*The 2014 World Congress on*
***Advances in Civil, Environmental, and Materials Research (ACEM14)***
*Busan, Korea, August 24-28, 2014*

# Influence of different etching time on microhardness of dental porcelain

Dan Liu[1)], Kit-Hon Tsoi[2)], and *Hai Ming Wong[2)]

[1), 2)] *Faculty of Dentistry, The University of Hong Kong, 34 Hospital Road, Hong Kong*
[2)] *wonghmg@hku.hk*

## ABSTRACT

The aim of this study was to evaluate the effects of different etching time on the surface properties of dental porcelain used for veneer restoration. Totally, 32 rectangle porcelain specimens were prepared and randomly divided into four groups according to the corresponding etching protocols: control group (without any treatment), group A (etched with an acid gel for 30 s and rinsed), group B (etched for 1 min and rinsed), group C (etched for 2 min and rinsed). All the porcelain specimens were measured with values of surface roughness (Ra) and then subjected to the test of microhardness (Vickers Hardness). EDX technique was used to assess the changes in surface chemical composition after etching and the surface topography was recorded under atomic force microscope (AFM). It was found that the values of surface roughness of four groups were 0.11, 0.25, 0.50, and 0.52, respectively. The difference between group B and group C was not significant. The application of acid etching for 30 s decreased the Vickers hardness number significantly from 652 (control group) to 489 (group A). With the extension of etching time, the Vickers hardness number was further reduced to 430 (group B) and 306 (group C). AFM examination showed that longer etching time resulted in the formation of more surface voids and pits on porcelain surface. The results of EDX assessment indicated that group B had the highest silicon content which might be beneficial for the function of silane coupling agent. The application of surface acid etching reduced the microhardness number of dental porcelain. Acid etching on dental porcelain for 1 min tended to produce the most appropriate surface condition for bonding with resin cement.

## 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 (Chen et al. 2005). 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% (Aristidis and Dimitra 2002; Murphy et al. 2005; Peumans et al. 1998; Walls 1995),

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[1)] Research Assistant
[2)] Assistant Professor

and the 10-year failure rate of porcelain veneer was 47% according to an investigation over 2,500 prostheses (Burke and Lucarotti 2009). Thus, many efforts have been done for improving its clinical performance in the past few decades.

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 (Calamia et al. 2007). 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 veneer restoration. Therefore, the conduction of an appropriate acid etching process on porcelain surface combined with the application of silane coupling agent should be carried out with great care (Peumans et al. 2000). The aim of acid etching on porcelain structure is to increase the surface roughness, promote the surface energy, and cleanse the bonding area (Van Noort 2013). Although, it was suggested that etching with HF could help to modify porcelain surface well, its influence on the reliability and stability of resin/porcelain bonding interface is still unknown. Since some of the surface contents are dissolved with the application of acid etching material, such as some silicates, porosities will be created on porcelain surface (Canay et al. 2001). Thus, the strength of porcelain material might be adversely affected, especially when a higher concentration of acid material or a longer etching duration time is adopted. Subsequently, the risk of failure might also be increased.

This study was aimed to investigate the effects of different etching protocols on the microhardness of dental porcelain and evaluate the most appropriate setting of acid etching for dental practice.

## 2. MATERIALS AND METHODS

### *2.1 Specimen preparation and surface treatments*

Thirty two porcelain specimens with a rectangle shape (2mm × 3mm × 10mm) were sectioned from commercial dental porcelain blocks (VITABLOCS® Mark II, Vita Zahnfabrik, Germany) using a diamond saw (Microslice, Metal Research limited company, England) with the cooling of running water. Subsequently, the porcelain specimens were polished with SiC abrasive paper under running water on a polishing platform (Lumn Major, Struers, Denmark). All the specimens were then divided into four experimental study groups (n=8) using the method of randomization, according to the different acid etching protocols applied:

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

Group B: the specimens in this group were etched with an acid gel (Vita Ceramics Etch, Vita Zahnfabrik, Germany) containing 5% hydrofluoric acid and 10% hydrosulfuric acid for 30 s and rinsed with de-ionized water. 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 Measurement of surface roughness*

All the thirty two specimens were measured with the values of surface roughness (Ra) after surface modification. Higher Ra value indicated the rougher surface. Each porcelain sample was evaluated 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.

*2.3 Measurement of microhardness*

The measurement of microhardness was carried out on a Vickers microhardness tester (Micro-hardness tester, Leitz, Germany) using a 200g 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.

*2.4 Observation with scanning electron microscope (SEM)*

A scanning electron microscope (Hitachi SU1510, Hitachi High-Tech, Japan) was utilized for the observation of surface morphology of porcelain specimen modified with different etching protocols. One additional porcelain specimen in each group was then prepared. 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.

*2.5 Examination of atomic force microscopy (AFM)*

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 small scale.

*2.6 Statistical analusis*

The final 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 conditions and mechanical strength.

## 3. RESULTS

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). Both group B, group C and group D had significantly ($p<0.05$) higher values than the control group. However, the difference between group C and group D was not significant ($p>0.05$).

The control group showed the highest mean value of microhardness (651.6 ± 29.6 HV). With the application of acid etching, the microhardness number of 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 ($p<0.05$).

For the morphological conditions of the control group, the porcelain surface was relatively smooth and with only some shallow grooves produced in the process of polishing treatment. After being acid 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.

The results of EDX evaluation showed that different acid etching treatments produced various changes in the surface elemental constitution. Group C had the highest weight percent number of silicon content (24.15%) and the group D had the lowest value (18.27%).

For the three-dimensional representative AFM images, 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 and 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 will lead to the dissolution of silica content and result in the formation of hexafluorosilicate (Soares et al. 2005). The surface roughness will also be significantly increased. This was confirmed in current SEM examination that acid 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 acid 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 adhesion of resin cement. On the other hand, it was reported in one study (Canay et al. 2001) that the resultant fluorosilicates of HF etching were insoluble which 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.

## 6. CONCLUSIONS

The application of surface acid etching reduced the microhardness number of dental porcelain. Acid etching on dental porcelain for 1 min tended to produce the most appropriate surface condition for bonding with resin cement.

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