



# The Effect of *in vitro* Aging and Fatigue on the Flexural Strength of Monolithic High-translucency Zirconia Restorations

<sup>1</sup>Hosain M Almansour, <sup>2</sup>Fawaz Alqahtani

## ABSTRACT

**Aim:** The aim of this *in vitro* study was to evaluate the effect of accelerated artificial aging (AAA) and fatigue on the biaxial flexural strength (BFS) of three types of monolithic high-translucency zirconia restorations compared with conventional low-translucency zirconia restorations.

**Materials and methods:** Four groups of 20 disc-shaped specimens (10 × 1.2 mm) were made from the following computer-aided design and computer-aided manufacturing (CAD/CAM) zirconia blocks: Low-translucency zirconia (Ceramill ZI-LT) as a control, and three brands of high-translucent zirconia (Lava Plus, Ceramill Zolid White, and Copran Monolithic HT). Ten discs from each group were subjected to the BFS test using the universal testing machine. The other 10 discs from each group were subjected to AAA (thermocycling, 3,500 cycles) and fatigue (250,000 cycles) before the fracture test. The definitive fracture load was recorded, and the BFS was calculated in accordance with International Organization for Standardization (ISO) 6872. The data were analyzed with one-way analysis of variance (ANOVA), Scheffe *post hoc*, and Mann–Whitney U test. Data analyses were evaluated at a significance level of  $p \leq 0.05$ .

**Results:** Significant differences were detected in the BFS among the four groups before AAA and fatigue. The mean BFS was highest with Ceramill ZI (935.3 ± 47.1 MPa), and least in Ceramill Zolid White (685.7 ± 32.6 MPa). After AAA and fatigue, significant differences were reported where the mean of BFS was highest with Copran Zr-i Monolithic HT (777.5 ± 21.2 MPa), and least in Ceramill Zolid White (576.0 ± 36.3 MPa).

Furthermore, Mann–Whitney U test showed that AAA and fatigue significantly affect the BFS of each material individually.

**Conclusion:** The AAA and fatigue significantly affected the BFS of the monolithic high-translucency zirconia restorations.

**Clinical significance:** Although monolithic high-translucency zirconia had significantly lower BFS than conventional zirconia tested in this study, they still have sufficient strength for clinical use.

**Keywords:** Accelerated artificial aging, Biaxial flexural strength, Fatigue, Monolithic high-translucency zirconia.

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**Conflict of interest:** None

## INTRODUCTION

All ceramic materials are getting more popularity due to the improvement in the mechanical properties and high esthetics outcome.<sup>1</sup> The recent development in dentistry has introduced a new high-strength alumina and zirconia-based all ceramic restorative materials which can be used for entire mouth.<sup>2,3</sup>

Furthermore, the development of CAD/CAM technologies makes it possible to mill the ceramic dental restorations from a solid ceramic block which has excellent mechanical properties.<sup>4-6</sup> The milled zirconia-based material is radiopaque and produce only the core which has to be veneered later, and the restoration fabricated using this type of ceramic suffers from chipping of porcelain veneer.<sup>7,8</sup>

A new monolithic zirconia ceramic block allows the milling of anatomic contour zirconia restorations without subsequent porcelain veneering which has significantly improved strength and resistance to chipping.<sup>9</sup> However, these monolithic zirconia restorations are radiopaque and

<sup>1</sup>Department of Prosthodontics, Riyadh Elm University, Riyadh Kingdom of Saudi Arabia; Saudi Board of Prosthodontics Program, Riyadh, Kingdom of Saudi Arabia

<sup>2</sup>Department of Prosthetic Dental Sciences, School of Dentistry Prince Sattam bin Abdulaziz University, Al-Kharj, Kingdom of Saudi Arabia

**Corresponding Author:** Hosain M Almansour, Department of Prosthodontics, Riyadh Elm University, Riyadh, Kingdom of Saudi Arabia; Saudi Board of Prosthodontics Program, Riyadh Kingdom of Saudi Arabia, Phone: +966558408889, e-mail: dr.hosain.2012@gmail.com

**Table 1:** Materials used in this study

Brand	Chemical composition	Manufacturer
Ceramill ZI-LT	ZrO <sub>2</sub> + HfO <sub>2</sub> + Y <sub>2</sub> O <sub>3</sub> : >99.0 Y <sub>2</sub> O <sub>3</sub> : 4.5–5.6, HfO <sub>2</sub> : <5 Al <sub>2</sub> O <sub>3</sub> : <0.5, other oxides: ≤1	Amann Girrbach, Koblach, Austria
Lava Plus HT	ZrO <sub>2</sub> + HfO <sub>2</sub> + Y <sub>2</sub> O <sub>3</sub> : ≥99% Y <sub>2</sub> O <sub>3</sub> : >4.5–≤6.0%, HfO <sub>2</sub> : ≤5% Al <sub>2</sub> O <sub>3</sub> : ≤0.5%, other oxides: ≤0.95%	3M ESPE, Seefeld, Germany
Ceramill Zolid White HT	ZrO <sub>2</sub> + HfO <sub>2</sub> + Y <sub>2</sub> O <sub>3</sub> : ≥99.0 Y <sub>2</sub> O <sub>3</sub> : 4.5–5.6, HfO <sub>2</sub> : ≤5 Al <sub>2</sub> O <sub>3</sub> : ≤0.5, other oxides: ≤1	Amann Girrbach, Koblach, Austria
Copran Zr-i Monolith HT	Y <sub>2</sub> O <sub>3</sub> : 5.15–5.55% Al <sub>2</sub> O <sub>3</sub> : 0.03–0.07% Iron hydroxide: 0–0.01% Other oxides: 0–0.02%	White Peaks, Essen, Germany

Data were collected from the technical product profile

cannot be used in patients demanding highly esthetic restorations.<sup>10</sup> To overcome the radiopacity, a new high-translucent monolithic zirconia block has been developed in recent years to fulfill the esthetic requirements of the patients. Mechanical properties, such as strength and fatigue are the main parameters taken into account when assessing the possible applications and clinical limitations of this new material.<sup>11</sup>

A study conducted by de Kok et al<sup>12</sup> showed that the flexural strength of the monolithic high-translucent zirconia crowns was 1,235 MPa under a three-point bending test. Another study conducted by Church et al<sup>13</sup> showed that the flexural strength of the monolithic high-translucent zirconia in beam-shaped specimens was 880 MPa.

Accelerated artificial aging is an experimental method used to simulate oral environmental conditions extra-orally.<sup>14</sup> Thermocycling through water baths at 5°C and 55°C or artificial saliva followed by fatigue or mechanical cycling was considered the most common method to assess the durability and the behavior of the dental materials.<sup>15</sup>

To date, only limited data are available regarding the effect of aging and fatigue on the BFS of high-translucent monolithic zirconia restorations. Therefore, the objectives of this *in vitro* study were:

- To evaluate the BFS of three different types of monolithic high-translucency zirconia restorations compared with the conventional low-translucency zirconia restorations.
- To study the effect of AAA and fatigue on the BFS among three types of monolithic high-translucency zirconia restorations and the conventional low-translucency zirconia restorations.

## MATERIALS AND METHODS

### Materials

Three brands of high-translucent yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics (Lava Plus HT,

Ceramill Zolid White HT, Copran Zr-i Monolith HT) and a low-translucent Y-TZP ceramic (Ceramill ZI-LT) were selected and used in this study. The brands, chemical compositions, and manufacturers of the materials used in this study are listed in Table 1. Eighty disc-shaped specimens and 20 discs from each ceramic brand were prepared from the pre-sintered zirconia blocks according to the manufacturer's directions.

## Methods

### Preparation of the Disk Specimens

A disc-shaped specimen with a diameter of 10 mm and a thickness of  $1.2 \pm 0.05$  mm (Fig. 1) was designed using three-dimensional software (Dental Wings, Montreal, Canada) and a stereolithography (STL) file was created.

Partially sintered blocks for each ceramic brand were used, and the STL file was uploaded to the specific milling machine for each brand to prepare the 20 ceramic discs ( $1.2 \pm 0.05$  mm in thickness and 10 mm in diameter) with machine incorporation to compensate for the sintering



**Fig. 1:** The disc-shaped design

shrinkage of 20 to 25% by CAD/CAM according to the manufacturer's instructions. Then, the specimens were finished by fine cross-cut tungsten carbide burs with low speed and sintered in the recommended furnace of each ceramic system according to the manufacturer's instructions.

The milling machine, the sintering oven, the sintering time, the sintering temperature, and the manufacturer for each system are summarized in Table 2. Digital caliper (Electronic Digital Caliper, Shan, China) was used to check the thickness and the diameter of the discs after sintering.

The specimens were divided into 4 groups of 20 specimens each. Furthermore, each group was divided into 2 subgroups of 10 specimens for testing the BFS before and after AAA and fatigue.

### BFS Test before AAA and Fatigue

Forty discs, 10 from the control (Ceramill ZI-LT) and 10 from each high-translucent zirconia ceramic system (Lava Plus HT, Ceramill Zolid White HT, and Copran Zr-i Monolith HT), were subjected to a single load-to-fracture using the universal testing machine (Instron 5965, Instron Corporation, USA) (Fig. 2).

According to ISO 6872:2008<sup>16</sup> and in order to carry out the BFS test, a custom-made mounting jig and loading pin were designed from stainless steel and attached to the Instron testing machine. The loading stainless steel pin was six sided in shape with a round end, 9 mm in length, and a diameter of 2 mm.

The custom-mounting jig had a circular opening at the center with a diameter of 10 mm and a circular margin at the base to support the disk during testing. The jig was fixed on the lower part of the Instron testing machine, and the discs were individually placed in the mounting jig manually by finger pressure which ensured the same relation between the supports and the applied load for all specimens. Then, the 2 mm diameter loading pin was mounted to the cross-head of the Instron testing machine and applied the load at the center of each specimen. The test was conducted at a crosshead speed of 0.5 mm/min until fracture (Fig. 3).

The maximum load (N) was recorded for each specimen, and the BFS (MPa) was calculated for each specimen according to the following equations according to ISO 6872:2008:

$$\sigma = -0.2387P(X - Y)/b^2$$

where  $\sigma$  is the BFS in MPa and P is the total load causing fracture in Newton.

X and Y are determined as follows:

$$X = (1 + \nu) \ln(r_2/r_3)^2 + [(1 - \nu)/2] (r_2/r_3)^2$$

$$Y = (1 + \nu) [1 + \ln(r_1/r_3)^2] + (1 - \nu) (r_1/r_3)^2$$

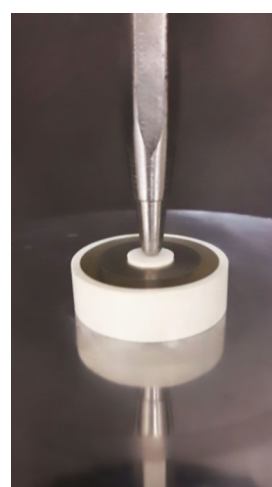
where  $\nu$  is the Poisson's ratio (if the value for the ceramic concerned is not known, the Poisson's ratio of 0.25 is used);  $r_1$  is the radius of the support circle/jig (5 mm);  $r_2$  is the radius of the loaded area (1 mm);  $r_3$  is the radius of the specimen (5 mm); b is the specimen thickness at the origin of fracture (1.2 mm).

**Table 2:** The milling machine, the sintering oven, and sintering time used for each system

Brand	CAD/CAM milling machine	Sintering oven, time, and temperature	Manufacturer
Ceramill ZI-LT	Ceramill Motion 2	Ceramill Therm 5 hours, 1,450°C	Amann Girrbach, Koblach, Austria
Lava Plus HT	Lava CNC 500	Lava Furnace 200 5 hours, 1,500°C	3M ESPE, Seefeld, Germany
Ceramill Zolid White HT	Ceramill Motion 2	Ceramill Therm 5 hours, 1,450°C	Amann Girrbach, Koblach, Austria
Copran Zr-i Monolith HT	CAM 5-S2	Tabco Furnace 5 hours, 1,450°C	vhf manufacture AG, Germany Mihm-Vogt, Germany



**Fig. 2:** Instron universal testing machine



**Fig. 3:** The loading pin and the disk on the mounting jig

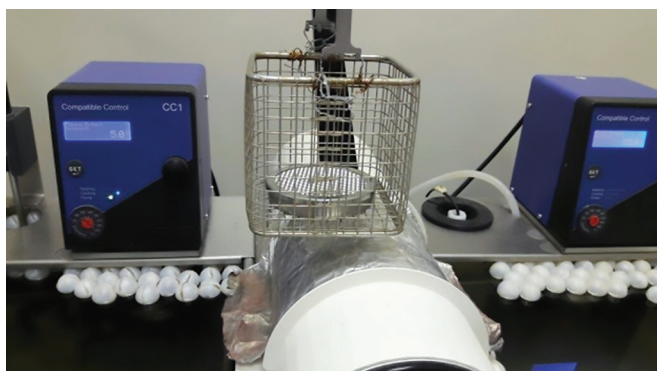


Fig. 4: Thermocycling machine and the samples on the tray

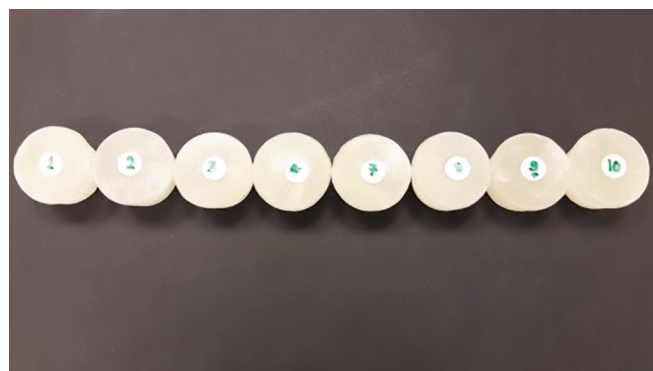


Fig. 5: The discs placed inside the self-curing resin

### BFS Test after AAA and Fatigue

**AAA test.** Forty discs, 10 from the control (Ceramill ZI-LT) and 10 from each high-translucent zirconia system (Lava Plus HT, Ceramill Zolid White HT, and Copran Zr-i Monolith HT), were placed on the thermocycling machine and subjected to 3,500 cycles (Thermocycler THE 1100 SD Mechatronik GmbH, Germany) between two water baths, 5°C and 55°C. Each cycle lasted 60 seconds in 5°C bath, 10 seconds to transfer samples to another bath, 20 seconds in 55°C bath, and 10 seconds to transfer samples back to 5°C bath (Fig. 4).

**Fatigue test.** After AAA, a holding jig was prepared using self-curing resin (Dentsply, Canada) and plastic mold with a circular opening (1.2 mm in thickness and 10 mm in diameter) and the 40 discs were placed manually inside that opening (Fig. 5).

The chewing simulator (CS-4.8, SD Mechatronik, Feldkirchen-Westerham, Germany) used contains eight test chambers, and each chamber has a loading bar with a diameter of 2 mm. Each mounted disk was placed at the center of the chamber with 37°C distilled water (Fig. 6); 250,000 cycles at a frequency of 1.6 Hz were selected with a load of 200 N at the center of the disc. During cyclic loading, the discs were inspected visually to detect any fracture. At the end of all cycles, all 40 discs had survived and were removed from the resin for the BFS test.

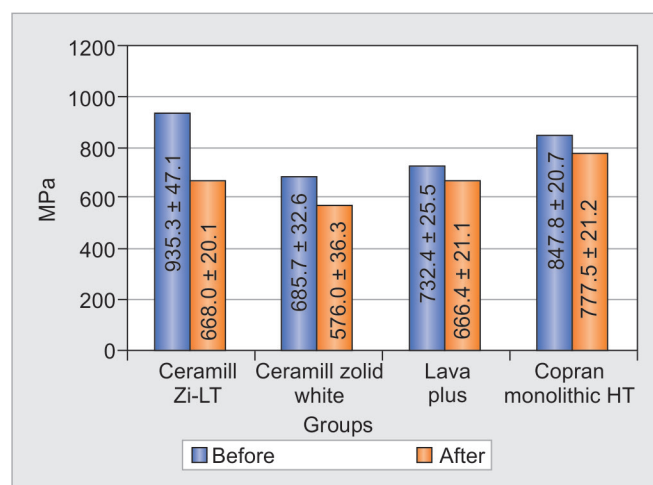
Finally, the 40 discs which were subjected to AAA and fatigue were subjected to single load-to-fracture using the universal testing machine (Instron 5965, Instron Corporation, USA) as described previously.

### Statistical Analysis

Means and standard deviations of the BFS were calculated. One-way ANOVA, Scheffe *post hoc*, and Mann-Whitney U test were performed for detecting the differences in BFS values between groups before and after AAA and fatigue. The data analyses were evaluated at a significance level of  $p \leq 0.05$ .



Fig. 6: Positions of the samples on the chewing simulator chambers



Graph 1: Mean (± standard deviation) of BFS before and after AAA and fatigue

### RESULTS

The mean and standard deviation of the BFS (MPa) values of Ceramill ZI, Ceramill Zolid White, Lava Plus, and Copran Zr-i Monolithic HT before and after AAA and fatigue are shown in Graph 1.

Before AAA and fatigue, the one-way ANOVA was used and revealed a statistically significant difference between groups ( $p < 0.05$ ). Then, Scheffe *post hoc* test was done (Table 3) and revealed a statistically significant difference in the mean BFS for all types of ceramic systems

**Table 3:** The Scheffe *post hoc* test for BFS (MPa) before AAA and fatigue

Groups		Mean difference	95% confidence interval		p-value
			Lower bound	Upper bound	
Ceramill ZI	Ceramill Zolid White	249.5*	206.3	292.8	0
	Lava Plus	202.9*	159.6	246.2	0
	Copran Zr-i Monolithic HT	87.5*	44.2	130.8	0
Lava Plus	Ceramill Zolid White	46.6*	3.4	90.0	0.030
Copran Zr-i Monolithic HT	Ceramill Zolid White	162.1*	118.8	205.3	0
	Lava Plus	115.4*	72.1	158.7	0

\*Significant difference with  $p \leq 0.05$ **Table 4:** The Scheffe *post hoc* test for BFS (MPa) after AAA and fatigue

Groups		Mean difference	95% confidence interval		p-value
			Lower bound	Upper bound	
Ceramill ZI	Ceramill Zolid White	92.0*	58.5	125.6	0
	Lava Plus	1.6	32.0	35.1	0.999
Lava Plus	Ceramill Zolid White	90.4*	56.9	124.0	0
Copran Zr-i Monolithic HT	Ceramill ZI	109.5*	76.0	143.1	0
	Ceramill Zolid White	201.5*	168.0	235.1	0
	Lava Plus	111.1*	77.6	144.7	0

\*Significant difference with  $p \leq 0.05$ 

tested in this study ( $p < 0.05$ ). The Ceramill ZI system had significantly higher BFS ( $935.3 \pm 47.1$  MPa) than Copran Zr-i Monolithic HT ( $847.8 \pm 20.7$  MPa), Lava Plus ( $732.4 \pm 25.5$  MPa), and Ceramill Zolid White ( $685.7 \pm 32.6$  MPa). Furthermore, the Lava Plus system had significantly higher BFS ( $732.4 \pm 25.5$  MPa) than Ceramill Zolid White ( $685.7 \pm 32.6$  MPa). The Copran Zr-i Monolithic HT system had significantly higher BFS ( $847.8 \pm 20.7$  MPa) than Lava Plus ( $732.4 \pm 25.5$  MPa) and Ceramill Zolid White ( $685.7 \pm 32.6$  MPa).

After AAA and fatigue, the one-way ANOVA was used and showed statistically significant difference between the groups ( $p < 0.05$ ). Then, Scheffe *post hoc* test (Table 4) was done and revealed that Copran Zr-i Monolithic HT had significantly higher BFS ( $777.5 \pm 21.2$  MPa) than Ceramill ZI ( $668.0 \pm 20.1$  MPa), Lava Plus ( $666.4 \pm 21.1$  MPa), and Ceramill Zolid White ( $576.0 \pm 36.3$  MPa) ( $p < 0.05$ ). The Ceramill ZI had significantly higher BFS ( $668.0 \pm 20.1$  MPa) than Ceramill Zolid White ( $576.0 \pm 36.3$  MPa) ( $p < 0.05$ ). The Lava Plus had significantly higher BFS ( $666.4 \pm 21.1$  MPa) than Ceramill Zolid White ( $576.0 \pm 36.3$  MPa) group ( $p < 0.05$ ). However, there was no significant difference in the BFS between Ceramill ZI ( $668.0 \pm 20.1$  MPa) and Lava Plus ( $666.4 \pm 21.1$  MPa) groups ( $p > 0.05$ ).

The Mann-Whitney U test was performed and showed a statistically significant difference in the BFS in all groups before and after AAA and fatigue ( $p < 0.05$ ).

## DISCUSSION

The main purpose of this *in vitro* study was to evaluate the BFS of three types of monolithic high-translucency

zirconia restorations compared with conventional low-translucency zirconia restorations and to study the effect of AAA and fatigue on the BFS.

The results of this experiment showed that the mean BFS before AAA and fatigue was the highest for Ceramill ZI ( $935.3 \pm 47.1$  MPa), followed by Copran Zr-i Monolithic HT ( $847.8 \pm 20.7$  MPa), Lava Plus ( $732.4 \pm 25.5$  MPa), and the least for Ceramill Zolid White ( $685.7 \pm 32.6$  MPa). Furthermore, the BFS of the conventional low-translucency zirconia restorations was significantly higher than that of the three types of monolithic high-translucency zirconia restorations before AAA and fatigue ( $p < 0.05$ ).

The mean BFS after AAA and fatigue was the highest for Copran Zr-i Monolithic HT ( $777.5 \pm 21.2$  MPa), followed by Ceramill ZI ( $668.0 \pm 20.1$  MPa), Lava Plus ( $666.4 \pm 21.1$  MPa), and the least for Ceramill Zolid White ( $576.0 \pm 36.3$  MPa). The Copran Zr-i Monolithic HT had significantly higher BFS ( $777.5 \pm 21.2$  MPa) than Ceramill ZI ( $668.0 \pm 20.1$  MPa), Lava Plus ( $666.4 \pm 21.1$  MPa), and Ceramill Zolid White ( $576.0 \pm 36.3$  MPa) ( $p < 0.05$ ). The Ceramill ZI had significantly higher BFS ( $668.0 \pm 20.1$  MPa) than Ceramill Zolid White ( $576.0 \pm 36.3$  MPa) ( $p < 0.05$ ). The Lava Plus had significantly higher BFS ( $666.4 \pm 21.1$  MPa) than Ceramill Zolid White ( $576.0 \pm 36.3$  MPa) group ( $p < 0.05$ ). However, there was no significant difference in the BFS between Ceramill ZI ( $668.0 \pm 20.1$  MPa) and Lava Plus ( $666.4 \pm 21.1$  MPa) groups ( $p > 0.05$ ).

In a study conducted by de Kok et al,<sup>12</sup> the mean flexural strength for the Lava Plus monolithic high-translucency zirconia crowns without AAA and fatigue was 1,235 MPa under 3-point bending test, which is higher

than the present study due to differences in specimen shape and the type of test. However, the BFS test is more accurate and similar to intraoral loading conditions compared with the other flexural strength tests.<sup>17</sup> In addition, the stress in the oral environment is considered biaxial.<sup>18</sup>

Regarding the flexural strength of Lava Plus, a comparable result (880 MPa) was found in a recent study conducted by Church et al,<sup>13</sup> with the variation that they had used beam-shaped specimens and a 3-point bending test in their study. Another study conducted by Bankoğlu Güngör et al<sup>19</sup> showed a higher BFS (1,100 MPa) for Lava Plus than the present study. Furthermore, that study showed no differences in the BFS of the tested material before and after thermocycling and mechanical cycling. Surprisingly, the BFS of Lava Plus was 1,257 MPa, which is higher than the control group. In addition, the study evaluated the effect of thermocycling and mechanical cycling individually, and they did not perform the test after subjecting the samples to both parameters to simulate the clinical condition.

The results of this study have shown that the AAA and fatigue have significantly affected the BFS of the three brands of high-translucent Y-TZP ceramics (Lava Plus HT, Ceramill Zolid White HT, and Copran Zr-i Monolith HT) and the low-translucent Y-TZP ceramic (Ceramill ZI-LT) tested. This finding is in agreement with previous studies,<sup>20,21</sup> in which they demonstrated that the flexural strength of zirconia bar specimens with a thickness of 0.2 mm was significantly decreased after aging.

Regarding the aging and fatigue effect on the flexural strength, our findings are in agreement with Cotes et al,<sup>22</sup> where they demonstrated that the flexural strength was decreased from  $955 \pm 108$  MPa to  $781 \pm 72$ ;  $771 \pm 108.5$  MPa as a result of monoclinic phase generation, which occurred when the zirconia discs were subjected to mechanical cycling and thermomechanical cycling (1,200,000 cycles/3.8 Hz/200 N) at temperatures ranging from 5°C to 55°C.

In contrast, the results of this study are not in agreement with a previous one, which has used air abrasion instead of thermocycling and few cycles as 100,000 cycles instead of 250,000 cycles.<sup>23</sup> In another study conducted by Pittayachawan et al,<sup>24</sup> only 10,000 and 20,000 cycles were used and no thermocycling was performed. The authors concluded that there was no significant effect on the flexural strength of zirconia after fatigue at 20,000 and 10,000 cycles.

In this study, disk specimens were used instead of clinical crowns which better simulate the clinical situation in the patient mouth. In addition, scanning electron microscopy analysis was not used to evaluate the material microstructure, transformation, and cracks before and after aging and fatigue.

## CONCLUSION

Within the limitations of the present study, the following conclusions were drawn:

- The BFS of the high-translucency monolithic zirconia used in this study was significantly less compared with the conventional zirconia only without AAA and fatigue.
- The AAA and fatigue protocols used in this study resulted in a significant decrease in the BFS of the zirconia systems tested.
- High-translucency monolithic zirconia has a BFS within acceptable clinical values.

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