ORIGINAL ARTICLE

Evaluation of roughness promoted by different concentrations and exposure times to hydrofluoric acid on ceramic surfaces reinforced by lithium dissilicate

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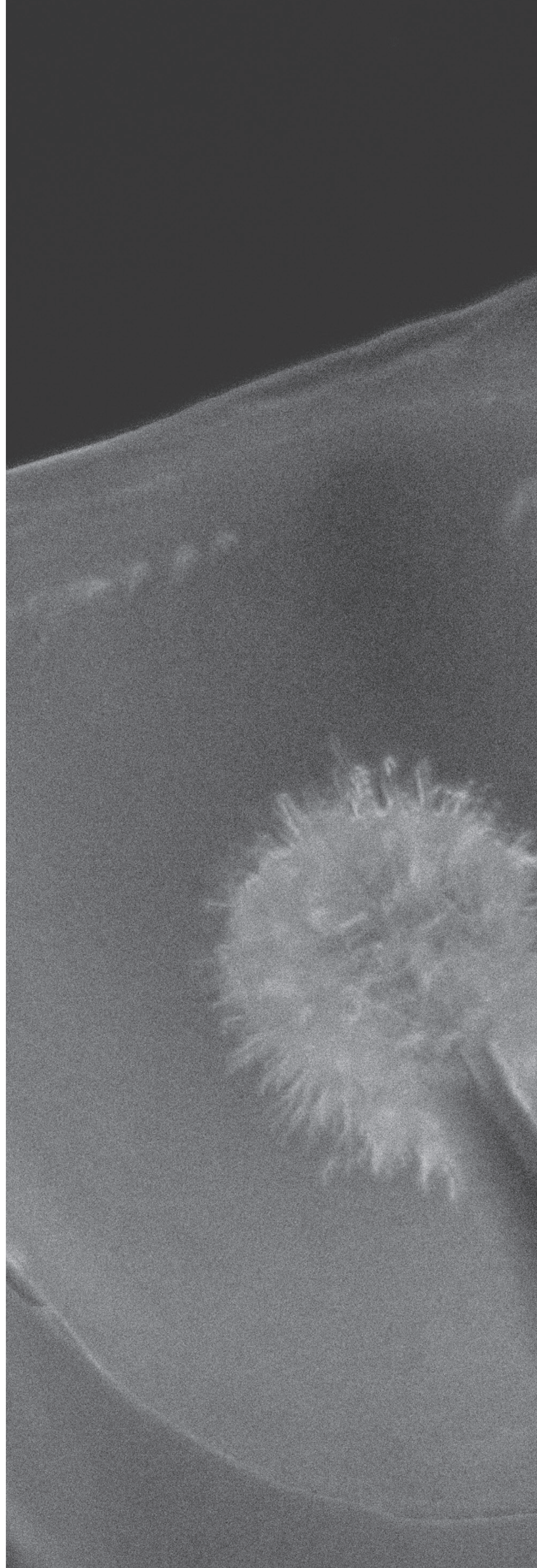

ABSTRACT: Introduction: Lithium disilicate-based ceramics have optical properties that make them stand out, and adhesion to the remaining dental structure is obtained from the sensitivity of this group of material to the action of hydrofluoric acid (AF), which allows the exposure of crystals of lithium disilicate and favors bonding to resin cementing agents. For the AF to be used without prejudice to the structural rigidity of the ceramic or to the quality of adhesion, care with its concentration and exposure time are essential. **Objective:** The study evaluated the influence of hydrofluoric acid in different concentrations and application times on the surface roughness of ceramic based on lithium disilicate. **Methods:** 32 circumferential specimens were made, divided into four groups according to the hydrofluoric acid application protocol, varying the concentration and conditioning time, namely: G1 - 5%, 40s; G2 - 5%, 80s; G3 - 10%, 20s; G4 - 10%, 40s. Surface roughness averages were obtained by three-dimensional profileometric analysis and evaluation of microphotographs by scanning electron microscopy, analyzed statistically, and with generation of images for qualitative analysis. Results: In groups 1, 2 and 4 there was a significant increase in surface roughness after the action of hydrofluoric acid. Three-dimensional images allowed us to infer that the specimens of group 1 showed a greater variation in peaks and valleys. **Conclusion:** It was concluded that the hydrofluoric acid promoted changes in the ceramic surface, being recommended its use for 40 seconds, in a concentration of 5%. **Keywords:** Hydrofluoric Acid; Dental Porcelain; Scanning Electron Microscopy.

INTRODUCTION

The advances in the field of dental ceramics in recent years have been very significant, both in terms of the properties of the material and in the manufacturing techniques. These advances include the introduction of glass-ceramics, which are highly aesthetic and have good mechanical properties,[1,2] which are related, in particular, to the addition of lithium disilicate crystals, which provided increased resistance, durability and improved optical properties compared to conventional ceramics.[3]

Another aspect highlighted in a systematic review by Valenti et al.[4] (2009) was the ability to join glass-ceramic systems to resin cementing agents, whose adhesion to dental tissues is well known, giving longevity to restorative treatment. For this union to take place, it is necessary to establish adequate adhesion between substrate and adherent.[5]

The adhesion process of these acid-sensitive ceramics to resinous materials, consolidated in the literature, is provided by the conditioning of their surface by hydrofluoric acid (HF).[6,7] Thus, the suggested protocol for joining the glass ceramic to the resin is the HF attack followed by the application of a silane coupling agent (chemical and micromechanical bonding).[8,9]

HF is considered an efficient surface modification agent, capable of dissolving the vitreous matrix and exposing lithium disilicate crystals,[10,11,12,13,14,15] with increased roughness and wettability, allowing for mechanical and chemical reactivity and favoring a long-term bond between resin cementing agents and lithium disilicate.[16,17]

In vitro studies[13,18,19] reported that conditioning with hydrofluoric acid has a positive effect on the surface topography, with increased roughness and removal and / or stabilization of surface defects.

Despite research[13,20] showing an increase in bond strength of resin cementing agents from conditioning with HF prior to cementation, Addison et al[21] (2007) observed that this conditioning could result in weakening of the ceramic due to changes that may occur on the surface depending on the conditioning time and concentration.

Bearing in mind that the adhesive procedure is dependent on the surface treatment, with interference in the prognosis of an aesthetic restoration, the objective of this research is to evaluate, the influence of the concentration and the time of exposure of the lithium disilicate ceramic surface to the action of the HF.

In the literature, several combinations have been reported for the conditioning and concentration periods of the acid, with influences on the level of surface roughness and strength.[20] As the different ceramics can be more or less sensitive, there is still controversy regarding the ideal concentration and time of exposure in order to achieve greater union with resin cementing agents and avoid possible deleterious effects.

MATERIALS AND METHODS

Selection and Preparation of Specimens

The material of choice was an IPS e.max Press lithium disilicate ceramic, color HT A2, manufactured by Ivoclar Vivadent® (Figure 1), with which 32 (thirty-two) circumferential 12 mm diameter and 2mm thick.

To achieve this, the ceramic was sintered at 910°C and injected for 90 seconds in a previously developed mold, generating specimens (CP) suitable for analysis, without cracks or amendments.

Following manufacturer's guidelines[22], the PCs were submerged in 1% AF, kept in an ultrasonic vat for 15 minutes, washed for 1 minute, dried for 30 seconds, and then sandblasted with 30psi particles of aluminum oxide (Al203).

To achieve standards that would generate sufficient stabilization, the samples were fixed to a circular base of colorless, self-curing acrylic resin through the sprue.

Experimental Design

The specimens were randomly divided into four experimental groups (n = 8; G1 to G4), in which the HF concentration varied (5% and 10%; BM4, Brazil) and the exposure time (20s, 40s, 80s), as shown in Table 1.

Table 1: Experimental Groups

GROUP	CONCENTRATION	EXPOSURE TIME
G1	5%	40s
G2	5%	80s
G3	10%	20s
G4	10%	40s

Distribution of specimens in 4 experimental groups, with variation in concentration and time of exposure of the ceramic surface to the action of HF.

Three-dimensional profilometry

Specimens from each experimental group, without surface conditioning, were subjected to three-dimensional profileometric analysis without contact, in order to determine the surface roughness (Nanovea PS50 Optical, NANOVEA®, Irvine, USA).

The assessment area of 1 mm^2 in the CP was standardized. The measurements were captured using a chromatic confocal sensor using an axial source of white light, at a scanning speed of 2mm / s. The means for three linear measurements of surface roughness (Ra) were obtained for each sample. The average structural loss corresponded to the size of the gap between the experimental areas (initial erosion and treatment phase).

After the analysis, the PCs were subjected to conditioning with HF following the concentration and exposure time of each experimental group, followed by cleaning with running water and drying with an air jet, and, subsequently, a new profile measurement..

Scanning Electron Microscopy

Each sample was placed under metallic stubs with the aid of carbon tape and the samples were metallized with a thin layer of gold-palladium alloy under high vacuum (Balzers-sputer coater, Germany) and taken for observation in a scanning electron microscope (SEM) (EVO 10, Carl Zeiss AG, Germany), in increments of 500 times and 5000 times. To assess the ceramic surface, random samples representative of each group were selected for qualitative analysis of the surface morphology.

Statistical analysis

Descriptive (mean, standard deviation, median) and exploratory analyzes of the roughness data were performed. Exploratory analyzes indicated that the data did not meet the assumptions of an analysis of variance (ANOVA) and were analyzed using generalized linear models, considering the design of repeated measures over time. The analyzes were performed in the R program (Foundation for Statistical Computing, Austria), with a significance level of 5%.

RESULTS

The results of the surface roughness (Ra) of the IPS e.max Press ceramic in the different concentrations and conditioning times with AF are shown in Table 2 and Figures 1 and 2.

Prior to conditioning with HF, groups 2 (G2; 5% AF, 80s) and 3 (G3; 10%, 20s), exhibited significantly higher Ra levels than the other groups ($p < 0.05$), followed by if observed in G4 (AF 10%, 40s).

After surface conditioning, G2 (5% AF, 80s) exhibited higher levels of Ra, with a statistically significant difference in relation to the others, followed by those observed in G3 (10% AF, 20s) and G4 (10% AF, 40s).

When comparing the averages previously displayed and after surface treatment, it was possible to verify that G3 (AF 10%, 20s) exhibited similar behavior in the two evaluations ($p > 0.05$), which was not the case in the other groups, which had higher Ra levels after PA action ($p < 0.05$).

Table 2: Means and standard deviations, median (minimum; maximum), observed in each experimental group, before and after conditioning with HF.

Hydrofluoric acid	TIME			
	Before		After	
	Mean (standard deviation)	Median (minimum; maximum)	Mean (standard deviation)	Median (minimum; maximum)
5% per 40s	1,29 (0,12) [Bc]	1,23 (1,18 - 1,51)	1,83 (0,30) [Ac]	1,83 (1,46 - 2,34)
5% per 80s	2,54 (0,32) [Ba]	2,61 (1,92 - 2,94)	3,76 (0,42) [Aa]	3,76 (3,14 - 4,52)
10% per 20s	2,78 (0,48) [Aa]	2,88 (1,72 - 3,28)	2,72 (0,08) [Ab]	2,70 (2,69 - 2,91)
10% per 40s	1,78 (0,19) [Bb]	1,77 (1,58 - 2,07)	2,69 (0,21) [Ab]	2,63 (2,46 - 3,06)

Different letters (uppercase in the horizontal and lowercase in the vertical) differ from each other ($p < 0.05$).

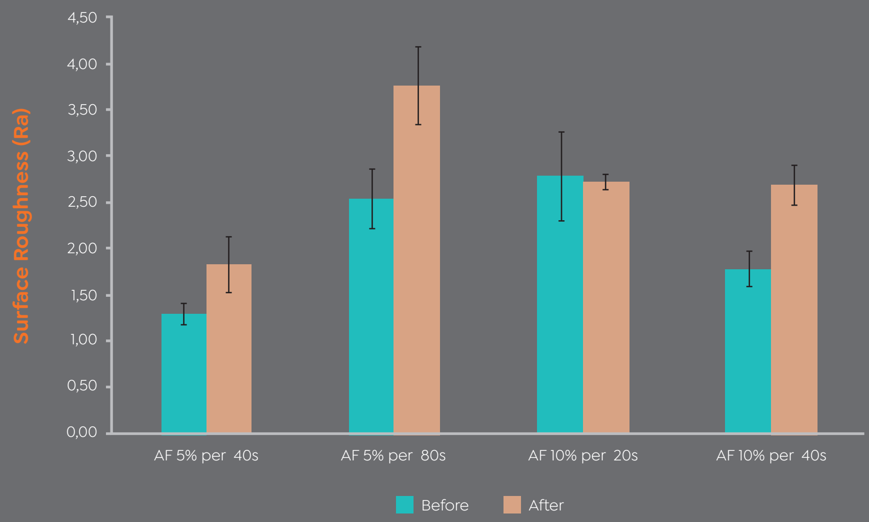

Figure 1: Means and standard deviations of surface roughness (Ra) as a function of groups and time.

Figure 2: *Boxplot* of surface roughness (Ra) as a function of groups and time.

Analysis of Surface Roughness by 3D Digital Profilometry

Three-dimensional profiling reconstructed the surface images before and after conditioning with HF. It can be noticed that the Ra levels increase proportionally with the conditioning time, making the discrepancy between peaks (red / pink tones) and valleys (blue tones) bigger and more disorganized. Intermediate tones show less discrepancy between peaks and valleys.

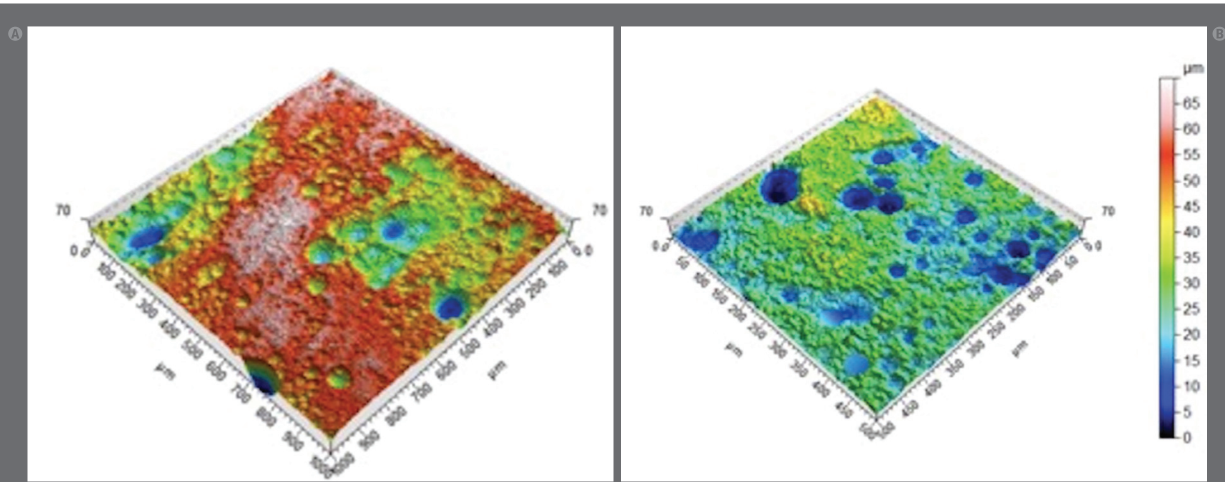

Figure 3: Three-dimensional profileometric analysis of G1 (5%, 40s) before (**A**) and after (**B**) conditioning with HF.

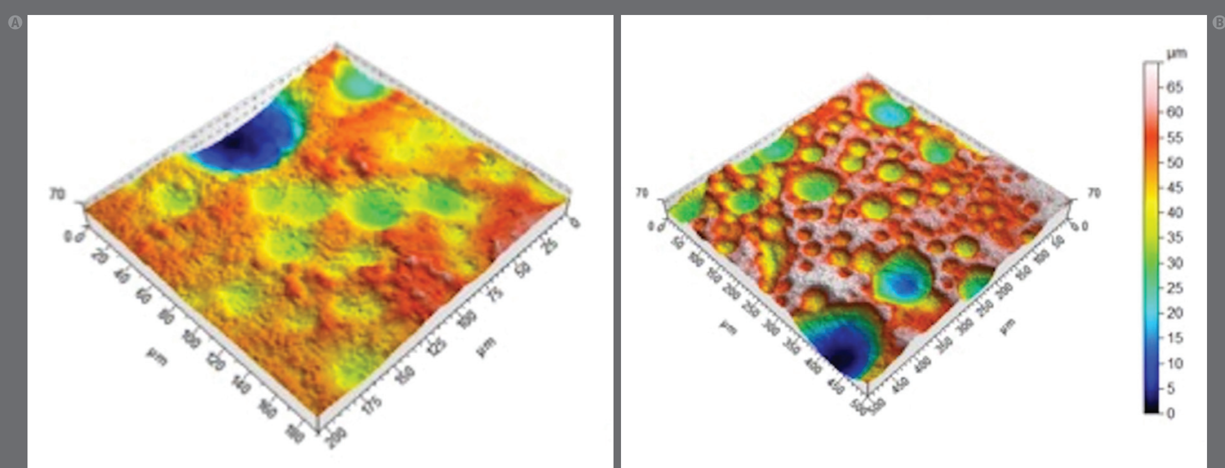

Figure 4: Three-dimensional profileometric analysis of G2 (5%, 80s) before (**A**) and after (**B**) conditioning with HF.

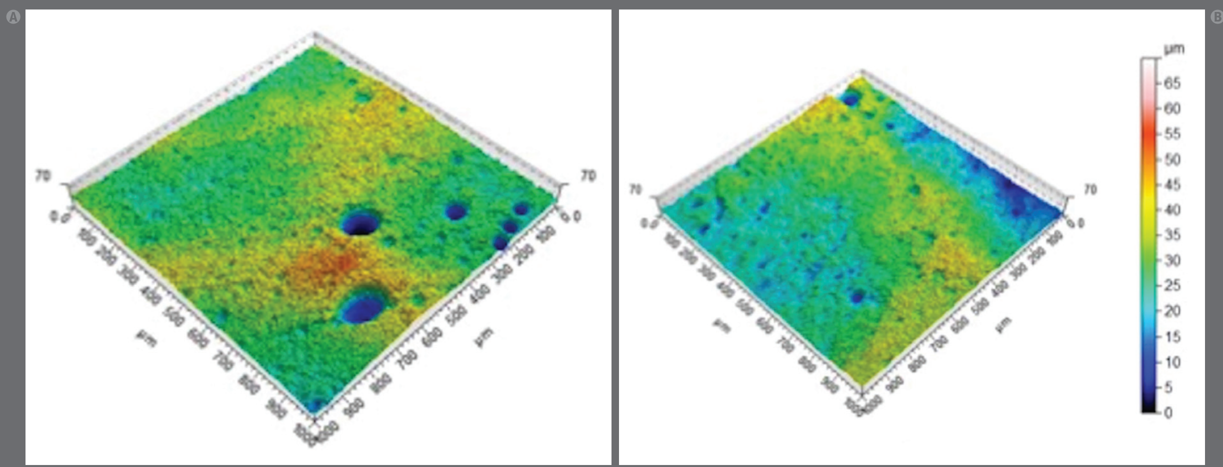

Figure 5: Three-dimensional profileometric analysis of G3 (10%, 20s) before (**A**) and after (**B**) conditioning with HF.

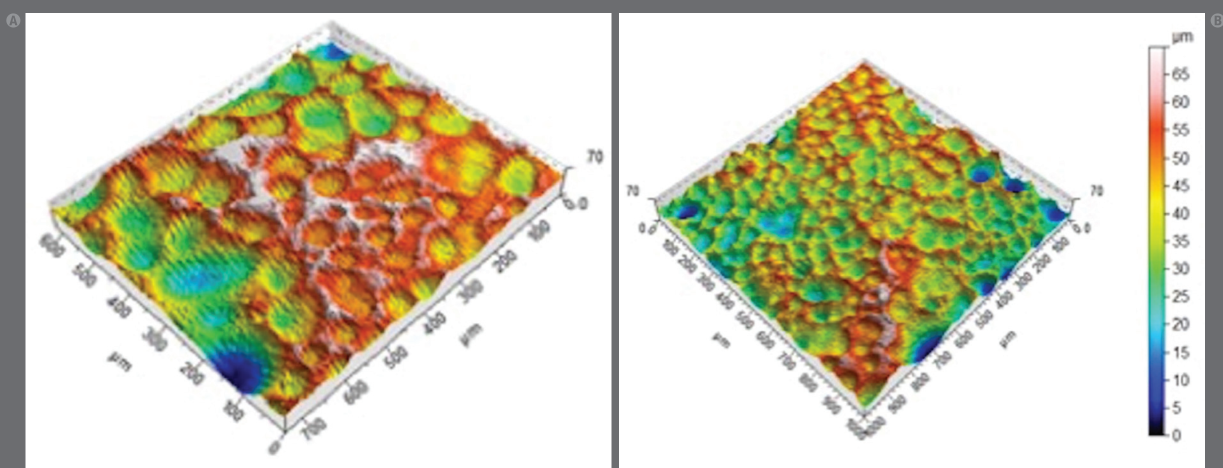

Figure 6: Three-dimensional profileometric analysis of G4 (10%, 40s) before (**A**) and after (**B**) conditioning with HF.

Analysis of Surface Roughness by Scanning Electron Microscopy

Analyzed the ceramic surfaces of lithium disilicate observed in SEM, they showed valuable information about the topography. A sample was randomly selected (in the same way adopted for the distribution of the groups), to characterize the resulting recording pattern.

The images resulting in an increase of 5000x, after conditioning with HF, show an increase in the degree of dissolution of the vitreous matrix and exposure of the lithium disilicate crystals with increasing concentrations and time in contact with the ceramics. Figure 8A, referring to group 1 (5% for 40s), shows a slight dissolution of the vitreous matrix, with a more regular surface. The figure equivalent to group 2 (5% for 80s) (8B), elucidates an increase in the degree of dissolution of the vitreous matrix, while 10% for 20s (8C), shows more evident patterns of these crystals.

In group 4 (10% for 40s), presenting topographic changes such as “craters” and microdefects, as shown in Figure 8D.

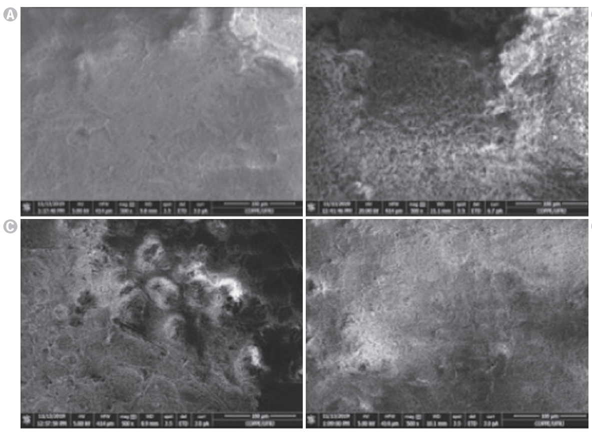

Figure 7: Images resulting from etching with hydrofluoric acid on the ceramic surface (IPS e.max Press), showing irregularities in the exposure of the vitreous matrix, for concentrations 5% / 40s, 5% / 80s, 10% / 20s, 10% / 40s (A, B, C, D, respectively), at 500x increase in SEM.

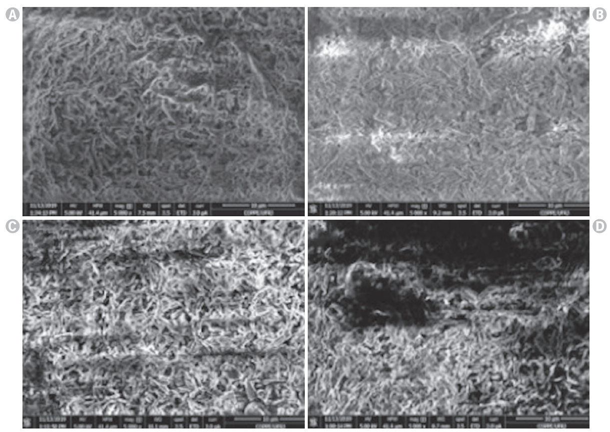

Figure 8: Images resulting from conditioning with hydrofluoric acid on the ceramic surface (IPS e.max Press), showing irregularities in the exposure of the vitreous matrix, for concentrations 5% / 40s, 5% / 80s, 10% / 20s, 10% / 40s (A, B, C, D, respectively), at 5000x increase in SEM.

DISCUSSION

It is undeniable the increase, in recent years, of the indications for restorative treatment using dental ceramics reinforced by lithium disilicate, which was due to the aesthetic and mechanical advances observed in this group of material.[23,24]

Adhesion to the remaining dental structure is obtained from the sensitivity of this group to the action of hydrofluoric acid (AF), which allows the exposure of lithium disilicate crystals and favors the bonding to resin cementing agents.[16,25]

For the AF to be used without prejudice to the structural stiffness of the ceramic or to the quality of adhesion, attention to its concentration and exposure time is essential,[25] however an ideal protocol is still not well understood in the literature, especially for this new technology IPS e.max Press.[25]

In this research, we sought to analyze the laboratory behavior of these two variables, through the surface roughness through three-dimensional profilometry[26] and scanning electron microscopy.[28]

First, the surface roughness (Ra) of the four experimental groups without HF action (initial averages) was analyzed in order to obtain a parameter for comparison after conditioning (final averages).[3]

Statistical analysis of the initial means, before the HF action, shown in Table 2, showed differences between the groups, except for the comparison between groups 2 and 3, which can be credited to the random distribution of the samples.

When analyzing the behavior of ceramic surfaces after the action of HF at different concentrations and exposure times, taking as a basis the surface roughness before conditioning, differences between the groups were identified, except for group 3 (PA 10%, 20s), which showed no statistically significant difference.

When comparing group 1 (AF 5%, 40s) with group 2 (AF 5%, 80s), it was observed that with the increase in the time of exposure to acid there was also an increase in surface roughness, and, along with this, characterized erosion sites, Figures 4 (B) and 7 (B), which was also observed in research by Zogheib et al.[3] (2011) and Veríssimo et al.[25] (2019).

In this study, it was found that the action of AF at a concentration of 5% for 40s was sufficient to promote dissolution of the vitreous matrix, corroborating with other authors,[13,15,17] as illustrated in Figures 3, 7 (A) and 8 (A).

Statistical comparisons between the final means of group 1 to groups 3 and 4 also showed a significant difference ($p < 0.05$), with higher levels of surface roughness in the groups that received 10% HF treatment (groups 3 and 4). Likewise, there was a significantly higher Ra in group 2 compared to the others.

The images of profile analysis and scanning electron microscopy confirmed higher levels of roughness in group 2 compared to the other groups evaluated, with more irregular peaks and valleys (Figures 4, 7B and 7B).

Prochnow et al.[15] (2017) concluded, in in vitro research, that less irregular ceramic surfaces tend to avoid brittleness, promoting micro retention of the resinous cementing agent by overlapping the treated surface.

Naves et al.[29] (2010), Zogheib et al.[3] (2011) developed research to evaluate the influence of the variation in the conditioning times of the ceramic surface by the HF, having concluded that the increase in the exposure time generated, proportionally, more irregular and with less adhesive resistance. Based on this finding, it is believed that by the groups tested in this study, group 1 is the best way to condition lithium disilicate-reinforced porcelain, even because the manufacturer of the AF 5% (BM4), prescribes a time limit as a recommendation average of 30 seconds.[30]

From this point of view, it is reasonable to assume that more adequate clinical results will be related to surfaces with less irregular valleys and peaks, such as those observed when used for 40s at a concentration of 5%. Thus, it can still be considered that the results presented by group 2 are less satisfactory, allowing us to understand that the increase in the exposure time was unfavorable.

Groups 3 and 4 received conditioning with 10% HF at different exposure times. It should be noted that the manufacturer of the used HF (BM4)[30] advises that this concentration should not be used in lithium disilicate ceramics, restricting it to feldspar and vitreous ceramics reinforced with leucite. The results obtained confirmed the orientation of the industry by demonstrating that these groups presented surfaces with higher levels of roughness than those observed in group 1 and, therefore, less convenient.

In the final analysis, among the groups evaluated, considering the concentration and the time of exposure to HF, it is feasible to deduce that the most indicated would be to perform conditioning with hydrofluoric acid at a concentration of 5% for 40s, where there is less clinical application time. and less risk of ceramic fragility.

CONCLUSION

Based on the parameters analyzed in this research, it is possible to infer that, hydrofluoric acid is able to change the surface of the lithium disilicate ceramic, generating changes in the surface roughness levels. The action of hydrofluoric acid for 40 seconds, in a concentration of 5%, produced ceramic surface with less irregularities in relation to the other groups analyzed, promoting less risk of fragility to ceramic.

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