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Effect of simulated gastric acid on the surface degradation of dental ceramics

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SUMMARY

Objective. The aim of this study was to compare the effect in vitro of simulated gastric acid solution on the roughness, microhardness and micro-structural topography surface of two dental ceramics: lithium disilicate glass-ceramic and monolithic zirconium dioxide (ZrO_2) at baseline and after different immersion time.

Materials and methods. Lithium disilicate and ZrO_2 glass-ceramic discs were fabricated (40 of each one) and was evaluated under microscope the roughness (Atomic force microscopy), microhardness (Vickers hardness tester) and micro-structural topography surface (Scanning electron microscopy) before immersion (baseline) and after three periods of immersion: 8 h 25 min, 46 h 2.5 min and 92 h 5 min in simulated gastric acid solution of hydrochloric acid (HCl, 0.06 M, pH 1.2) at 37 ºC. The ceramics were compared over time with a repeated measures analysis of variance (ANOVA).

Results. Statistically significant changes ($P<.05$) were found in the microhardness values between both ceramics, which decreased after all times points of immersion in simulated gastric acid solution, ZrO2 showed higher microhardness mean values ($P<.05$) but lithium disilicate showed significant changes ($P <.05$) in the surface microhardness at baseline versus the three immersion times. Lithium disilicate had higher roughness values than ZrO_2 ($P<.05$), which increased after 8 h 25 min of immersion; however, after 46 h 2.5 min and 92 h 5 min of immersion, the roughness decreased. More microporosities were observed in the lithium disilicate surface than with ZrO_2.

Conclusion. The simulated gastric acid solution of HCl affected the roughness, microhardness and micro-structural topography surface of both lithium disilicate and ZrO_2, but greater surface degradation was presented lithium disilicate.

Keywords: lithium disilicate; zirconium dioxide; surface topography; microhardness, simulated gastric acid.

INTRODUCTION

Gastro-esophageal reflux disease (GERD), eating and psychological disorders such as bulimia nervosa cause accumulation of gastric acid in the oral cavity (1, 2), degrading the physical and mechanical characteristics of the restorative materials, and causing surface degradation that negatively influences in the integrity of the materials (3, 4). The principal components of gastric acid are hydrochloric acid (HCl), potassium chloride, and sodium chloride, these acids from GERD present a pH of less than 2 (5). Gastric acid has been reported to cause chemical degradation of the surface of prosthetic restorations, increasing surface roughness from chemical corrosion of the glass after diffusion of water molecules that form hydroxyl ions that react with the water molecules to form hydroxyl ions with nonbridging oxygen atoms (6).

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Restorative materials for patients with gastrointestinal diseases should be selected based on the mechanical and esthetic properties of the ceramic after acid exposure (7, 8), as the acid generated by GERD and vomiting caused by bulimia nervosa reduce stability, decrease flexural strength, and may lead to crack development in ceramics (9). Changes in surface properties by the erosion process alter mechanical properties, therefore, the survival and durability of ceramic restorations in the oral cavity will depend on their resistance to acid attack (10-12).

Dental ceramic materials have become popular because of their biocompatibility, esthetics, and wear resistance in the oral cavity. Ceramics are used to produce prostheses replacing the morphology and function of damaged teeth (13-16). The most widely used ceramics today include lithium disilicate glass and zirconium dioxide (ZrO_2), with acceptable optical characteristics (17). Despite the excellent mechanical and surface properties of dental ceramics, conditions such as GERD may cause damage to the microstructural topography of the ceramic surface (18).

Lithium disilicate glass is widely used to restore lost tooth tissue in patients with erosion, abrasion, or attrition; however, ZrO_2 is also used because of its outstanding mechanical properties (19, 20). Strong acids, including HCl, have been reported to increase surface roughness, but there is distinct evidence that a smoother surface is a characteristic of resistant to acidic challenge (21). The time all ceramic materials are exposed to a chemical agent affects their durability and stability of their mechanical and surface properties. The most common erosive substances are gastric acid in GERD patients, citric acid from fruits and many bicarbonated beverages, so the time of consumption will determine the bonding strength and hardness values of ceramic materials (22). Therefore, it is necessary to understand how acidic agents affect long and short-term the ceramic materials, due to the fact that in the microenvironment conditions in the oral cavity the acidic foods or beverages are in contact with the ceramics surfaces for only a short time before being washed away by saliva. There is a significant interaction between the ceramic surface and an acidic aqueous medium but the time after exposure has been established to affect its optical and surface properties (23, 24).

In addition, although the exposure of ceramic materials to gastric acid has been assumed to alter their chemical stability and surface texture, generalized conclusions are lacking (25). The objective of this *in vitro* study was to compare the effect of simulated gastric acid solution on the surface microtopography, roughness, and microhardness of two dental ceramics: lithium disilicate glass-ceramic and ZrO_2 at baseline and after different exposure times. The null hypothesis was that the simulated gastric acid solution not generate the degradation of the surface microtopography and changes in the roughness and microhardness surfaces of dental ceramics after different exposure times of immersion.

MATERIALS AND METHODS

This in vitro study was performance at the Faculty of Stomatology, Autonomous University of San Luis Potosí, San Luis Potosí, Mexico. Two ceramic dental materials were evaluated in this *in vitro* study: a pressable lithium disilicate ($Li_2Si_2O_5$, LS2) glass-ceramic and a monolithic ZrO_2 (Table 1). Forty discs (6.0 mm in diameter and 1.0 mm in thickness) of each ceramic were produced according to the manufacturer's instructions. The lithium disilicate material was hot-pressed in a furnace (EP600, Ivoclar Vivadent), and the ZrO_2 glass-ceramic discs were milled (Zenotec mini, Wieland), sintered (Infire HTC speed, Dentsply Sirona), and finished with abrasive paper (320-, 500-, 800-, 1000-, 2000-grit silicon carbide papers) and diamond paste with 0.5 μm particles (Diamond Polish Mint, Ultradent) for 60 secs by the same operator. Two layers of glaze were applied to simulate fabrication processes of the restoration. The ceramic discs were cleaned ultrasonically in distilled water for 15 min to remove debris and air dried for 20 secs before immersion in the simulated gastric acid solution, prepared according to Hunt and McIntyre (26) with HCl 0.06 M (0.113% solution in deionized water, pH 1.2).

Each disc was immersed individually in 2 mL of the simulated gastric acid solution and maintained at 37 °C during the periods evaluated: at baseline and after 18 h 25 min, 46 h 2.5 min, and 92 h 5 min of immersion. This was equivalent to 2, 5, and 10 years respectively of exposure based on a patient with bulimia or GERD regurgitating 3 times a day (27) with an estimated contact time of the acidic gastric juice with the restorations of 30 secs (28). The pH of the simulated gastric acid solution was monitored, and the solution was changed daily. Once the immersion periods were completed, the discs were removed and cleaned ultrasonically with distilled water and air dried for 20 secs. All procedures were performed by the

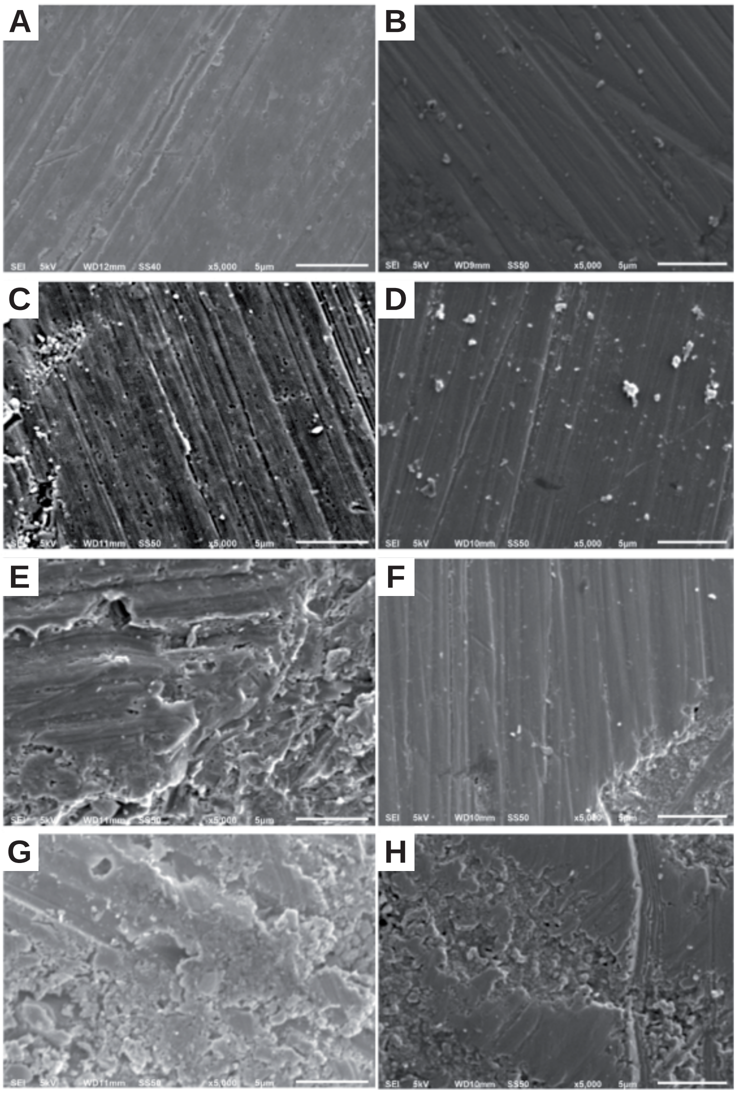

Fig. 1. Representative SEM micrographies of surface change in lithium disilicate glass-ceramic discs at baseline (A) and after 18 h 25 min (B), 46 h 2.5 min (C), 92 h 5 min (D) of immersion and in ZrO_2 discs at baseline (E) and after 18 h 25 min (F), at 46 h 2.5 min (G) and at 92 h 5 min (H) of immersion in simulated gastric acid solution.

same researcher (N.M.M) and all the observations and measure were made by the operator of each of the equipment used.

Surface morphology analysis

The glass-ceramic discs from each group of immersion times were randomly selected (n =5) for morphological assessment under Scanning electron microscopy (SEM, JSM-6510, JEOL). The discs were critically dried at 30 min intervals with alcohol at 25, 40, 50, 75, 80, 90, and 100% and were gold sputter-coated (SPI-SUPPLIES).

Table 1. Materials used and composition

Brad name	Manufacturer	Composition	Temperature firing (ºC)
NexxZr S (Zirconium Oxide)	Sagemax Biocer-amics, Inc. (USA)	3Y-TZP (Zirconium dioxide)	920
Upcera (Lithium disilicate glass dental ceramic)	Shenzhen Upcera Dental Technology Co., Ltd (China)	Silicon dioxide (58.5-72.5%), lithium oxide (13-15%), potassium oxide (3-5%), other oxides (7.5-25%)	725

Surface microhardness testing

Surface microhardness were evaluated with a Vickers hardness tester (Digital Microhardness Tester, Sinowon) with a 100 g load for 10 secs. Five indentations per test were carried out for each glass-ceramic disc (n=30), and the average was considered as the surface microhardness. Mean differences in surface microhardness values (N/mm^2) were calculated.

Surface roughness measurements

The surface roughness analysis in the glass-ceramics discs (n=5) were carried out through Atomic force microscopy (AFM, Dimension Edge, Bruker) along with the NanoScope Analysis ver. 1.40r1 software, all observations were performed by the same examiner, and the measurements were repeated blindly and at random. The AFM images were obtained in the tapping mode by using an antimony (n) doped Si probe (model TESPA) with a resonance frequency of 320 kHz and spring constant of k=42 N/m. The roughness parameters evaluated were Rq (maximum roughness depth), Rz (maximum roughness), and Ra (arithmetical average of surface heights), the scan areas were 20×20 µm and 50 readings were collected from the surface of each ceramic disc.

Statistical analysis

The statistical analysis was carried out with the IBM SPSS statistics v. 22 program. The Shapiro-Wilk test and the test of homogeneity of variances with the Levene statistical test were performed. A two-way analysis of variance (ANOVA) was performed and the Tukey's post hoc test was used to search for statistically significant differences in the mean of the study variable among the study groups (α=0.05).

RESULTS

Surface microtopography analysis

A smooth and homogeneous topographic surface was observed in

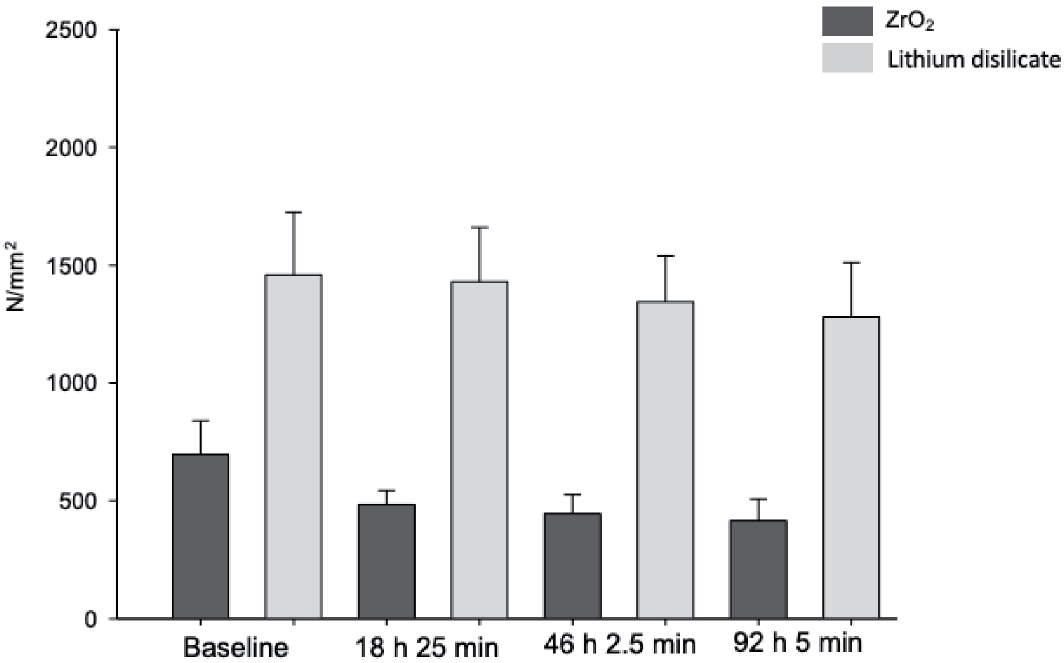

Fig. 2. Surface microhardness values (N/mm2) at baseline and after 18 h 25 min, 46 h 2.5 min and 92 h 5 min of immersion in simulated gastric acid solution

the lithium disilicate glass-ceramic discs at baseline, without depressions and with a continuous linear pattern produced by the polishing of the surface (Figure 1A). After 18 h 25 min, the linear pattern continued with minimal reliefs and depressions and with microporosities distributed on the surface (Figure 1B). At 46 h 2.5 min, the linear pattern of the surface disappeared, and an irregular homogeneous degradation was observed, with increases in the roughness of different dimensions (Figure 1C). In the final immersion period of 92 h 5 min, the irregular, rough, and porous degradation pattern continued to be observed in localized areas (Figure 1D). A surface with a linear pattern and without microporosities was observed in the ZrO_2 ceramic discs at baseline (Figure 1E). After 18 h 25 min, a topographic pattern similar to that at baseline continued to be observed, without any surface changes (Figure 1F). At 46 h 2.5 min, localized areas of microroughness and microporosities were observed on the surface, with some reliefs and depressions in the surface topography (Figure 1G). In the final immersion period of 92 h 5 min, the loss of the linear pattern was observed with localized areas of irregular and smooth surfaces (Figure 1H).

Surface microhardness testing

The mean microhardness values decreased for both lithium disilicate and ZrO_2 discs with increased immersion periods in the simulated gastric acid solution. Microhardness was higher for both ceramics at baseline and lower on the lithium disilicate discs than in the ZrO_2 discs. A statistically significant difference was found between the mean microhardness values of the lithium disilicate and ZrO_2 discs (P<.05) at all time points. At baseline, the microhardness values for the lithium disilicate discs were 697.6 (141.4) N/mm^2, at 18 h 25 min 492.1 (60.6) N/mm^2, at 46 h 2.5 min 48.6 (81.4) N/mm^2, and in the final immersion period of 92 h 5 min, microhardness values of 444.8 (90.9) N/mm^2, a significant difference was found between baseline and

Table 2. Microhardness values for lithium disilicate and ZrO_2 discs

Periods	Lithium disilicate			
	Microhardness N/mm² (SD)	Rq µm (SD)	Ra µm (SD)	Rz µm (SD)
Baseline	697.62 (141.43)	0.069 (0.02)	0.053 (0.01)	0.650 (0.13)
18 h 25 min	492.14 (60.62)*	0.288 (0.07)*	0.206 (0.06)*	0.729 (0.12)*
46 h 2.5 min	481.66 (81.43)*	0.215 (0.03)*	0.151 (0.02)*	0.43 (0.08)*
92 h 5 min	444.81 (90.91)*	0.20 (0.10)*	0.135 (0.069)*	0.345 (0.09)*
Periods	**ZrO_2**			
	Microhardness N/mm² (SD)	**Rq µm (SD)**	**Ra µm (SD)**	**Rz µm (SD)**
Baseline	1502.43 (265.70)	0.050 (0.01)	0.040 (0.01)	0.510 (0.09)
18 h 25 min	(1466.32) 230.30)	0.197 (0.58)*	0.086 (0.02)*	0.744 (0.11)*
46 h 2.5 min	1352.54 (194.80)	0.047 (0.01)	0.044 (0.01)	0.338 (0.05)*
92 h 5 min	1269.21 (230.70)	0.035 (0.01)	0.049 (0.01)	0.200 (0.68)*

Mean ± Standard Deviation (SD) for surface ceramic roughness values (µm) Rq, Ra and Rz and (N/mm^2) for microhardness surface values before and after immersion in simulated gastric acid solution. *Indicates statistically significant difference between lithium disilicate and ZrO_2 discs at baseline versus the immersion time points (vertical). P <.05 was accepted as significance level.

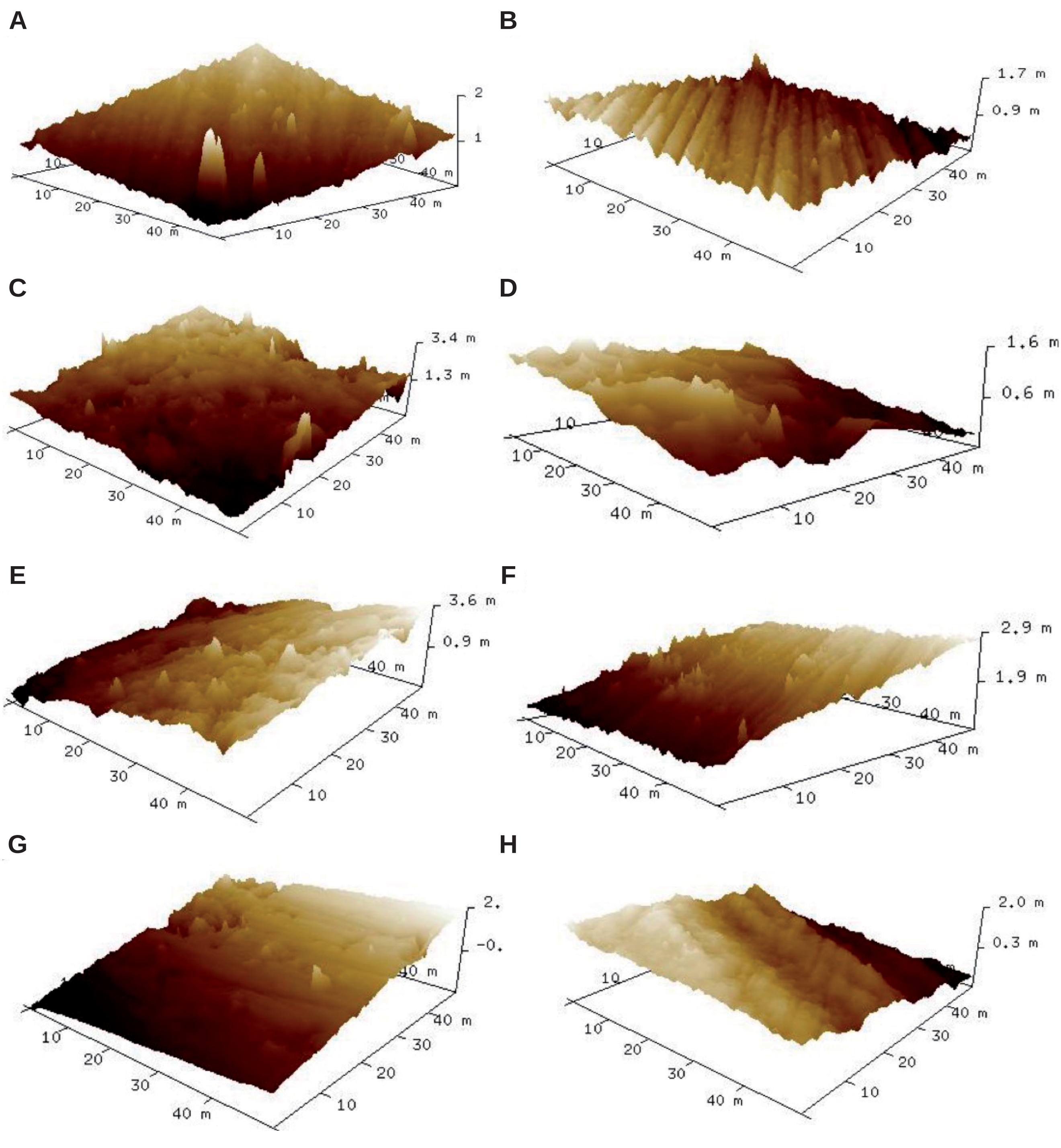

Fig. 3. Representative AFM pictures of surface change lithium disilicate glass-ceramic discs at baseline (A) and after 18 h 25 min (B), 46 h 2.5 min (C), 92 h 5 min (D) of immersion, and ZrO_2 discs at baseline (E) and after 18 h 25 min (F), 46 h 2.5 min (G), 92 h 5 min (H) of immersion in simulated gastric acid solution

all time points ($P<.05$). For the ZrO_2 discs, the value obtained at baseline was 1502.4 (265.7) N/mm^2, at 18 h 25 min 1466.3 (230.3) N/mm^2, at 46 h 2.5 min 1352.5 (194.8) N/mm^2, and at 92 h 5 min 1269.2 (230.7) N/mm^2, the values between baseline and all time points were statistically similar ($P>.05$) (Table 2, Figure 2).

Surface roughness measurements

Table 2 shows the means, standard deviations, and statistical comparison among the values of the roughness parameters (Rq, Ra, Rz) of lithium disilicate and ZrO_2 discs. The lithium disilicate discs had higher roughness values than the ZrO_2 discs at all time points of immersion, and the values of the parameters evaluated were higher after 18 h 25 min than at baseline, indicating that the roughness increased after the first period of immersion. Nevertheless, after 46 h 2.5 min and 92 h 5 min of immersion in simulated gastric acid, the lithium disilicate and ZrO_2 roughness decreased (Figure 3A-3H).

DISCUSSION

The surface characteristics of ceramic materials under acidic conditions in the oral cavity are affected by the low pH and the degradation of the microtopography according to the chemical composition of the dental ceramics (29). The objective of this *in vitro* study was to evaluate the surface degradation of lithium disilicate and ZrO_2 after immersion in simulated gastric acid solution, which has been reported to cause more severe damage than that generated by an acidic diet (30). The results obtained in this study showed that both lithium disilicate and ZrO_2 ceramic underwent modifications in their surface microtopography after 8 h 25 min, 46 h 25 min, and 92 h 5 min of immersion in simulated gastric acid. ZrO_2 showed less porosity after all time points of immersion versus lithium disilicate. Also, the microhardness values were higher for ZrO_2, and the average roughness values (Rq, Ra, Rz) were lower compared with lithium disilicate glass-ceramic. The null hypothesis was rejected because significant changes in surface degradation were found in the surface microtopography of dental ceramics after immersion in simulated gastric acid.

The effect of acid solutions on the chemical degradation of ceramic surfaces after an acidic challenge has been reported, and the assumption has been made that this altered the surface texture (31, 32). However, it has not been previously determined whether a smooth surface promotes resistance to chemical corrosion after an acidic challenge. The present *in vitro* study simulated the corrosive process with HCl (pH 1.2) at 37ºC, and we have demonstrated that the simulated gastric acid solution changed the topography of the lithium disilicate and ZrO_2 ceramics after 8 h 25 min, 46 h 25 min, and 92 h 5 min, equivalent to 2, 5, and 10 years of use in a patient suffering from bulimia or GERD (24, 25). The changes in the surface microtopography were more evident in the lithium disilicate glass-ceramic, possibly because of its chemical composition (33, 34). The effects on the ceramic surfaces will also depend on the acid concentration and immersion time and temperature (35, 36).

The average values of surface microhardness obtained in this study were lower in lithium disilicate than in ZrO_2, possibly because of increased surface changes. The modifications in the surface microhardness of the ceramics changes the flexural strength, generating surfaces with lower compressive strength (37-39). The altered microhardness may also be caused by chemical composition, amount of dopant, size, arrangement of polycrystalline grains, temperature, pH, and sintering (40). Kukiattrakoom *et al.,* (37) reported that potassium, aluminum, and silicon decreased significantly in the chemical composition of dental ceramics after an acidic challenge, reducing their microhardness values.

The results obtained in the present study were consistent with those of Kulkarni *et al.*, (25) who compared the exposure of dental ceramics to artificial gastric acid combined with toothbrush abrasion. Their results showed that monolithic ZrO_2 had better mechanical properties than feldspathic porcelain or lithium disilicate glass-ceramic, with no significant changes in color, translucency, and surface roughness. Also, the results of the present study were consistent with those of Sulamain *et al.*, (24) who reported that lithium disilicate glass-ceramic had increased weight loss after 96 h immersed in 0.06 M of HCl at 37ºC compared with ZrO_2, the increased weight loss was explained by the different microstructures of lithium disilicate and ZrO_2 ceramic. The lithium disilicate glass-ceramic was found to be more vulnerable to chemical corrosion degradation, and the ZrO_2 ceramic showed evidence of resistance, with a smooth surface and no change in initial roughness. Moreover, the crystalline phase of lithium disilicate glass-ceramic appears to dissolve at a slower rate than the glass matrix, creating a rougher surface in the first hours of incubation in simulated gastric acid. Also, Vasiliu *et al.*, (38) explain that the different results reported in articles evaluating ceramic surfaces in acidic environments may be influenced by the pH of the acid solution. A higher acidity has a greater capacity to dissolve the glass phase and affect the chemical composition of the ceramics. In the present study, HCl with a pH of 1.2 was used, and the longest incubation time was 92 h 5 min. Sulaiman *et al.*, (24) used HCl with a pH of 1.2 for 96 h to simulate clinical exposure over 10 years, Backer *et al.*, (39) used gastric juice with a pH of 1.2 for 6 h and 18 h to simulate 2 and 8 years of exposure to vomiting, Matsou *et al.*, (40) exposed dental ceramics to an acidic solution with a pH of 3.8 for 24 h, and Švančárková *et al.*, (41) determined that the pH of the acidic solution significantly affected the ion leaching process; all these studies used different parameters but all reported that the surfaces of dental ceramics are affected by acidic solutions. Moreover Gulakar *et al.*, conclude that the exposure of gastric acid affects the hardness and flexural strength properties of dental restorative ceramic materials (42).

The SEM images obtained in the present study showed scratches from the polishing of the surface of the lithium disilicate and ZrO_2 ceramic before immersion in the simulated gastric acid solution. After all immersion times, these scratches appeared less sharp, which may explain the decrease in the roughness values obtained. After the final period of immersion of 92 h 5 min, the ZrO_2 ceramic appeared smoother, with microporosity areas caused by the corrosive process. In the lithium disilicate, the microporosities were observed at all time points. Cruz *et al.*, (43) concluded that the simulated gastric juice significantly decreased the roughness of monolithic esthetic restorations after acid exposure in HCl at a pH of 1.2, changing the surface microtopography, consistent with the results obtained in the present study. After 46 h 25 min and 92 h 5 min of immersion in simulated gastric acid, the roughness values decreased. Alnasser *et al.*, (33) reported findings contrary to those of our study that lithium disilicate and ZrO_2 showed no statistically significant change in surface roughness after exposure to HCl for 45 h and 91 h at 37ºC. In contrast, Milleding *et al.*, (44) reported that the composition and microstructure of the restorative material were the main reasons for surface roughness changes. Other important factors to be considered include the patient's saliva and diet and the temperature of the oral cavity, which can vary from 0°C to 67°C.

The topographic pattern exhibited by dental ceramics when exposed to acidic challenges at times equivalent to 1 year has been reported to result in smoother surfaces being generated over longer exposure periods, consistent with the findings of the present study (45). The SEM images of ZrO_2 showed localized microporosities, grooves, and smooth areas from corrosive degradation. However, Osama *et al.*, (45) reported that despite finding an increase in the surface roughness of the ceramics, the surface area of the ZrO_2 was not significantly affected; however, they used a single incubation time of 96 h. This was in agreement with Harryparsad *et al.*, (46) who reported a linear relationship between surface area and surface roughness, determining that a longer period of immersion time results in a smoother material.

Finally, an adequate polishing system that achieves a smooth and homogeneous surface to avoid changes in the surface of the ceramics should be chosen, since an acidic environment will cause micromorphological changes. Before selecting the restorative material, the chemical properties of restorative materials should be evaluated, as chemical stability will largely determine restoration longevity. Limitations of the present study included that only two ceramics were evaluated and that its *in vitro* design may not have fully replicated the complex intraoral conditions that include the capacity of saliva to compensate for changes in pH and temperature in the oral cavity.

CONCLUSION

According to microscopy results lithium disilicate and ZrO_2 exhibited significant changes in the microstructures of the surface topography, with increased porosity and smoother surfaces after 92 h 5 min exposure to HCl. Lithium disilicate glass-ceramic showed a changed surface topography and was affected more than ZrO_2. The microhardness values and roughness parameter decreased after all times points in both ceramics.

CONFLICTS OF INTERESTS

The author declares no potential conflicts of interest with respect to the authorship and/or publication of this article.

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None

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