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Effect of Accelerated Aging on High-Translucity Zirconia: an *in Vitro* Study of Phase Transformation and Fracture Resistance

Efeito do Envelhecimento Acelerado na Zircônia de Alta Translucidez: um Estudo *in Vitro* da Transformação de Fase e Resistência à Fratura

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Abstract

The aim of the study is to assess the effect of accelerated aging on high translucency zirconia, through an in vitro investigation on phase transformation and fracture resistance. Blocks of conventional zirconia (Y-TZP N=05) and high translucency zirconia (HTZ N=05) were sectioned to generate specimens with dimensions of 12mm x 1.2mm. The specimens were evaluated for morphological analysis using a Scanning Electron Microscope (SEM) and structural analysis using X-Ray Diffraction (XRD), before and after thermal aging for 10,000 cycles with baths (5°C±1°-55°C±1°). Following aging, the experimental groups underwent biaxial flexural strength testing. One-way ANOVA ($p<0.05$) was employed. Morphological analysis post-aging revealed an increase in grain size in both groups: in the Y-TZP group, from 762.69nm to 1,215.25nm; in the HTZ group, from 671.56nm to 768.44nm. Chemical analysis confirmed the presence of Zirconium and Oxygen in both ceramics. XRD analysis revealed distinctive post-aging behavior, indicating a potential phase change in the Y-TZP group. Mechanical strength showed that Y-TZP presented a higher value compared to HTZ, with a statistically significant difference ($p=0.000$). Accelerated aging in high translucency zirconia does not seem to cause phase changes, presenting grain sizes similar to the non-aged condition. Conversely, conventional zirconia exhibited changes in grain size and post-aging morphological condition, yet still demonstrated mechanical superiority compared to high translucency zirconia.
Keywords: Ceramics. Zirconia. Accelerated Aging. Fracture Resistance.

Resumo

O objetivo do estudo é avaliar o efeito do envelhecimento acelerado na zircônia de alta translucidez, por meio de um estudo in vitro sobre a transformação de fase e resistência à fratura. Blocos de zircônia convencional (Y-TZP N=05) e de alta translucidez (HTZ N=05) foram seccionados gerando espécimes com dimensões de 12mm x 1,2mm. Os espécimes foram avaliados quanto à análise morfológica usando um Microscópio Eletrônico de Varredura (MEV) e análise estrutural usando Difração de Raios-X (DRX), antes e após o envelhecimento térmico 10.000 ciclos com banhos (5°C±1°-55°C±1°). Após o envelhecimento, os grupos experimentais foram submetidos ao teste de resistência à flexão biaxial. Anova 1 Fator (p<0,05) foi adotado. A análise morfológica após o envelhecimento, os grãos em ambos os grupos aumentaram de tamanho: no grupo Y-TZP, de 762,69nm para 1.215,25nm; no grupo HTZ, de 671,56nm para 768,44nm. A análise química confirma a presença de Zircônio e Oxigênio em ambas as cerâmicas. A análise de DRX revela comportamento distintivo pós-envelhecimento, indicando uma possível mudança de fase no grupo Y-TZP. A resistência mecânica mostra que o Y-TZP apresenta um valor mais alto em comparação com o HTZ, além de apresentar uma diferença estatística (p=0,000). O envelhecimento acelerado na zircônia de alta translucidez não parece promover mudanças de fase, apresentando tamanhos de grãos semelhantes à condição sem envelhecimento. A condição oposta foi encontrada com o tamanho de grão e a condição morfológica da zircônia convencional após o envelhecimento, entretanto, ainda se mostrou mecanicamente superior em relação à zircônia de alta translucidez.
Palavras-chave: *Cerâmicas. Zircônia. Envelhecimento Acelerado. Resistência à Fratura.*

1 Introduction

The tetragonal zirconia partially stabilized by yttrium oxide (Y-TZP) has been widely used in clinical practice as infrastructure for bridges and crowns, due to its excellent mechanical properties and characteristics such as durability, resistance to wear and corrosion, in addition to biocompatibility[1,2]. There has been an evolution of this ceramic material over the years, in an attempt to improve translucency without compromising the mechanical integrity[3].

Recently, a new type of high-translucency Zirconia, 5 mol% Zirconia partially stabilized with yttrium with a larger fraction of the cubic zirconia phase, has become commercially available[4]. Monolithic Y-TZP with different levels of translucency has been applied in indirect dental restorations. The composition and microstructure of Zirconia are modified to improve translucency[5].

Papers have observed different behaviors of this material under simulated oral aging[4-9], such as identification of phase transformation (tertragonal to monoclinic), reduced fracture resistance, increased roughness and changes in the dimensions of Zirconia grains[4,6-9]. The relationship between composition, microstructure and sintering procedures seems to determine its properties, thus reflecting on clinical performance, being a complex problem that needs to be resolved[6,8,9].

Therefore, it is essential that dentists and technicians are attentive when working with High-translucency Zirconia restorations, especially in the adjustment phase before and after cementation, as they can affect the monoclinic phase of the material, thereby altering surface roughness, mechanical properties, wear of the antagonistic enamel[7]. Therefore, it is important to know and consider the limitations of translucent monolithic Y-TZP before determining its clinical use[6]. Furthermore, it is still unclear how this material responds to aging[5], as it is not fully predicted in the literature[4].

The emergence of High Translucency Monolithic Zirconia in Dentistry generated a challenge in the scientific community to understand the behavior of this material and thus recommend it for clinical use. Research on the long-term properties of this material is scarce and presents conflicting results in the literature. Thus, it was observed that there is a greater prevalence of studies in which high translucency Zirconia presents grains with smaller dimensions, after aging they tend to present monoclinic phase transformation, improving optical properties and reducing mechanical resistance[7-9]. However, there is research that demonstrates some results opposite to those mentioned previously[4,6,9].

Based on the above, we objectively evaluated the effect of accelerated aging on high-translucency Zirconia, through an in vitro study on phase transformation and fracture resistance. The expected results for this research, based on the proposed objective, are: Null Hypothesis (H0): There are no statistically significant differences between conventional and high-translucency zirconia in terms of mechanical resistance values after aging; Alternative Hypothesis 1 (H1): There will be statistically significant differences between conventional and high-translucency zirconia in terms of mechanical resistance values after aging.

2 Material and Methods

2.1 Specimen preparation

Pre-sintered zirconia blocks for high-translucency (Katana HT, Kuraray Noritake Dental, Japan) and conventional CAD-CAM (VITA In-Ceram Zirconia, VITA Zahnfabrik, Bad Saackingen, Germany), with dimensions of 20 x 19 mm were machined into cylinders and with the aid of a cutting machine (ISOMET 1000, Buehler Ltd., IL, USA), specimens with approximate dimensions of 15 mm in diameter and 1.6 mm in height were obtained. Excesses and surface regularization of the specimens were carried out using a polishing machine using 600, 800, 1,200 grit sandpaper and the aid of a caliper to check dimensions[6,7,9].

Then, the specimens were sintered in a ZYrcomat furnace (Vita Zahnfabrik, Germany), according to the sintering cycle recommended by the manufacturer. After the sintering process, the specimens contracted, presenting final dimensions of approximately 12 mm in diameter and 1.2 mm in height (ISO 6872)[10]. The prepared specimens were stored in distilled water in an oven (FANEM, Orion Greenhouse 502), at a temperature of 37° and after 24 hours, test analyzes began.

2.2 Experimental design

2.2.1 Experimental groups and sample value

Two experimental groups were formed, Y-TZP for Conventional Zirconia and HTZ for High Translucency Zirconia. These groups presented 05 specimens for the analyzes under study. The sample value for this study was calculated (Minitab version 16.1 for Windows, Pennsylvania, USA) based on the standard deviation of similar research, thus N=05 achieves a sampling power of 80.0% in relation to the maximum for mechanical resistance.

2.2.2 Superficial analysis

The surface analysis of representative specimens (N=01) from each group was analyzed using Scanning Electron Microscopy (SEM) (HITACHI, Model TM300), using a magnification of 30,000 X, in order to mainly identify the grain size of the Zirconia[6]. Grain size was determined from SEM images using the mean equivalent circular diameter method[8]. This analysis was carried out before and after aging.

In the surface chemical analysis, chemical elements were identified using Energy Dispersive X-ray Spectroscopy (EDS-Electron Dispersive Spectroscopy) (Bruker).

2.2.3 Structural analysis

The phase transformation analysis was performed to elucidate the relative amount of monoclinic phase present in each experimental group before and after aging, through X-ray Diffraction analysis (XRD – Rint 2000, Rigaku, Tokyo, Japan)[6-8].

The parameter adopted was voltage of 40.0 kV, current of 30.0 mA, Theta-2Theta axes scanning range = 25,000 - 80,000, scanning mode = continuous scanning, scanning speed = 2.0000 (degrees/min), sampling = 0, 0200 (degrees) and predefined time = 0.60 (sec) and N sample equal to 05 specimens.

2.2.4 Accelerated aging

The specimens from both experimental groups were subjected to thermal aging in a thermocycler (Nova Ética, São Paulo, Brazil), for 10,000 cycles with baths of 5 °C ± 1° and 55 °C ± 1°. The immersion time in each bath was 30 seconds and the transfer time between the two baths was at 2-second intervals.

2.2.5 Mechanical resistance

The biaxial flexural strength test was performed after aging. The specimens were positioned on a circular metal base with three 3.2 mm diameter spheres in distilled water, equidistant from each other, forming a plane (ISO 6872)[10].

A blunt tip of 1.6 mm in diameter was attached to a testing machine (Emic DL-1000, Emic, São José dos Pinhais, PR, Brazil), and the load was applied at a speed of 1 mm/min and cell load of 1,000 Kgf. During the test, the specimen was covered with tape on the compression side, in order to prevent contact with the load applicator tip from producing defects and to make it possible to keep the fragments in position[6,8].

The calculation of biaxial flexural strength (σ) (MPa) of the discs was obtained in accordance with the description of the ISO 687210 standard (Formula 1): where P is the load in kgf, X and Y are parameters related to the elastic properties of the material (Poisson's Ratio and Elastic Modulus) and b is the thickness of the specimen at the origin of the fracture in mm[6,8]. Poisson's Ratio and Elastic Modulus values were sought in the literature[11,12].

Formula 1: Calculation of Biaxial Flexural Strength.

$$\sigma = -0{,}2387P\frac{(X-Y)}{b^2}$$

2.2.6 Fractography

The fractured specimens from both groups were analyzed using a stereomicroscope (Discovery V20, CarlZeiss, Germany) to determine fracture characteristics[8].

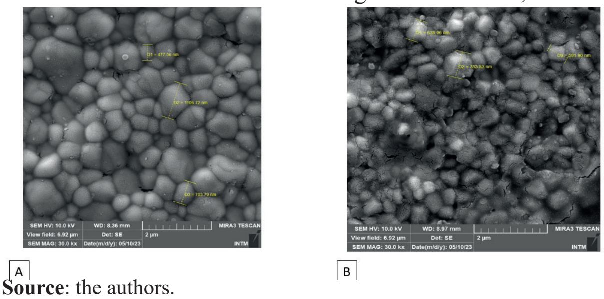
2.2.7 Analysis of results

The results were tabulated and analyzed in Minitab (version 16.1 for Windows, Pennsylvania, USA), with a significance level of 5% adopted. Mechanical resistance data after aging were subjected to the Anova 1 Factor statistical test ($p < 0.05$). The findings of the morphological, structural and fractographic analysis were presented qualitatively. Previously, the Komolgorov Smirnov Normality Test was applied to the data and experimental groups, which presented a significance level greater than 1%.

3 Results and Discussion

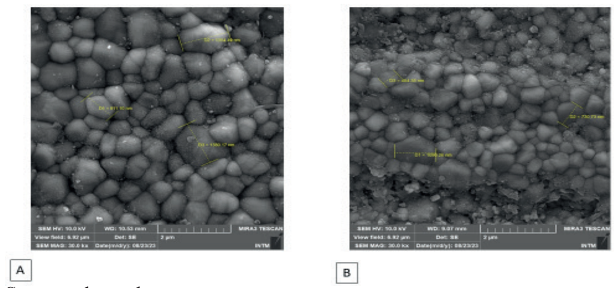
The findings regarding morphological analysis show the same pattern with the presence of grains between the two experimental groups (Figure 1, 2). The grains were measured before and after aging, reaching an average of 762.69 nm and 1,215.25 Y-TZP before and after aging, respectively. For the HTZ group, the grains presented an average of 671.56 nm and 768.44 nm before and after aging, respectively. The chemical analysis shows the presence of Zirconium and Oxygen for both ceramics (Figure 3). The spectrum obtained by XRD analysis shows differences between the behavior among ceramic materials and after aging, in terms of the observed peaks that may suggest a phase change for the Y-TZP group (Figure 4).

Figure 1 - Surface images before aging, 30x magnification in SEM and measurements of different grains: A- Y-TZP; B - HTZ



Source: the authors.

Figure 2 - Surface images after aging, 30x magnification in SEM and measurements of different grains: A- Y-TZP; B - HTZ



Source: the authors.

Figure 3 - EDS spectra, A – Y-TZP group before aging, B- Y-TZP group after aging, C- HTZ group before aging, the presence of Carbon can be explained by remnants of the tape to perform the SEM, D - HTZ group after aging

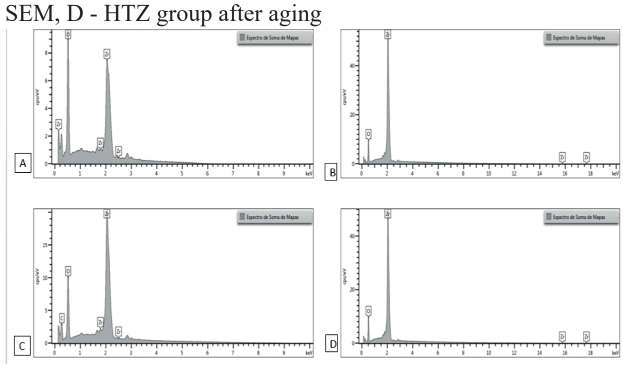


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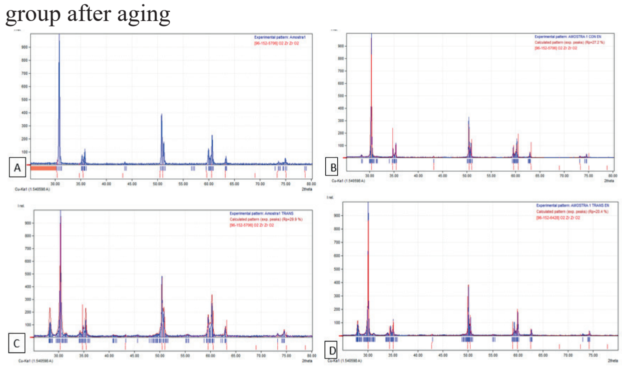
Figure 4 - XRD spectra, A – Y-TZP group before aging, B- Y-TZP group after aging, C- HTZ group before aging, D - HTZ group after aging



Source: the authors.

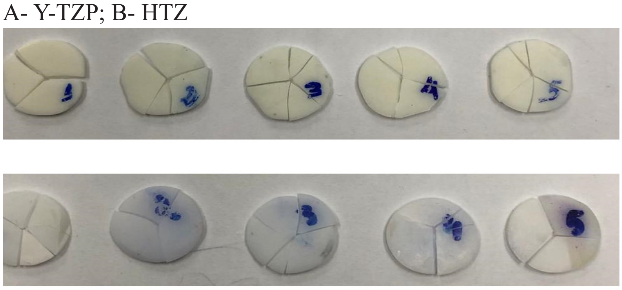
The mechanical resistance findings show that the Y-TZP group presents a higher value when compared to the HTZ group, in addition to presenting a statistical difference (p=0.000) (Table 1). As for the number of fragments after fracture, there is a similarity in the fracture pattern between the experimental groups (Table 1). The number of fragments ranged from 3 to 5 for the Y-TZP group and from 3 to 6 for the HTZ group (Figure 5).

Table 1 - Data after the mechanical resistance test

Experimental Group	Average Strength (N) (standard deviation)	Average Resistance (Mpa) (standard deviation)	Average Number of fragments after fracture
Y-TZP	903,886	29.919,88 (3735)	4,4
HTZ	844,694	5.831,823 (765)	4

Source: the authors.

Figure 5 - Fractured specimens after the flexural strength test, A- Y-TZP; B- HTZ



Source: the authors.

Based on the research findings, Alternative Hypothesis 1 (H1), there as statistically significant differences between conventional and high-translucency zirconia, in terms of mechanical resistance values after aging, it was accepted. The literature shows that Translucent Zirconia has significantly lower mechanical resistance than Conventional Zirconia[6,8], corroborating the findings of this research. The resistance values of Zirconia with translucency may vary depending on the manufacturer[4]. However, they are in line with other studies that showed no statistical difference between different zirconia, translucent and conventional, or that high-translucency zirconia did not suffer mechanical changes throughout aging, in terms of resistance to biaxial bending[9,13].

The studies that address the behavior of zirconia-based ceramics apply different means of aging specimens, such as hydrothermal, mechanical, thermomechanical, autoclave, hydrothermal reactor. The diversity of methods for aging specimens may interfere with flexural strength findings, making comparison and discussion between findings difficult, since the behavior of the ceramic material is modified according to aging methods[6]. Also, aging had little effect on roughness, but produced a significant reduction in flexural strength in translucent zirconia[7].

The minimal alteration of zirconia grains with high translucency was also observed in other papers[9]. Perhaps the size of the zirconia grain after aging and a distinct pattern in the morphological analysis spectrum suggests a possible phase change for the Y-TZP group and not for the HTZ group, corroborating the findings of similar studies[4,6,8]. However, the phase change was observed in translucent zirconia with surface treatment after accelerated aging[7], but in the present research the specimens did not receive any surface change. In addition to a previous high translucency monolithic Zirconia having presented a larger average grain size after aging[6], it is worth highlighting that it is aging and different manufacturers that seem to influence the properties of the ceramic.

From the results obtained, it is observed that the limitations of this research were the small sample number, not evaluating the mechanical resistance before aging and applying different accelerated aging methods. Further studies should be carried out to identify the predominant phase in specimens after aging, present anatomical specimens for mechanical testing, verify surface roughness after aging, in order to obtain long-term data to explore the results for daily clinical practice.

4 Conclusion

From the data obtained, accelerated aging on high-translucency Zirconia seems not to promote phase changes, presenting grain sizes similar to the condition without aging. The opposite condition was found with the grain size and morphological condition of Conventional Zirconia after aging, however it still proved to be mechanically superior in relation to high-translucency zirconia.

Data Availability

All data are available in the Federal University of Pernambuco (UFPE) repository.

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