Original Article

Effect of Aging Procedures on Phase Transformation of Posterior 3-Unit Zirconia Frameworks Used in Fixed Partial Dentures

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Abstract

Background: Zirconia is an essential material for dentistry, and its properties should be investigated in all aspects. **Aim:** The primary objective of this study was to assess the impact of aging procedures on the phase transformation of posterior 3-unit zirconia frameworks used in fixed partial dentures. **Setting and Designs:** The study considered three aging procedures: Group T underwent thermocycling with 20,000 cycles between 5°C and 55°C for 30 seconds, Group M experienced mastication cycles with 500,000 cycles at 1.3 Hz and 49 N force, and Group T + M was subjected to consecutive thermocycling (20,000 cycles/5–55°C/30 s) and mastication cycles (500,000 cycles/1.3 Hz/49 N). **Methods:** Each group comprised 12 specimens from manufactured presintered zirconia blocks (inCoris ZI, Sirona, Salzburg, Austria). The evaluation of phase transformation was performed through X-ray diffraction (XRD) analysis. **Statistical Analysis:** The obtained data were subjected to statistical analysis using one-way ANOVA and the Tukey tests ($\alpha = 0.05$). **Results:** The study revealed statistically significant differences in the effects of aging processes on phase transformation in zirconia frameworks ($P < 0.05$). Group T + M demonstrated the highest phase transformation compared to other groups ($P < 0.05$). Moreover, significant differences were observed between the distal connector and pontic regions ($P < 0.05$). **Conclusion:** All aging processes applied to zirconia increased the amount of monoclinic phase.

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INTRODUCTION

In recent times, zirconia has garnered significant attention within dentistry.[1,2] This material boasts remarkable mechanical properties, including high fracture toughness, flexural strength, and exceptional wear resistance. Zirconia has been deemed a safe and reliable option in posterior fixed partial dentures (FPDs).[3,4] Moreover, its excellent biocompatibility is attributed to its unique properties, particularly the absence of metals that might induce allergic reactions, discoloration, or corrosion.[5] Additionally, it is noteworthy that all generations of zirconia demonstrate a wear-friendly nature when interacting with various antagonists.[6] These qualities have led to the wide-ranging application of zirconia, positioning it as a viable alternative to traditional metal and ceramic restorations.[4]

Zirconia is an oxide ceramic in three crystallographic forms: monoclinic, tetragonal, and cubic.[7] Monoclinic zirconia, the weakest phase, exists at room temperature and transforms into the tetragonal phase at temperatures higher than 1170ºC.[8] The cubic phase is observed at higher than 2370ºC.[5] Temperature changes caused by various laboratory procedures and intraoral use weaken the zirconia by causing monoclinic phase transformation from the tetragonal phase.[9-12] Tetragonal zirconia has superior mechanical and physical properties compared to other phases. Therefore, manufacturers stabilize the

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zirconia in the tetragonal phase at room temperature to use it as a dental material. Oxides such as magnesium, calcium, cerium, and yttrium are added to the zirconia, and partially stabilized zirconia is obtained at room temperature.[7,13] The 3% molyttrium is the most commonly used oxide. So, zirconium oxide is partially stabilized in the tetragonal phase at room temperature and has developed mechanical characteristics.[14,15]

Zirconia is considered a metastable material, meaning that while it exists in the tetragonal phase at room temperature, it retains internal energy, which can cause it to transform into the monoclinic phase. External stresses, such as grinding, aging, sandblasting, and high forces, can induce the tetragonal-to-monoclinic phase transformation.[16-19] Under certain conditions, such as exposure to water vapor, body fluids, and sterilization procedures, zirconia can transform from the tetragonal to the monoclinic phase, a phenomenon known as long-term degradation (LTD).[20,21] Uncontrolled and spontaneous phase transformations during hydrothermal aging of Y-TZP (yttria-stabilized tetragonal zirconia polycrystal) can lead to surface roughening, grain pull-out, and microcracks.[22,23]

Various methods have been established for assessing the aging of zirconia, focusing on the crystallographic form changes that result in surface roughness. In the literature, the most commonly employed techniques include X-ray diffraction (XRD), atomic force microscopy (AFM), Raman spectroscopy, scanning electron microscopy (SEM), and optical interferometry (OI). Diffractogram shows the patterns formed by X-rays sent to a material being refracted through atomic planes. The X-axis (2θ) represents the diffraction angle. The Y-axis (intensity) represents the intensity (density) of the X-ray signals reaching the detector, and the peaks represent the diffraction signals from a particular crystal plane. The positions and heights of these peaks are used to determine the crystal structure and phases of the material.[21] Quantitative XRD determines both the percentage of transformation and the depth distribution of the monoclinic phase. Peak points on diffraction patterns are used to determine the relative amount of the phase-modified monoclinic zirconia (Xm). The most dominant peaks are found in the XRD spectrum at $2\theta = 28.2°$ and $2\theta = 31.5°$ (monoclinic) and at $2\theta = 30.2°$ (tetragonal), respectively.[24] Since zirconia restorations are exposed to cyclic loading, temperature fluctuations, and moisture in the oral cavity, *in vitro* tests are necessary to evaluate the material's long-term success under similar conditions.[25]

The objective of this study was to investigate the impact of aging procedures and different measurement points on the phase transformation of posterior FPD zirconia frameworks. Zirconia frameworks are widely used in dental prosthetics due to their high strength and esthetic appeal. However, their long-term durability can be compromised by phase transformation, which may lead to mechanical failure and reduced lifespan of dental restorations. The study aimed to address the following problem: How do aging procedures and different measurement points affect the phase transformation of zirconia frameworks, specifically in terms of increasing the percentage of monoclinic phases, which can negatively impact the material's properties? The first null hypothesis posited that aging procedures would increase the percentage of monoclinic phases at comparable rates. The second null hypothesis suggested that there would be no significant difference in phase transformation ratios between the various measurement points.

Materials And Methods

A phantom model of the mandible (Frasaco, Tettnang, Germany) was utilized to replicate the clinical scenario of a 3-unit FPD replacing the first molar. The left mandibular second premolar and the left mandibular second molar were prepared with shoulder margins, mimicking the complete crown preparation following their anatomical shapes. To facilitate the design and manufacturing process, a CAD-CAM scanner (Yena D30, Yenadent, Istanbul, Turkey) was used to digitize the model. Subsequently, the final cast for the designed restorations was obtained from a Cr-Co (Chromium-Cobalt) block (Bego, Bremen, Germany) and it is shown in Figure 1a. In the subsequent steps, Cr-Co abutments were explicitly produced for simulating mastication and were scanned using a CAD-CAM scanner (MCXL, Sirona, Salzburg, Austria). The digital casts were transferred into a virtual environment, enabling further analysis and evaluation of the restorations and abutments.

Zirconia FPD frameworks were designed using a CAD-CAM device on the digital casts (MCXL, Sirona, Salzburg, Austria), and designed frameworks are shown in Figure 1b. The cement gap was disregarded during the design process, and the occlusal surfaces were rendered flat. For the production of the frameworks, 36 examples of this design were manufactured utilizing presintered zirconia blocks (inCoris ZI, Sirona, Salzburg, Austria) designed explicitly for CAD-CAM applications. The chemical composition and technical properties of the ceramic specimens used in the study are presented in Table 1.

After milling, the obtained framework specimens were separated from the rods and any remaining residues. Subsequently, the specimens were subjected to a sintering process using a sintering furnace (inFire HTC, Sirona, Bensheim, USA) for 2 hours, following the fast sintering mode per the manufacturer's instructions [Figure 1c and d].

Following the sintering, the specimens were randomly divided into three groups, each containing 12 specimens. The division was based on the factor of "aging procedure." The three groups were labeled as follows: Thermocycling (Group T), Mastication cycle (Group M), and Thermocycling and mastication cycle combined (Group T + M)

The crystalline structure analysis of the zirconia specimens was conducted before and after subjecting them to aging procedures using an X-ray diffractometer (X-Pert, Nottingham, UK) with monochromatic CuK α ray. XRD analysis was performed at three points on the specimens: the mesial connector, distal connector, and pontic center [Figure 2].[16] During the analysis, the specimen was securely placed in the instrument's specimen holder, and the XRD scans were performed on the specimen surface over the range of 20–60 degrees, with angles measured at 0.01-degree intervals. Density values obtained from the XRD analysis were recorded. The regions with increased density in each specimen were identified, and the corresponding 2ϴ angles were determined. The diffraction patterns obtained from the scans were analyzed using computer software compatible with the XRD system (High Score Plus; Malvern Panalytical). The software helped identify the highest peak values and the corresponding diffraction angles where these peaks were observed. Predictor variables of this study are aging procedures and measurement points (mesial connector, distal connector, pontic center).

To determine Xm on the treated surfaces of the specimens close to the tetragonal phase, the Garvie and Nicholson method[26] was employed. The monoclinic phase fraction was expressed as the percentage of the tetragonal phase that had transformed into the monoclinic phase. The amount of monoclinic phase was calculated with the following formula[27] using the area of intensity for the monoclinic peaks divided by the total area of intensity for both monoclinic and tetragonal peaks:

$$X_m = [I_m(-111) + I_m(111)]/[I_m(-111) + I_m(111) + I_t(101)]$$

In this study, three groups of specimens were subjected to different aging procedures to assess their effects on the phase transformation in the mesial, distal, and pontic centers of the zirconia FPD frameworks. Group T: The specimens in this group underwent thermocycling using a thermocycler (SD Mechatronics, Offenburg, Germany). The thermocycling involved subjecting the specimens to 20,000 cycles in water, with temperatures alternating between 5°C and 55°C for 30-second periods. The transition time between cycles was set to 5 seconds. Each 10,000 thermal process in this procedure corresponded to approximately 1 year of clinical use.[28] Group M: The specimens in this group were exposed to 500,000 cycles in a mastication simulator (SD Mechatronics, Offenburg, Germany). The mastication simulation involved applying a chewing frequency of 1.3 Hz and a force of 49 N to the specimens. On average, 240,000 to 250,000 mastication simulator cycles were considered equivalent to 1 year of *in vivo* wear.[29] As in Group T, a 6 mm steatite antagonist was used to replicate tooth enamel's physical properties and wear characteristics.[30] Group T + M: The specimens in this group were subjected to a combination of thermal cycling and mastication simulation. First, the thermal cycle (20,000 cycles/5–55°C/30 s) was applied, followed by the mastication simulator (500,000 cycles/1.3 Hz/49 N).

According to the data obtained, the effects of the thermocycling, mastication simulator, and both test methods on the phase transformation in the mesial, distal, and pontic centers of the specimens were analyzed by SPSS 22.0 (SPSS Inc, Chicago, Illinois, USA) computer software. Statistical analysis was performed using 1-way ANOVA, Tukey HSD, and Kruskal–Wallis tests (α =,05). These statistical analyses aimed to determine if there were any significant differences in the phase transformation among the different aging procedures and measurement points within the zirconia FPD frameworks.

RESULTS

Figure 3 illustrates the phase change rates for the different aging types and measurement regions. Table 2 shows the results of the 1-way ANOVA and Kruskal–Wallis tests for the phase transformation rates concerning the aging procedures. Table 2 shows statistically significant differences between the effects of the different aging methods on the phase transformation ($P < 0.05$). When analyzing the aging procedures without considering the measurement regions, it was observed that Group M had a higher phase transformation rate than Group T ($P < 0.05$). Moreover, the group where both the mastication cycle and the thermal cycle were applied (Group T + M) showed higher phase transformation than the groups where only the thermal cycle or the chewing simulator was used ($P < 0.05$). Finally, the specimens subjected to thermocycling alone

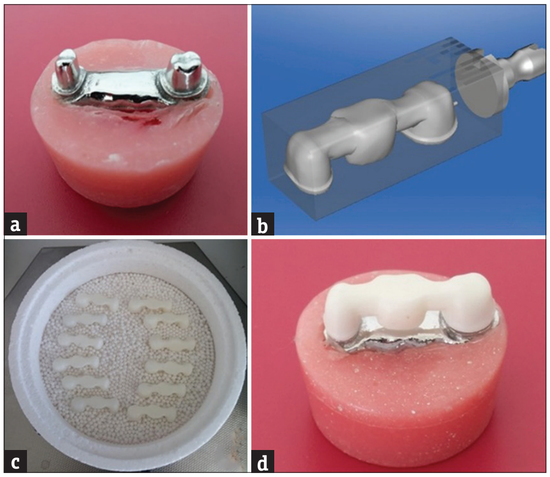

Figure 1: Production process of specimens (a: Cr-Co abutment model, b: Image of digital framework design placed on zirconia block, c: Zirconia frameworks after sinterization, d: Final restoration and abutment fitting control)

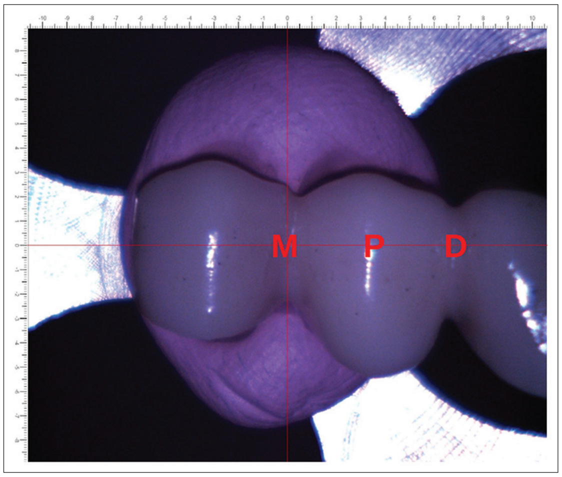

Figure 2: Measurement points on the XRD camera image (M: mesial connector, P: pontic, D: distal connector)

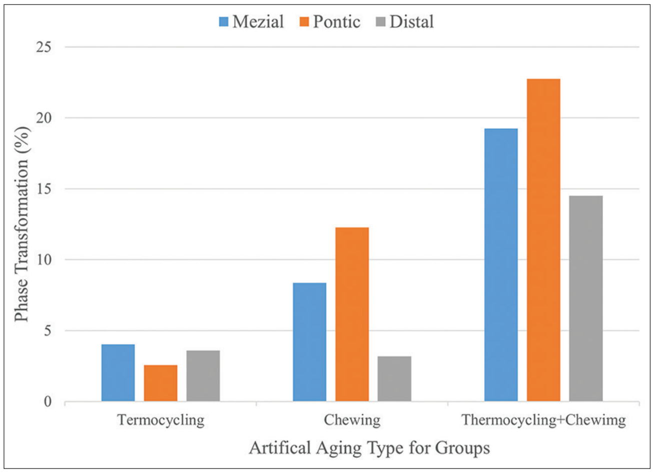

Figure 3: According to region and aging procedures phase transformation

Table 1: The chemical composition and technical properties of inCoris ZI

Chemical Composition	$ZrO_2 + HFO_2 + Y_2O_3 \geq 99.9\%$ $Y_2O_3 = 5.4\%$ $Al_2O_3 \leq 0.35\%$ $Fe_2O_3 \leq 0.01\%$ Other oxides ≤0.2%
Technical Properties	Density (ρ)=6.08 g cm^{-3} Fracture toughness (K_{IC})=6.4 MPa $m^{1/2}$ Thermal expansion coefficient (TEC)=10.4 10^{-6} K^{-1} Flexural strength >900 MPa

Table 2: Relative amount (%) of the monoclinic phase in three experimental groups

Group	*n*	Mean	±SD	*P*
T	36	3.4^a	0.64	79.00*
M	36	7.93^b	3.84	0.00
T+M	36	18.8^c	3.67	

*Kruskal-Wallis T, Thermocycling; M, Mastication Cycle; T+M, Thermocycling + Mastication Cycle; SD, standard deviation. Different superscripted letters in the same column show significant differences ($P<0.05$)

Table 3: Relative amount (%) of the monoclinic phase in three experimental groups

Group	*n*	Mean	±SD	*P*
M	36	10.53^{ab}	6.54	7.992*
P	36	12.5^a	8.40	0.018
D	36	7.58^b	5.34	

D, Distal; M, mesial; P, pontic; SD, standard deviation. Different superscripted letters in the same column show significant differences ($P<0.05$). *Kruskal–Wallis

exhibited the lowest Xm value among all groups. These findings demonstrate that the applied aging procedures significantly impact the phase transformation rates of the zirconia FPD frameworks, and the combination of the mastication cycle and thermal cycle led to the highest phase transformation levels.

Table 3 presents the results of the one-way ANOVA and Kruskal–Wallis tests for the phase transformation ratios of the specimens, considering different regions of the zirconia FPD frameworks. The analysis focused on evaluating the monoclinic phase change independently of the aging method group to which each specimen belonged. According to the findings in Table 3, significant differences were observed in the phase transformation among different parts of the FPD ($P < 0.05$). Specifically, the phase transformation ratio at the pontic point was higher than that in the mesial connector region. However, no statistically significant difference was noted between these two regions ($P > 0.05$). Similarly, the phase transformation ratio in the pontic region was higher than that in the distal connector region, with the differences being statistically significant ($P < 0.05$). However, no significant differences were observed when comparing the mesial and distal connector points regarding phase transformation ($P > 0.05$). The results indicate that the pontic region exhibited the highest Xm value, indicating a higher percentage of phase transformation toward the monoclinic phase in this area compared to the other regions.

Discussion

The main objective of this study was to investigate the impact of aging procedures and different measurement points on the phase transformation of posterior three-unit zirconia fixed partial denture (FPD) frameworks. The study findings revealed that all aging procedures increased the monoclinic phase in all specimens. However, when the mastication cycle and the thermal cycle were applied together, a statistically higher phase transformation was observed compared to the group where only the thermal cycle or the chewing simulator was used ($P < 0.05$). As a result, the first null hypothesis was partially rejected, indicating that the combination of mastication and thermal cycling had a more significant effect on the phase transformation than either method used in isolation. Furthermore, the study demonstrated that the phase transformation varied across different regions of the FPD frameworks. Specifically, the phase transformation ratio in the pontic region was statistically higher than in the distal connector region ($P < 0.05$), leading to the rejection of the second null hypothesis.

To evaluate the long-term success of all-ceramic systems, they should be examined under various conditions for long-term durability *in vitro* before clinical applications. For this purpose, 3-unit zirconia FPD frameworks, frequently applied in the mandibular posterior region, were tested for long-term phase transformations. The effect of different aging procedures on phase transformation in the zirconia frameworks was investigated. Since the mandibular first molars are permanent teeth that are first lost in the mouth and lost the most frequently, the study was planned on the lack of this tooth.[31]

Various laboratory types of research have been conducted to evaluate the feature of dental materials, temperature variations, to simulate the impact of the mastication load, and a combination of these factors.[15,25,32] Fatigue devices are commonly used to study the feature of these materials.[15,25,33,34] The Y-TZP has a mechanism for phase transformation that hinders crack progression when it is subject to aging. On the other hand, there is insufficient information about its behavior when exposed to prolonged fatigue and aging. This study applied a thermal cycle and mastication simulator to evaluate the phase transformation due to thermal and mechanical factors in FPD frameworks with zirconia frameworks. Zirconia stability was evaluated to determine whether aging occurred. XRD was used to compare the corresponding diffractograms before and after the aging procedures and to characterize the crystalline phases detected.[16] According to current studies, the most appropriate method seems to be thermal cycle and mechanical load fatigue assessment.[20,35-37] Therefore, the use of the thermal and mastication cycles, both separately and together, was preferred in this study.

The analysis of the diffractograms before aging procedures showed that all specimens had similar diffraction patterns in this study. However, after aging procedures, it was observed that the specimens had different diffraction patterns and contained a monoclinic phase. As a result of this study, it was observed that more monoclinic phases occur in the pontic region. This can be explained by positioning the tip of the chewing simulator in the occlusal center of the pontic. Since the chewing force is concentrated on the pontic, it was observed that the amount of monoclinic phase was higher in the pontic than in the connector regions. Recent literature supports these findings. A study observed that nearly all 3-unit monolithic zirconia restorations subjected to an aging procedure fractured at the connector area during the fracture test.[38]

Kawai *et al*.[39] found that zirconia stabilized with yttrium undergoes phase transformation with hydrothermal effects but less in zirconia reinforced with alumina. This study measured monoclinic contents ranging from 2.5% to 20% in regions where there are applied thermal cycles. Also, it was found that applying the thermal cycle to the models with a chewing simulator increased the monoclinic content more than the specimens used only with the thermal cycle. A study evaluating the stability of 3Y-TZP zirconia abutments after thermal and mechanical cycles reported that signs of aging were not observed after a 5-year simulation of its clinical use.[20] On the contrary, Peampring *et al*.[18] stated hydrothermal aging caused phase transformation and increased surface roughness.

XRD is the most commonly used nondestructive method to evaluate stability by characterizing crystallographic phases from a qualitative and quantitative perspective.[20] The analysis of the phases expressing the shapes of the crystal structures that make up the material is done by the XRD method. Guazzato *et al*.[40] applied different surface treatments to zirconia-reinforced alumina-based ceramic material and evaluated the material's crystal structure change with XRD analysis. Borchers *et al*.[25] estimated phase conversion rates by XRD analysis after applying different thermal and mechanical tests to zirconia stabilized with 3 mol yttria. This study used XRD analysis to evaluate the phase transformation from tetragonal to monoclinic in zirconia FPD frameworks, similar to previous studies.

XRD gives fewer precise results, especially in small changes in fractions of less than 5% of the transformed phase, which occurs in the early stages of aging.[30] Another limitation is that the XRD is limited only to the surface that does not exceed a surface depth. Therefore, the results obtained from the analyzes may differ slightly depending on the position of the scanning beam in the specimen.[41] Arata *et al*.[42] reported that the Toraya equation showed more monoclinic phase fractions than the Rietveld method. Therefore, when comparing the results of the previous studies, it should be considered which equation is used to calculate the monoclinic phase fraction.

The application of long-term dynamic loads, such as chewing, is the main factor in the failures of ceramic restorations.[32] This study showed that the mastication simulation caused more phase transformation than the thermal cycle. The results of this study support these statements. In principle, phase transformation does not permanently damage dental zirconia. However, as the amount and frequency of load application increase in a humid environment, especially in thermal variation, problems may occur for prostheses made of zirconia.

The limitations of this study are as follows: It is an *in vitro* study, zirconia material of different brands was not included in the study, no veneer ceramics were applied to the frameworks, aging procedures were applied for a short time, and only XRD analysis was used to evaluate the phase transformation. These limitations do not invalidate the results and conclusions of this study but provide a basis for further future research.

CONCLUSION

Within the limitations of this study, the following conclusions can be drawn. Mechanical factors affect phase transformation more than thermal factors in zirconia frameworks. Thermal and mechanical effects cause much more phase transformation when applied together. More *in vitro* and *in vivo* studies are needed to evaluate the long-term success of the material and for more detailed results.

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Conflicts of interest

There are no conflicts of interest.

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