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Effect of Thermal and Mechanical Aging on Flexural Strength of Zirkonzahn and Mamut Zirconia Ceramics

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Abstract

Objective: Despite the high strength of zirconia restorations, aging in the oral environment and masticatory loading may result in transformation of tetragonal to monoclinic phase and decrease their strength. Statements in this regard are controversial. This study sought to compare the flexural strength (FS) of Zirkonzahn (ZirkonZahn, Cercon, Ceramill) and Mamut (Dubai Medical Equipment LLC, Dubai, UAE) zirconia ceramics and assess the effect of thermal and mechanical aging on their FS.

Methods: In this *in vitro* experimental study, 40 bar-shaped specimens measuring 20×5×2 mm were cut from Zirkonzahn and Mamut zirconia blocks and polished. Specimens in the aging groups were subjected to thermocycling (12,000 cycles, 5-55°C, dwell time of 20 seconds). Next, they were subjected to mechanical stress in a chewing simulator (40,000 cycles, 200N force). The three-point flexural strength (TPFS) was determined in megapascal (MPa) using a Universal Testing Machine at a crosshead speed of 0.5 mm/min. Data was analyzed using two-way ANOVA.

Results: The mean and standard deviation (SD) of TPFS of Zirkonzahn and Mamut specimens in the no aging group was 809.57 (205.95) and 708.53 (158.72) MPa, respectively. These values were 810.53 (158.96) and 839.06 (217.49) MPa for the Zirkonzahn and Mamut specimens subjected to aging, respectively. Type of zirconia (Zirkonzahn or Mamut) and exposure to aging process (p=0.27) had no significant effect on TPFS of specimens.

Conclusion: Within the limitations of this study, the results showed that the process of aging did not decrease the TPFS of Zirkonzahn and Mamut specimens. Thus, these ceramics may be successfully used in the clinical setting.

Key words: Aging, Ceramic, Strength, Zirconia.

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Introduction:

Zirconia-based ceramics are the most-recent generation of high-strength ceramics. Favorable mechanical properties, high esthetics, excellent biocompatibility, insignificant plaque accumulation and low thermal conductivity are among the advantages of zirconia ceramics. All these factors play a role in selection of these ceramics for esthetic tooth restorations (1).

Mechanical properties of dental materials such as their strength are the main parameters taken into account when assessing the possible applications and clinical limitations of dental restorations. Despite the favorable mechanical properties of zirconia ceramics, the surface of zirconia specimens may undergo changes. Occasionally, in humid environments at relatively low temperatures, the tetragonal phase of zirconia may transform into the monoclinic phase (2). This phenomenon, known as the low temperature degradation, is related to the partial stability of the tetragonal phase at room temperature and may lead to the formation of small and large cracks, decrease strength and impair mechanical properties of zirconia (3-5). This mechanism occurs very slowly in the oral environment but may decrease the strength and density of zirconia restorations (6, 7). Moreover, the strength may further decrease due to cyclic stresses attributed to the masticatory loading in the clinical setting (8, 9). Thermal stresses may also decrease the zirconia strength and create strain inside the ceramic materials (10, 11). Some studies have demonstrated that the process of aging of yttria-stabilized zirconia specimens at low temperatures causes the transformation of tetragonal to monoclinic phase and creates small cracks in the zirconia structure (12). The speed of phase transformation is related to factors such as temperature, size of grains, dopant concentration (13) and surface polishing of specimens (14). Rapid phase transformation leads to the destruction of ceramic specimens and decreases their strength.

Dopants, available in the form of solid solutions, are added to zirconia ceramics in small amounts to change their chemical properties. Dopants like silica and alumina directly alter the morphology, substructure and stability of zirconia ceramics (15, 16). Ceria, also added to zirconia ceramics, plays a role in the process of sintering and improves resistance to aging (17, 18).

Due to functional loading in the oral environment, zirconia-based restorations may develop fatigue in spite of high strength (19). Over time, these stresses accumulate and cause tiny defects (20) and small cracks (21) in the material.

The process of aging may affect the strength of dental ceramics (22). Thus, it is necessary to assess the effect of aging on the strength of zirconia ceramics. Considering the existing concerns regarding the adverse effects of aging on mechanical properties and strength of zirconia ceramics, this study aimed to compare the FS of Zirkonzahn and Mamut zirconia specimens and evaluate the effect of thermal and mechanical aging on their FS.

Methods:

This was an *in vitro*, experimental study. Specimens were prepared in a laboratory and those with inaccurate dimensions were excluded and replaced with accurate ones. Specimens were evaluated in four groups of 10. Sample size was calculated to be 10 specimens in each group by a statistician according to similar previous studies (22, 23, 24). Specimens were randomly divided into four groups of Zirkonzahn and Mamut zirconia specimens with and without aging.

Twenty specimens were fabricated of each type of zirconia according to the manufacturers' instructions. Bar-shaped specimens, 4 (0.25) mm in width, 1.2± 0.2 mm in thickness and 20mm in length, were cut from ceramic blocks via the machining process using 30-40µabrasive diamond discs. Specimens were then polished with 15-20µ diamond discs and the opposing surfaces were paralleled. Next, all Zirkonzahn and Mamut specimens were rinsed with distilled water to remove any remaining debris. Zirkonzahn and Mamut zirconia specimens in the aging groups were thermocycled (Thermocycler, Dorsa, Iran) for 12,000 cycles. At each cycle, specimens were immersed in a distilled water bath at 5°C for 20 seconds, followed by 20 seconds of dwell time in the air and another 20 seconds in a distilled water bath at 55°C. After thermocycling, bar-shaped specimens were placed in CS-4 chewing simulator (SD Mechatronik, Feldkirchen-Westerham, Germany) to receive 40,000 cycles at a load of 200N=20Kg in order to simulate masticatoryloads in the clinical setting.

Three-point flexural strength testing was performed according to ISO6872 standards. This test was performed under dry conditions at room temperature by Universal Testing Machine (Santam, Iran). Bar-shaped specimens were placed on the two jigs of the machine. The jigs had a semi-circle cross-section with 1.6mm diameter. The span length was 15mm. Compressive strength was applied by the upper blade (with a semi-circle cross-section and1.6mm diameter) from the top at a crosshead speed of 0.5 mm/min in between the two jigs (Figure 1) until fracture.

Figure 1- Bar-shaped specimen placed on the two jigs. Load was applied from the top by the blade for TPFS testing

To calculate mechanical parameters by the software of Universal Testing Machine, three different parameters are defined for each specimen: thickness of specimen, width of specimen and length of specimen. These parameters were accurately measured for each specimen using a digital micrometer (Mitutoyo Ltd., Andover, England) and entered in the software as the characteristics of the specimen. After TPFS testing, the load at failure for each specimen was calculated in MPa and the FS was calculated using the following equation:

$TPFS=3wl/2bd^2$

Where w is the load at failure (N), l is the length of span (mm), b is the width of specimen and d is the thickness of specimen (mm)

TPFS data of specimens in different groups were statistically analyzed using two-way ANOVA.

Results:

Based on the results, the mean and SD of TPFS of Zirkonzahn specimens was 809.57 (205.95) MPa in the no aging and 810.53 (158.96) MPa in the aging group. For Mamut specimens, these values were 708.53 (158.72) MPa and 839.06 (217.49) MPa, respectively (Table 1).

Type of zirconia ceramic (Zirkonzahn or Mamut) (p=0.54) and the aging process (p=0.27) had no significant effect on the FS of specimens. Moreover, the interaction effect of the type of zirconia and aging on flexural strength was not statistically significant (p=0.28).

Table 1- The mean and SD of TPFS of Zirkonzahn and Mamut specimens with and without aging

Group	Aging	Number	mean	SD	Minimum	Maximum
Zirkonzahn	Aging	10	810.53	158.96	482.94	1111.64
	No aging	10	809.57	205.95	592.23	1242.65
Mamut	Aging	10	839.06	217.49	572.74	1340.44
	No aging	10	708.53	158.72	461.98	964.49

Discussion:

In this study, Mamut and Zirkonzahn specimens were subjected to thermal and mechanical cycles to assess the effect of these processes on TPFS of ceramics. Based on the results, aging or type of zirconia had no significant effect on FS of specimens. Moreover, the two types of zirconia were not significantly different in terms of FS. The obtained FS values for Zirkonzahn and Mamut zirconia were not similar to the values claimed by the manufacturers (1400 and 1100 MPs, respectively). This difference may be explained by the general differences in preparation of specimens, sintering conditions and consequent differences in microstructures and sum of cracks (25).

Our study showed that aging had no adverse effect on FS of zirconia specimens. This result has also been confirmed by several previous studies (26). In a study by Papanagiotou *et al*, in 2006, the process of thermal aging at low temperatures had no negative effect on FS of Y-TZP ceramics cut from In-Ceram YZ ceramic blocks (26). Ardlin (2002) demonstrated that FS of Y-TZP high-strength ceramics was not influenced by the process of thermal aging at low temperatures or chemical aging (immersion in 4% citric acid for 168 hours at 80°C)(27). In a study by Picconi, *et al*. (1998) zirconia specimens were stored in Ringer's solution at 37°C for different time periods and after one year no change occurred in the FS of specimens. In their study, hydrothermal procedures at 120°C for 120 hours in a humid environment had limited effects on the FS of Y-TZP ceramic specimens (28).

Yilmaz *et al*. in 2011 evaluated the effect of aging on FS of Lava and Cercon zirconia ceramics (22). In their study, specimens were subjected to 20,000 cycles of 200N load with 2Htz frequency and no significant difference was noted in the FS of aging and no aging groups. Vult Von Steyern *et al*. (1993) assessed the effect of 10,000 mechanical cycles and 5000 thermal cycles on FS of specimens (23) and reported no significant difference in FS of specimens with and without aging.

Pittayachawan *et al*. (2007) and Curtis *et al*. (2006) calculated the FS of Lava zirconia with and without mechanical cycles (10,000 and 20,000 cycles at 250N, 2000 cycles at 500N, 700N and 800N and 10,000 and 100,000 cycles at 80N load, respectively) and reported no significant effect on zirconia strength (21, 29).

In a study by Brochers *et al*. (2010), the effects of different environments and loading conditions on the FS of In-Ceram YZ and Lava zirconia specimens were evaluated. Mechanical loading with 100N stress for 1 and 5 million cycles, thermocycling for 10,000 cycles and storage in distilled water at 36°C for 200 days, 80°C for 64 days and 134°C for 8 hours were found to have no significant effect on zirconia strength (25).

It appears than zirconia is resistant to aging during functional loading cycles for at least 75 months with 800,000 masticatory cycles per year (11). In other words, this level of aging during this time period cannot cause zirconia destruction (29).

In our study, similar to many previous ones, aging had no significant effect on FS. However, some others have reported reduction in FS of zirconia ceramics. If the phase transformation does not continue into the material mass, the FS would not decrease significantly. In other words, transformation of tetragonal to monoclinic phase starts at the material surface and propagates inward. This phase transformation at the surface is associated with increased volume but does not cause significant internal stress. Only after formation of a significant amount of monoclinic phase on the surface, stresses may accumulate and cause microcracks. When microcracks reach adequate length, they can cause fracture and decrease the strength of the ceramic (25). This case scenario did not occur in any of our understudy specimens.

Kim *et al*. in 2009 investigated the effects of aging at low temperatures on mechanical properties and phase stability of Y-TZP ceramics and reported that these effects were influenced by the temperature at which the specimens were stored (30). Chevalier *et al*. (1999) reported that by increased temperature and duration of aging, the rate of phase transformation increased and this issue was related to the size of grains (8). Simultaneous with an increase in tetragonal grain, the stable phase gradually disappears. In the current study, the FS of zirconia groups increased following the process of aging. Theoretically, aging at 134°C for one hour corresponds to 3-4 years of clinical service (31, 32). Itinoche *et al*. (2006) measured the FS of In-Ceram zirconia with and without mechanical cycles (20,000 cycles at 50N load) and demonstrated that mechanical cycles decreased the FS of specimens; however, this reduction was not statistically significant (24).

Chevalier *et al*. (1999) indicated that as the result of aging at 130°Cfor 7 hours, for a monoclinic content higher than 30%, the rate of nucleation reached the saturation level (13). Thus, one possible explanation for increased FS may be the concentration of the monoclinic phase. The transformation of tetragonal to monoclinic phase starts at the surface of zirconia specimens. As long as the monoclinic phase is limited to a small area on the surface, changes in grain size and physical properties are insignificant (21). On the other hand, residual stresses are eliminated and the expansion created by the phase transformation prevents crack propagation (6). This process may be related to the increased FS of specimens. On the other hand, when the saturation phenomenon occurs at the surface of zirconia in the monoclinic phase, the transformation phase occurs in the body of ceramic. In this situation, internal cracks may occur in the critical zone and cause inevitable reduction in FS (6).

Vásquez *et al*. (2008) investigated the effects of mechanical and thermal cycling on FS of glass ceramics fused to titanium and reported that application of mechanical and thermal cycles decreased the FS of specimens in comparison with the control samples (31).

Att *et al*. (2007) compared the FS of Vita YZ, Cerec In-Lab and Procera specimens with and without aging. Specimens in the aging group were subjected to 120,000 thermo-mechanical cycles (corresponding to 5 years of clinical service) with 49N force, which resulted in a reduction in strength of specimens particularly in the Procera group (32).

The controversial results obtained by different studies may be due to the different aging processes or type of strength testing. Density of presintered zirconia blocks (due to correlation with critical crack size), the sinterability of the powder (due to correlation with the size of primary gains), mechanically created cracks, residual compressive stresses during the preparation of specimens and the yttria content (due to its significance in transition of tetragonal to monoclinic phase) also play a role in ceramic strength.

Previous studies on the phase stability of zirconia ceramics mostly showed no reduction in strength as the result of aging. Tinschert in 2000 and Tanaka in 2003 reported that zirconia ceramics fabricated in early 1980 were less stable than the currently used Y-TZP ceramics (33, 34). Moreover, it appears that standard Y-TZP ceramics with high density and adequate concentration of Yttria did not experience any reduction in strength related to phase transformation even after long-term clinical service. Also, researchers reported that commercial Y-TZP ceramics still had adequate stability after being subjected to long-term clinical and experimental aging procedures and no aging-related mechanical destruction was noted (35). The results of the current study regarding no significant change in FS of Zirkonzahn and Mamut specimens following aging was in accord with the findings of previous studies (27, 35).

Conclusion:

Aging had no significant effect on TPFS of

Zirkonzahn and Mamut zirconia ceramics. Thus, they seem to have adequately high strength for application in the clinical setting and they are expected to show favorable results in long-term clinical service. However, clinical studies are required to cast a definite judgment in this respect.

Conflict of interest: "None Declared"

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