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Morphology, roughness, and surface loss of sound enamel submitted to different acid etching agents and different durations of application

Morfologia, rugosidade e perda superficial do esmalte hígido submetido a diferentes condicionadores ácidos e diferentes tempos de aplicação

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ABSTRACT

Objective: Knowledge about the effects of different phosphoric acids can help clinicians choose the appropriate acid etchant, preserving dental structure without compromising bond strength. This study aimed to analyze the morphology, surface loss, and roughness of different acid etching agents, especially a self-limiting phosphoric acid, at different time intervals. **Material and Methods**: Bovine enamel specimens were allocated into experimental groups (n = 5). The surfaces were treated with acid etchant (UE: UltraEtch; C37: Condac37; PE37: Power Etching 37%; DGC: Dental Gel Conditioner; SE: Scotchbond Etchant) for 15, 30, 60, 90, and 120 s. Surface loss and roughness were evaluated with an optical profilometer, and morphological analyses was performed by Scanning Electron Microscopy. Data were statistically analyzed (α=0.05). **Results:** The surface loss increased for all acids over time, except for UE. Up to 60 s, none of the acids promoted different surface loss. For 90 s, UE showed the smallest loss, and SE exhibited the greatest loss; for 120 s, UE had the smallest loss, while DGC and SE had the highest. The roughness increased for all acids over time, including UE, except for 90 s. For 90 s, DGC promoted the highest surface roughness, while UE, PE37, and SE showed the smallest. Compared to the control, all acids showed significant differences in structural loss and roughness. Finally, the type 2 etching pattern predominated. **Conclusion:** UltraEtch exhibited a self-limiting etching behavior, even though it causes an increase in surface roughness, it does not promote a significantly increasing surface loss over time.

KEYWORDS

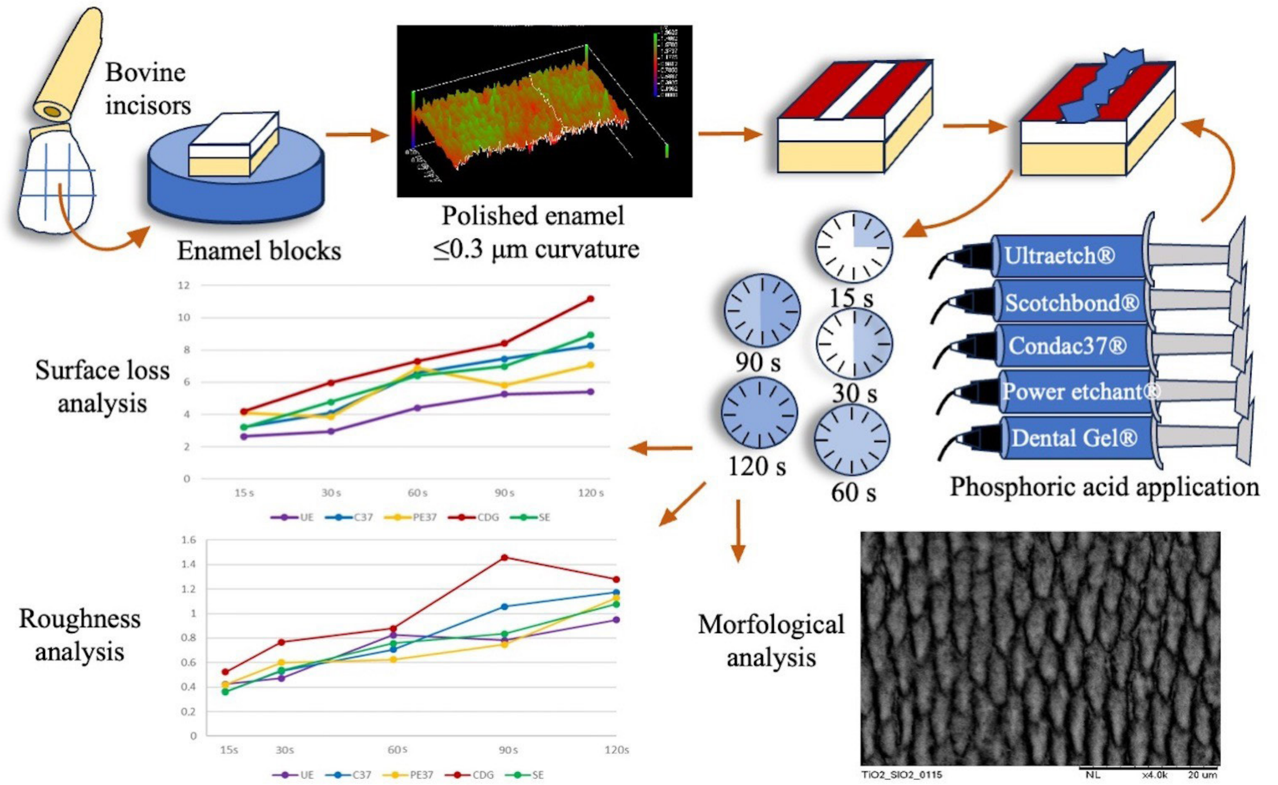
Acid Etching; Enamel; Morphology; Phosphoric Acid; Surface Properties.

RESUMO

Objetivo: Compreender os efeitos de diferentes ácidos fosfóricos pode auxiliar os clínicos na escolha do condicionador ácido, preservando a estrutura dentária sem comprometer a resistência adesiva. Este estudo avaliou a morfologia, a perda superficial e a rugosidade promovidas por diferentes condicionadores ácidos, com ênfase em um agente autolimitante, em diferentes tempos de aplicação. **Material e Métodos:** Incisivos bovinos foram divididos em grupos experimentais (n = 5). As superfícies foram tratadas com UltraEtch (UE), Condac37 (C37), Power Etching 37% (PE37), Dental Gel Conditioner (DGC) e Scotchbond Etchant (SE), por 15, 30, 60, 90 e 120 segundos. A perda superficial e a rugosidade foram medidas com perfilômetro óptico, e a morfologia foi avaliada por Microscopia Eletrônica de Varredura (MEV). Os dados foram analisados estatisticamente (α = 0,05). **Resultados:** A perda superficial aumentou com o tempo para todos os ácidos, exceto para o UE. Até 60 segundos, nenhum dos ácidos promoveu perda superficial significativamente diferente. Aos 90 segundos, o UE apresentou a menor perda, enquanto o SE apresentou a maior; aos 120 segundos, o UE manteve a menor perda, e o DGC e o SE apresentaram as maiores perdas. A rugosidade aumentou com o tempo para todos os ácidos, inclusive o UE, com exceção do tempo de 90 segundos. Em comparação ao grupo controle, todos os ácidos demonstraram diferenças significativas na perda estrutural e na rugosidade. O padrão de condicionamento do tipo 2 foi predominante. **Conclusão:** O UltraEtch demonstrou comportamento autolimitante, aumentando a rugosidade, mas sem provocar perda significativa de estrutura ao longo do tempo.

PALAVRAS-CHAVE

Condicionamento Ácido; Esmalte; Morfologia; Ácido Fosfórico; Propriedades da superfície.



INTRODUCTION

Minimally invasive treatments aimed at enhancing smile aesthetics through the cementation of ceramic veneers on teeth, without any preparation or with minimal enamel wear, have become an increasingly common clinical practice [1]. The success of the treatment is closely related to the improvement of ceramic materials, but even more to that of resinous and adhesive materials, which allow ultra-thin ceramic veneers to remain in place without frictional or macromechanical retention between them and the substrate [2]. Therefore, micromechanical interactions between enamel and cement, after the application of an intermediate agent (adhesive system), and between the adhesive system and the restorative material, are considered fundamental [3,4].

However, adhesion to enamel, a highly mineralized tissue, depends on its proper prior preparation [5]. Among the various methods of preparing enamel, including sandblasting and laser irradiation, phosphoric acid etching is the gold standard in adhesive dentistry due to its proven effectiveness in creating surface irregularities on the substrate [6,7]. Undoubtedly, the enamel-restoration interface represents an important factor in the longevity of adhesive restorations; therefore, the inability of etched enamel to provide adequate material retention may be the main reason for failure, compromising its durability [8,9].

Hence, etching the enamel, as an isolated step, with an acidic agent is essential [10], even when using self-etching/self-adhesive resin materials containing functional monomers [11]. This agent, typically phosphoric acid [12], increases surface free energy, wettability, and roughness of the enamel [13,14]. It also demineralizes a portion of the enamel's inorganic component, leading to the formation of micro-pores and retention sites, thereby establishing micromechanical adhesion between the substrate and the resinous materials [15,16]. Enamel treated with phosphoric acid results in an irregular surface, accompanied by a reduction in surface hardness [17] and the removal of approximately 10 μm of its thickness in a non-uniform manner [18-20].

In vitro, the application of phosphoric acid at concentrations between 35 and 37% for 15, 20, or 30 seconds has been determined as the ideal standard for enamel etching to enhance the retention of aesthetic restorative materials [18,21]. However, this seems to depend not only on the duration the acid remains acting, but also on its various properties, such as concentration, composition (organic-inorganic), viscosity, thixotropy, pH, and buffering capacity, and even the duration of the rinse [22,23]. Zhu et al. [23] demonstrated that agents containing phosphoric acid at concentrations below 30% are inadequate for proper enamel etching, while concentrations above 50% result in insignificant changes in surface morphology. Furthermore, variable etching times ranging from 15 to 60 s, and even less, produce similar clinical outcomes on microtensile bond strength to enamel.

On the other hand, there is no evidence of a demineralization pattern in enamel with overetching for more than 60 s [24], nor for enamel treated for less than 30 s. Currently, the cementation of multiple ceramic veneers has become increasingly common, resulting in simultaneous etching of several teeth, which leads to variable durations of acid exposure due to differences in the time from application to washing. Therefore, situations like these require further studies since the properties of the enamel surface and roughness can be altered within a few seconds [17].

Similarly, the characteristics of each acid etching agent should be investigated for their effects on enamel, as they have been shown to be relevant when etching dentin [25]. Products with similar concentrations of phosphoric acid but different thickeners, when applied for the same duration on dentin, promoted demineralization at varying depths and with distinct morphological patterns [26]. Aggressive demineralization can render the substrate more susceptible to subsequent acid challenges, such as cariogenic ones; however, enamel requires a certain degree of etching for proper adhesion [27]. Even if enamel etching could uniformly act in depth, the formation of insoluble by-products may clinically compromise adhesion, potentially leading to early adhesive interface failure [28,29].

In order to balance adequate properties with reduced concern regarding etching time, 35% phosphoric acid formulations with self-limiting capacity were developed (Opal Etch® and Ultraetch®; Ultradent Corporation, Ultradent, South Jordan, UT). According to the manufacturer, such capacity implies a shallower etching depth. Inopportunely, one of the self-limiting etchants, Opal Etch, exhibited shear bond strength values similar to those of a traditional agent (Caulk Tooth Conditioner Gel - 34% Phosphoric Acid®; Dentsply Caulk, Milford, DEL), even when overetching. Both acids resulted in lower bond strength values on enamel at the longest etching time (120 s) compared to 30, 60, and 90 s. The highest bond strength values were obtained with a 90-second etching time [30].

Regarding the depth of the etched enamel, evaluated by means of confocal laser scanning microscopy, the self-limiting etchant with 35% phosphoric acid gel (Opal Etch®) was always inferior to the traditional 34% Phosphoric Acid® (Dentsply) [31]. The self-limiting etchant behaved similarly to the traditional one over time: at 15 and 30 s, it promoted equivalent depths to each other and significantly lower than those achieved at any of the times - 60, 90, and 120 s – which were also similar to each other [31]. According to the scanning electron microscopy analysis of a single specimen, the images of the etch patterns revealed that for both agents, they were of type 1 for 30 s and of type 2 for 120 s [31]. Since the etch pattern can influence micromechanical bonding [21], there is no clear advantage in using the self-limiting agent (Opal Etch®) over the traditional one [30,31]. However, it is important to note that only one sample was studied, and the morphological aspect of the substrate treated with different etching times was not evaluated. Finally, the literature has rarely studied the loss of structure and surface roughness in etched enamel, and it is even rarer to analyze them simultaneously.

Thus, this study evaluated the loss of structure (depth), roughness, and surface morphology of bovine enamel etched with phosphoric acid agents of varying characteristics, including an acid with self-limiting capacity (UltraEtch®), for varying durations. The null hypothesis was that the various phosphoric acid agents and the different application times would not influence the results.

MATERIAL AND METHODS

Experimental design

This randomized in vitro study evaluated the loss of structure (depth), surface roughness, and micromorphology of bovine enamel etched with phosphoric acid agents at 5 levels (UE: UltraEtch®/Ultradent; C37: Condac37®/FGM; PE37: Power Etching 37%®/BM4; DGC: Dental Gel Conditioner®/Dentsply; SE: Scotchbond Etchant®/3M ESPE), and the etching time at 5 levels (15 s; 30 s; 60 s; 90 s; 120 s). Separately, a control group (C) did not undergo enamel etching.

Specimens preparation

After the research protocol received approval from the Ethics Committee on Animals Research (CEUA - 012/2018) at the Faculty of Dentistry, University of São Paulo, ninety bovine incisors were obtained from a slaughterhouse; they were cleaned with periodontal curettes and then stored in 0.1% thymol at 4ºC. Twenty teeth were used for conducting pilot tests, and seventy for the experimental groups.

The crown portion was sectioned to obtain 5 × 5 mm slabs using an automatic cutting machine (Isomet Low Speed Saw; Buehler Ltd., Lake Bluff, IL, USA) under water irrigation at a speed of 300 rpm. The fragments were fixed on acrylic bases, flattened, and polished in a metallographic manual polisher (Buehler Ltd., Lake Buff, IL, USA) with SiC paper (Buehler Ltd., Lake Buff, IL, USA) of decreasing granulation (120-, 180-, 240-, 800-, 1200-, 2500-, and 4000-grit), under water-cooling at speed of 250 rpm. Then, the specimens were immersed in distilled water and taken to an ultrasonic cleaner for 10 min.

The initial surface curvature of all specimens was analyzed with an optical profilometer (Proscan 2100, Scantron, Venture Way, Taunton, UK) along with the appropriate software (Proscan Application Software version 2.0.17). Specimens with curvature less than 0.3 μm were selected and randomly divided into the experimental groups (n = 5). The mean and standard deviation of the baseline values were 0.1199 μm (± 0.0825).

In the same profilometer, the initial roughness of each specimen was analyzed as a parameter for determining the difference (delta/Δ) in final roughness. Such initial roughness was calculated as the average of three different readings taken on each specimen. The same approach was applied to determine the final roughness.

After determining initial curvature and surface roughness, all specimens, except for the control group, were etched with different phosphoric acids (UE, C37, PE37, DGC, SE) for different durations (15 s,30 s, 60 s, 90 s, 120 s). For this procedure, unplasticized polyvinyl chloride (UPVC) tape was affixed to the surface of the samples to obtain a central window of 5 × 1 mm, exposing it to the acid etching. The exposed enamel area of each specimen was then washed with an air-water spray and thoroughly dried using a water/oil-free air flow from a triple syringe. Subsequently, acid etchant was applied to the enamel according to the experimental group, using an applicator tip from the respective manufacturer, while ensuring there was no surface contact and confirming the free flow of the material on sterile gauze beforehand. After the etching time according to each experimental group, the surface was thoroughly washed with an air-water spray for 30 s and dried with an air flow for 5 s, always maintaining a distance of approximately 1 cm from the surface.

Surface loss and roughness analysis

After acid etching, the UPVC tape was removed, and the specimens were analyzed for loss of structure (depth) and final surface roughness (*Ra*).

For surface loss, a central area of 2 mm in length (x-axis) × 1 mm in width (Y-axis) of each specimen was scanned by a non-contact optical profilometer (Proscan 2100 – Sensor Model S11/03). This area was selected to include the etched region as well as two reference areas. The equipment was set in 200 steps with a size of 0.01 mm on the X-axis, and 10 steps of 0.1 mm on the Y-axis. The depth of phosphoric acid etching, in μm, was calculated by the software (Proscan Application Software version 2.0.17) using the difference between the average height of the test area and the average of the two reference areas, considering a 3-point height tool.

Roughness, the numerical expression of surface irregularities, was determined as the average *Ra* value from three scans performed on each specimen. The baseline roughness (average of the three initial readings) was subtracted from the final average to assign a roughness value to each sample. Each scan was running for a 4-mm long line using a non-contact optical profilometer (Proscan 2100 – Sensor Model S11/03) with the scanning parameters: Step Size X = 0.003 and Number of Steps X = 1333; Step Size Y = 0.001 and Number of Steps Y = 0; and analysis parameters: Cut off = 0.8 mm and Surface Filter = 99.

Morphological analysis

Morphological analysis of the etched enamel surface of all specimens was performed using a Toshiba TM3000 low-vacuum tabletop scanning electron microscope (SEM, Hitachi, Japan), operating at an accelerating voltage of 5 kV with magnifications of ×2000 and ×4000.

This low-vacuum SEM enables direct observation of specimens without the conventional preparation procedures required for high-vacuum SEMs, such as sputter coating or dehydration.

The specimens were mounted on aluminum stubs and fixed with a cyanoacrylate-based double-sided tape. The enamel surface was positioned parallel to that of the stub, allowing the electron current to pass through the sample for reading via electron beam scanning.

Each specimen was classified according to the typical patterns of surface micromorphology of etched enamel (type 1, type 2, or type 3) to determine the frequency within each experimental group. When different etching patterns were observed in the same specimen, classification was assigned based on the most prevalent pattern, since a mixed type or patterns other than 1, 2, or 3 were not recognized.

Statistical analysis

As for the quantitative response variables, surface loss and roughness, considering the acid agent for enamel etching (at 5 levels) and the duration of its action (at 5 levels), the obtained data were statistically evaluated using 2-way ANOVA and Tukey tests. To compare each of the experimental groups with themselves and with the control group, after verifying non-normal distribution using Shapiro-Wilk test, the Kruskal-Wallis and Tukey tests were applied.

As for the nominal qualitative response variable, to verify the association of each experimental condition with the frequency of patterns of enamel etched, the chi-square test was applied. In this case, comparisons with the control group are meaningless since they were not etched, therefore, they did not show any pattern frequency.

The statistical software used was SigmaPlot™ 13 (Systat Software, Inc., San Jose/CA, USA), and a level of significance of 5% was always adopted.

RESULTS

The etchant acid ($p<0.001$) and etching time ($p<0.001$) had significant influence on surface loss, and there was an interaction between them ($p=0.033$). Over time, all acids, except for UltraEtch, showed an increase in surface loss values, although not always in a regular or proportional way. The enamel surface loss of UltraEtch acid did not differ regardless of etching time. All phosphoric acids, when applied for up to 60 s, resulted in similar surface loss and did not differ from each other. By acting for 90 s, UltraEtch and Scotchbond Etchant promoted the lowest and highest surface loss, respectively; and Condac37, Power Etching 37%, and Dental Gel Conditioner, intermediate loss. When etched for 120 s, UltraEtch resulted in the lowest surface loss, Dental Gel Conditioner and Scotchbond Etchant led to the highest, while Condac37 and Power Etching 37% showed intermediate levels of loss. The means and standard deviation of enamel surface loss are presented in Table I.

Regarding the roughness results, the acid agent ($p<0.001$) and the etchant time ($p<0.001$) also exhibited a significant influence. Interactions were also significant ($p=0.018$). For all acids, roughness increased over time, including UltraEtch. All acids showed similar surface roughness when etched for 15, 30, 60, and 120 s. When etched for 90 s, Dental Gel Conditioner promoted the highest surface roughness, UltraEtch, Power Etching 37%, and Scotchbond Etchant led to the lowest, while Condac37 showed intermediate levels of roughness (Table II).

Table I - Mean ± SD of enamel surface loss (in μm) of the etchant (UE, C37, PE37, DGC, SE) applied on the enamel and the duration (15, 30, 60, 90, 120 s) of their application*

	UE	C37	PE37	DGC	SE
15 s	2.642 ± 0.321^{Aa}	3.212 ± 0.189^{Aa}	4.106 ± 0.730^{Aa}	4.191 ± 0.462^{Aa}	3.205 ± 0.726^{Aa}
30 s	2.948 ± 0.567^{Aa}	4.088 ± 0.468^{ABa}	3.845 ± 0.837^{Aa}	5.968 ± 1.125^{ABa}	4.776 ± 1.177^{ABa}
60 s	4.417 ± 0.490^{Aa}	6.587 ± 1.308^{Ba}	6.881 ± 2.393^{Ba}	7.316 ± 0.715^{Ba}	6.408 ± 0.701^{BCa}
90 s	5.268 ± 0.532^{Aa}	7.447 ± 0.540^{BCab}	5.798 ± 0.954^{ABab}	8.409 ± 1.230^{BCb}	6.981 ± 1.287^{BCab}
120 s	5.412 ± 0.436^{Aa}	8.274 ± 1.159^{Cab}	7.067 ± 0.906^{Bab}	11.176 ± 3.816^{Cb}	8.923 ± 1.646^{Cb}

*Different superscript capital letters show statistically significant difference between times at each row ($p<0.05$), and different superscript lower case letters show statistically significant difference between acid treatments at each column ($p<0.05$).

Table II - Mean ± SD of surface roughness of the etchant (UE, C37, PE37, DGC, SE) applied on the enamel and the duration (15, 30, 60, 90, 120 s) of their application*

	UE	C37	PE37	DGC	SE
15 s	0.423±0.116^{Aa}	0.363±0.055^{Aa}	0.420± 0.159^{Aa}	0.522±0.072^{Aa}	0.362± 0.197^{Aa}
30 s	0.471±0.106^{ABa}	0.529±0.088^{Aa}	0.601± 0.103^{Aa}	0.767±0.303^{Aa}	0.537±0.115^{ABa}
60 s	0.824±0.116^{ABa}	0.707±0.174^{ABa}	0.624± 0.066^{Aa}	0.879±0175^{Aa}	0.758±0.223^{ABa}
90 s	0.781±0.118^{ABa}	1.058±0.125^{Bab}	0.748±0.188^{ABa}	1.457± 0.284^{Bb}	0.834±0.181^{Ba}
120 s	0.948±0.171^{Ba}	1.173±0.063^{Ba}	1.128±0.329^{Ba}	1.280±0.411^{ABa}	1.076±0.150^{Ba}

*Different superscript capital letters show statistically significant difference between times at each row ($p<0.05$), and different superscript lower case letters show statistically significant difference between acid treatments at each column ($p<0.05$).

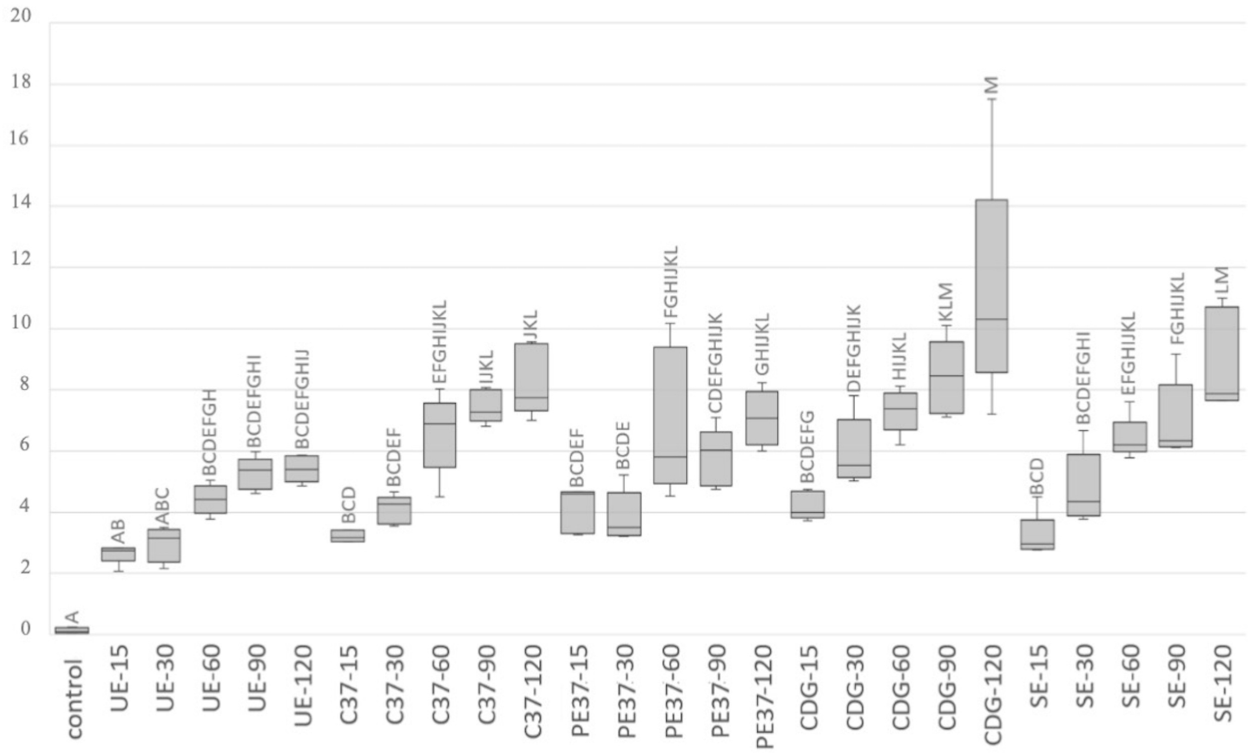


Figure 1 - Scores data (25%/median/75%) of the surface loss (in μm) in each experimental group compared to the control group. Different letters indicate a statistically significant difference between experimental groups.

In the comparison of each of the experimental groups, individually, among themselves, and with the control, a statistically significant difference was detected for surface loss ($p<0.001$) and roughness ($p<0.001$). All acids at any application time were able to promote significant alteration of the enamel surface in terms of both structure loss and roughness compared to the control.

Only UltraEtch, when applied for 15 and 30 s, did not cause a surface loss different from the control (Figure 1). DGC for 15 s, UE and SE for 15 and 30 s, C37 and PE37 for 15, 30, and 60 s did not promote roughness different from the control (Figure 2).

Finally, there was a significant association between the experimental conditions and the enamel etching pattern ($p<0.001$). Type 2 etching pattern prevailed for most acids and times used. Type 1 was more common when UE, C37, and PE37 were applied for 15 s, PE37 for 30 s, and C37 for 60 s. An equivalent frequency of types 1 and 2 was observed when SE was applied for 15 s. Type 3 etching patterns were rarely detected when DGC was applied for 60 and 120 s, and SE for 15 s. Frequency data for the etching pattern types as well as the micrographs are shown in Table III and Figures 3 to 5, respectively.

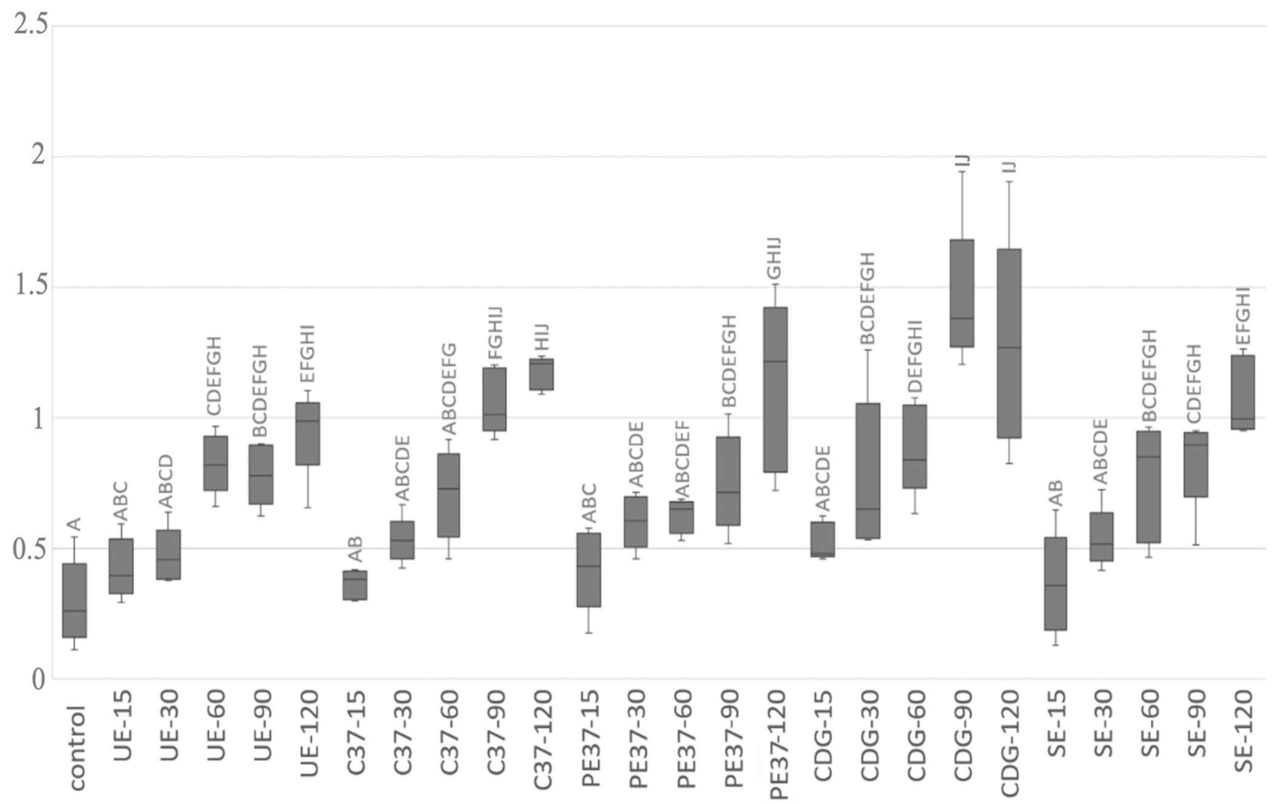


Figure 2 - Scores data (25%/median/75%) of the surface roughness in each experimental group compared to the control group. Different letters indicate a statistically significant difference between experimental groups.

Table III - Percentages (%) of enamel etching pattern of the acids (UE, C37, PE37, DGC, SE) applied over time (15, 30, 60, 90, 120 s)

		Type 1	Type 2	Type 3
UE	**15 s**	100.00	0	0
	30 s	20.00	80.00	0
	60 s	0	100	0
	90 s	20.00	80.00	0
	120 s	0	100.00	0
C37	**15 s**	60.00	40.00	0
	30 s	40.00	60.00	0
	60 s	100.00	0	0
	90 s	20.00	80.00	0
	120 s	20.00	80.00	0
PE37	**15 s**	0	100.00	0
	30 s	60.00	40.00	0
	60 s	0	100.00	0
	90 s	0	100.00	0
	120 s	0	100.00	0
DGC	**15 s**	20.00	80.00	0
	30 s	20.00	80.00	0
	60 s	0	60.00	40.00
	90 s	0	100.00	0
	120 s	0	80.00	20.00
SE	**15 s**	40.00	40.00	20.00
	30 s	0	100.00	0
	60 s	0	100.00	0
	90 s	0	100.00	0
	120 s	0	100.00	0

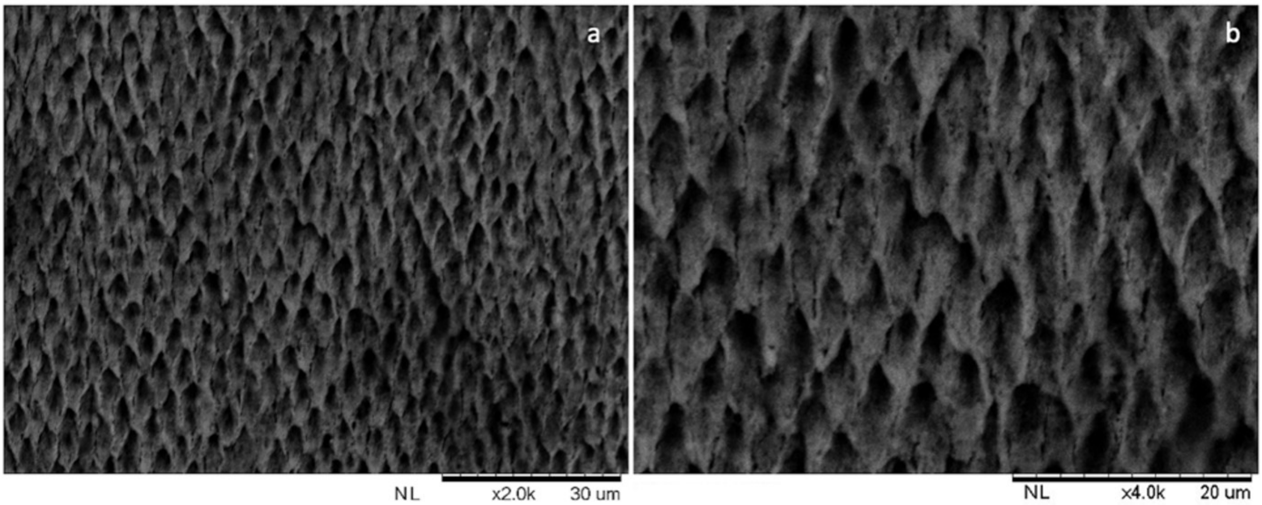


Figure 3 - Scanning electron micrograph images representative of etching pattern type 1 (preferential demineralization of enamel prism cores) on the etched enamel (a: 2000× and b: 4000× magnification).

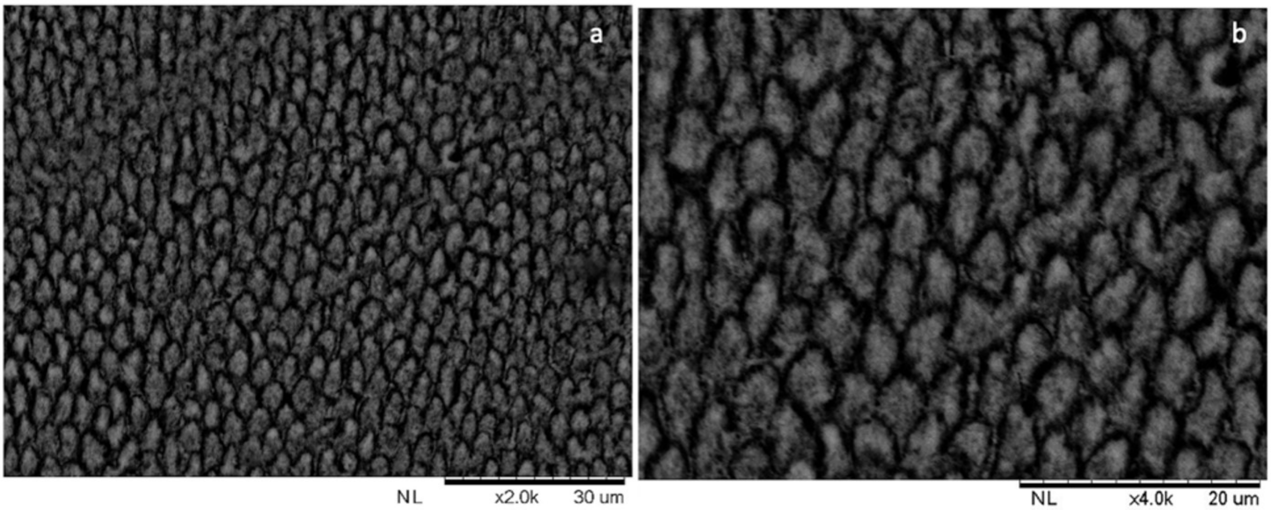


Figure 4 - Scanning electron micrograph images representative of etching pattern type 2 (preferential demineralization of enamel prism peripheries) on the etched enamel (a: 2000× and b: 4000× magnification).

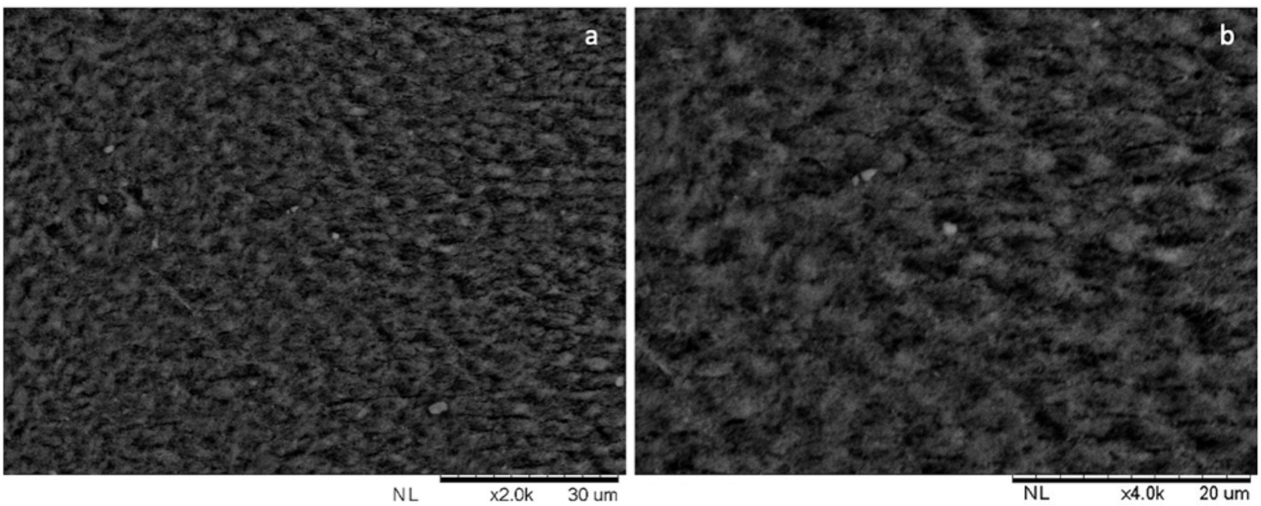


Figure 5 - Scanning electron micrograph images representative of etching pattern type 3 (disorderly demineralization) on the etched enamel (a: 2000× and b: 4000× magnification).

DISCUSSION

In the present study, the null hypothesis was rejected. Only the self-limiting acid behaved differently in surface loss over time, including at 90 and 120 s.

According to the manufacturer, UltraEtch® is a 35% phosphoric acid solution with excellent viscosity. Its consistency allows precise application and ensures coverage even in areas with poor enamel coalescence. Additionally, the agent is glycerin-free permitting its quick and complete removal after rinsing. The self-limiting capacity refers to the etching depth after a 20 s application on dentin, averaging 1.9 μm, compared to 5.0 μm for traditional agents [32]. What attributes all these properties to it, and in particular its self-limiting capacity, remain undisclosed.

When comparing what the manufacturer claims for dentin and what the present study has revealed, the self-limiting acid also resulted in surface losses for enamel almost twice as low as those observed for the other acids. Moreover, even in the event of overetching (for 90 and 120 s), the UltraEtch® phosphoric acid promoted losses that did not exceed much more than 5.0 μm, almost half of the thickness typically reported for traditional phosphoric acid agents [18-20].

Even more surprising than the lower aggressiveness, UltraEtch® did not promote a significant increase in structure loss over time, a phenomenon that was not observed with any of the other acids. Wilson et al. [31] had previously noticed that the depth of enamel etched by Opal Etch®, the first self-limiting version, was always less than that induced by a traditional acid, when both were applied for the same duration. However, this version promoted the same enamel changes over time as those caused by traditional acid, making it not an advantage to use since it did not show a self-limiting behavior itself [30,31].

In the present study, the UltraEtch® self-limiting acid has demonstrated the ability to fulfill its designated function, proving to be less invasive when the enamel etching time exceeds 60 s. In addition, this phosphoric acid has shown, except for 90 s, surface roughness equivalent to that promoted by all other evaluated acids. Hence, even less invasive, UltraEtch® does not fail to meet expectations when compared to the other acids in creating surface irregularities on the substrate for the proper impregnation of adhesive material [33].

Hypothetically, all phosphoric acid agents have a self-limiting effect on enamel, since the calcium released during demineralization starts to function as a buffer for the reaction, capable of resisting changes in hydrogen ion concentration [31]. In fact, most acids stop increasing further tooth structure loss, especially when they have been applied for a longer duration. Moreover, at a certain stage of etching, the by-products resulting from enamel demineralization become insoluble, probably because of a controlled diffusion mechanism [28,34]. Nevertheless, it seems common to observe significant differences regarding the loss of enamel structure when different agents are applied for 15, 30, 60, 90, and even 120 s.

Then, it could be suggested that the lower loss of structure is a consequence of the slightly lower concentration (35%) of this agent compared to most traditional agents (37%) such as Condac37®, Power Etching 37%®, and Dental Gel Conditioner® [35]. Only Scotchbond Etchant®, one of the evaluated counterparts, is a low viscosity gel that also contains 35% phosphoric acid by weight.

To explain the diversity of etching effects, the higher the viscosity, usually due to the addition of colloidal silica and other polymeric spheres, even though it makes the application easy and precise on a certain area [36], the effectiveness of the agent is reduced [37]. However, it later was verified that the use of acidic agents with different viscosities does not promote distinct alterations of the enamel surface [38]. Although viscosity has not been evaluated in this study, the manufacturers of UltraEtch®, Condac37®, Power Etching 37%®, Dental Gel Conditioner®, and Scotchbond Etchant® define them, respectively, as a phosphoric acid solution with excellent viscosity, a low viscosity water-based gel with thixotropic properties, a water-based product with a good level of viscosity, a phosphoric acid-based gel thickened with colloidal silica, and a low viscosity gel. Perhaps UltraEtch® has the lowest viscosity because it is a solution rather than a gel. This could explain the effectiveness of enamel etching in increasing surface roughness and in never showing a type 3 etching pattern, as well as the minor, non-progressive loss of structure. Previously, Guba et al. [39] demonstrated a more uniform and better-defined etching pattern when using a low-viscosity phosphoric acid agent compared to a high-viscosity one.

On the other hand, very thick formulations led to the formation of air bubbles between the agent and the substrate during application, promoting a non-etched area [36]. The acids evaluated in this study are unlikely to have significantly different viscosities among themselves since it was not observed in any area that etching did not occur in any of the specimens belonging to any of the experimental groups. The micrographs obtained from the control group specimens, with smooth and polished surfaces, are clearly distinct from all others. Furthermore, even though the recommendation to obtain as many types 1 or 2 etching patterns as possible may not be clinically relevant [23], micrographs of etched enamel revealed that a type 3 pattern was rare.

It remains important to further investigate the formulation of the self-limiting acid to understand the basis of its efficacy. The information provided by the manufacturer indicates that dimethicone (< 1%) is present in its composition, a viscoelastic polymer potentially soluble in water with antifoaming and surfactant properties. In contrast, Dental Gel Conditioner®, which differed most from the self-limiting acid in all analyses, contains an unspecified surfactant and hydrophilic fumed silica (Aerosil® 200), which functions as an anti-settling, thickening, and anti-sagging agent. These compositional differences may account for the distinct etching characteristics observed among the acids.

Further studies are therefore needed to investigate viscosity, thixotropy, chemical composition, as well as pH and buffering capacity. Additionally, evaluation of the adhesion of various resin materials to enamel pretreated with a self-limiting acid is important, as their performance can vary depending on the etchant used [40].

While this in vitro study highlights the potential advantages of self-limiting acids, several limitations must be considered. Being an in vitro study, it cannot fully replicate intraoral conditions such as salivary flow, masticatory forces, temperature fluctuations, or long-term exposure to oral biofilms. Only a limited number of phosphoric acid formulations and etching times were tested, and subsequent adhesive procedures, restorative material interactions, and bond durability after extended aging were not evaluated. Consequently, direct clinical extrapolation is restricted. Nevertheless, the results suggest that self-limiting acids could reduce excessive enamel loss during etching, especially when treating multiple teeth or when etching times are inadvertently prolonged. Clinically, this may allow preservation of dental tissue without compromising the establishment of the adhesive interface, provided that bond strengths remain within the effective range of 15 to 25 MPa. Such insights could guide practitioners in selecting etching agents that optimize adhesion while minimizing structural loss, supporting more conservative and predictable restorative procedures.

CONCLUSION

All phosphoric acid agents are capable of etching the enamel surface, increasing surface roughness and structural loss, and predominantly induce a type 2 etching pattern, regardless of the duration of their application. On the other hand, the UltraEtch® acid exhibited a self-limiting etching behavior when applied for 15 to 120 seconds, even though it increased surface roughness, it did not lead to a significant increase in structural loss.

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Author's Contributions

KL: Data Curation, Investigation, Methodology, Writing – Original Draft Preparation, Writing – Review & Editing. ICP: Data Curation, Investigation, Writing – Review & Editing. JFOA: Conceptualization, Investigation, Methodology, Project Administration, Writing – Review & Editing. BOI: Data Curation, Investigation, Writing – Review & Editing. DMZ: Methodology, Validation, Writing – Review & Editing. LFFR: Conceptualization, Formal Analysis, Funding Acquisition, Project Administration, Resources, Supervision, Writing – Original Draft Preparation, Writing – Review & Editing.

Conflict of Interest

The authors have no conflicts of interest to declare.

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Regulatory Statement

The Ethics Committee on Animals Research (CEUA) approved the study, providing the following protocol and assent number (012/2018) for the use of bovine teeth in this study.

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