

EFFECT OF DIFFERENT ACIDITY CONDITIONS ON THE STRUCTURAL PROPERTIES OF ZIRCONIA CERAMICS

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ABSTRACT

In this study, two groups of dental ceramics exposed to acidic conditions were evaluated: Group 5Y-TZP (Zirconia stabilized by 5mol% Y₂O₃ and obtained experimentally by chemical synthesis), and Ceramill® Zirconia Group, commercially acquired. Both materials were compacted in the form of pellets, using a mold with a diameter of 6 mm and a hydraulic press, applying a uniaxial load of 3 tons for 60 seconds. After the pressing stage, the pellets were sintered at 1500°C for 2 hours. All the tablets produced (N=30) were weighed on an analytical scale and then divided according to the acid immersion solutions into 4 media: SA (artificial saliva), SL (orange juice), CC (cola [Coca-Cola] and C (coffee). The tablets were stored in an oven at 37°C for 72 hours and after the end of this time they were washed in water and dried on absorbent paper. Their masses were again measured in order to evaluate the possible loss of mass during storage. The effect of the acid medium on the crystallinity of the ceramics was evaluated by X-ray diffraction (XRD) analysis, while the chemical bonds were evaluated by Infrared Vibrational Spectroscopy (FTIR). The XRD analyses indicated that, regardless of the acidic medium, none of the ceramic groups observed a change in the crystallinity pattern. The FTIR spectra indicated the presence of new absorption bands, possibly due to the permeability of the acid solutions on the surface of the ceramic samples.

Keywords: Zirconia. Bioceramics. Acid degradation. Crystallinity.

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INTRODUCTION

Dental aesthetics has become increasingly important in contemporary society (MOTRO *et al.*, 2012; WRIGHT *et al.*, 2004). The demand for ceramic restorations has increased due to their improved aesthetics and greater durability when compared to other restorative materials (dos SANTOS *et al.*, 2016; DELLA BONA, 2009; SARAC *et al.*, 2006). However, the development of restorations that allow mimicking the natural appearance of teeth is still one of the greatest clinical challenges in dentistry (NOGUEIRA *et al.*, 2013; SON *et al.*, 2010; BREWER *et al.*, 2004). Thus, ceramics must reproduce the natural characteristics of the teeth, including color, surface texture, and translucency, to achieve aesthetic levels that accurately correspond to the natural structure of the tooth (NOGUEIRA *et al.*, 2013; SON *et al.*, 2010; HARIANAWALA *et al.*, 2014).

Due to their superior mechanical properties, biocompatibility with human tissues, and improved optical properties, yttria-stabilized tetragonal zirconia (Y-TZP) ceramics have become increasingly important in recent decades as biomaterials for restorations, being widely used commercially in the manufacture of crowns, fixed dental prostheses, and dental implants (MIYAZAKI *et al.*, 2013; CAMPOSILVAN; TORRENTS; ANGLADA, 2014; DENRY and KELLY, 2014; PRESENDA *et al.*, 2015; TONG *et al.*, 2016; ZHAO; ZHU; LUO, 2016). Recently, Y-TZP ceramics have undergone microstructure and composition modifications to improve translucency, however, without causing damage to mechanical properties (ZHANG *et al.*, 2018; MATSUZAKI *et al.*, 2015).

Regarding the properties of ceramic materials, it is known that the chemical composition of the materials, the manufacturing technique, the application method, among other factors affect the properties (DELLA BONA, 2009). The properties can also be affected when the ceramic is subjected to a process of surface degradation, which occurs when the surface is exposed to aqueous solutions and/or changes in pH (ESQUIVEL-UPSHAW *et al.*, 2013; KUKIATTRAKOON *et al.*, 2010; KUKIATTRAKOON *et al.*, 2009; SWAIN, 2014; CCAHUANA *et al.*, 2010). In addition, this process can be intensified by temperature variations (ESQUIVEL-UPSHAW *et al.*, 2013; KUKIATTRAKOON *et al.*, 2009) and has undesirable consequences for restoration, for example the accumulation of biofilm and restorations with changes in color and appearance (KUKIATTRAKOON *et al.*, 2010; MINAMI *et al.*, 2007).

Many *in vitro* studies have shown that commonly used beverages such as coffee, tea, red wine, cola soft drinks, and fruit juice can cause significant changes in the color of restorative materials (REN *et al.*, 2012; GULER *et al.*, 2005; FUJITA *et al.*, 2006; ASHCROFT *et al.*, 2008; TUNC *et al.*, 2009; FONTES *et al.*, 2009). However, studies



evaluating the influence of acidic diets on the crystal structure of ceramics are scarce. In view of the above, this *in vitro* study evaluated the alterations of the crystal structure and chemical bonds in ceramics of 5Y-TZP (Zirconia stabilized by 5mol% of Y₂O₃, obtained experimentally by chemical synthesis process) and Ceramill® zirconia, commercially acquired, when exposed to potentially acidic solutions. The null hypothesis was that the proposed acid solutions would not be able to alter the properties of the evaluated ceramics.

MATERIALS AND METHODS

CHEMICAL SYNTHESIS OF EXPERIMENTAL 5Y-TZP CERAMICS

The chemical synthesis process was initiated by weighing, on an analytical scale, the masses of zirconium butoxide and yttrium nitrate hexahydrate necessary for the reaction. Separately, citric acid was dissolved in distilled water and kept under stirring and heating at 60°C for 30 min. The citric acid solution was added to the vial containing the reagents and the mixture was kept in stirring and heating at 90°C for 1 h. Only then was ethylene glycol added, the temperature being raised to 130°C and the resulting solution kept in stirring for 1 h at this temperature.

Then, the solution was preheated in a muffle furnace at 350°C for 3 h, followed by heat treatment at 800°C for 6 h in order to promote the formation of the crystalline phases.

SELECTION OF COMMERCIAL CERAMIC SYSTEM

In this study, the pre-sintered ceramic block of the commercial system Ceramill® zirconia (Amangirrbach, Koblach – Austria) introduced in the Dental market was used for the manufacture of infrastructures by CAD/CAM (*Computer Aided Design / Computer Aided Manufacturing*).

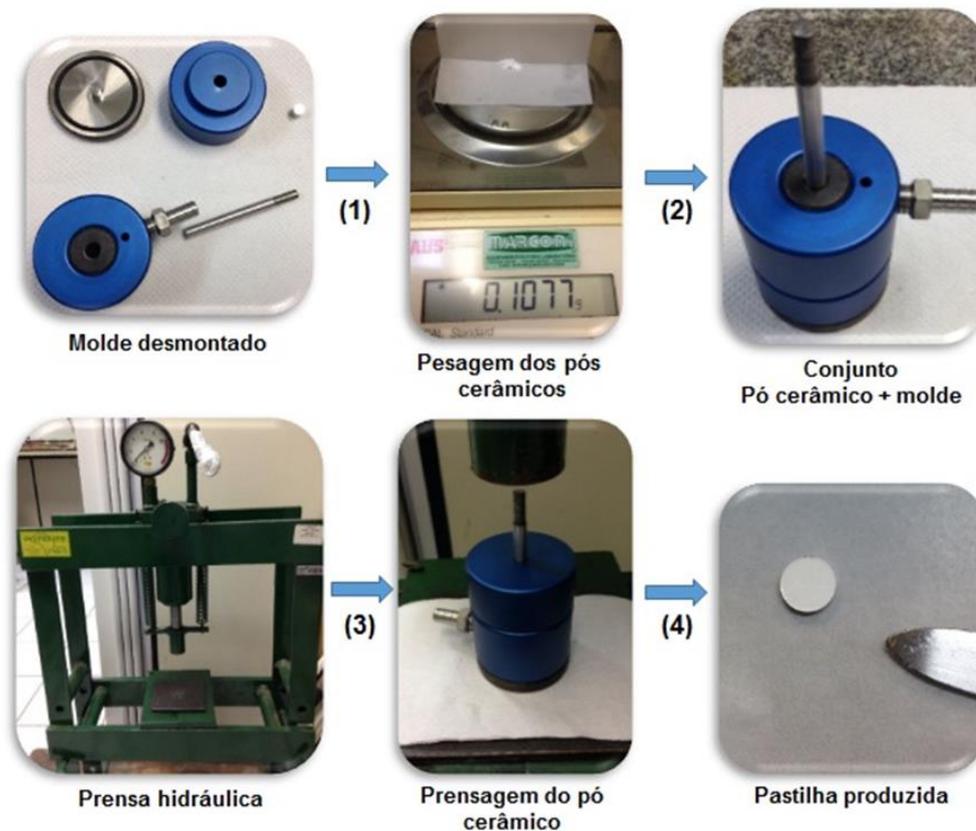
The product Ceramill® zirconia is described as a super-high translucency zirconium oxide. Indicated for monolithic or anatomically reduced restorations in the anterior region, as well as bridges of up to 3 elements in the molar area. This ceramic combines the excellent processing properties of zirconium oxide with a translucency comparable to that known so far only in lithium disilicate. The result is a highly aesthetic dental prosthesis of long-lasting strength and stability.

The pre-sintered Ceramill® zirconia block was cut with a diamond blade coupled to a precision cutter with a cutting speed of 400 rpm under abundant water cooling, in order to obtain bar-shaped specimens. The sectioned specimens were disaggregated with the aid of a mortar and pestle.

UNIAXIAL PRESSING OF CERAMIC MATERIALS AND SINTERING

The powders of the experimental ceramics 5Y-TZP and commercial ceramics Ceramill® zirconia were pressed uniaxially in a stainless steel mold for the manufacture of pellets. The inserts were pressed according to the conditions: applied load of 3 tons and load application time equal to 60 seconds. From the pressing, pellets of 6 mm in diameter by 1.45 mm in height were obtained, and approximately 0.10 g in mass. Figure 1 illustrates the sequence of steps in the manufacture of the tablets.

Figure 1 - Sequence of steps for making ceramic tiles.



Source: Authored by the author.

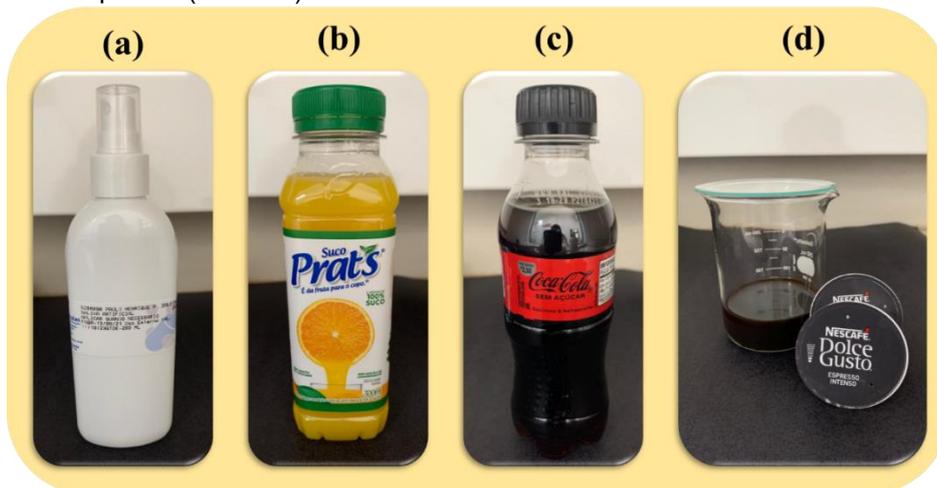
The pellets produced (N = 30) were taken to an Amanngirrbach furnace, model Ceramill® therm 3 following the guidelines indicated by the furnace manufacturer: sintering temperature 1500°C and dwell time of 2 h. After the sintering stage, the pellets were weighed on an analytical scale.

STUDY OF EXPOSURE TO DIFFERENT PH CONDITIONS

After weighing the tablets, the samples of each of the systems, 5Y-TZP and Ceramill® zirconia, were divided according to the immersion solutions into 5 groups (n = 3): group GC (Control), group SA (artificial saliva), group SL (orange juice), group CC (cola

[Coca-Cola]) and group C (coffee). Figure 2 illustrates the image of the solutions used in this study.

Figure 2 - Solutions used in the acid degradation test: (a) Artificial saliva (Bauru Formulas Laboratory); (b) Whole orange juice without sugar and preservatives (Prat's); (c) Coca-Cola sugar-free soft drink (FEMSA); (d) Dolce Gusto intense espresso (Nescafé).



Source: Authored by the author.

The samples of the GC group were stored in a falcon tube, in the absence of liquid medium, in an incubator at 37°C for 72 hours. In turn, the samples of the SA, SL, CC and C groups were immersed in a volume of 5 mL of their respective solutions and, likewise, were stored in falcon tubes and kept in an incubator at 37°C for 72 hours.

The description of the groups studied is summarized in Table 1.

Table 1 - Groups submitted to the acid degradation test in an oven at 37°C and a stay of 72 h.

Group	Description
5Y-TZP_GC	Experimental 5Y-TZP wafers stored in absence of solution
5Y-TZP_SA	Experimental 5Y-TZP pellets stored in artificial saliva
5Y-TZP_SL	Experimental 5Y-TZP lozenges stored in orange juice
5Y-TZP_CC	Experimental 5Y-TZP pellets stored in Coca-Cola
5Y-TZP_C	Experimental 5Y-TZP pellets stored in coffee
Ceramill_GC	Ceramill zirconia wafers® stored in the absence of solution
Ceramill_SA	Ceramill® zirconia tablets stored in artificial saliva
Ceramill_SL	Ceramill zirconia lozenges® stored in orange juice
Ceramill_CC	Ceramill zirconia lozenges® stored in Coca-Cola
Ceramill_C	Ceramill® zirconia tablets stored in coffee

Source: Authored by the author.

After the end of the storage time, the samples were washed in distilled water, dried on absorbent filter paper and taken to drying in an oven at 37°C for 5 h. Their masses were again determined on an analytical scale in order to evaluate the possible loss of mass during the acid degradation process. Then, the wafers were characterized for changes in the crystal structure and chemical bonds.



CHARACTERIZATIONS

Determination of pH

The solutions used in the acid degradation test, i.e., artificial saliva, orange juice, Coca-Cola soft drink and coffee were evaluated for their pH (hydrogen potential).

pH determination was performed using a pH meter (pH meter) (Akso, model AK95) previously calibrated with known pH buffer solutions (pH = 4, pH = 7 and pH = 10). The analysis is based on the measurement of the potential difference between two suitable electrodes, immersed in the solution under examination. One of these electrodes is sensitive to hydrogen ions and the other is the reference electrode, with constant potential. The measurement was carried out in a room with a controlled temperature of 22°C. The measurements were performed in triplicate.

Mass Loss

The mass of each pellet was determined before and after the acid degradation test in order to determine the possible mass loss that occurs in the process. Thus, the percentage of mass loss ($\% \Delta m$) was calculated using equation (1), where m_i corresponds to the mass before the degradation test and m_f to the mass after degradation.

$$\% \Delta m = \frac{m_i - m_f}{m_i} \quad (1)$$

X-ray diffraction

The wafers were examined by X-ray diffraction (XRD) using a Rigaku DMAX Ultima+ X-ray diffractometer. The experimental conditions were: interval from 20° to 80° with increment $\Delta 2\theta = 0.02^\circ$, scanning speed of 2°/min, CuK radiation α , wavelength 1.54 Å, power 40 kV, current 20 mA. The data obtained from XRD were processed by *software* using the ICDD (*International Centre for Diffraction Data*) database.

Fourier Transform Infrared Spectroscopy

The samples were examined by Fourier Transform Infrared Spectroscopy (FTIR) using a Bruker Fourier Spectrophotometer, model Vertex-70. The analyzed range was 4000-400 cm^{-1} , 64 accumulation scans, and 4 cm^{-1} resolution. The position of the main absorption bands was indicated in the spectra in order to facilitate the attribution of the identified chemical bonds.



RESULTS AND DISCUSSION

PH DETERMINATION

Table 2 summarizes the pH data of the solutions used in the degradation study.

Table 2 - pH data of the solutions analyzed in the acid degradation study.

Group	ph
Saliva artificial	5.04 ± 0.01
Whole Orange Juice	3.61 ± 0.02
Sugar-free Coca-Cola soda	3.18 ± 0.01
Intense espresso café	5.29 ± 0.00

Source: Authored by the author.

The pH or hydrogen potential of a solution is represented on a scale ranging from 0 to 14 and this scale measures the acidity or basicity of a solution. Thus, if the pH value is equal to 7 (pH of water), the medium of the solution (or liquid) will be neutral, while if the pH is less than 7, it will be acidic, and if it is greater than 7, basic (ATKINS, 2011). In view of the above, the characterization of the pH values of the solutions involved in the degradation study aimed to evaluate the erosive potential of the solutions.

To facilitate the discussion of the results obtained, the pH data were classified as: potentially erosive (solutions with pH lower than 5.5) and potentially non-erosive (solutions with pH equal to or greater than 5.5), following the attribution made by LIMA *et al.* (2005) which considers that the dissolution of tooth enamel begins when the pH on the tooth surface reaches values below 5.5 (PRISTA, 2015). Thus, according to the data presented in Table 2, the pH values of the solutions ranged from 3.18 (Coca-Cola) to 5.29 (Coffee) and, therefore, all the solutions used in the study were classified as potentially erosive.

MASS LOSS

In view of the pH data obtained, it was decided to evaluate the possible loss of mass (% Δ m) of the ceramic tiles associated with the acid degradation process. The data on mass loss are presented in Table 3.

It was found that all the pellets submitted to the degradation test showed a loss of mass. However, the highest values of mass loss correspond to the groups of ceramic tablets stored in Coca-Cola Soda medium, i.e., groups 5Y-TZP_CC and Ceramill_CC, with losses of 0.43% and 1.50%, respectively.

According to DRUMMOND *et al.* (2019), Coca-Cola Soft Drink has in its composition the phosphoric acid content (H₃PO₄) of 0.059 g/ 100 mL of soft drink. Considering the acid force, that is, the force that determines the degree of ease with which acids ionize in water and other solvents, phosphoric acid is defined as a moderate or semi-strong acid, while

citric acid (C₆H₈O₇), present in orange juice, is considered a weak acid (ATKINS, 2011). Thus, it is believed that the property related to acid strength justifies the fact that orange juice, which presented pH = 3.61, did not prove to be potentially erosive in relation to Coca-Cola Soft Drink.

Table 3 - Percentage of mass loss after acid degradation.

Group	% δ m
5Y-TZP_GC	0.00 \pm 0.00
5Y-TZP_SA	0.05 \pm 0.02
5Y-TZP_SL	0.06 \pm 0.03
5Y-TZP_CC	0.43 \pm 0.14
5Y-TZP_C	0.05 \pm 0.03
Ceramill_GC	0.01 \pm 0.01
Ceramill_SA	0.06 \pm 0.02
Ceramill_SL	0.09 \pm 0.05
Ceramill_CC	1.50 \pm 0.10
Ceramill_C	0.08 \pm 0.05

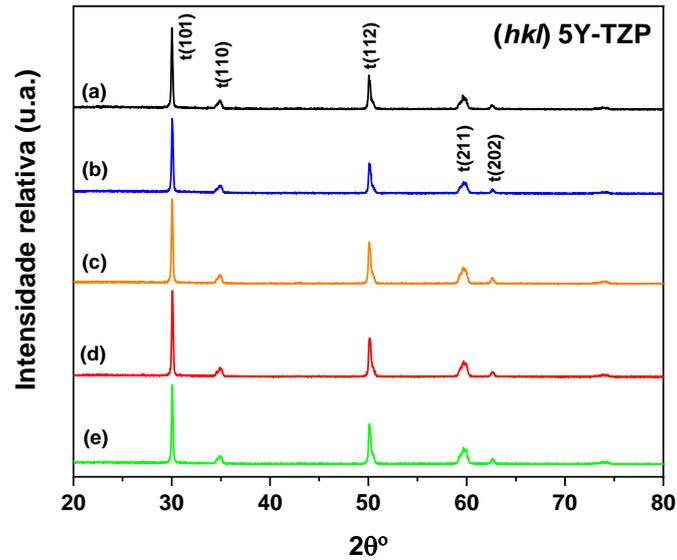
Source: Authored by the author.

X-RAY DIFFRACTION

The XRD characterization made it possible to identify the crystalline phases formed, as well as the effect of acid degradation on the crystallinity of zirconia ceramics. Figure 3 illustrates the XRD patterns of the 5Y-TZP groups studied, while Figure 4 illustrates the patterns of the Ceramill® zirconia groups.

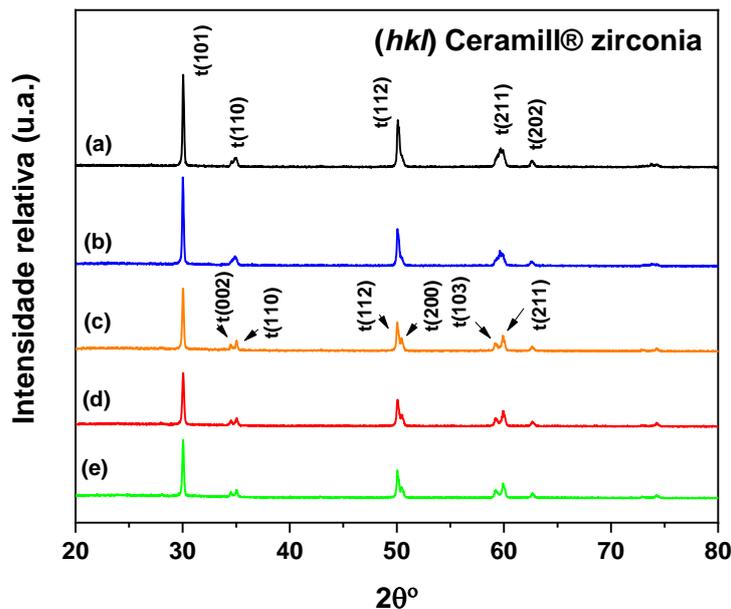
The analysis of the crystalline phases, as well as the indication of the crystallographic planes (*hkl*) in Figures 3 and 4 was performed using as reference the crystallographic file 79-1769 of the PCPDFWIN database of the *International Centre for Diffraction Data* (ICDD), illustrated in Figure 5. The crystallographic record was chosen based on the crystal structure of zirconia (ZrO₂) evidenced in the diffraction patterns obtained. According to this factsheet, the pattern represents the tetragonal cell of ZrO₂.

Figure 3- Diphratograms of roots X two groups 5Y-TZP subjected to acid degradation: (a) 5Y-TZP_GC; (b) 5Y-TZP_SA; (c) 5Y-TZP_SL; (d) 5Y-TZP_CC; (e) 5Y-TZP_C.



Source: Authored by the author.

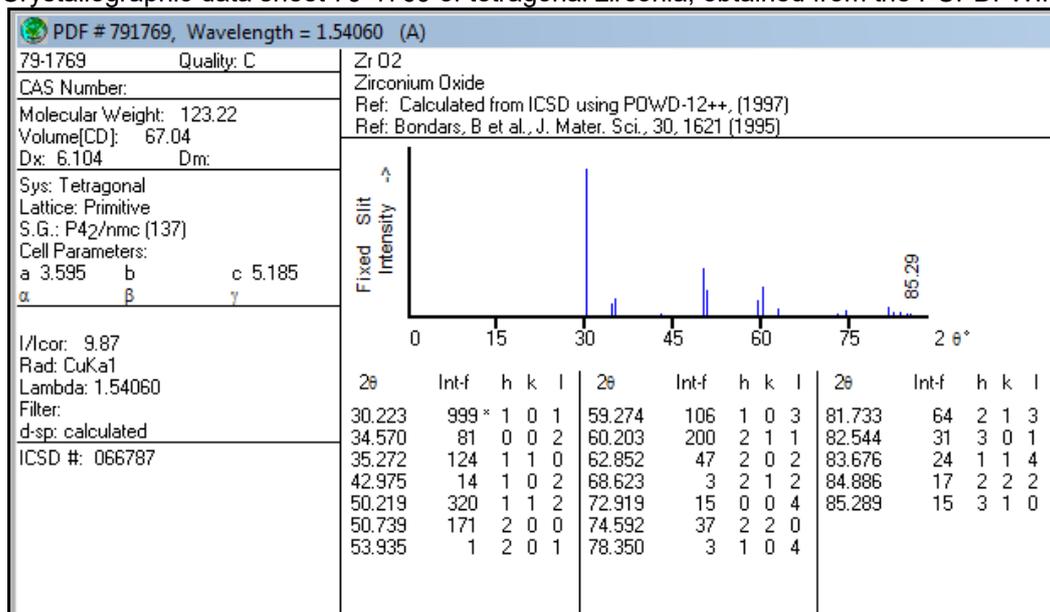
Figure 4- X-ray diffractograms of Ceramill® zirconia groups subjected to acid degradation: (a) Ceramill_GC; (b) Ceramill_SA; (c) Ceramill_SL; (d) Ceramill_CC; (e) Ceramill_C.



Source: Authored by the author.

The XRD data indicated that both control groups, 5Y-TZP_GC and Ceramill_GC, which were not exposed to the acidic environment, showed diffraction peaks associated with the tetragonal crystalline phase of zirconia. In addition, it was found that regardless of the acid solutions used in this study, they did not promote the change in the crystallinity pattern of the ceramics.

Figure 5- Crystallographic data sheet 79-1769 of tetragonal zirconia, obtained from the PCPDFWIN database.

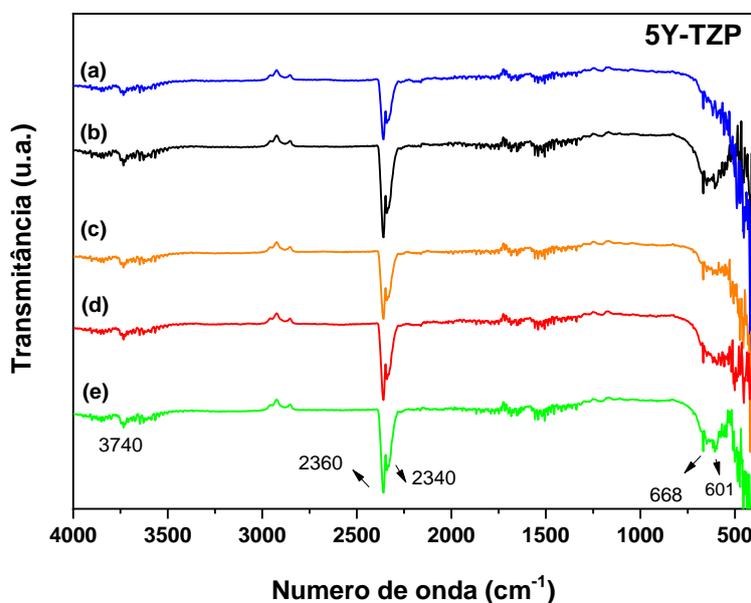


Source: Authored by the author.

FOURIER TRANSFORM INFRARED SPECTROSCOPY

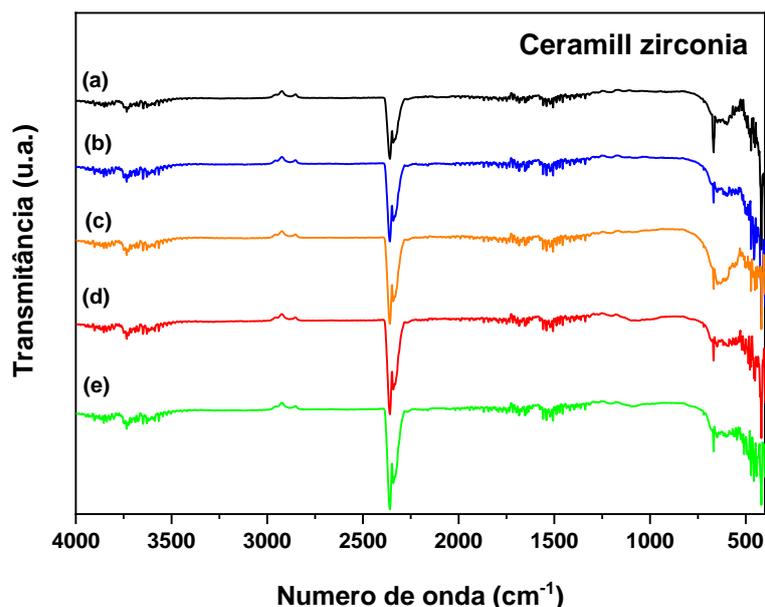
The results obtained by FTIR from the analysis of the chemical bonds present in zirconia, 5Y-TZP and Ceramill® zirconia ceramics, after the acid degradation test are illustrated in Figures 6 and 7, respectively.

Figure 6- FTIR spectra of two groups of 5Y-TZP subjected to acid degradation: (a) 5Y-TZP_GC; (b) 5Y-TZP_SA; (c) 5Y-TZP_SL; (d) 5Y-TZP_CC; (e) 5Y-TZP_C.



Source: Authored by the author.

Figure 7- FTIR spectra of Ceramill zirconia groups subjected to acid degradation: (a) Ceramill_GC; (b) Ceramill_SA; (c) Ceramill_SL; (d) Ceramill_CC; (e) Ceramill_C.



Source: Authored by the author.

Table 4 summarizes the attributions of the chemical bonds identified in the zirconia ceramics studied.

Table 4 - Data obtained by FTIR for the groups of zirconia ceramics submitted to the degradation test. Abbreviations: ν , bond stretch, and δ , angular deformation.

Wavenumber (cm^{-1})										Connect ion
5Y-TZP					Ceramill zirconia					
GC	HIS	SL	CC	C	GC	HIS	SL	CC	C	
3741	3741	3741	3738	3740	3734	3736	3736	3736	3736	$\nu(\text{OH})$
2360	2360	2360	2360	2360	2360	2360	2360	2360	2360	νCO_2
2341	2341	2341	2341	2340	2340	2340	2340	2340	2340	νCO_2
667	668	668	668	668	668	668	668	668	668	$\nu(\text{M-O})$
-	605	-	-	601	-	-	601	-	-	$\delta(\text{C-H})$

Source: Authored by the author.

The FTIR spectra revealed that all the evaluated groups indicated the presence of the hydroxyl band (O-H) in the region of 3740 cm^{-1} , which can be attributed to water molecules adsorbed on the surface of the pellets during the execution of the analyses. Similarly, the bands at 2360 cm^{-1} and 2340 cm^{-1} can be attributed to the presence of carbon dioxide (CO_2) in the environment during the tests. As expected, all spectra presented the band at 668 cm^{-1} associated with M-O (metal-oxygen) bonds. However, in some of the spectra, bands in the region of 600 cm^{-1} were identified, attributed to C-H (carbon-hydrogen) bonds. According to the data already discussed on the percentage of mass loss, which confirmed the degradation of the ceramic tiles, it is believed that the erosive character of the solutions used may have caused porosity in the tiles. In turn, erosion may have



promoted the permeability of the acid solutions on the surface of the ceramic samples, which justifies the C-H bonds identified in the spectra.

CONCLUSIONS

Under the experimental conditions evaluated, the acid degradation study carried out confirmed that all the acid solutions used, i.e., artificial saliva, orange juice, Coca-Cola soft drink and coffee, promoted the wear of the 5Y-TZP ceramics.

All the acid solutions used presented pH values considered potentially erosive and, therefore, may represent a risk to dentin enamel demineralization.

Coca-Cola soft drink was responsible for the greatest loss of mass in the tablets, possibly due to the presence of phosphoric acid in its composition. The erosive wear of the inserts may have caused greater porosity of the surface of the ceramics, which justifies the chemical bonds C-H identified by FTIR. On the other hand, it was found that acid degradation had no effect on the crystallinity of the 5Y-TZP ceramics evaluated.



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