# INFLUENCE OF COMPACTION PRESSURE AND HEATING RATE ON DENSIFICATION AND MECHANICAL PROPERTIES OF 5Y-PSZ ZIRCONIA

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## ABSTRACT

Yttria-stabilized zirconia is widely used as a biomaterial in dentistry due to its excellent mechanical properties, corrosion resistance, and biocompatibility. In this study, samples of 5Y-PSZ zirconia were prepared using different compaction pressures and heat treatment conditions, with sintering temperatures ranging from room temperature to 1500 °C. The methodology involved uniaxial powder compaction followed by controlled sintering under specific heating rates. To characterize the specimens, relative density was measured using the Archimedes method, while microhardness (Vickers), surface roughness, and wettability were evaluated to assess mechanical and surface behavior. The results showed that compaction pressure directly influences material densification, and higher densification is associated with improved mechanical performance, reduced surface porosity, and enhanced surface quality. Furthermore, samples subjected to a lower heating rate exhibited more homogeneous microstructures, greater mechanical strength, and superior wettability, suggesting enhanced surface energy and potential for better cell adhesion. These findings indicate that both the heating rate and compaction pressure play a major role in determining the final microstructural, mechanical, and functional properties of the ceramic. Therefore, proper control of these processing parameters is essential to optimize densification and improve the performance of 5Y-PSZ zirconia, making it a highly promising and reliable material for dental and biomedical applications.

**Palavras-chave**: Bioceramics. 5Y-PSZ. Sintering.

# 1 Introduction

Yttria-stabilized zirconia (YSZ) is an advanced ceramic material extensively employed in structural and biomedical applications due to its excellent mechanical strength, thermal stability, and biocompatibility [1]. In modern dentistry, depending on the generation of the material, zirconia is used in a broad range of prosthetic and restorative applications, including inlays/onlays, veneers, crowns, fixed partial dentures, abutments, implant components, fully ceramic anterior fixed prostheses bonded with resin cement, and full-arch implant-supported rehabilitations (Brånemark protocols) [2].

Among the compositional variants, zirconia stabilized with 5 mol% yttria (5Y-PSZ) is particularly noteworthy for its significant cubic phase content, which enhances translucency compared to the conventional 3Y-TZP (3 mol% $Y_2O_3$). This improved translucency makes 5Y-PSZ particularly suitable for aesthetic dental applications, such as anterior crowns and prostheses. However, the increase in cubic phase is accompanied by a reduction in mechanical performance, notably in flexural strength and fracture toughness [3,4]. The 5Y-PSZ grade typically exhibits larger grain sizes (~1.33 μm) and approximately 69% cubic phase, while offering lower flexural strength (~641 MPa) and fracture toughness (~2.4 MPa·m$^{0.5}$) compared to 3Y-PSZ (~878 MPa and 5 MPa·m$^{0.5}$, respectively) [5].

Porosity, a critical factor influencing the integrity of ceramic components, corresponds to the proportion of voids within a compacted powder mass. These voids arise naturally during the agglomeration of fine particles—often at nanometric scales—under the influence of pressure and temperature, sometimes aided by binders or moisture. Depending on the intended application, porosity may either be beneficial or detrimental. In applications requiring dense ceramics, pore elimination is essential to achieve optimal mechanical behavior [6].

Optimizing processing parameters is thus vital for enhancing the performance of 5Y-PSZ. Defects such as pores and inclusions introduced during manufacturing, as well as those resulting from handling or clinical use, serve as stress concentrators that compromise material strength. The relatively low fracture toughness of ceramics, combined with the presence of such flaws, leads to strength variability that depends on the distribution and size of inherent cracks [7].

Porosity directly affects the mechanical response of dense ceramics, which is of particular importance in dental applications where the ability to withstand occlusal forces without failure is critical [8].

In this context, the present study aims to investigate the effect of compaction pressure and sintering heating rate on the densification behavior and mechanical performance of 5Y-PSZ zirconia. By conducting relative density measurements (Archimedes method), Vickers microhardness testing, surface wettability analysis, and exploring distinct heating profiles during sintering, the study seeks to identify optimal processing conditions for achieving a balance between mechanical reliability and aesthetic quality in dental zirconia ceramics.

## 2 Materials e Methods

In this study, 5 mol% yttria-stabilized zirconia (5Y-PSZ) powder (G2DY-020OO, SINOCERA, China) was used as the ceramic raw material. Table 1 summarizes the main characteristics of the powder, including chemical composition (by weight), density, particle size, and specific surface area.

**Table 1. Summary of characteristics of the raw ceramic powder used in this study (as provided by the manufacturers). Source: SINOCERA and BYK.**

| Parameter | Typical value provided by manufacturer |
|---|---|
| $Y_2O_3$ content | 9.3 wt% ($\approx$ 5 mol%) |
| $Al_2O_3$ content | 0.05 wt% |
| $ZrO_2$ content | Balance |
| Average particle size | 90 nm |
| Crystallite size | 36 nm |
| Specific surface area | 10 m²/g |
| Sintered density | ~6.04 g/cm³ |

A total of 48 samples of 5Y-PSZ were prepared and divided into two experimental groups of 24 samples each, differentiated by the sintering heating rate. Each sample was composed of 1 g of ceramic powder, uniaxially pressed into cylindrical pellets with 10 mm diameter and 1 mm thickness.

During the compaction stage, the ceramic powder was subjected to four different uniaxial loads (1, 2, 3, and 4 tons) using a hydraulic press. For each pressure value, three samples were pressed, resulting in 12 specimens per group. This variation in compaction pressure aimed to evaluate its influence on the final densification and mechanical properties of the sintered material.

The first group underwent a **multi-step sintering cycle** with the following heating profile:

From room temperature to 800 °C at 1 °C/min, followed by a 30-minute dwell;

From 800 °C to 1100 °C at 2 °C/min, with a 1-hour dwell;

From 1100 °C to 1500 °C at 5 °C/min, followed by a 2-hour dwell;

Cooling from 1500 °C to room temperature at 5 °C/min.

The second group underwent a **single-step sintering cycle**: heating from room temperature to 1500 °C at a constant rate of 5 °C/min, held for 2 hours, followed by cooling at the same rate.

After sintering, the following characterization techniques were applied:

Relative Density

Relative density was determined by the Archimedes method, following standards such as ASTM C373-88. Each sample was measured under three conditions: dry (weighed in air), immersed (weighed underwater), and saturated (fully water-absorbed). These measurements allowed accurate determination of apparent density.

Surface Roughness

Surface roughness was analyzed using non-contact laser interferometry (Zygo NewView 7100, Zygo Corporation, Middlefield, CT, USA). The measured parameters were:

**Ra**: arithmetic average of the absolute profile height deviations over the evaluation length;

**Rq**: root mean square of height deviations;

**PV (Peak-to-Valley)**: difference between the highest and lowest points within the measured area.

## Wettability

Wettability was assessed by contact angle measurements using an optical goniometer. Three measurements were taken per sample using a 5 µL drop of distilled water applied to the polished surface. The contact angle was automatically calculated by the equipment software at room temperature.

## Microhardness

Vickers microhardness was measured in accordance with ASTM C1327 and ASTM E384 standards for advanced ceramics. A representative sample from each experimental group (2 total) was selected. Eleven indentations were made on each sample using a 1 kgf load (HV1) for 30 seconds. The first indentation was discarded to minimize the effect of surface irregularities, and the average of the remaining ten measurements was used for hardness calculation.

The definitions of the roughness parameters followed ISO 21920-2:2021. Three distinct areas were measured per sample, and the mean values were reported. The 3D interferometric analysis provided enhanced insight into surface morphology compared to traditional contact profilometers.

## 3 Results and Discussion

Figure 1 presents the relative density results obtained through Archimedes' method, assuming a theoretical density for zirconia of 6.05 g/cm³. Group 2, which was subjected to the higher heating rate, exhibited lower densification compared to Group 1. This result supports previous studies indicating that rapid heating rates can lead to incomplete sintering, resulting in increased porosity and poor densification [9].
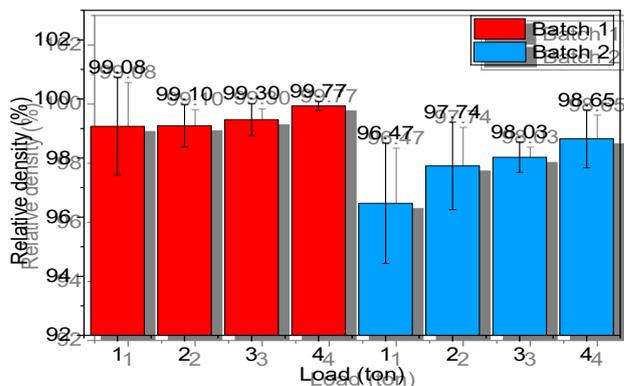


Figure 1. Relative density of the synthesized groups of samples

The roughness results indicated that the samples subjected to the lowest compaction pressure and the highest heating rate exhibited the highest surface roughness values. This outcome aligns with the greater porosity observed in these samples, as a more porous microstructure tends to produce more irregular surfaces [10]. Figure 2 (a–d) presents the roughness values for each sample.

The lower density values observed are associated with a higher amount of open and closed pores, which compromise mechanical properties such as fracture toughness due to stress concentration at structurally weak points [11]. It was noted that Group 1, which underwent a more controlled thermal cycle, showed relative densities ranging from 99.08 to 99.77 g/cm³, increasing with applied compaction pressure. Nevertheless, all four values were statistically similar within a 5% confidence interval. This densification effect as a function of compaction pressure was more pronounced in the group subjected to the rapid heating cycle, with relative densities ranging from 96.47 to 98.65 g/cm³.

The literature clearly establishes that surface roughness significantly influences the clinical performance of dental ceramics. Rougher surfaces can enhance the micromechanical interlocking of resin cements, which is beneficial for retention [14]. However, excessive roughness may promote bacterial colonization and impair the material's aesthetics, in addition to generating stress concentration zones that facilitate crack initiation [15].



(a)



(b)
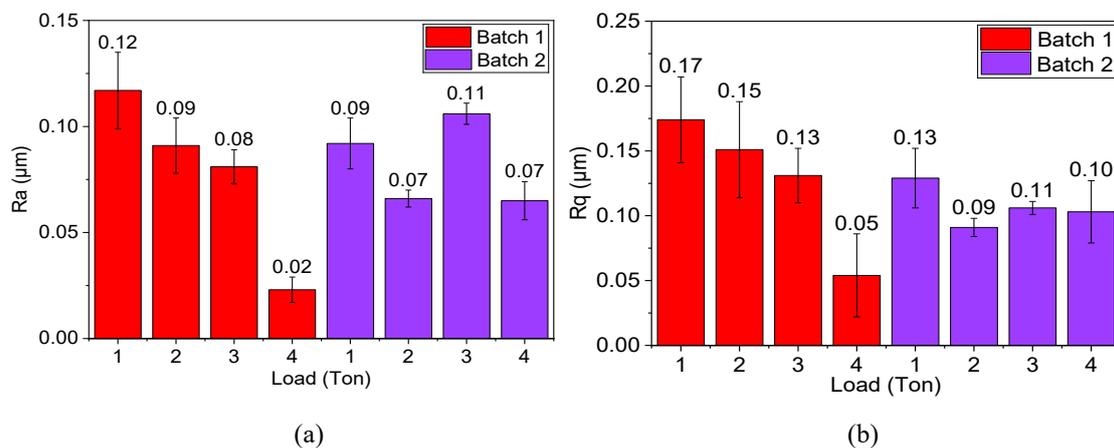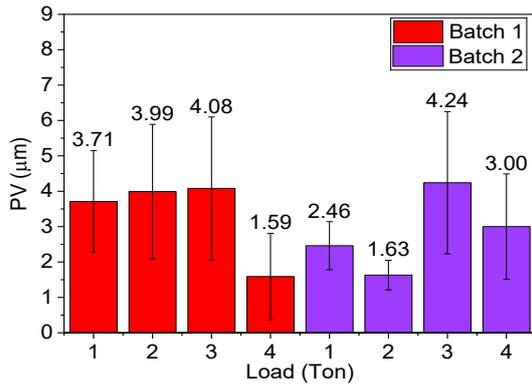
(c)

Figure 2. Surface roughness parameters obtained by interferometry: a) Ra; b) Rq; c) PV

By analyzing the Ra and Rq parameters from the first heating group, it was evident that increasing the compaction load reduced the final surface roughness, with values ranging from 0.12 to 0.02 µm for Ra and from 0.17 to 0.05 µm for Rq. However, no clear trend of roughness reduction was observed in Group 2, where average values ranged from 0.07 to 0.11 µm for Ra and from 0.09 to 0.13 µm for Rq. Regarding the peak-to-valley (PV) values, Group 1 exhibited values between 1.59 and 4.08 µm, while Group 2 ranged from 1.63 to 4.24 µm. Although overall values were similar between groups, the analysis of individual compaction loads revealed significant differences, primarily attributed to surface irregularities. One such surface is depicted in Figure 2-d.

Moreover, the heating rate also influenced wettability, with Group 2 showing a higher wetting capability. Increased porosity contributes to a larger available surface area and greater apparent surface energy, thereby enhancing hydrophilicity [12]. In dental applications, this feature may be beneficial for improving adhesion between restorative materials. However, excessive porosity may impair the structural integrity and long-term durability of the restoration [13]. Figure 3 presents the average values of contact angle and surface energy parameters obtained from the wettability analysis.
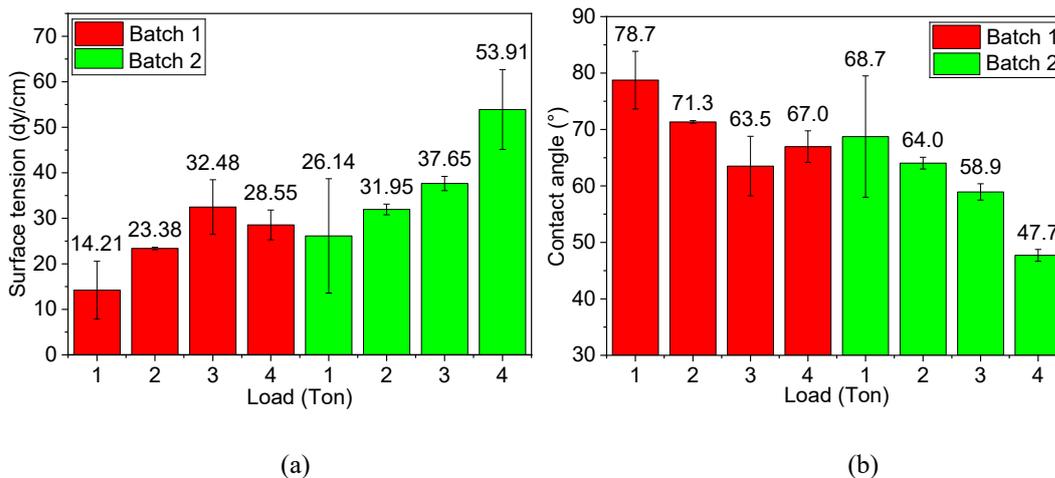


(a)



(b)

Figure 3. Wettability surface parameters of the sintered samples

Surface tension exhibited an inverse relationship with the contact angle. As the compaction load increased, surface tension tended to rise. However, in Group 1 at 4 tons, this trend was not followed, resulting in a surface tension value of 28.55 dy/cm. Group 1 displayed surface tension values ranging from 14.21 to 32.48 dy/cm, while Group 2, characterized by greater

porosity, showed values between 26.14 and 53.91 dy/cm—indicating that increased porosity leads to higher surface tension.

Meanwhile, contact angles tended to decrease as compaction load and porosity increased. Both groups demonstrated hydrophilic behavior, more pronounced in Group 2, with contact angles ranging from 68.74° to 47.71°, compared to Group 1, which ranged from 78.74° to 63.52°.

The results from mechanical testing demonstrate that control over compaction pressure and heating rate during 5Y-TZP fabrication is crucial to ensuring adequate densification, fracture resistance, and other key mechanical properties necessary for clinical applications. These findings highlight the importance of optimizing processing parameters to balance densification and microstructure, thus ensuring functional performance in dental applications [14]. Figure 4 presents the average values of hardness and fracture toughness obtained from Vickers microindentation testing.

Microhardness values varied slightly but were statistically similar within a 5% confidence interval, regardless of sintering cycle or applied load. In Group 1, values ranged from 1297.61 to 1338.68 HV, while in the group subjected to higher heating rates, hardness values ranged from 1299.39 to 1304.07 HV. All results are consistent with values reported in the literature for these densification levels [1,3].
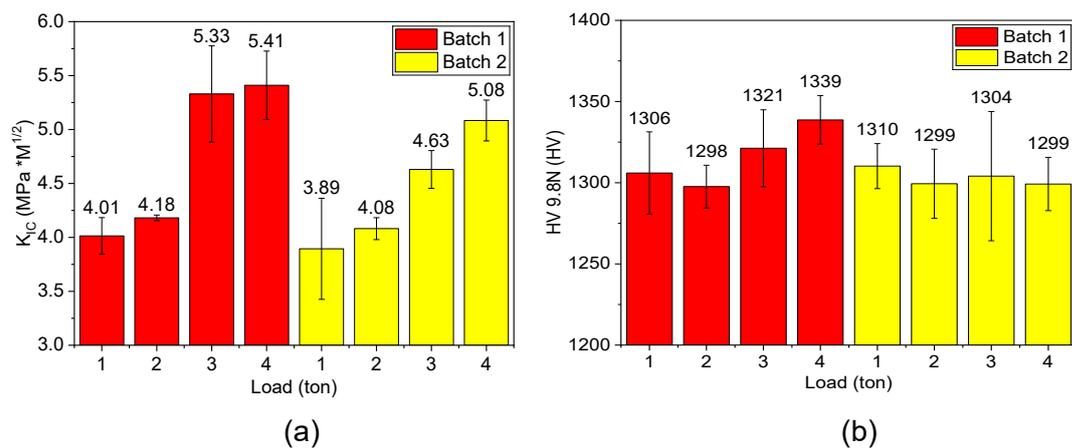


Figure 4. Vickers microindentation results: a) Hardness; b) Toughness

The analysis of fracture toughness (KIC) data clearly reveals the effect of residual porosity the mechanical properties of zirconia samples. This influence is particularly evident with respect to the compaction load applied and the heating rate used during the sintering process. Group 1 exhibited values ranging from 4.01 to 5.41 MPa·m$^{1/2}$, whereas Group 2 ranged from 3.89 to 5.08 MPa·m$^{1/2}$, consistently showing slightly lower toughness values at all pressure levels compared to Group 1.

Notably, the compaction load had a greater influence on fracture toughness than the heating rate. The highest KIC values were achieved in samples pressed at 3 and 4 tons, underscoring the importance of simultaneously controlling initial densification (via compaction) and the heating rate during sintering in order to achieve a denser and mechanically stronger microstructure.

## 4 Conclusion

The samples subjected to higher compaction pressures exhibited greater density and mechanical strength, reinforcing the importance of minimizing porosity to achieve improved mechanical properties. The results of the present study demonstrated that both compaction pressure and heating rate during the sintering process significantly influence the final densification and mechanical performance of 5Y-PSZ zirconia.

Furthermore, a controlled heating rate proved essential for obtaining homogeneous microstructures, with slower heating promoting better densification and superior mechanical properties. In contrast, higher heating rates increased residual porosity, compromising strength and altering surface characteristics such as roughness and wettability.

Therefore, careful adjustment of these processing parameters is crucial to optimize both the functional and aesthetic performance of 5Y-PSZ zirconia, ensuring its effectiveness in dental prosthetic applications. This study highlights the need for strict control over fabrication conditions to maximize the durability and quality of ceramic biomaterials.

## Referências

[1] Almohammed SN, Alshorman B, Abu-Naba'a LA. Mechanical properties of five esthetic ceramic materials used for monolithic restorations: a comparative in vitro study. *Ceramics, Basel*. 2023;6(2):1031–1049.

[2] Cesar PF, Sato F, Miyazaki T, Zhang Y. Avanços recentes na zircônia odontológica: 15 anos de evolução do material e do processamento. *Dental Materials*. 2024;40:824–836.

[3] Aboushelib M, Kleverlaan C, Feilzer A. Microtensile bond strength of different components of core veneered all-ceramic restorations. Part II: Zirconia veneering ceramics. *Dental Materials*. 2006;22(9):857–863.

[4] Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. *Dental Materials*. 1999;15(6):426–433.

[5] Santos C, Koizumi MD, Santos J, Elias C, Ramos A. Properties of Y-TZP/$Al_2O_3$ ceramic nanocomposites obtained by high-energy ball milling. Materials Science and Engineering: A. 2009;502:6–12.

[6] Borba M, Araújo MD, Fukushima KA, Yoshimura HN, Cesar PF, Griggs JA, Della Bona A. Effect of the microstructure on the lifetime of dental ceramics. *Dental Materials*. 2011;27:710–721.

[7] Callister WD Jr, Rethwisch DG. Ciência e engenharia dos materiais: uma introdução. 8. ed. Rio de Janeiro: LTC; 2013. 817 p

[8] Askeland DR, Phulé PP. Ciência e engenharia dos materiais. 1. ed. São Paulo: Cengage Learning; 2008. 594 p.

[9] Ingh P, Kumar D. Influence of sintering temperature and heating rate on densification and grain growth of 5Y-PSZ. Materials Today: Proceedings. 2021;38:869–874.

[10] Li J, Zhang Y, Lawn BR. Fracture resistance of ceramic structures: a strategy for interface design. Journal of the Mechanical Behavior of Biomedical Materials. 2016;54:153–161.

[11] Zhang Y, Pajares A, Lawn BR. Fatigue and damage tolerance of Y-TZP zirconia ceramics. J Dent Res. 2014;93(3):219–224

[12] González-Martín ML, Labajos-Broncano L, Jańczuk B, Bruque JM. Wettability and surface free energy of zirconia ceramics and their constituents. J Mater Sci. 1999;34(24):5923–5926.

[13] Patel NP, Chauhan KV, Prajapati RN. Anti-icing, wettability and structural characterization of Zirconia thin films. Materials Today: Proceedings. 2023;73:3524–3530

[14] Kong MC, Park MG. Effect of sintering condition and low-temperature degradation on the flexural strength and phase transformation of zirconia. *J Prosthet Dent*. 2021;126(5):692.e1–692.e14.