

# PHYSICOCHEMICAL PROPERTIES OF ENAMEL AFTER MICROABRASION TECHNIQUE

## **ABSTRACT**

AIM: This study aimed to evaluate the enamel microhardness, surface roughness, and chemical composition after microabrasion technique, followed by polishing and different immersion times in artificial saliva. MATERIALS AND METHODS: Ninety enamel blocks (25 mm2) from bovine incisors were divided into two groups (G1 and G2), and then subdivided in five subgroups (n = 9)according to their microabrasion treatment and polishing with a diamond paste: 35% phosphoric acid and pumice (H3PO4+Pum) and polishing; just H3PO4+Pum; 6.6% hydrochloric acid and silica (HCl+Sil) and polishing; just HCl+Sil; and control (no treatment). For G1, roughness and microhardness analyses were performed before (L1) and after (L2) microabrasion. After 15 (L3) and 30 (L4) days of immersion in artificial saliva, microhardness analysis was also performed. After (L4) analysis, the specimens were subjected to SEM analysis. G2 was used for the chemical analysis using energy dispersion testing (EDS). The data was subjected to statistical analysis ( $\alpha = 5\%$ ). For roughness, L2 presented higher values than L1, except for the polished groups. For microhardness, L2 presented higher values than L1, except for unpolished groups. The L3 and L4 did not differ and were higher than L1. RESULTS: No changes were observed in the phosphorus concentrations of the microabrasioned enamel. However, the specimens treated with HCl+Sil were observed as having decreased calcium and increased chlorine and silica on the EDS test. CONCLUSION: Microabrasion followed by polishing and immersion in artificial saliva for 15 days is enough to increase the microhardness of microabrasioned enamel surfaces, although the microabrasion procedure can alter the mineral content of the enamel.

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**KEYWORDS** 

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## **INTRODUCTION**

One of the most challenging aspects of dentistry involves the esthetic resolution of most of the spots on the dental enamel, which can have different colors that affect the esthetics. Such spots may have different depths and ranges of extension with or without surface irregularities, and they can be intrinsic or extrinsic, depending of the etiology.<sup>1-3</sup>

These enamel stains can be treated with the microabrasion technique that selectively abrades the discolored areas, giving them a healthy clinical aspect compared with the dilapidated enamel. Initially, the microabrasion technique consisted of the application of acids to the surface of the spotted enamel.<sup>4,5</sup> However, advances in technology and abrasive agents have resulted in a significant improvement of this technique's effectiveness. This technique is used for the esthetic treatment of fluorosis white spots, 1-4 inactive white spots by demineralization, orthodontic post-treatment, hypoplasia arising from dental trauma or infection, and idiopathic hypoplasia in which the discoloration is limited exclusively to the most superficial layer of the dental enamel. 1,6,7

Numerous studies have evaluated the effects of microabrasion, and most have been performed through microscope evaluations and clinical cases.<sup>1,8-12</sup> Despite the benefits of using this technique to remove spots on superficial enamel,<sup>2,4</sup> little is known about its

remaining effects on enamel surface<sup>12-14</sup> and its interaction with saliva.<sup>15</sup> Although microabrasion and commercially available products are widely used clinically, only limited data is available about their effects on teeth,<sup>16</sup> being important to perform newer studies.

Based on this data, and given the lack of studies assessing the effects of abrasion on enamel's structure and morphology, a method of chemical analysis was applied in this work that produced effective results for the dental structure. This method is called chemical analysis by energy dispersion (EDS) and is used to quantify the amount of calcium (Ca) and phosphorus (P) by a semi-quantitative method based on measuring the intensity of characteristic x-rays emitted by elements in the sample. 17 The intensity of the energy emitted by the components is related to the concentration of each element in the sample. Only in past 10 years has the analysis by x-ray fluorescence been applied in biological tissues.<sup>18</sup>

Thus, the purpose of this study was to analyze the physicochemical properties of enamel after microabrasion treatments with phosphoric acid and pumice or with hydrochloric acid and silica, evaluating the effects of the polishing technique and artificial saliva on the microabrasioned enamel.

## **MATERIALS AND METHODS**

Ninety bovine incisive teeth were cleaned and disinfected with thymol (Dinâmica; Piracicaba, SP, Brazil) and submitted to an initial polishing with pumice (SS White Ltda; Rio de Janeiro, RJ, Brazil) and water. After separating the coronary portion by means of a double-face diamond disc (KG Sorensen Ind. Com. Ltda.; Barueri, SP, Brazil), 25 mm<sup>2</sup> enamel blocks were obtained using a precision saw (Isomet 1000, Buehler; Lake Bluff, IL, USA) and a diamond disc (4"  $\times$  012  $\times$ ½, Buehler). The fragments were planed using silicon carbide abrasive paper of decreasing granulation (400-, 600-, and 1200-grit) and surface polished with felt (TOP, RAM, and SUPRA) (Arotec; Cotia, SP, Brazil) along with a diamond paste of decreasing granulation (1 and ¼ µm), greased with specific oil (Arotec). The specimens were washed for 15 min. in an ultrasonic washer (Marconi Equipamentos para Laboratórios; Piracicaba, SP, Brazil) to remove rubbish on the enamel surface.<sup>19</sup> The specimens were stored in distilled water at 37°C until the experiment began.

Initially, the specimens were divided into two groups with 45 specimens each (G1 and G2) and five subgroups (n = 9): phosphoric acid (35%) and pumice ( $H_3PO_4+Pum$ ) and polishing; just  $H_3PO_4+Pum$ ; hydrochloric acid (6.6%) and silica (HCl+Sil) and polishing; just HCl+Sil; control (no treatment).

The specimens for G1 were submitted to surface roughness (Ra) and Knoop microhardness (KHN) analyses. Both tests were performed in 2 time frames: before (L1) microabrasion treatment and after (L2) microabrasion with or without polishing step. KHN was also evaluated after 15 (L3) and 30 (L4) days of immersion in artificial saliva. For the roughness test, three readings of roughness were made using a roughness tester (Surftest 211, Mitutoyo; São Paulo, SP, Brazil). For the microhardness test, five indentations were made on center of the enamel surface using a Knoop indenter with a static load of 25g for five seconds using a microhardness tester (HMV-2000, Shimadzu; Tokyo, Japan). The final results of the roughness and microhardness readings were the mean of the values obtained.

After the initial readings, microabrasion proceedings were initiated, with equal parts of 35% phosphoric acid (Ultra-Etch, Ultradent Products Inc.; South Jordan, UT, USA) and pumice (SS White Ltda; Rio de Janeiro, RJ, Brazil) H<sub>3</sub>PO<sub>4</sub>+Pum; or with 6.6% hydrochloric acid and silica (Opalustre, Ultradent Products Inc.) HCl+Sil, both measured with a dosage spoon (0.184 g), and a portion of the agent was applied to each specimen. The enamel microabrasion was performed using a lowrotation micro-motor (LB-2000, Beltec Indústria e Comércio de Equipamentos Odontológicos Ltda; Araraquara, SP, Brazil),

with standardized rotation about 13,000 rpm. The same application technique was used for all groups and each specimen at 10 applications of microabrasive product for 10 s. <sup>14,15</sup> After each application of the abrasive agent, the specimens were washed with flow water and placed into an ultrasonic washer (Marconi Equipamentos para Laboratórios) for 15 min.

The polishing procedure for designed groups was performed using a diamond paste for a composite finish (Diamond Excell, FGM Produtos Odontológicos Ltda; Joinville, SC, Brazil) with felt discs (FGM Produtos Odontológicos Ltda). The polishing was performed using the same low-rotation micromotor used for microabrasion for a period of two minutes with intervals of 30 s so there was no heating of the specimen. Next, the specimens were washed for 15 min. in an ultrasonic washer (Marconi Equipamentos para Laboratórios) to remove any rubbish from polishing.

The specimens were stored in artificial saliva (Proderma; Piracicaba, SP, Brazil) for 15 and 30 days. The components of artificial saliva are shown in Table 1. After roughness and microhardness tests, representative specimens of each group were dehydrated, placed in a metal stub under aluminum tape, subjected to a vacuum in a sputter (Balzers-SCD 050 sputter coater, Germany), formed into a plasma, and then a thin layer gold was

deposited on each specimen. Then, the stubs containing the specimens were placed under a scanning electron microscope (JSM 5600LV, JEOL; Tokyo, Japan) to obtain photomicrographs of the representative areas of each specimen studied with increases of 3700x.

To determine the chemical composition of the enamel, specimens of G2 were dehydrated, placed in a metal stub under aluminum tape, subjected to vacuum in a sputter coater (Balzers-SCD 050), formed into plasma, and a thin layer of vaporized carbon (Carbon Yarn Part. No. YRN001-0001, Denton Vacuum; Moorestown, NJ, USA) was deposited on each specimen. Increases of 75x were made to obtain areas to calculate the elements' rates (calcium, phosphorus, chlorine, and silica).

The data were submitted to analysis of variance (ANOVA) for repeated measures and Tukey's test. The control group was compared with the other groups by the Dunnett's test. The significance level was of 5%. However, the chlorine and silica data did not meet the assumptions of parametric analysis and were analyzed by a Mann Whitney test at 0.05 of significance level.

# **RESULTS**

# **ROUGHNESS:**

The abrasives agents tested did not differ significantly in all of the situations tested (p = 0.5847). There was a significant difference

after polishing (p  $\leq$  0.05). The reductions on the roughness (Table 2) were obtained exclusively with a diamond paste after microabrasion.

# MICROHARDNESS:

\_\_\_\_\_The abrasives products evaluated did not differ significantly in all of the situations tested (p = 0.5847). There was a significant difference after polishing, which was observed in the highest hardness values (p  $\leq$  0.05). However, there was not a significant difference after the different times of immersion in artificial saliva (Table 3).

## **ENERGY DISPERSION:**

The results for calcium and phosphorus are shown in Table 4 and chlorine and silica are presented in Table 5.

For phosphorus, the microabrasives tested did not differ significantly in all of the situations tested (p = 0.4414), and the polishing (p = 0.8681), and group x polishing interaction was not significant (p = 0.9607). The groups did not differ from the control (p > 0.05). For calcium, none abrasive evaluated differed significantly in all of the situations tested (p = 0.4414), there was no difference between the polishing (p = 0.6021), and group x polishing interaction was not significant (p = 0.4545). The HCl+Sil group presented lower means than the control with or without polishing (p  $\leq$  0.05). The groups treated with

Opalustre presented chlorine and silica content  $(p \le 0.05)$ .

SCANNING ELECTRON MICROSCOPY (SEM ANALYSIS):

SEM showed that the application of a microabrasive generated the formation of morphological patterns distinct and different from control group for tested abrasive products. The polishing procedures were efficient in reestablishing the characteristics of the enamel (Figures 1–5).

#### DISCUSSION

The microabrasion technique consists in applying a compound that combines the chemical action of an acid (in this study, 35% phosphoric or 6.6% hydrochloric acid) and an abrasive action (in this study, pumice or silica microparticles). The procedure has proven efficacy as an alternative technique for the esthetic resolution of surface stains. However, it's possible effects on the structure of enamel, such as microhardness, roughness, and chemical changes have not been extensively studied nor have the ways to alleviate these effects.

The enamel surface structure is composed of hydroxyapatite, and each crystal of  $Ca_2(PO_4)6(OH)_2$  is surrounded by a layer of tightly bound water. The presence of this hydration shell shows that the crystal is electrically charged and can attract ions that

play a part in remineralization.<sup>19</sup> As a result, it is apparent that, despite the external appearances, enamel is porous and ion migration is possible.<sup>20</sup> The acid used in microabrasion procedures can penetrate the

enamel and displace ions, and this can cause an increase in porosity levels, thereby facilitating acid transport and demineralization.<sup>21</sup>

Table 1. Artificial saliva's composition.

	Material	Manufacturer	Lot #	Composition				
F	Artificial Saliva	Proderma (Piracicaba, SP, a 10 Brazil)	SP, 100807	Sodium	c h l o r i d e	(0.674	g),	magnesium
				chloride hexahydrate (0.041g), calcium chloride di-hydrate (0.274				
				g), methylparaben (1.500 g), propilparaben (0.200 g), saccharin (1				
				g), distilled w	ater (1000 mL)	, pH 6.9		

Table 2. Surface roughness (Ra) means followed by the standard deviation of the experimental groups.

Croun	Time	Polishing	Polishing			
Group	Time	Polished	Unpolished			
II. DO . Dum	L1	0.20 (0.04) Aa	0.20 (0.03) Ab			
H <sub>3</sub> PO <sub>4</sub> +Pum	L2	0.21 (0.04) Ba	*0.32 (0.06) Aa			
HCl Cil	L1	0.21 (0.03) Aa	0.20 (0.03) Ab			
HCl+Sil	L2	0.24 (0.02) Ba	*0.31 (0.06) Aa			
Combuol	L1	0.18 (0.03)				
Control	L2	0.19 (0.03)				

There was no significant difference between abrasives agents (p = 0.5847). Means followed by different letters (uppercase in horizontal and lowercase in vertical comparing time within each category of abrasive) differ statistically (p  $\leq$  0.05). \*Differs from control group at the time after polishing (p  $\leq$  0.05).

Table 3. Knoop microhardness (KHN) means followed by the standard deviation of the experimental groups.

Group	Time	Polishing			
Group	Tille	Polished	Unpolished		
	L1	339.7 (35.9) Ac	342.1 (16.7) Ab		
H <sub>3</sub> PO <sub>4</sub> +Pum	L2	*389.0 (38.4) Ab	355.0 (18.8) Bb		
H3FO4+Fulli	L3	443.8 (31.0) Aa	440.9 (40.2) Aa		
	L4	437.8 (31.7) Aa	444.0 (25.4) Aa		
	L1	338.2 (14.7) Ac	337.2 (25.5) Ab		
HCl+Sil	L2	*420.9 (18.3) Ab	360.2 (27.2) Bb		
HCI+3II	L3	439.1(19.5) Aa	436.7 (48.3) Aa		
	L4	442.4 (21.2) Aa	437.4 (37.2) Aa		
	L1	340.0 (19.3)			
Control	L2	335.7 (24.0)			
Control	L3	440.2 (40.8)			
	L4	437.8 (30.0)			

There was no significant difference between abrasive agents (p = 0.5847). Means followed by different letters (uppercase in horizontal and lowercase in vertical comparing time within each category of abrasives) differ (p  $\leq$  0.05). \*Differs from control group at the time after polishing (p  $\leq$  0.05).

Table 4. Means of phosphorus and calcium element wt% (standard deviation).

Group	Polishing	Phosphorus	Calcium
H <sub>3</sub> PO <sub>4</sub> +Pum	Polished	29.80 (1.81) a	7019 (1.81) a
	Unpolished	29.90 (1.03) a	70.09 (1.01) a
HCl+Sil	Polished	29.50 (1.25) a	*68.92 (1.56) b
	Unpolished	29.54 (0.77) a	*69.49 (0.72) b
Control	-	28.63 (0.32)	71.37 (0.32)

Means followed by same lowercase letters in vertical did not differ between themselves (p > 0.05). \*Differs from control group at the time after polishing ( $p \le 0.05$ ).

Table 5. Median (minimum value; maximum value) from chlorine and silica.

Group	Polishing	Chlorine	Silica
II.DO - Dum	Polished	0.00 (0.00; 0.00) Ba	0.00 (0.00; 0.00) Ba
H <sub>3</sub> PO <sub>4</sub> +Pum	Unpolished	0.00 (0.00; 0.00) Ba	0.00 (0.00; 0.00) Aa
HCl+Sil	Polished	*1.18 (0.88; 1.44) Aa	*0.27 (0.08; 0.85) Aa
1CI+3II	Unpolished	*0.98 (0.70; 1.16) Ab	0.00 (0.00; 0.00) Ab
Control	-	0.00 (0.00; 0.00)	0.00 (0.00; 0.00)

Distinct letters (uppercase in horizontal and lowercase in vertical) differ between themselves ( $p \le 0.05$ ). \*Differ from control group ( $p \le 0.05$ ).

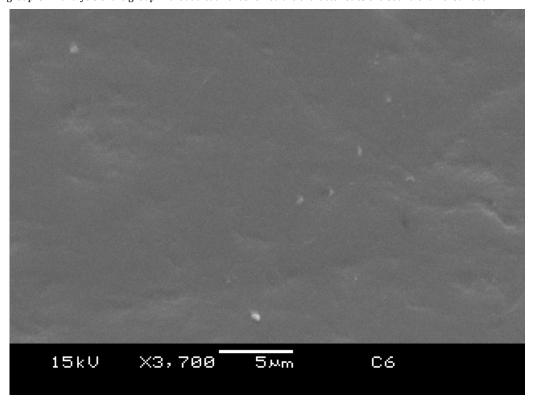
In this research, the group treated with H<sub>3</sub>PO<sub>4</sub>+Pum did not present significant statistical differences in terms of surface roughness compared with the group treated with (HCl+Sil), according to other studies. 14,15 This result may be related to the acids used. Hydrochloric acid demonstrated greater erosive power and is more erosive than phosphoric acid; however, as its concentration was reduced (to 6.6%), it is suggested that an equalization occurs in relation to erosive power that could create a conditioning pattern of the enamel, taking into account a lower formation of porosity on the enamel surface, making the observed amount of roughness equal to that of phosphoric acid, which is a weak acid in higher concentrations.<sup>22,23</sup>

The enamel's roughness increased after the microabrasion treatment following the initial abrasion, which is in agreement with the results present in the literature. 12,14,15,24 The particle size of the different agents used were analyzed by Loguercio et al. (2007),16 who observed the greater granulation associated with Opalustre's micro-abrasive system at 20-160 μm, followed by Prema Compound's 30–60 µm abrasive system, and pumice with particle sizes varying between 0.8 μm and 3.0 μm. According to these authors the higher roughness found were obtained with Opalustre's (6.6% hydrochloric acid) abrasive system in comparison with Prema Compound's (10% hydrochloric acid) abrasive system, which has the same abrasive agent in its composition with fewer granulations. Although Opalustre's particle size was larger than that of pumice, which could possibly contribute to more of the roughness of the enamel, this effect was not significant in this study, perhaps due to the acid concentration used in each abrasive

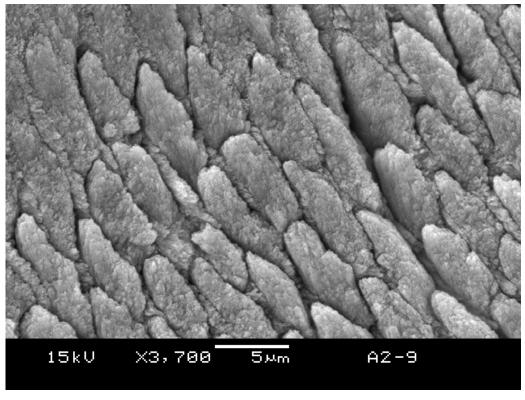
system. Opalustre has 6.6% hydrochloric acid in its composition, and despite it being a strong

acid; it was used in a low concentration.

Figure 1. Control group. SEM analysis of the group without treatment showed the characteristics of a sound enamel surface.



Figure~2.~Group~unpolished~treated~with~H3PO4+Pum.~SEM~analysis~showed~the~exposition~of~interprismatic~spaces.



Figure~3.~Group~unpolished~treated~with~HCl+Sil.~SEM~analysis~showed~the~nonspecific-etching~pattern.

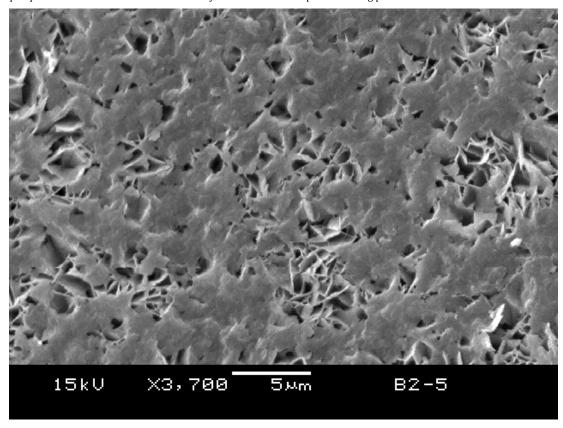


Figure 4. Group polished treated with H3PO4+Pum and polished. SEM analysis showed reestablishment of normal enamel characteristics, similar to control.

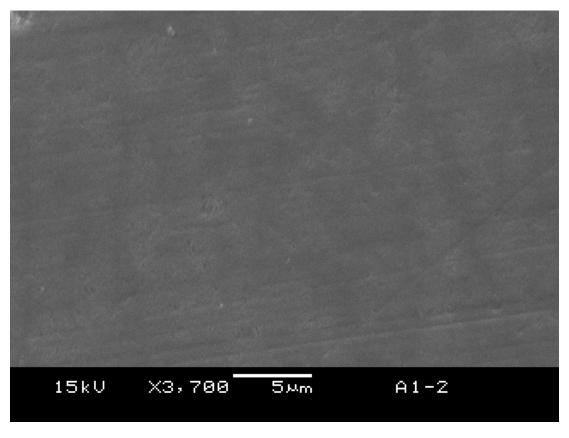
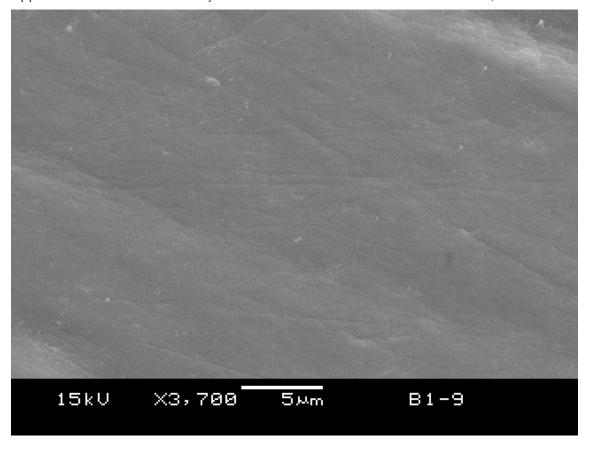


Figure 5. Group polished treated with HCl+Sil. SEM analysis showed reestablishment of normal enamel characteristics, similar to control.



Roughness is intimately connected with brightness and light reflection, as well as to the accumulation and retention of dental plaque. Thus, enamel polishing is important, as otherwise the roughness may interfere directly with the esthetics of the teeth and the health of the adjacent tissues.<sup>25</sup> In this study, polishing techniques on the abrasioned enamel were used with the assistance of materials usually used for the polishing and restoration of compound resins. Such materials have excellent results on enamel.

It was noted that with respect to polishing after microabrasion, the diamond polishing paste led to a decrease on the enamel surface's roughness in all groups. This data is consistent with recent studies.<sup>14,15</sup> According to Jefferies (2006)<sup>26</sup> this fact could be due to the small sizes of the diamond granulation in the diamond paste (2–4 µm). The results in this research still match the results obtained by Scheibe et al. (2009)<sup>27</sup> when comparing the surface roughness of the specimens of the compounds polished by aluminum oxide discs, silicone tips, and diamond pastes. The best results were obtained when using such materials. According to Chung (1998),<sup>28</sup> such procedures could reduce the surface roughness from 74%, thus justifying the use of these polishing methods.

The results showed that the values of the microhardness of the enamel increased after microabrasion followed by polishing. The slight superficial abrasion of the enamel rods with acid erosion obtained by simultaneous microabrasion systems causes the compression of mineralized tissue within the region's organic enamel, replacing the outer region's prisms or even the micronized diamond from diamond paste in these regions. <sup>10</sup> The pattern of conditioning can also provide a way for the dissolution of the enamel's subsurface that, along with the minerals in the external fluid, may precipitate and increase the hardness.

In this study, the specimens were stored with artificial saliva for 15 days and 30 days.

The results show that for both groups, polished and unpolished, there are observed changes after 15 days of exposure. But in a comparison of the two times of immersion, no changes were detected in the amount of enamel microhardness, indicating that the artificial saliva for periods over than 15 days did not increase the hardness of enamel. A study by Fragoso et al. (2011)<sup>15</sup> related that times of immersion of 24 h or even seven days after microabrasion did not increase the abrasioned enamel's hardness. In this study, the specimens were stored in artificial saliva only after the microabrasion and polishing procedures, so it was possible to evaluate the isolated effects of these procedures in order to determine if exposure to saliva altered the results in this phase.

According to energy dispersion (EDS) results, no alteration was observed for phosphorus means after microabrasion, with or without polishing. But for calcium, the groups treated with HCl+Sil presented lower means than the control. This can be explained because the specimens used for this test did not stay immersed in artificial saliva, so the fluid could not precipitate and increase the EDS values. However, it was observed, for HCl +Sil groups, showed the presence of chlorine on the enamel, and when the procedure was associated to polishing, some silica was incorporated on the substrate. Thus, it is possible that this incorporation may alter the hardness values, as observed in Table 3.

According to the SEM analysis, the application of H<sub>3</sub>PO<sub>4</sub>+Pum acid showed a morphological enamel etching patterns similar to those described in the literature, <sup>11,19</sup> which are characterized by the dissolution and homogeneous region of interprismatic clear enamel (Fig. 2). The surface morphology of treatment with HCl+Sil was slightly irregular, with a gritty appearance (Fig. 3). Both treatments presented morphological patterns that differed from the control group (Fig. 1).

However, this procedure does not lead to abraded enamel that the technique suggests.

The groups polished with diamond polishing paste (Figs. 4 and 5) had a morphology closer to that of the control group and improved the surface smoothness after

microabrasion, confirming the need to perform this step after the microabrasion procedure.

## **CONCLUSION**

According to the results obtained from this research, it can be concluded that the abrasion followed by polishing can increase the microhardness of enamel, leaving a better surface smoothness. The immersion in artificial saliva for a minimum of 15 days is sufficient to increase the surface hardness of the abrasioned enamel. In addition, it can be conclude that microabrasion procedures can alter the mineral content of dental enamel by incorporating microabrasive chemical elements on this substrate.

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