



# Surface treatment of feldspathic porcelain: scanning electron microscopy analysis

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**PURPOSE.** Topographic analysis of treated ceramics provides qualitative information regarding the surface texture affecting the micromechanical retention and locking of resin-ceramics. This study aims to compare the surface microstructure following different surface treatments of feldspathic porcelain. **MATERIALS AND METHODS.** This *in-vitro* study was conducted on 72 porcelain discs randomly divided into 12 groups (n=6). In 9 groups, feldspathic surfaces were subjected to sandblasting at 2, 3 or 4 bar pressure for 5, 10 or 15 seconds with 50 µm alumina particles at a 5 mm distance. In group 10, 9.5% hydrofluoric acid (HF) gel was applied for 120 seconds. In group 11, specimens were sandblasted at 3 bar pressure for 10 seconds and then conditioned with HF. In group 12, specimens were first treated with HF and then sandblasted at 3 bar pressure for 10 seconds. All specimens were then evaluated under scanning electron microscopy (SEM) at different magnifications. **RESULTS.** SEM images of HF treated specimens revealed deep porosities of variable sizes; whereas, the sandblasted surfaces were more homogenous and had sharper peaks. Increasing the pressure and duration of sandblasting increased the surface roughness. SEM images of the two combined techniques showed that in group 11 (sandblasted first), HF caused deeper porosities; whereas in group 12 (treated with HF first) sandblasting caused irregularities with less homogeneity. **CONCLUSION.** All surface treatments increased the surface area and caused porous surfaces. In groups subjected to HF, the porosities were deeper than those in sandblasted only groups. [J Adv Prosthodont 2014;6:387-94]

**KEY WORDS:** Air abrasion; Dental porcelain; Hydrofluoric acid; Scanning electron microscopy; Surface treatment

## INTRODUCTION

A reliable bond between the tooth structure and restorative materials has always been a concern in restorative dentistry.<sup>1</sup> Dentists are always searching for methods to increase the longevity and function of restorations.<sup>2</sup> A durable bond

between the resin cement, tooth structure and restorative material is required to form a uniform coherent structure and ensure the long-term survival and clinical success of restorations.<sup>3</sup>

Both micromechanical and chemical bonding of ceramic surfaces reinforce the fracture resistance of the restored teeth and restorations, provide high retention, prevent microleakage and improve marginal adaptation.<sup>4</sup> Resin-ceramics bond strength is influenced by the ceramic surface treatment technique depending on its chemical composition.<sup>5-7</sup>

Feldspathic porcelains are a type of silica-based porcelains containing quartz and feldspar.<sup>6</sup> Feldspar bonds to some metal oxides to form a glass phase with an amorphous structure.<sup>6,8</sup> This type of porcelain has a high glass content and can best simulate optical properties of enamel and dentin and thus, is commonly used for manufacturing indirect restorations namely laminates, inlays, onlays and overlays.<sup>6,8</sup> They are also used for veneering of ceramic copings and metal frameworks.<sup>9</sup> In these porcelains, quartz

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constitutes the crystalline phase.<sup>6</sup> In addition to high esthetics, these ceramics have high resistance to wear and compressive loads and are biocompatible.<sup>6,9</sup> However, their mechanical strength is lower than other ceramic types.<sup>6</sup>

These ceramic restorations require internal surface preparation in order to bond to tooth structure with the use of adhesive cements<sup>8</sup> and may also undergo chipping or fracture due to various reasons.<sup>10,11</sup> Therefore, they require surface treatment before repair with composite resin to prevent replacement of restoration, further damage to the tooth and high cost for patients<sup>2</sup> and a simpler, more conservative and quicker treatment is performed as such.<sup>12</sup> On the other hand, with increasing number of adults requiring orthodontic treatment, there is a need for a reliable method for bonding brackets to ceramic surfaces.<sup>13,14</sup>

In all conditions mentioned above, a strong resin bond should be achieved by pre-treatment and formation of micromechanical interlocking to ceramic surfaces. This process requires micro-roughness and cleaning of the surface for adequate retention.<sup>15,16</sup>

Acid etching with HF, sandblasting with alumina particles or sometimes a combination of both are among the most common surface treatments for silica-based ceramics. The bond strength following these surface treatments has been assessed in several studies.<sup>10,17,18</sup>

HF affects the feldspathic porcelain through reaction with the glass phase<sup>2</sup> and its optimal concentration and application time have been specified.<sup>19</sup> The mechanism of action of sandblasting is abrasion of the surface with aluminum oxide particles under air pressure through a chair-side device that increases surface area.<sup>20</sup> Function of this process depends on various parameters namely size of particles, applied pressure and duration of application.<sup>21,22</sup> According to Dravell's study,<sup>23</sup> large abrasive particles can create greater surface abrasion because surface abrasion is increased by the square of particle diameter. Studies regarding the effect of other parameters of sandblasting on surface roughness of feldspathic porcelain are scarce.<sup>24</sup> Moreover, using a combination of HF etching and sandblasting, may be the order of applied methods due to them different mechanisms of action, may yield variable results in terms of porcelain surface roughness and micromechanical retention and thus are in need of further investigation.

By analyzing the surface topography of treated ceramics, we can obtain qualitative data regarding the surface texture that can affect the mechanism of micromechanical retention.<sup>25</sup> Scanning electron microscopy (SEM) provides high-resolution information regarding the surface texture for the assessment of surface topography.<sup>26</sup>

This study aims to compare the surface texture of feldspathic porcelain following different treatment techniques. The null hypothesis was that the surface roughness following the use of different sandblasting pressures and times and HF acid etching is not significantly different. Another hypothesis was that surface roughness in the HF acid etching and sandblasting combined techniques is not influenced by the order of applied methods.

## MATERIALS AND METHODS

For the assessment of surface topography, 72 porcelain discs measuring 6 mm × 2 mm (CeramCo 3, Dentsply, Burlington, NJ, USA) with metal base (Ni-Cr alloy) were prepared, fired and polished according to the manufacturer's instructions (Fig. 1A). Specimens were evaluated under a magnifier at 10× magnification to ensure absence of cracks or chipping. Specimen surfaces were polished with 400 and 600 grit SiC discs under running water, rinsed and dried with oil-free air. Specimens were randomly divided into 12 groups of 6 and coded. The following surface treatments were performed (Table 1):

Group (G) 1. The surface of specimens was sandblasted using microsandblaster (Microetcher II, Danville Materials Inc., San Ramon, CA, USA) (Fig. 1B) with 50 µm alumina particles (Ronvig, Denmark) at 2 bar pressure for 5 seconds from a 5 mm distance at 90° angulation of the nozzle in a circular motion. (For the surface preparation, a special jig was fabricated for this purpose for all specimens in 12 groups.) Specimens were then rinsed with oil-free water and dried with oil-free air.

G 2. Specimens were sandblasted similar to G 1 but sandblasting was performed at 2 bar pressure for 10 seconds.

G 3. Specimens were sandblasted similar to G 1 but sandblasting was performed at 2 bar pressure for 15 seconds.

G 4. Specimens were sandblasted similar to G 1 but sandblasting was performed at 3 bar pressure for 5 seconds.

G 5. Specimens were sandblasted similar to G 1 but sandblasting was performed at 3 bar pressure for 10 seconds.

**Table 1.** Surface treatment parameters in different study groups

Group	Sandblasting pressure (bar)	Sandblasting time (seconds)	HF etching time (seconds)
G1	2	5	-
G2	2	10	-
G3	2	15	-
G4	3	5	-
G5	3	10	-
G6	3	15	-
G7	4	5	-
G8	4	10	-
G9	4	15	-
G10	-	-	-
G11*	3	10	120
G12**	3	10	120

\*In this group, specimens were first sandblasted and then etched with HF acid gel.

\*\*In this group, specimens were first etched with HF acid gel and then sandblasted.

G 6. Specimens were sandblasted similar to G 1 but sandblasting was performed at 3 bar pressure for 15 seconds.

G 7. Specimens were sandblasted similar to G 1 but sandblasting was performed at 4 bar pressure for 5 seconds.

G 8. Specimens were sandblasted similar to G 1 but sandblasting was performed at 4 bar pressure for 10 seconds.

G 9. Specimens were sandblasted similar to G 1 but sandblasting was performed at 4 bar pressure for 15 seconds.

G 10 (control). Buffered 9.5% HF gel (Porcelain etchant gel, Bisco, Schamburg, IL, USA) (Fig. 1C) was applied to the specimen surfaces for 2 minutes and then rinsed with oil free water and dried with oil free air spray.

G 11. Specimens were first sandblasted as in G 5 and then HF acid was applied as in G 10.

G 12. Specimens were first treated with HF acid gel as in G 10 and then sandblasted as in G 5.

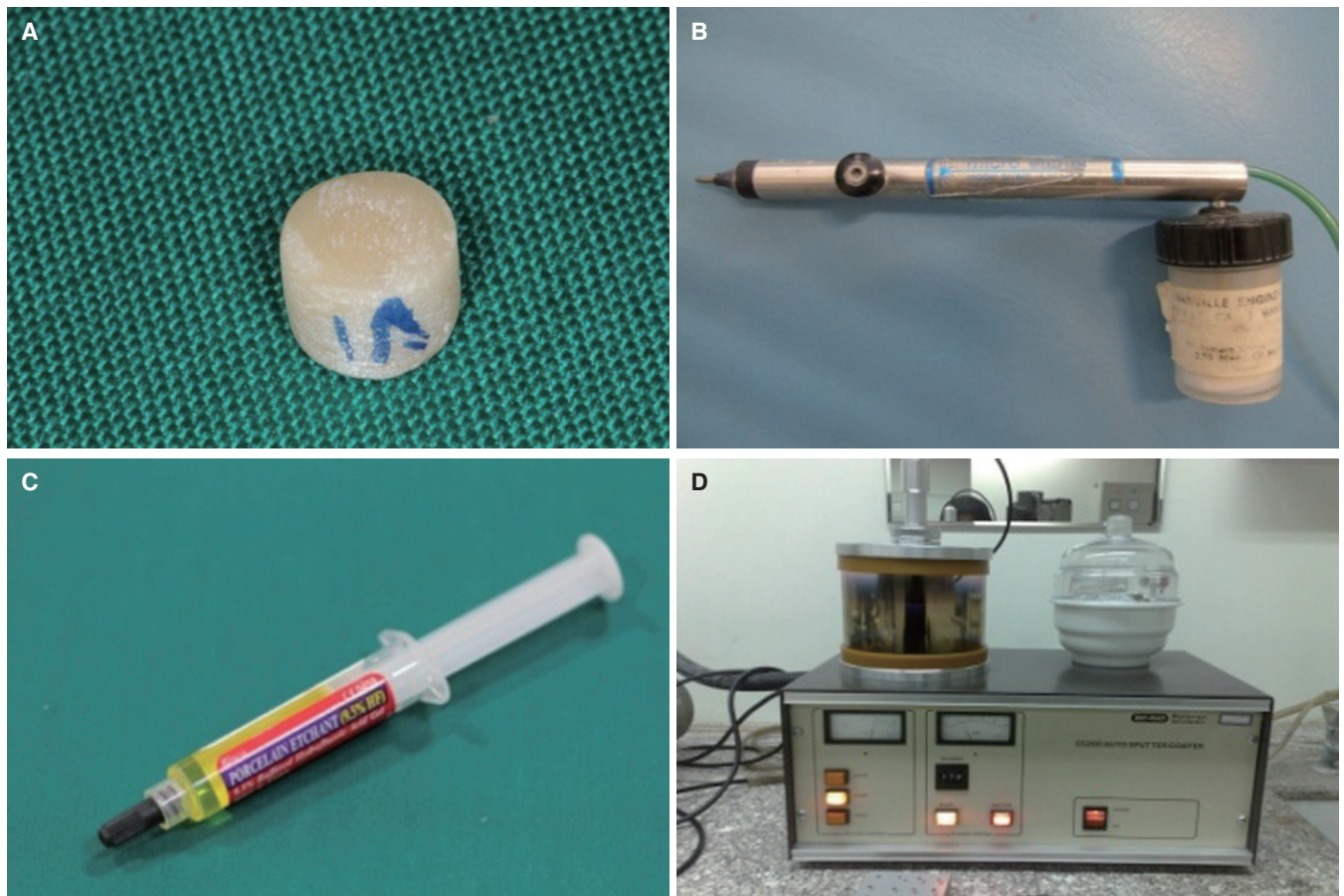
All specimens were then gold-coated by sputter-coater machine (K950X, EMTECH, England) (Fig. 1D) with 15 nm thickness and SEM images were obtained of 5 random areas in each specimen under electron microscopy (VEGA, TESCAN, Czech Republic) at 500 $\times$ , 2,000 $\times$ , 4,000 $\times$  and 10,000 $\times$  magnifications.

## RESULTS

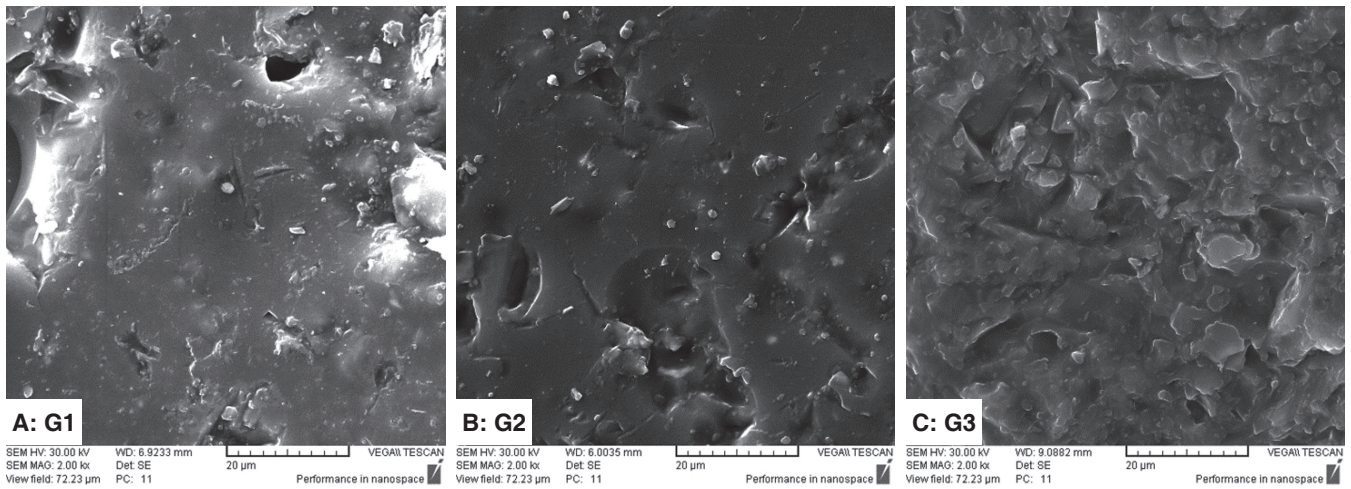
SEM analysis showed that the sandblasted surfaces were porous with steep peaks. Increasing the pressure and duration of sandblasting increased surface roughness. In some SEM images of sandblasted surfaces, tiny alumina chips were evident. By increasing the sandblasting pressure from 2 to 4 bar surface porosities increased as well and a slight increase was observed in the depth of the porosities (Fig. 2, Fig. 3, Fig. 4).

SEM analysis of the HF treated feldspathic porcelain surfaces revealed a porous surface of variable-sized porosities (Fig. 5). SEM showed that 9.5% HF for 2 minutes caused deeper porosities than sandblasting even at 4 bar pressure for 15 seconds. Sandblasted surfaces yielded a rougher microstructure with deep and uniform size porosities and steeper peaks than the surfaces treated with HF acid. Increasing the pressure and duration of sandblasting enhanced uniformity of the size of the porosities.

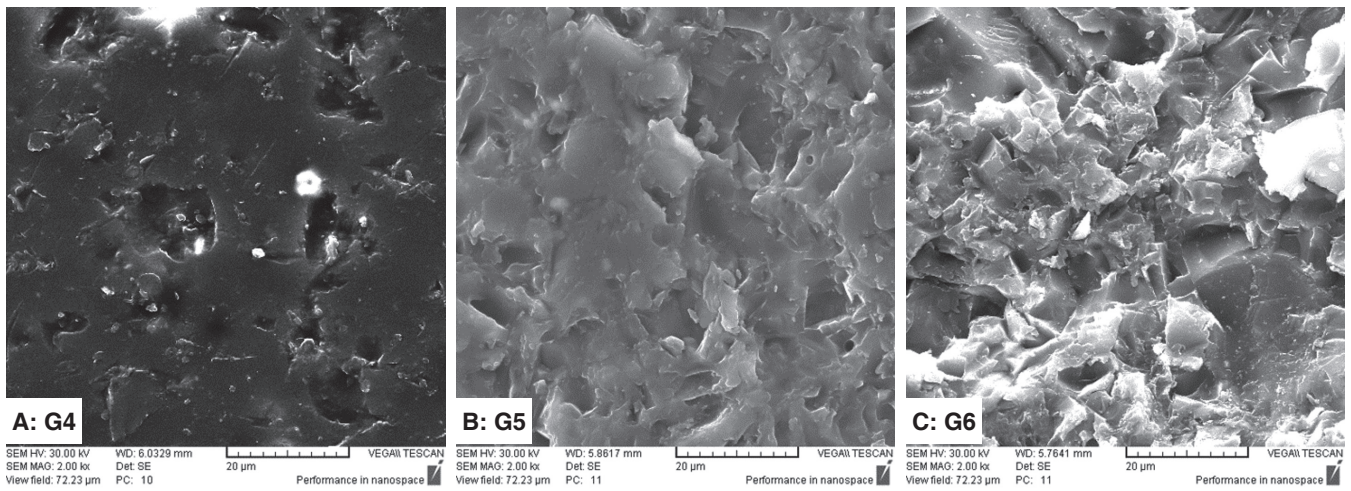
SEM analysis of specimens treated with the combined technique of etching with HF and subsequent sandblasting showed increased surface roughness and porosities (Fig. 6,



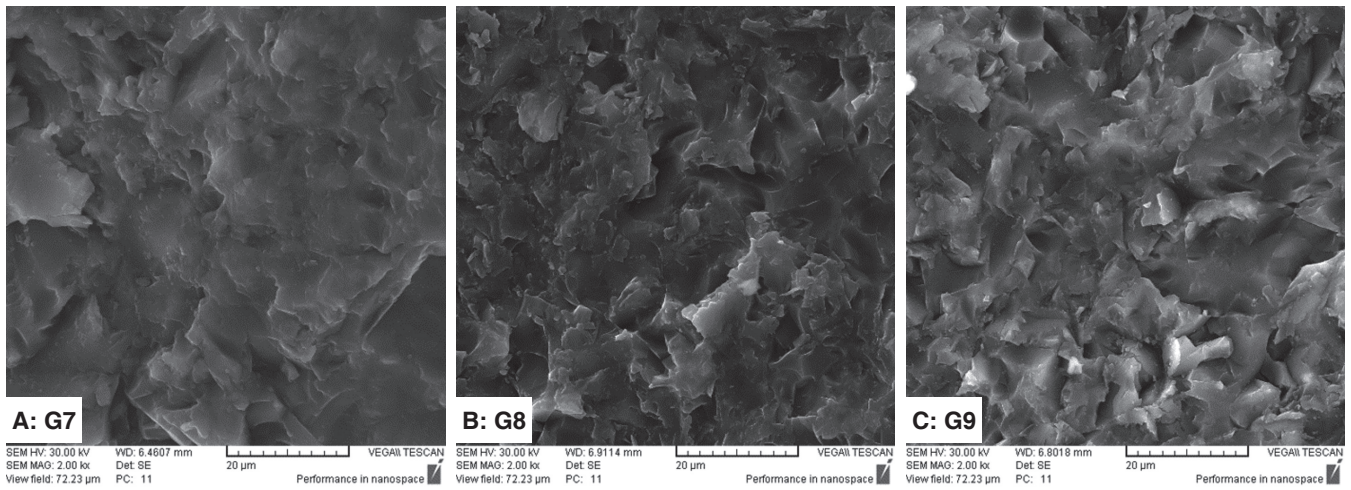
**Fig. 1.** Materials and equipment used. (A) feldspathic porcelain disc measuring 6 x 2 mm, (B) microsandblaster, (C) buffered 9.5% HF gel, (D) sputter-coater machine.



**Fig. 2.** SEM analysis of the feldspathic porcelain surface topography following sandblasting with 50 μm alumina particles from a 5 mm distance at 2 bar pressure for 5 s (A), 10 s (B) and 15 s (C). (2,000X magnification).



**Fig. 3.** SEM analysis of the feldspathic porcelain surface topography following sandblasting with 50 μm alumina particles from a 5 mm distance at 3 bar pressure for 5 s (A), 10 s (B) and 15 s (C). (2,000X magnification).



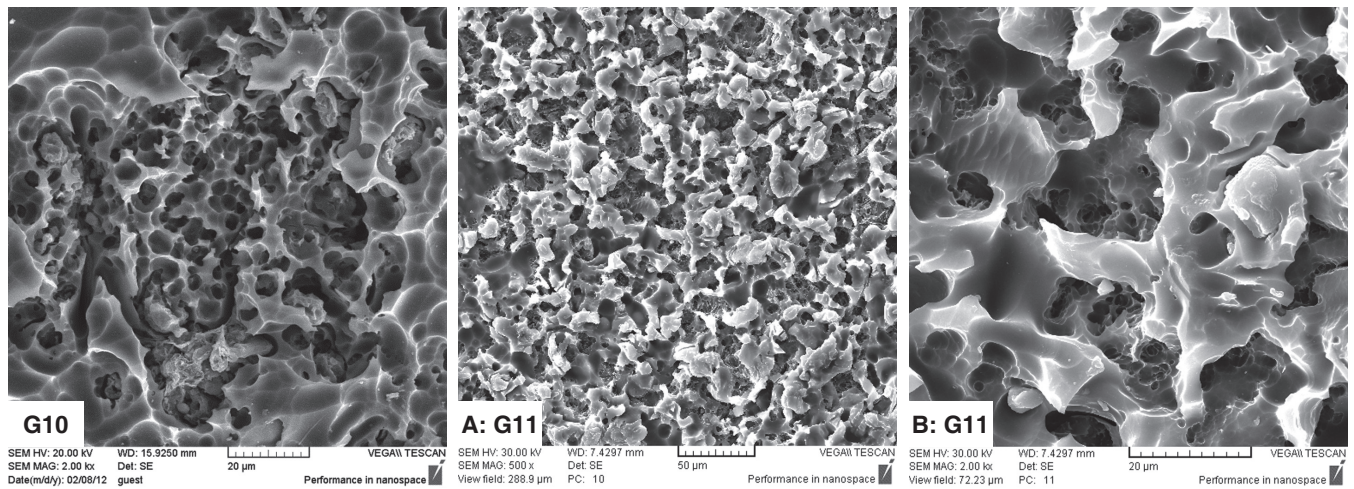
**Fig. 4.** SEM analysis of the feldspathic porcelain surface topography following sandblasting with 50 μm alumina particles from a 5 mm distance at 4 bar pressure for 5 s (A), 10 s (B) and 15 s (C). (2,000X magnification).

Fig. 7). SEM analysis of specimens treated with the combined technique of sandblasting and subsequent treatment with HF acid showed more prominent deep porosities due to HF acid. In the former technique (HF application and subsequent sandblasting), sandblasting removed a greater surface area and provided less uniform porosities compared to the other sandblasted groups.

## DISCUSSION

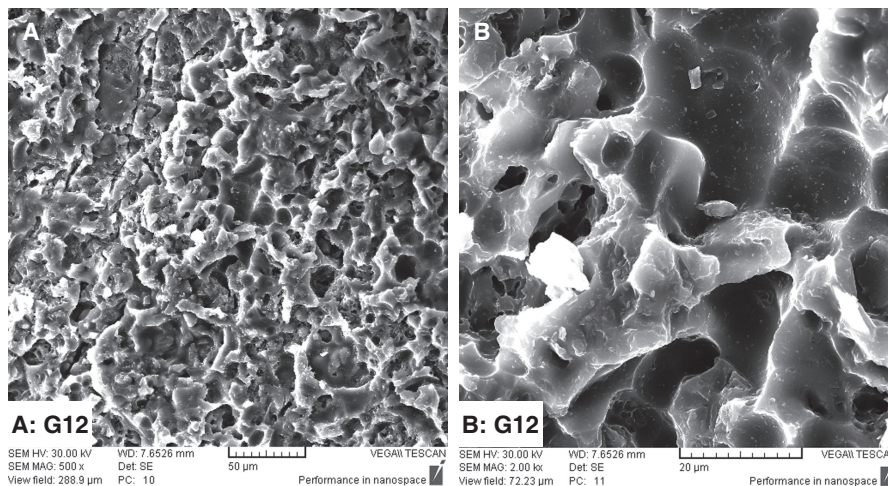
Microscopic analysis can greatly help study the characteristics of materials.<sup>25</sup> Qualitative assessment of micro-mor-

phologic surface changes following different ceramic surface treatments can enhance our understanding of the surface changes affecting the bond strength.<sup>27</sup> Surface treatments roughen the porcelain and enhance the formation of optimal micromechanical bond between the porcelain and resin.<sup>28</sup> Thus, ceramic surface preparation (by etching or sandblasting)<sup>14</sup> is a critical part for clinical success of indirect bonded ceramic restorations, direct ceramic repair procedures<sup>25</sup> and bonding of orthodontic brackets to ceramic surfaces.<sup>13</sup> This study aimed to assess the quality of microstructure obtained by different surface treatments of feldspathic porcelain.



**Fig. 5.** SEM analysis of the feldspathic porcelain surface topography following acid etching with 9.5% HF acid for 120 s. (2,000X magnification).

**Fig. 6.** SEM analysis of the feldspathic porcelain surface topography following sandblasting with 50  $\mu$ m alumina particles at 3 bar pressure for 10 s and then HF acid etching for 120 s; (A): 500X magnification; (B): 2,000X magnification.



**Fig. 7.** SEM analysis of the feldspathic porcelain surface topography following 9.5% HF acid etching for 120 s and then sandblasting with 50  $\mu$ m alumina particles at 3 bar pressure for 10 s from a 5 mm distance; (A): 500X magnification; (B): 2,000X magnification.

SEM images following the application of HF acid to feldspathic porcelain surface (control group) showed small and large micro-porosities as a three-dimensional network of canals and voids, increasing the surface microroughness. These porosities had a tunnel-like micro-retentive pattern resembling a honey-comb pattern. This finding is in accord with the results of studies by Borges *et al.*,<sup>29</sup> Bottino *et al.*<sup>27</sup> and Kukiattrakoon and Thammasitboon.<sup>2</sup> This pattern results from chemical interaction of HF with glass matrix forming insoluble hexafluorosilicate and partially removing the glass matrix. Subsequently, the crystalline structure is exposed and a surface containing tunnel-like undercuts is created.<sup>30</sup> Due to the increased surface energy, the wettability of silane improves as well.<sup>27</sup> Sandblasting is another technique for successful surface treatment of feldspathic porcelain.<sup>31</sup> SEM images of sandblasted surfaces in our study revealed increased surface irregularity and micro-roughness and porosities with sharper peaks but shallower than those of surfaces treated with HF acid alone. Different SEM patterns in sandblasted and HF treated groups are due to the different types of surface treatment<sup>28</sup> because, as stated earlier, HF chemically reacts with the silica phase and penetrates into the depth<sup>30,32</sup> while in sandblasting, the surface is mechanically bombarded by alumina particles under air pressure,<sup>20,33</sup> resulting in removal of weak ceramic phases<sup>25</sup> and subsequent surface irregularity and increased surface area.<sup>25,32</sup> A clean active bonding area is formed as such in the porcelain increasing resin and ceramic wettability and improving the micromechanical interlocking and bond strength.<sup>34</sup>

SEM images revealed that by increasing the pressure and duration of sandblasting, irregularities and surface microhardness increased as well. Moreover, increased pressure slightly increased the depth of impact. Increased micro-roughness is enhanced by the mechanism of action of the microsandblaster device. In a study by Aminsalehi *et al.*, increased time of sandblasting revealed slight increase in feldspathic porcelain surface porosities.<sup>24</sup> SEM analysis in our study demonstrated that surface roughness caused by sandblasting especially at higher pressures and longer time periods was more than that following HF acid application. Other studies have demonstrated greater surface roughness due to sandblasting rather than HF acid etching in glass matrix ceramics using profilometry, SEM and AFM.<sup>15,34,35</sup> However, some researchers like Saraç *et al.*<sup>28</sup> stated that air abrasion caused less surface roughness than application of 9.6% HF gel for 2 minutes. Thus, different sandblasting parameters should be taken into account in such studies. Saraç *et al.*<sup>28</sup> used 25 µm alumina particles at 2.5 bar pressure and a 10 mm distance for 4 seconds. Such sandblasting settings might be responsible for the less surface roughness achieved in their study in comparison to ours.

SEM images in our study showed different micro-morphology and higher surface roughness due to sandblasting compared to HF application. However, some studies found no significant difference in bond strength following sandblasting and HF acid treatment.<sup>13,15,24</sup> Ersu *et al.*<sup>15</sup> stated that

although sandblasting provides a rougher surface, this roughness does not improve bond strength. Results in this regard are controversial and some others have reported higher bond strength following sandblasting compared to HF application.<sup>28</sup>

Considering all the above, a few points should be taken into account: First, aside from surface roughness, there are several other factors that affect the resin-ceramics bond strength.<sup>2</sup> More importantly, although sandblasting increases surface roughness, such surface irregularities are not in the form of retentive undercuts (like in HF-treated surfaces) and bond strength may even decrease in the long-term<sup>2,36</sup> because hydrolytic changes are intensified by aging. This effect is more significant in sandblasted specimens compared to the HF-treated ones because porosities caused by HF application are deeper and thus, resin is better protected from the degradation process.<sup>37</sup> However, application of silane as the promoter of adhesion, in the clinical process of reinforcing the chemical resin-ceramic bond can enhance the clinical success of mechanical surface treatment methods<sup>38</sup>; because silane helps achieve a durable resin-ceramic bond.<sup>38</sup> The bond strength following HF application or sandblasting has been reported to be clinically acceptable.<sup>24,39,40</sup> On the other hand, it should be noted that there is a threshold for surface porosity limiting its effect on bond strength.<sup>2</sup> In some studies like the one by Amin Salehi *et al.*,<sup>24</sup> increasing the sandblasting time of feldspathic porcelain increased surface roughness but could not increase the bond strength. Kern *et al.*,<sup>41</sup> in their study in 2009 reported that by increasing the zirconia sandblasting pressure, surface roughness increased but no increase in bond strength was noted. Thus, in case of presence of adequate bond strength, there is no need to increase surface roughness because porcelain roughness may decrease porcelain restoration strength<sup>28</sup> and even its flexural strength because superficial cracks can lead to failure at lower stress levels.<sup>42</sup> On the other hand, bond strength depends on its indication. For example, for bonding orthodontic brackets, 6 to 8 MPa bond strength is clinically sufficient<sup>43</sup> because porcelain roughness following debonding of orthodontic brackets may increase plaque accumulation, gingival inflammation and soft tissue adverse reactions.<sup>28</sup> Surface roughness can affect the gloss and color of porcelain and lead to staining. Porcelain esthetics can be negatively affected as such.<sup>2</sup> Thus, there is no need for high pressure or long duration of sandblasting either alone or in combination with HF despite increasing the surface roughness because based on previous studies, excessive sandblasting can induce chipping or significant loss of porcelain material.<sup>7,34</sup>

On the other hand, although HF is effective for porcelain surface treatment and creation of micromechanical retention,<sup>2,44</sup> some previous studies show that treatment with HF provides statistically higher bond strength than other types of surface treatments.<sup>2</sup> However, it has an acidic and corrosive nature<sup>4</sup> and can decrease the flexural strength of ceramics.<sup>45</sup> It has the potential to severely traumatize soft tissue and tooth structure.<sup>38</sup> Intraoral applica-

tion of HF acid is not easy and its contact with the tooth is sometimes inevitable.<sup>46</sup> It has been shown that exposure of the dentin surface to HF can cause collapse of collagen fibers and formation of a hybrid layer with 3 µm thickness. Deposition of relatively insoluble CaF<sub>2</sub> on dentin prevents the effect of phosphoric acid on the tooth surface and negatively affects the process of bond formation.<sup>46</sup> HF can also cause soft tissue burns and rash.<sup>2</sup> Its inhalation is toxic<sup>47</sup> and in the long-term it can even adversely affect the nerves.<sup>46</sup> Thus, care must be taken when using HF acid<sup>38</sup> and some studies even suggest that this method not be used for intra-oral repair of restorations.<sup>18,34,47</sup> In our study, SEM images showed that the combined method of HF etching and sandblasting with alumina particles increased the surface roughness and porosities related in both procedures. In some studies, application of HF after sandblasting caused no significant difference in surface roughness compared to sandblasting alone.<sup>28,35</sup> Thus, it seems that sandblasting may be superior to HF only because of the harmful nature and stimulatory effects of HF.<sup>28</sup> On the other hand, in our study, following primary application of HF and then sandblasting for surface preparation of feldspathic porcelain, sandblasting was found to be more aggressive than other methods (based on SEM images) and removed more volume from the porcelain surface. Because HF reacts with silica phase, removes some of porcelain surface and weakens porcelain structure.<sup>30</sup> Since sandblasting impact is on loose phases of ceramic,<sup>25</sup> application of sandblasting following HF etching results in more degradation of surface.

All methods used in this study increased the surface roughness but HF was also able to cause micro-retentive areas in addition to increasing the surface roughness and surface area compared to those of sandblasting. Sandblasting at higher pressure and longer periods caused greater surface roughness. Use of sandblasting following HF etching yielded more aggressive results. Although surface roughness, to some extent, promotes the micromechanical retention, but excessive roughness should be avoided due to adverse effects.

Limitation of this study is the lack of quantitative evaluation of surface texture. Future studies are needed to evaluate other surface treatments such as various lasers and their parameters.

## CONCLUSION

Within the limitations of this study, the following results were obtained:

SEM analysis showed that the porosities caused by HF acid etching were deeper and of various sizes while sandblasting porosities were uniform in size and more homogeneous. The sandblasted surface was rougher with sharper peaks.

Increasing the sandblasting time increased surface roughness. Increasing the pressure increased surface roughness and depth of pores.

Combination of HF etching and sandblasting increased

surface roughness and porosities. More deeper-pores were evidently observed in surfaces that were subjected to sandblasting. In case of primary HF etching, sandblasting removed a greater volume of porcelain surface and the porosities were less uniform in size and shape.

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