Original Article

Technological Optimization of Sintering Protocols for Dental Zirconia: In Vitro Evaluation of Structural and Mechanical Properties

Optimization of Sintering for Dental Zirconia Properties

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ABSTRACT

Objective: This study compared the effects of a conventional sintering cycle (1500°C for 2h, total 11h) versus a short cycle (1500°C for 2h, total 4h) on the properties of a commercial 3Y-TZP (Upcera YZ HT).

Study Design: Experimental analytical study

Place and Duration of Study: This study was conducted at the Department of Prosthetic Dental Science, College of Dentistry, University of Najran, Saudi Arabia, from 1st June 2025 to 30th November 2025.

Methods: Thirty bar-shaped specimens (n=15/group) were milled, sintered, and analyzed for flexural strength, grain size, and monoclinic phase content. Intergroup comparisons were performed using Student's t-test and Mann–Whitney U-test. A p-value < 0.05 was considered statistically significant.

Results: Results showed no statistically significant differences between groups: flexural strength (Conventional: 1309.42 ± 329.88 MPa; Short: 1187.23 ± 326.35 MPa; p=0.208), monoclinic phase content (Conventional: 47.92 ± 12.18%; Short: 50.42 ± 8.03%; p=0.84), or grain size (Conventional: 0.64 ± 0.18 µm; Short: 0.46 ± 0.08 µm; p=0.11). Correlations among these parameters were also non-significant.

Conclusion: Within the study's limitations, the short sintering cycle yielded zirconia with comparable mechanical properties, phase stability, and a clinically acceptable microstructure to the conventional cycle. This supports accelerated sintering as a viable, technology-driven, time-efficient alternative for dental laboratories without compromising core material performance.

Key Words: Yttria-stabilized zirconia, Dental ceramics, Sintering protocols, Flexural strength, Digital dental technology

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INTRODUCTION

Tooth loss, predominantly resulting from dental caries and periodontal disease, continues to represent a significant global oral health concern.[1] Although the incidence of complete edentulism is gradually declining, the prevalence of partial edentulism is on the rise.[2,3] Conventional treatment modalities encompass removable partial dentures, fixed dental prostheses (FDPs), and implant-supported crowns.[4]

Among the various FDP options, metal-ceramic restorations, characterized by a durable metallic framework overlaid with a veneering ceramic layer, have long been favored due to their mechanical strength and resistance to fracture.[5,6] Nevertheless, longitudinal clinical data indicate that veneering ceramic fractures occur in approximately 5–10% of cases within ten years.[7] Increasing patient demands for superior esthetics, heightened awareness of potential hypersensitivity to metal components, and the preference for more conservative tooth preparations have collectively driven the development and adoption of metal-free alternatives, thereby contributing to the growing popularity of all-ceramic restorations.[8,9]

Dental ceramics are generally classified into three main categories: glassy ceramics (e.g., feldspathic porcelain), reinforced glass-ceramics, and polycrystalline ceramics.[10] While glass-ceramics offer excellent aesthetics, their limited strength led to the development of high-strength oxides like alumina and zirconia.[11,12] Among these, 3Y-TZP zirconia is widely used for frameworks due to its superior mechanical properties, biocompatibility, and chemical stability.[13]

Pure zirconia exhibits polymorphism, existing in monoclinic (room temperature to 1170°C), tetragonal (1170°C to 2370°C), and cubic (>2370°C) phases.[14]

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The incorporation of 3–6 mol% yttria (Y_2O_3) stabilizes the tetragonal phase at room temperature in a metastable form.[15] This metastability enables transformation toughening—a stress-induced phase shift from tetragonal to monoclinic, accompanied by ~4% volumetric expansion.[15] The resulting compressive stresses around cracks inhibit their propagation,[16] enhancing fracture toughness (5–10 MPa√m) and flexural strength (800–1300 MPa). These properties support the use of Y-TZP in crowns, FDPs, implant abutments, and endodontic posts.[11,15]

The sintering densifies a ceramic compact, critical for zirconia frameworks, as it transforms the porous, granular structure into a dense, cohesive polycrystalline solid.[17] Conventional sintering typically involves prolonged heating (8–12 hours) between 1350°C and 1550°C.[18] The sintering parameters such as temperature, duration, and heating/cooling rates profoundly influence the final microstructure (grain size, density) and phase composition of Y-TZP, directly dictating its mechanical performance and long-term stability.[19] Grain size is particularly crucial as larger grains facilitate the stress-induced t→m transformation, enhancing toughness, but grains exceeding a critical size can undergo spontaneous transformation during aging (e.g., in the oral environment), potentially leading to low-temperature degradation (LTD) characterized by surface roughening, microcracking, and strength reduction.[20]

While Y-TZP offers significant advantages, optimizing its processing, particularly sintering, is essential for maximizing clinical performance, cost-effectiveness, and sustainability. The use of accelerated sintering technology, characterized by shortened heating durations and precise thermal control, offers significant workflow and energy-efficiency benefits, especially in digital dentistry and same-day fabrication protocols. However, concerns remain regarding the potential compromise of mechanical properties and phase stability. Despite the widespread adoption of CAD/CAM systems and high-temperature furnaces in dental technology, studies evaluating the effect of short-cycle sintering protocols as a technological innovation are limited and sometimes contradictory.[20] Understanding these relationships is crucial for integrating advanced processing technologies into routine clinical and laboratory workflows.

Therefore, this study aimed to evaluate and compare the effect of a conventional sintering cycle versus a commercially available short sintering cycle on the flexural strength, grain size, and phase transformation behavior of a widely used yttria-stabilized presintered zirconia (3Y-TZP). The null hypothesis was that the sintering cycle (short vs. conventional) would have no significant effect on the flexural strength, grain size, or monoclinic phase content of the material.

METHODS

This study was conducted at the Department of Prosthetic Dental Science, College of Dentistry, University of Najran, Saudi Arabia, from 1st June 2025 to 30th November 2025. Zirconia blanks (Upcera YZ HT; Shenzhen Upcera Dental Technology, China) were used for specimen fabrication. The blanks primarily comprised zirconium oxide (94–96 wt%), yttrium oxide (4–6 wt%), and trace amounts of hafnium oxide (1–2 wt%), alumina, and silica (0–0.1 wt%). 30 bar-shaped specimens (25 mm × 4 mm × 1.2 mm) were designed using CAD software and milled from the pre-sintered zirconia blanks using a CAD/CAM system (MC XL, Dentsply Sirona, Germany). The STL file was digitally transferred to the milling unit, illustrating the integration of computer-aided design and manufacturing (CAD/CAM) technology in dental prosthesis production. All specimens were randomly divided into two groups (n = 15 per group) based on sintering protocol: Group 1 (Conventional sintering): 1500 °C for 2 h with a total cycle time of 11 h. Group 2 (Short sintering): 1500 °C for 2 h with a total cycle time of 4 h.

Sintering: Sintering was performed in a high-temperature furnace according to the protocols specified. In both groups, the heating rate was 10 °C/min, with a holding temperature of 1500 °C. Samples were cooled to room temperature at a controlled rate.

Flexural Strength Testing: All specimens were subjected to three-point bending tests using a universal testing machine (model no. 3369, Instron, Canton, MI, USA) at a crosshead speed of 1 mm/min. The flexural strength (σ) was calculated using the formula:

$$\sigma=3NI/2bd^2$$

where N is the fracture load (N), I is the span length (mm), b is the width (mm), and d is the thickness (mm) of the specimen.

Grain Size Analysis: Five samples from each group were polished up to 1 μm using diamond suspension, ultrasonically cleaned in isopropanol, and sputter-coated with gold. Surface morphology and grain size were examined using a Scanning Electron Microscope (JEOL JSM-6490, Tokyo, Japan) at 30,000× magnification.

Phase Transformation Analysis: X-ray diffraction (XRD) analysis was conducted on five samples per group using Ultima IV diffractometer (Rigaku Corporation, Tokyo, Japan) with Ni-filtered CuKα radiation. Scans were performed in the 2θ range of 25°–35° with a step size of 0.01° at 0.5 s/step. The monoclinic phase fraction (Xm) was calculated using the Garvie and Nicholson formula:

Statistical Analysis: Data were analyzed using SPSS version 11.0. Descriptive statistics (mean ± standard deviation) were calculated for all variables. Intergroup comparisons were performed using Student's t-test or Mann–Whitney U-test, depending on data normality. A p-value < 0.05 was considered statistically significant.

RESULTS

The table presents the flexural strength of the study groups subjected to different sintering cycles. Group I exhibited a higher mean flexural strength (1309.42 ± 329.88 MPa) compared to Group II, which had a mean of 1187.23 ± 326.35 MPa. Although Group I showed a higher median value (1382 MPa) and narrower range, the statistical comparison between the two groups yielded a non-significant p-value of 0.208. The 95% confidence interval for the mean difference ranged from 1125.6 – 1372.1 MPa, suggesting overlapping variability.

The mean phase transformation percentages for the study groups are summarized in Table 2. Group I exhibited a mean of 47.92 ± 12.18%, while Group II (conventional sintering) demonstrated a mean of 50.42 ± 8.03%. Median values remained comparable, and the minimum and maximum values in both groups showed overlapping distributions. The 95% confidence interval for the comparison (32.85–59.76%) reflects the observed variability. Statistical analysis showed no significant difference between the groups (p = 0.84).

Table 3 and Figure 1 illustrate the comparison of grain size (µm) between zirconia samples subjected to short and conventional sintering protocols. Group I (short sintering) demonstrated a lower mean grain size (0.46 ± 0.08 µm) in contrast to Group II (conventional sintering), which exhibited a mean of 0.64 ± 0.18 µm. The medians and observed range further support the trend of finer-grain structure in the short sintering group. The 95% confidence interval for the comparison (0.36–0.87 µm) indicates partial overlap between the two groups. The statistical comparison showed a non-significant difference (p = 0.11).

Table No. 1: Comparison of Flexural Strength (MPa) Between Short and Conventional Sintering Cycles of Zirconia Cores

Group	Mean (MPa) ± SD	Median (MPa)	Min	Max	95% CI (Lower–Upper)	Significance
Group I	1309.42 ± 329.88	1382	822	1956		0.208 (Non-significant)
Group II	1187.23 ± 326.35	1094	912	2280		
Group I vs II					1125.6 – 1372.1	

Table No. 2: Comparison of Phase Transformation Percentages Between Short and Conventional Sintering Cycles of Zirconia Cores

Group	Mean ± SD (%)	Median (%)	Min (%)	Max (%)	95% CI (Lower–Upper)	Significance
Group I	47.92 ± 12.18	49.1	31.24	60.85		0.84 (Non-significant)
Group II	50.42 ± 8.03	50.31	38.22	60.12		
Group I vs II					32.85 – 59.76	

Table No. 3: Comparison of Grain Size (µm) Between Zirconia Cores Subjected to Short and Conventional Sintering Cycles

Group	Mean ± SD (µm)	Median (µm)	Min (µm)	Max (µm)	95% CI (Lower–Upper)	Significance
Group I	0.46 ± 0.08	0.45	0.37	0.58		0.11 (Non-significant)
Group II	0.64 ± 0.18	0.66	0.43	0.85		
Group I vs II					0.36 – 0.87	

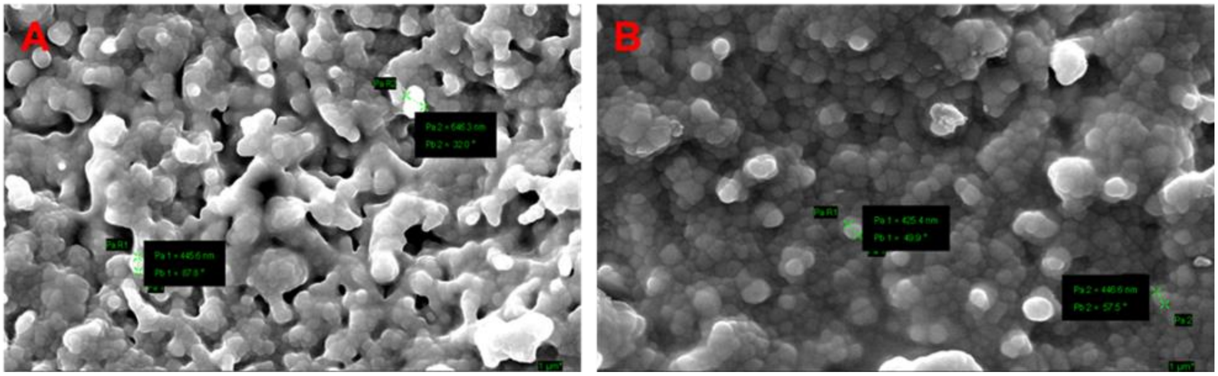

Figure No. 1: SEM images (30,000× magnification) showing the surface microstructure of zirconia specimens: (A) Specimen from the short sintering group showing comparatively smaller and regular grain morphology, while (B) shows a specimen from the conventional sintering group with larger and more irregular grain morphology

Table No. 4: Spearman's correlation coefficients (ρ) and significance levels for relationships among flexural strength, grain size (SEM), and monoclinic phase fraction (XRD) in zirconia cores subjected to short and conventional sintering cycles

Group	Comparison	ρ (rho)	p-value	Significance
Group I (Short Sintering)	SEM vs Flexural Strength	0.382	0.269	Non-significant
	SEM vs XRD	–0.231	0.502	Non-significant
	Flexural Strength vs XRD	–0.041	0.912	Non-significant
Group II (Conventional Sintering)	SEM vs Flexural Strength	0.096	0.882	Non-significant
	SEM vs XRD	–0.387	0.518	Non-significant
	Flexural Strength vs XRD	0.057	0.927	Non-significant

Table 4 summarizes Spearman's correlation analysis. No statistically significant associations were found between flexural strength, grain size, and monoclinic phase content in either sintering group. In Group I (short sintering), a weak positive correlation was observed between SEM and flexural strength ($\rho = 0.382$), while a weak negative correlation existed between SEM and XRD ($\rho = –0.231$). Similarly, Group II (conventional sintering) demonstrated negligible to weak correlations, with the most notable being a weak negative correlation between SEM and XRD ($\rho = –0.387$). However, all p-values remained > 0.05, confirming the absence of statistically significant relationships.

DISCUSSION

Sintering critically influences the mechanical properties of yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP), the most commonly employed form of dental zirconia.[10,17] While manufacturing, design, and milling also affect its properties, the sintering temperature and duration directly impact the grain size and phase composition.[19] Kulyk et al. investigated the effects of sintering temperature and yttria content on 3Y- and 6Y-doped zirconia ceramics, reporting that specimens sintered at 1550 °C for 2 h exhibited significantly larger average grain sizes (~0.5–0.8 µm) and higher flexural strengths (~1600 MPa), compared to those sintered at 1450 °C with finer grains (~0.1–0.4 µm) and lower strength (≈1080 MPa),[21] while Casellas et al. reported reduced grain size correlating with increased flexural strength; though both effects were statistically insignificant. Optimizing the sintering cycle is therefore essential for enhancing YTZP's performance.[22]

Phase transformation behavior was assessed using XRD, revealing no significant difference in monoclinic phase content between the two groups (p = 0.854). While controlled tetragonal-to-monoclinic transformation remains a critical mechanism for enhancing fracture toughness, commonly referred to as transformation toughening, excessive transformation can compromise long-term integrity. Recent study affirms that the stress-induced t→m transformation, with its associated ~4–5% volumetric expansion, generates compressive stresses around crack tips that impede crack propagation, thereby increasing toughness.[23] The comparable monoclinic content observed in this study suggests that both sintering protocols preserved phase stability in 3Y-TZP zirconia without risking detrimental over-transformation

Grain size analysis indicated a marginally larger grain size in the conventional sintering group (0.64 µm) relative to the short-sintering group (0.46 µm), with the difference lacking statistical significance (p = 0.11). This trend aligns with recent findings by Shahmiri et al., who demonstrated that prolonged sintering durations and higher temperatures in 3Y-TZP lead to increased grain growth, while careful control of thermal profiles maintains microstructures within optimal size ranges (< 0.8 µm) conducive to phase stability and mechanical integrity.[24] Both groups remained well within the clinically acceptable grain size threshold as defined by ISO 13356:2008, and below the critical limit (~1 µm) where transformation-induced degradation becomes more likely. Additionally, another study reported average grain sizes around 0.65 µm for specimens sintered at 1475 °C for 2 h, consistent with the dimensions observed in our study, further confirming the adequacy of our sintering protocols.[25]

The short sintering cycle employed a rapid heating rate (70°C/min) without dwell time and reached the sintering temperature of 1500°C in about 4 hours and 20 minutes, whereas the conventional cycle required approximately 11 hours with a total 4-hour dwell time. Despite these differences, mechanical and microstructural outcomes were comparable. A study by Luo and Pan shows that fast heating quickly surpasses the temperature range where surface diffusion dominates. Consequently, densification proceeds via grain boundary diffusion much earlier, contributing to efficient neck formation and densification.[26] While Pan et al., reported that even minimal dwell time could yield favorable outcomes, corroborating the present findings that effective sintering is possible without prolonged thermal holding.[27] These findings reinforce the potential of advanced sintering furnaces equipped with precise thermal control to optimize processing protocols in dental technology applications.

Cooling time was standardized to 4 hours in both groups, ensuring gradual thermal reduction and minimizing the risk of residual stresses. The previous studies emphasized the importance of controlled cooling in preserving strength and reducing crack propagation in zirconia, highlighting the relevance of uniform cooling in the present methodology.[17,19]

Although the correlation between flexural strength, phase transformation, and grain size was statistically non-significant, some trends were noted. The group subjected to short sintering exhibited slightly higher flexural strength, accompanied by reduced grain size and lower monoclinic phase content. These trends align with a study reporting that flexural strength initially rises with grain size before declining beyond a critical limit.[28]

This in vitro study did not evaluate long-term aging, bonding behavior, or clinical performance. Only flexural strength was assessed, excluding other key mechanical properties like fracture toughness or fatigue resistance. Findings are limited to one zirconia type and sintering furnace. Future studies should incorporate thermocycling, aging protocols, and more comprehensive mechanical tests. Evaluating different zirconia grades and their bonding potential after varied sintering cycles is recommended. Clinical trials and cost–benefit analyses will help validate the practicality of rapid sintering protocols.

CONCLUSION

Within the limitations of this in vitro study, it can be concluded that the short sintering cycles produced yttria-stabilized zirconia with mechanical and microstructural properties comparable to conventional sintering. Hence the null hypothesis is accepted. However, time-efficient sintering protocols can be adopted in clinical and laboratory workflows without compromising material performance. As zirconia continues to play a central role in restorative dentistry, optimizing processing parameters through the use of digital design and controlled thermal technologies can significantly enhance productivity, cost-effectiveness, and consistency in laboratory workflows.

Author Contributions: The author solely conceived, designed, and conducted the study; collected, analyzed, and interpreted the data; and wrote and approved the final manuscript.

Ethical Approval: This study was based on the synthesis and testing of dental materials; ethical approval was not required in accordance with institutional and national research ethics guidelines.

Author's Contribution:

Concept & Design or acquisition of analysis or interpretation of data:	Khalid Dhafer Alhendi
Drafting or Revising Critically:	Khalid Dhafer Alhendi
Final Approval of version:	The above author
Agreement to accountable for all aspects of work:	The above author

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