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RESEARCH ARTICLE

EFFECT OF AGING ON TZP ON HARDNESS AND FLEXURAL STRENGTH – AN IN VITRO STUDY

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ABSTRACT

Objective: The purpose of this study was to evaluate the hardness and flexural strength of yttrium stabilised zirconia ceramics after various low temperature aging using computerised micro hardness tester and universal testing machine respectively. **Materials and methods:** The study included blocks of yttrium stabilized tetragonal zirconia from two manufacturers (n=15) Group A-AIDITE (Qinhuangdao Technology Co., Ltd, Hebei, China) and Group B-UPCERA (Shenzhen Upcera Dental Technology Co., Ltd, Guangdong, China), for each brand were machined, sintered and glazed according to manufacturer's specifications to mimic a final polished crown. The sintered blocks were of 40mm (length) ×5mm (width) ×3mm (height) in dimensions approximately. Specimens were artificially aged in distilled water by heat treatment at temperatures of 100°, 150° and 200°C for 10 hours each in order to induce phase transformation. These specimens after aging were individually evaluated for hardness using computerised microhardness tester (Reichert Austria microhardess tester) and flexural strength using universal testing machine (Unitest-10). **Results:** The results from hardness testing showed that the hardness was highest without aging. There was a considerable decrease in hardness in group B when compared to group A. The results from flexural strength evaluation showed a decrease in the mean flexural strength with aging in Group A. On the contrary, flexural strength was higher without aging. Furthermore, the flexural strength in group B kept fluctuating and was the highest when aged at 100°C. A sudden decrease in the flexural strength was noted when aged at 150°C. **Conclusion:** The in vitro tests conducted reveal that when low temperature aging was carried out at a temperature of 100°C for 10 hours, the hardness of Y-TZP ceramics decreased whereas the flexural strength increased. However, beyond this temperature of 100°C especially at 150°C and above, the flexural strength of both the materials started to decrease.

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INTRODUCTION

Zirconia, emerging as a supreme material with great value of strength and esthetics is being widely used in the field of dentistry. The array of applications of zirconia in dentistry include fabrication of posts, crowns, bridges, implants, implant abutments and esthetic orthodontic brackets (Manicone *et al.,* 2007). Zirconia exists in three crystal phases at different temperatures namely cubic, monoclinic and tetragonal. Zirconia is in the monoclinic phase at room temperature and transforms into tetragonal phase above 1070°C. This tetragonal phase of zirconia is stable between the temperature of 1170°C and 2370°C. The cubic phase is stable only at very high temperatures (Kim *et al.,* 2009). Clinically, the tetragonal phase of zirconia is important as it is hard, has good fracture toughness and is corrosion resistant. In order to maintain this clinically significant tetragonal phase at room temperature, stabilisers such as yttria, magnesia, ceria etc are added. Occlusal and lateral forces along with added influence of saliva bring about a transformation from the tetragonal to monoclinic phase which would be undesirable normally. Interestingly, during clinical use this transformation is beneficial as this exhibits a volume expansion of 3-4% (Subbarao *et al.,* 1974). This expansion inhibits the crack propagation that is initiated by various stresses. This transformation toughening phenomenon brings about high strength and fracture toughness of zirconia in vivo. Though zirconia has excellent mechanical properties, various phenomena that decrease the life time of zirconia have been reported. Kobayashi et al were the first to observe the degradation after aging process at 150-400°C (Kobayashi *et al.,* 1981). Since then, various studies have been carried out on low temperature aging phenomenon affecting the mechanical properties (Chevalier *et al.,* 1999). To test the clinical performance of yttrium stabilised tetragonal zirconia (Y-TZP) in vitro, a process known as low temperature aging is carried out. Low temperature aging of zirconia is tetragonal to

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monoclinic transformation under simulated clinical conditions. This mimics the clinical conditions in the oral cavity and tests the performance of Y-TZP over a period of time. For example, aging at 134°C for 1 hour is theoretically similar to 3-4 years in vivo (Kim *et al.,* 2009; Chevalier *et al.,* 1999; Cales, 2000; Deville *et al.,* 2005; Anehosur *et al.,* 2017).

MATERIALS AND METHODS

Blocks of Y-TZP from two manufacturers (n=15) Group A-AIDITE and Group B-UPCERA for each brand were machined, sintered and glazed according to the manufacturers specifications to mimic a final polished crown that is ready for cementation (Fig-1a and 1b). These sintered blocks were of 40mm (length) × 5mm (width) × 3mm (height) in dimensions approximately. Control specimens (n=10) of each group were evaluated in the as-received condition.

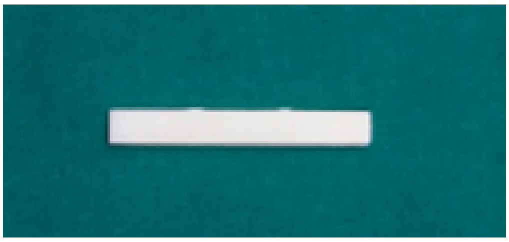

Fig 1a. Y-TZP block- Group A:-AIDITE

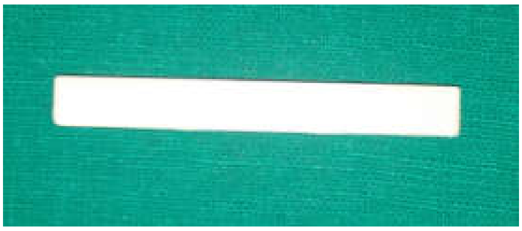

Fig 1b. Y-TZP block- Group B-UPCERA

Low temperature aging: Specimens were artificially aged in distilled water by heat treatment at temperatures of 100, 150 and 200°C for 10 hours each in order to induce phase transformation (Fig-2). Further these specimens were evaluated for hardness using a microhardness tester and flexural strength using a universal testing machine.

Hardness: The microhardness in (HV) was tested using a Reichert Austria make microhardness tester at an applied load of 100g (Fig 3).

Flexural strength: The flexural strength of the specimens was tested using model no Unitest-10 universal testing machine having a crosshead speed of 0.5mm/minute and a span length of 25mm (Fig-4a and 4b). The following equation was used to calculate the flexural strength.

Flexural Strength = $3PL / 2bd^2$

Where P is Fracture load; L is span between supports; d is thickness; b is width

Statistics: Statistical analysis was performed with SPSS (Statistical Packages for Social Sciences) software. Descriptive statistics including the mean, standard deviation, median, minimum and maximum values of flexural strength were calculated for each group. Kolmogorov- Smirnov test was conducted to assess the normality of variance. The flexural score in each group followed a normal distribution. Therefore, the data of the experimental groups were analysed by one way analysis of variance (ANOVA).

Fig Low temperature aging

Fig. 3. Microhardness tester

Fig. 4a. and Fig. 4b. Universal testing machine

RESULTS

Hardness: The hardness testing consisted of making a dent in the Groups with a load of 1N (100g). A gradual decrease in hardness was noted as the aging temperature increased. The hardness was highest without aging as compared to when aging was carried out (Table-1). There was a considerable decrease in hardness in group B when compared to group A.

Flexural strength: In Group A, there was a decrease in the mean flexural strength when aging was carried out. The flexural strength decreased from room temperature to 150°C and increased slightly at 200°C. However, the flexural strength was the highest without aging. Furthermore, the flexural strength in Group B kept fluctuating and was the highest when aged at 100°C. A sudden decrease in the flexural strength was noted when aged at 150°C (Table 2).

Table 1. Hardness in HV before and after aging in group A and group B

	Equivalent to	Microhardness (in HV) Group A (AIDITE)	Microhardness (in HV) Group B (UPCERA)
Without Aging		1198	1088
Aging 100ºC	8-10 yrs	1103	843.66
Aging 150ºC	10-15 yrs	917.66	825
Aging 200ºC	15-20 yrs	882.3	740.66

Table 2. Flexural strength before and after aging in group A and group B

Groups	Group-A (Flexural Strength in MPa)	Group-B (Flexural Strength in MPa)
Without Aging	1002.66	715.97
Aging at 100°C	827.77	827.77
Aging at 150°C	826.38	637.77
Aging at 200°C	899.02	720.55

DISCUSSION

The phase transformation from tetragonal to monoclinic (up to 12%) is favourable in clinical situations as it does not allow crack to propagate resulting in an increase in the flexural strength hence demonstrating an improved clinical performance of the material. As the percentage of monoclinic crystal increases beyond this i.e. 12-54%, there is a decrease in the flexural strength. In vitro phase transformation from tetragonal to monoclinic starts taking place from 100°C onwards. There is an increase in the monoclinic phase as the temperature increases. Therefore, to test the clinical performance of the material over a period of time, temperatures of 100°C, 150°C and 200°C were used. Also, the three temperatures used in the study correspond to the duration of the material in the oral cavity which signifies the longevity of the material. For instance, aging at 134°C for 1 hour is theoretically similar to 3-4 years in vivo. Hence aging at 100°C, 150°C and 200°C for 10 hours corresponds to approximately 8-10, 10-15 and 15-20 years in vivo respectively (Kim *et al.,* 2009). A study conducted by Maria et al concluded upto 30% reduction in the hardness of Y-TZP that was seen when subjected to low temperature degradation which was produced by aging Y-TZP at 140°C for 24, 96, 168 hours.[9] In contrast to this study, our test groups when subjected to low temperature aging at 150°C for 10 hours, there was a decrease in hardness by 23% in group A and decrease in hardness by 27% in group B. The study conducted by Kim et al showed an increase in flexural strength from room temperature to 125°C and was highest at 125°C after which it started to decrease (Kim *et al.,* 2009). However, in our study, even though the flexural strength of group A kept decreasing as aging was carried out till temperature of 200°C, there was no reduction in the flexural strength below 700 MPa, thus indicating that group A will give good clinical performance in both the anteriors as well as the posteriors for approximately 15-20 years. In group B, however, the flexural strength was fluctuating. The highest flexural strength was observed when aging was carried out at 100°C which suddenly decreased on aging at 150°C to 637.77 MPa, thus indicating that group B will give good clinical performance when used in the anteriors to a maximum of 8-10 years. The results from above confirm group A being a more reliable material for clinical use when compared to group B (Anehosur *et al.,* 2017).

Conclusion

(i) Positive and negative effects were noted on the mechanical properties of Y-TZP ceramics after low temperature aging, depending on the various temperatures used.
(ii) The tetragonal to monoclinic transformation was caused by low temperature aging at temperatures of 100°C, 150°C and 200°C. After aging treatment upto 100°C, monoclinic phase started to appear. The fraction of monoclinic crystals decreased with the higher temperatures above 100°C, at 150°C and 200°C.
(iii) When low temperature aging was carried out at a temperature of 100°C for 10 hours, the flexural strength of Y-TZP ceramics increased. However, beyond this temperature of 100°C especially at 150°C and above, the flexural strength started to decrease both in group A and group B.

Clinical implications of the study: The in vitro tests conducted reveal that Aidite shows sufficient flexural strength and hardness to be used as a reliable clinical material for the anterior as well as posterior restorations for 15-20 years. On the other hand, based on the fluctuating flexural strength and hardness of Upcera, it can give good clinical performance in the anterior teeth for 8-10 years but its clinical reliability as a posterior restoration is questionable.

Scope of the study

(i) The need to study the percentage of monoclinic crystals.
(ii) We recommend an in vivo simulation of lateral and vertical forces after low temperature aging.

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