasive paper on a polishing platform (Lunn Major, Struers, Denmark). All the specimens were then randomly divided into four experimental study groups according to the different etching protocols:

Group A (control group): no surface treatment was carried out in this group.

Group B: the specimens in this group were etched with an gel etchant (Vita Ceramics Etch, Vita Zahnfabrik, Germany) containing 5% hydrofluoric acid and 10% sulfuric acid for 30 s and rinsed with de-ionized water thoroughly. They were then dried with clean air flow.

Group C: the porcelain samples were etched with the same acid gel as group B for 1 min, then rinsed and dried.

Group D: the porcelain samples were etched with the same acid gel as group B for 2 min, then rinsed and dried.

2.2. Preparation and shear bond strength testing of resin/porcelain composite specimens

A silane coupling agent (RelyX^{TM} Ceramic Primer, 3M ESPE, Germany) was applied on the surface of each porcelain specimen. They were left for drying and chemical reaction for 3 min. A transparent polyethylene mould with the inner diameter of 3.6 mm was utilized in the bonding process. It was fixed on the porcelain surface and filled with a self-adhesive resin cement (RelyX^{TM} Unicem Aplicap, 3M ESPE, USA) which was prepared according to instructions of the manufacturer. Then, the resin stubs were light cured using a halogen curing lamp unit (Elipar^{TM} 2500, 3M ESPE, USA) from the up and lateral sides of the mould for 40 s, respectively . The polyethylene mould was removed carefully after curing process was finished. Each resin stub was prepared with a diameter of 3.6 mm and the thickness of 3 mm. There were 25 resin/porcelain specimens produced in each group. All the specimens were preserved for 24 h at room temperature before shear bond strength (SBS) testing.

The SBS testing was conducted on a universal testing machine (ElectroPuls ™ E3000, Instron, USA). Each specimen was mounted with a metal jig and subjected to the loading until the occurrence of failure. The speed of crosshead was set at 1 mm/min. The shear bond strength was obtained by dividing the load of failure with adhesion area.

2.3. Measurement of surface roughness

Furthermore, in each group, eight extra porcelain specimens (3mm×2mm×10mm) were prepared and etched with the same procedure. All these porcelain specimens were measured with the values of surface roughness (R_a) after surface modification. Higher R_a value indicated the rougher surface. Each porcelain sample was measured for three times and the mean value was adopted as the indicative value. The process of measurement was conducted on a flat surface using a profilometer (Surtronic 3+, Taylor Hobson Ltd, UK). The cut-off value was 0.8 mm. The mean value of each group was then calculated. There were eight measurements of surface roughness in each group.

2.4. Measurement of microhardness

The measurement of microhardness was carried out on a Vickers microhardness tester (Micro-hardness tester, Leitz, Germany) using a 200 g load for each porcelain specimen. The loading time was 20 s. Three measurements were conducted on the modified surface of each sample and the mean value was adopted as the indication of corresponding sample.

2.5. Evaluation of biaxial flexural strength

Biaxial flexural strength test was conducted according to ISO 6872: 2008 [18]. Forty Mark II CAD/CAM ceramic discs were prepared with the size of 12 mm in diameter and 1.2 mm in thickness. There were ten specimens in each group. Each sample was placed concentrically on three supporting metal balls which were arranged equally apart on a circle. The load was applied on the center of each ceramic disc until fracture happened. The speed of cross-head was 1.0 mm/min. The biaxial flexural strength was calculated using the following equation:

$$\sigma = -0.287P(X-Y)/b^2 \quad (1)$$

where σ is the maximum center tensile stress (MPa); P is the load of failure (N); b is the thickness of ceramic sample (mm); $X=(1+v)\ln(r_2/r_3)^2 + ((1-v)/2)(r_2/r_3)^2$; $Y=(1+v)(1+\ln(r_1/r_3)^2) + (1-v)(r_1/r_3)^2$;.

In which v is the Poisson's ratio (0.25 was taken for the ceramic examined); r_1 is the radius of supporting circle (mm); r_2 is the radius of loaded region (mm); r_3 is the radius of ceramic sample (mm).

2.6. Surface observation with atomic force microscopy

The examination of atomic force microscopy (AFM, ScanAsyst™, Bruker, Germany) technique on porcelain surface was performed with PeakForce Tapping mode. Zones with the size of 10 µm × 10 µm were scanned at a slow speed of 0.1 Hz. Images were taken in opening environment for the analysis at a small scale.

2.7. Scanning electron microscopy (SEM) observation

A scanning electron microscope (Hitachi SU1510, Hitachi High-Tech, Japan) was utilized in the observation of surface morphology of porcelain specimens modified with different etching protocols. After gold sputtering, the examination was performed at a voltage of 40 kV with ×1000 magnification. The examination with energy dispersive X-ray spectrometry (EDX) technique was performed for the analysis of surface elemental composition.

2.8. Statistical analysis

The results of surface roughness and Vickers microhardness number were analyzed with a statistical software (SPSS 16.0, SPSS Inc, USA). One-way ANOVA was adopted in the comparison of effects of different etching duration times on porcelain surface morphology and mechanical strength.

The shear bond strength values were analyzed with two-parameter Weibull distribution. The calculation process was carried out according to the instructions of ISO standard 20501:2003 [19].

The cumulative distribution expression is defined as below:

$P_f = 1 - \exp[-(\sigma/\sigma_\theta)^m]$ (2)

where P_f denotes the probability of failure; σ_θ indicates the characteristic strength and it corresponds to the value when P_f is 63.2%; m is the Weibull modulus, the higher m value indicates a closer grouping of the shear bond strength data; and σ is the shear bond strength value of each sample measured. The value of P_f corresponds to the ranking of each specimen.

All the specimens in each group were ranked in ascending order and the probability of failure was calculated based on the following equation:

$P_f(\sigma_i) = (i - 0.5) / N$ (3)

where i is the ith datum and N is the number of specimens in each group.

The following equation is derived from function (2):

$\ln\ln[1/(1-P_f)] = m\ln\sigma - m\ln\sigma_\theta$ (4)

By plotting $\ln\ln[1/(1-P_f)]$ (as the ordinate) against lnσ (as the abscissa), a slope with value m and the intercept which is equal to $m\ln\sigma_\theta$ can then be obtained. Therefore, the parameters of the Weibull distribution and fitted lines have been provided by maximum likelihood analysis. The stresses at which 1% and 90% specimens failed were calculated for each group. The accumulative failure probability curves were produced to evaluate the distribution of resin porcelain shear bond strengths.

3. Results

It is shown in Table 1 that the lowest mean value of surface roughness (0.11 ± 0.02 μm) was obtained in the control group and group D had the highest mean value (0.52 ± 0.09 μm). In particular, group B, group C and group D had significantly higher($p<0.05$) roughness than the control group. However, the difference between group C and group D was not significant ($p>0.05$).

Table 1 also demonstrates that the control group had the highest mean value of microhardness (651.6 ± 29.6 HV). With the application of HF etching, the microhardness number of the porcelain was reduced to 488.7 ± 26.1 HV in group B, 430.1 ± 23.4 HV in group C, and 305.7 ± 16.5 HV in group D. The differences among these four groups were significant ($p<0.05$). In the biaxial flexure test, it was found that the highest mean biaxial flexural strength was produced in the control group (90.0±5.5 MPa), followed by group B (89.0 ± 4.8 MPa), group D (86.8 ± 9.6 MPa), and group C (86.3±7.4 MPa). Nonetheless, the differences among these four groups were not significant ($p>0.05$).

The results of EDX evaluation are shown in Table 2. Different etching treatments produced various changes in the surface elemental constitution. Group C had the highest weight percentage of silicon content (24.15%) and the group D had the lowest value (18.27%).

The mean shear bond strength values are listed in Table 3. The control group had the highest mean shear bond strength (11.60 ±1.93 MPa). With the application of HF etching, a slight decrease in bond strength was found. The lowest value was obtained in group D (10.26 ±1.46 MPa). The difference between these two groups was significant ($P<0.05$). The results of Weibull analysis show that the characteristic strength of control group was the highest among all the four experimental groups. However, significant difference ($p<0.05$) could only be found between the control group and group D since there was an overlap in the 95% confidence intervals of these two groups. Group B had the highest Weibull modulus (9.99) which indicates a closer distribution of data than other groups. R^2-values of linear regression varied from 0.86 to 0.96. Therefore, the fitting of experimental data to Weibull distribution was acceptable. The stresses for the failure probability of 1% and 90% in each group were also listed. In Figure 1, the fitting lines (stress against probability of failure) were plotted on Weibull probability paper with the two-sided 90% confidence bounds for demonstrating the failure points of shear bond strength testing. Each dot in the graph represents an actual experimental value. The observation of failure mode showed that the cohesive destruction in porcelain base was the only mode of failure that could be found.

The morphological changes of porcelain surface after different surface treatments are shown in Figure 2. In the control group, the porcelain surface was relatively smooth and only some shallow grooves produced in the process of polishing treatment could be found. After being etched for 30 s, the formation of porous structures, such as pits and craters, could be observed because of the dissolution of surface contents. With the extension of etching time to 1 min, more porous structures on porcelain surface were found to be created. Further increase in etching time to 2 min resulted in the generation of excessive surface destructions.

Three-dimensional representative AFM images are displayed in Figure 3. The comparison of feature depth among four experimental groups revealed that the pattern of porcelain surface of control group was the most uniform. The etched surfaces were much rougher than the control group. Longer etching time tended to produce more and deeper porous structures with the dissolution of glassy matrix. Group C and group D exhibited more retentive structures for the infiltration of flowable resin cement than the other groups.

4. Discussion

The dental porcelain investigated in the current study is in fact a composite material consisted of glassy matrix and feldspathic crystalline components. It was designed to be applied in the production of veneer restorations in oral esthetic zones. As an essential surface treatment process in cementation procedures, etching with hydrofluoric acid would lead to the dissolution of silica content and result in the formation of hexafluorosilicate [20]. The surface roughness would also be significantly increased. This was confirmed in current SEM examination that HF etching generated porous structures on porcelain surface due to the chemical reaction between hydrofluoric acid and silica. The longer etching time helped to produce higher values of surface roughness due to the dissolution of more silica contents. However, the surface roughness was not significantly increased further when the HF etching was extended from one minute to two minute. This result might indicate that etching process longer than one minute could not generate more retentive structures for the infiltration of resin cement. On the other hand, it was reported in one study [17] that the resultant fluorosilicates of HF etching were insoluble and could only be removed by ultrasonic cleaning and the resin porcelain bonding might be impaired by the generation of these precipitates. However, the deposition of fluorosilicates could not be observed by SEM examination in our study. Furthermore, no fluorine content was detected in EDX examination which might be explained by that the resultant fluorosilicates could be effectively removed by rinsing with de-ionized water and the application of ultrasonic cleaning was not necessary.

The porous structures on porcelain surface produced by HF etching could play an important role in the establishment of micro-interlocking between porcelain and resin composite cement and helped to increase the shear bond strength [21]. AFM observation results in this study also revealed that etching for 1 and 2 min tended to produce the most effective surface topographical pattern for the infiltration of flowable resin cement as well as the formation of micro-retentive resin tags.

Nonetheless, the negative influence of HF etching on the mechanical properties of porcelain should not be underestimated since such porous structures might also act as the surface flaws. It was demonstrated that HF etching could cause the significant reduction in the flexural strength and reliability of feldspathic porcelain [22]. As seen from the current results, the control group exhibited the highest microhardness number of 651.6 HV and the decrease in microhardness was induced by the application of HF etching gel. Especially, the value of group D was even less than 50% of the control group. The significant decrease in microhardness on adhesion surface was due to the loss of glassy matrix and might also be the indication of lower reliability and stability of resin bonded veneer. In clinical situation, this might lead to increased risk of failure. The results of shear bond strength testing could also add to this point. It was showed that the mean shear bond strength value of the control group, the characteristic strength of Weibull distribution, as well as the stress for 90% failure rate were the highest among all the four testing groups. Furthermore, the difference between the control group and group D (etching for 2 min) was significant. Such findings were different from the previous studies. The reason may be partially answered by the observation of failure mode. Indeed, the cohesive failure in porcelain was the only mode of failure found in the current study, *i.e.* the propagation of cracks developed within the felspathic porcelain. This is to say the bond strength between resin and porcelain was higher than the cohesive strength of the porcelain. Thus, the destruction in porcelain occurred before the debonding between resin and porcelain. Therefore, the tested bond strength was in fact determined by the surface mechanical strength of porcelain. Accordingly, the reduction in the surface microhardness which might be caused by any surface treatments could lead to the decrease in resin-to-porcelain shear bond strength. The reliability of porcelain veneer restoration could then be significantly impaired. In order to reduce the risk of failure, the application of HF etching should be carried out with great care and the overtreatment of the porcelain surface should be avoided.

On the other hand, the biaxial flexure test also showed that the differences among four experimental groups were not significant. Such results might indicate that the deleterious influence of HF etching was only limited to the superficial layer of porcelain and the mechanical strength of the bulk of porcelain sample was still maintained. Therefore, the etching reduced the surface mechanical strength of the porcelain instead of flexural strength of the integral specimen which might lead to the decrease in resin-to-porcelain shear bond strength.

The conduction of silanization treatment in adhesion process was reported to significantly enhance the bond strength between porcelain and resin cement [23]. The hydrolyzed silane molecules contain both silanol groups which could react with the hydroxyl groups on silica surface and the vinyl groups that could combine with organic reactive sites in resin composite cement. Therefore, silane coupling agents was reported to produce a linkage function for improving the bond strength between organic resin cement and inorganic restoration surface [24]. According to the results of EDX examination, all the four groups had high concentration of silicon content on porcelain surface which might be beneficial to the linkage function of silane coupling agents and a relatively strong resin to porcelain bond strength could also be expected. Although the surface roughness of the control group was much lower than the other three groups, the higher mean resin porcelain bond strength was still produced with the application of silane coupling agent. In other words, the linkage function of silane coupling agent helped to make up the loss in the formation of surface porous structure in the control group and played an important role in maintaining the reliability of resin porcelain integration.

Higher Weibull modulus values show the less scattering of the resultant bond strengths [25].Therefore, group B with the highest Weibull modules among the four experimental groups might represent the more valid and reliable approach. The R^2 values of four experimental groups in Weibull analysis indicate the relatively good fit to Weibull distribution. This is important for a valid prediction of mechanical behaviors with different etching treatments. Therefore, the Weibull plots could present the failure probability at different stress levels rather than merely the mean value of shear bond strength. However, in this study, only the initial value of resin porcelain bond strength was evaluated and only one type of porcelain was investigated. Therefore, the current results may not represent all the situations under clinical conditions. In the further investigation, the evaluation of bond strengths between dental porcelain with different resin cements under various aging conditions should be carried out. Nonetheless, it can still provide some indications about the negative influence of HF etching on the reliability of porcelain veneer restoration.

5. Conclusions

Within the limitations of this laboratory study, the following conclusions were drawn: Etching of the CAD/CAM feldpathic porcelain surface reduced the microhardness of dental porcelain. Etching with 5% hydrofluoric acid on dental porcelain for more than 1 min might impair the reliability of resin bonded CAD/CAM porcelain veneer.

References

[1] Chen JH, Shi CX, Wang M, Zhao SJ, Wang H. Clinical evaluation of 546 tetracycline-stained teeth treated with porcelain laminate veneers. J Dent 2005;33: 3-8.

[2] Aristidis GA, Dimitra B. Five-year clinical performance of porcelain laminate veneers. Quintessence Int 2002;33:185-9.

[3] Murphy E, Ziada HM, Allen PF. Retrospective study on the performance of porcelain laminate veneers delivered by undergraduate dental students. Eur J Prosthodont Restor Dent 2005;13:38-43.

[4] Peumans M, Van Meerbeek B, Lambrechts P, Vuylsteke-Wauters M, Vanherle G. Five-year clinical performance of porcelain veneers. Quintessence Int 1998;29: 211-21.

[5] Walls AW. The use of adhesively retained all-porcelain veneers during the management of fractured and worn anterior teeth: Part 2. Clinical results after 5 years of follow-up. Br DentJ 1995;178:337-40.

[6] Burke FJ, Lucarotti PS. Ten-year outcome of porcelain laminate veneers placed within the general dental services in England and Wales. J Dent 2009;37:31-8.

[7] Miyazaki T, Hotta Y. CAD/CAM systems available for the fabrication of crown and bridge restoration. Aust Dent J 2011;56 Suppl 1:97-106.

[8] Höland W, Schweiger M, Watzke R, Peschke A, Kappert H. Ceramics as biomaterials for dental restoration. Expert Rev Med Devices 2008;5:729-45.

[9] Calamia JR, Calamia CS. Porcelain laminate veneers: reasons for 25 years of success, Dent Clin North Am 2007;51:399-417, ix.

[10] Morig G. Aesthetic all-ceramic restorations: a philosophic and clinical review. Pract Periodontics Aesthet Dent 1996; 8:741-9; quiz 50.

[11] Peumans M, Van Meerbeek B, Lambrechts P, Vanherle G., Porcelain veneers: a review of the literature. J Dent 2000; 28:163-77.

[12] Van Noort R. Introduction to Dental Materials4th. Elsevier Health Sciences 2013.

[13] Tian T, Tsoi JK, Matinlinna JP, Burrow MF. Aspects of bonding between resin luting cements and glass ceramic materials. Dent Mater 2014;30:e147-62.

[14] Ayad MF, Fahmy NZ, Rosenstiel SF. Effect of surface treatment on roughness and bond strength of a heat-pressed ceramic. J Prosthet Dent 2008;99:123-30.

[15] Jang HK, Chung YD, Whangbo SW, Lee YS, Lyo IW, Whang CN, Lee SJ, Kim G. Effects of chemical etching with sulfuric acid on glass surface. J Vac Sci Technol A 2000;18:401-4.

[16] Chen JH, Matsumura H, Atsuta M. Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain. J Dent 1998;26:53-8.

[17] Canay S, Hersek N, Ertan A. Effect of different acid treatments on a porcelain surface. J Oral Rehabil 2001;28:95-101.

[18]ISO, Dentistry – Ceramic materials (2008).

[19] ISO, Fine ceramics (advanced ceramics, advanced technical ceramics)-Weibull statistics for strength data (2003).

[20] Soares CJ, Soares PV, Pereira JC, Fonseca RB. Surface treatment protocols in the cementation process of ceramic and laboratory-processed composite restorations: a literature review. J Esthet Restor Dent 2005;17:224-35.

[21] Özcan M, Vallittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. Dent Mater 2003;19:725-31.

[22] Addison O, Marquis PM, Fleming GJ. The impact of hydrofluoric acid surface treatments on the performance of a porcelain laminate restorative material. Dent Mater 2007; 23:461-8.

[23] Brentel AS, Ozcan M, Valandro LF, Alarca LG, Amaral R, Bottino MA. Microtensile bond strength of a resin cement to feldpathic ceramic after different etching and silanization regimens in dry and aged conditions. Dent Materi 2007;23: 1323-31.

[24] Lung CY, Matinlinna JP. Aspects of silane coupling agents and surface conditioning in dentistry: an overview. Dent Mater 2012;28:467-77.

[25] Tian T, Tsoi JKT, Matinlinna JP, Burrowa MF. Evaluation of microtensile bond strength on ceramic-resin adhesion using two specimen testing substrates. Int J Adhes Adhes 2014;54:165-71.

Table1 Comparison between different acid etching treatments

Group	Surface treatment	Surface roughness (μm)	Microhardness (HV)	Biaxial flexural strength (MPa)
A	Control	0.11± 0.02a	651.6± 29.6^{a}	90.0 ± 5.5^{e}
B	Etching for 30 s	0.25 ± 0.04b	488.7± 26.1^{b}	89.0 ± 4.8^{e}
C	Etching for 1 min	0.50± 0.07^{c,d}	430.1± 23.4^{c}	86.3 ± 7.4^{e}
D	Etching for 2 min	0.52±0.09^{d}	305.7± 16.5^{d}	86.8 ± 9.6^{e}

(Different superscript letters indicate the significant differences ($p<0.05$))

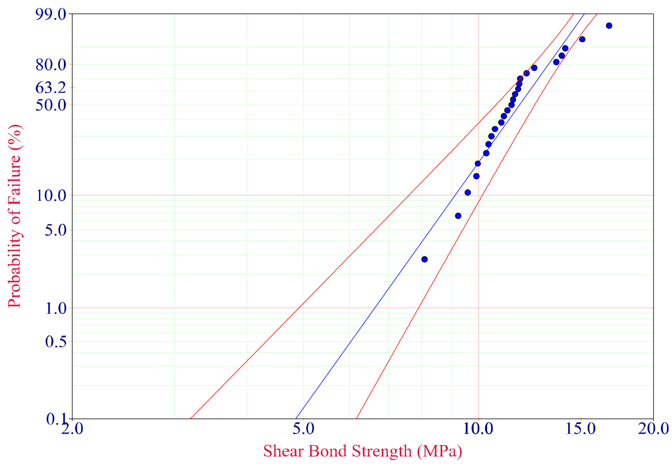
Table 2 The EDX analysis of porcelain surface(wt %)

	C	O	Na	Al	Si	K
Group A	14.97	43.50	5.00	10.40	21.68	4.45
Group B	20.74	42.96	3.70	8.14	19.81	4.64
Group C	10.02	46.49	4.22	9.30	24.15	5.82
Group D	24.24	42.36	3.16	7.46	18.27	4.51

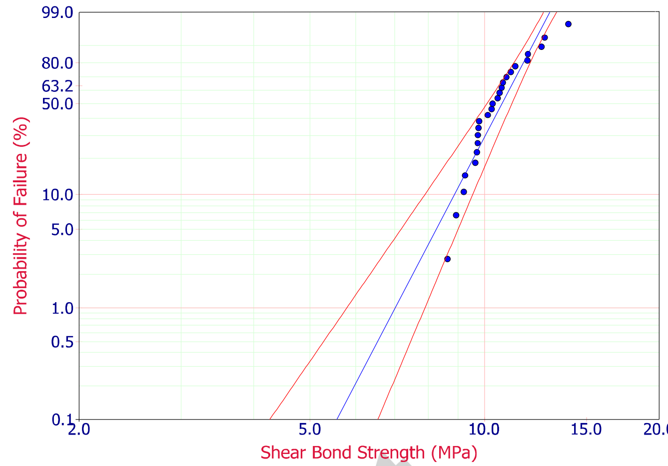
Table 3 Weibull analysis of shear bond strengths (n=25)

	Mean shear bond strength(MPa)	Weibull modulus (m)	Characteristic strength (MPa) σ_θ	95% Confidence intervals of σ_θ	R^2	Stress for 1% failure probability (MPa) $\sigma_{0.01}$	Stress for 90% failure probability (MPa) $\sigma_{0.90}$
Group A	11.60 ±1.93 [a]	7.38	12.37	11.63-13.16	0.90	6.63	13.85
Group B	10.58 ±1.27 [a,b]	9.99	11.12	10.61-11.66	0.86	7.02	12.09
Group C	10.37 ±2.32 [a,b]	5.36	11.25	10.40-12.18	0.92	4.77	13.15
Group D	10.26 ±1.46 [b]	8.11	10.88	10.34-11.46	0.96	6.17	12.06

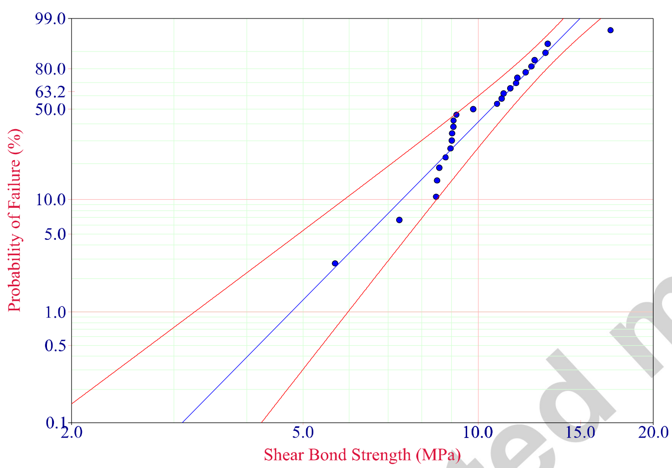
Different superscript letters indicate the significant differences ($p<0.05$). (A) Control; (B) Etching for 30 s; (C) Etching for 1 min; (D) Etching for 2 min



Group A: Control



Group B:Etching for 30 s



Group C:Etching for 1 min

Group D:Etching for 2 min

Figure. 1 Weibull plots for resin porcelain bonding (Shear stress vs probability of failure) with 90% confidence bounds. The fitted lines were produced by maximum likelihood analysis of experimental data according to Weibull distribution equation. Black dots in the graph are actual experimental data points.

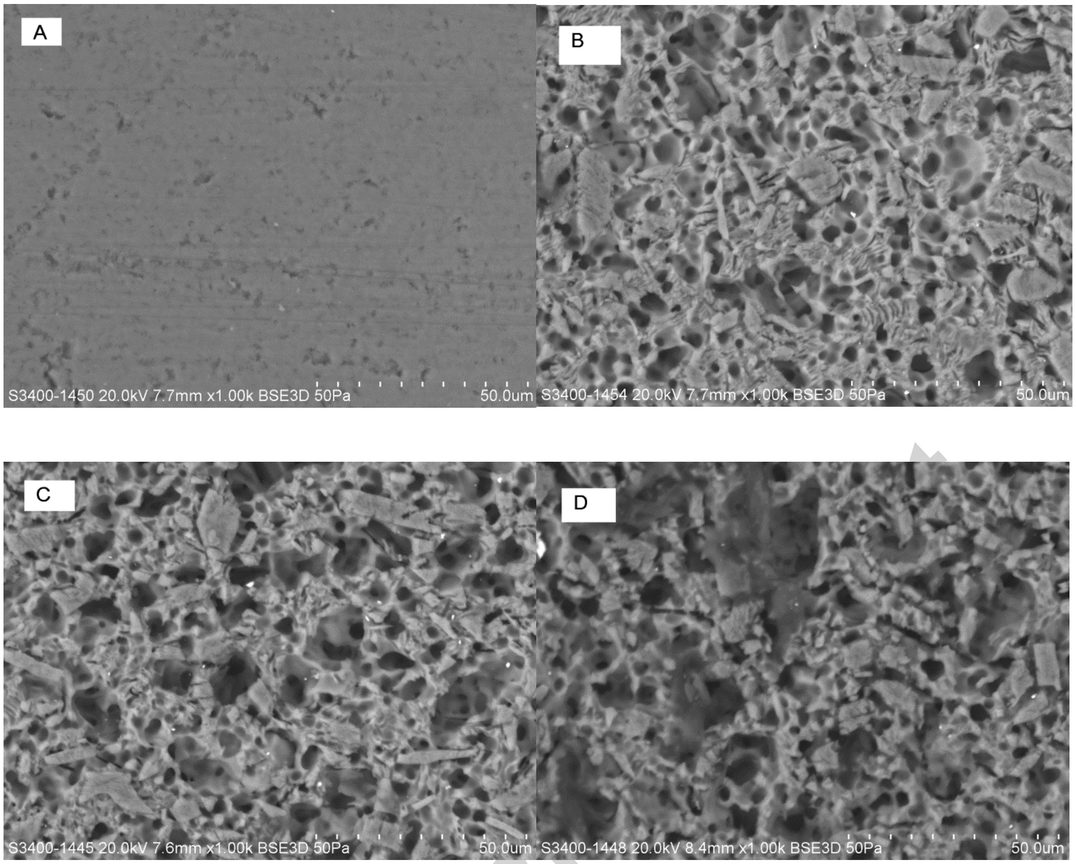


Figure 2 Representative images of SEM examination (magnification × 1000) (A) Control; (B) Etching for 30 s; (C) Etching for 1 min; (D) Etching for 2 min

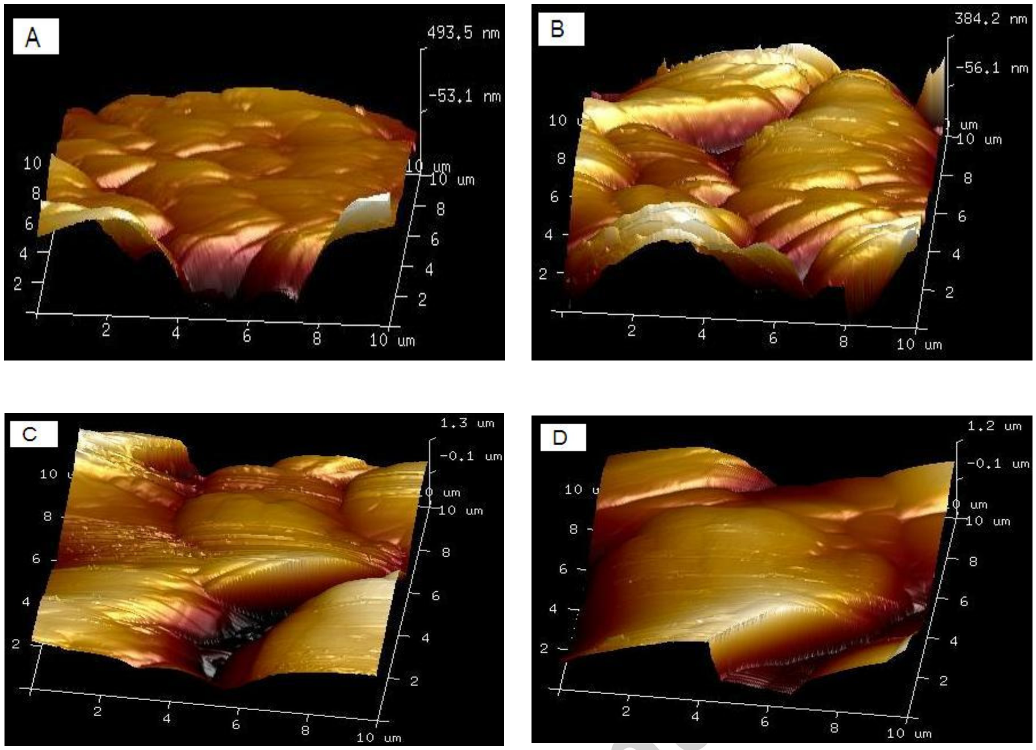


Figure 3 Representative images of AFM observation (A) Control; (B) Etching for 30 s; (C) Etching for 1 min; (D) Etching for 2 min