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Effect of Cyclic Loading on the Fracture Resistance of Bilayered 3Y-TZP and Monolithic Gradient 5Y-TZP/3Y-TZP Zirconia Based Fixed Partial Denture (In-Vitro Study)

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ABSTRACT

Background**:* Developments in dental materials have led to the creation of an innovative strength-gradient zirconia, which merges 3-mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) with 5-mol% yttria-stabilized tetragonal zirconia polycrystal (5Y-TZP). Despite these advancements, there is a lack of information on its fracture resistance behavior. ***Aim of the Study: This study aims to measure the fracture resistance of a four-unit fixed partial denture constructed from gradient monolithic zirconia 3Y-TZP/5Y-TZP compared to bilayered zirconia 3Y-TZP four-unit fixed partial denture. ***Material and Methods:*** A standardized stainless-steel master die was designed to replicate a mandibular first premolar and second molar, prepared to receive 4-unit monolithic and bilayered zirconia FPDs. Ten zirconia fixed partial dentures were milled and divided into two groups (n=5): one with 3Y-TZP that was layered by 0.4 mm ceramic veneer, the other group is monolithic gradient zirconia 5Y-TZP and 3Y-TZP. The FPDs underwent cyclic loading in a mastication simulator to simulate six months of clinical use, then fracture resistance was measured using a universal testing machine at a crosshead speed of 1 mm/min, and fractographic analysis was conducted using a scanning electron microscope. ***Results:*** The highest statistically significant fracture load was recorded in monolithic 5Y-TZP and 3Y-TZP gradient zirconia (844 ±156 N), while the lowest statistically significant fracture load was recorded in bilayered zirconia 3Y-TZP (601 ±89 N). ***Conclusion:*** The fracture strength of 4-unit gradient multilayered zirconia 5Y-TZP/3Y-TZP was significantly higher than bilayered zirconia 3Y-TZP when both were subjected to fracture resistance tests after mastication simulation.

Keywords: Gradient Zirconia, Bilayered Zirconia, Fracture resistance, Cyclic loading.

INTRODUCTION

For decades, metal-ceramic restorations have been regarded as a benchmark for the fabrication of fixed partial dentures due to their superior mechanical properties, remarkable long-term survival rates, and satisfactory aesthetics.[1, 2] Nevertheless, the increasing demand for enhanced aesthetics and biocompatibility of metal-free

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alternatives has sparked significant recent interest.[3, 4]

Zirconia dental restorations have become an alternative to metal-ceramic restorations due to their biocompatibility, low thermal conductivity, satisfactory optical properties, and excellent mechanical properties.[5-8] Zirconia's classification is based on yttria content, influencing its mechanical properties and translucency. 3Y-TZP zirconia offers high fracture toughness and flexural strength but suffers from high opacity, often necessitating a veneering ceramic, which has been prone to chipping and fractures.[9]

Statistically, the fracture rates of the veneer are observed to be between 2-9% for crowns for 2 to 3 years and between 3-36% for dental bridges throughout 1 to 5 years.[10] To improve translucency, manufacturers have increased yttria content, creating partially stabilized zirconia with higher cubic phase content, such as 5Y-TZP and 4Y-TZP.[11] These variations offer improved translucency but at the cost of reduced transformation toughening and, consequently, lower mechanical strength, restricting their use primarily to the anterior region.[12]

The development of multilayered and gradient zirconia technologies has further diversified the application of zirconia in dentistry.[13] This innovative material has been found useful in monolithic restorations for FPDs with limited pontics, offering a balance of strength and aesthetics.[14-17]

Despite the undeniable advantages of monolithic zirconia ceramics, scientific research to develop proper clinical and laboratory guidelines for their effective application is needed.[18, 19] The first multilayered zirconia system in dentistry featured three zirconia variants— Multi-Layered, Super Translucent, and Ultra Translucent Multi-Layered Zirconia by Katana Noritake Dental Japan.[20-22] Typically, an increase in yttria content enhances the cubic phase and thus improves translucency at the expense of reduced strength.[23, 24] The manufacturer claims these materials are suited for all monolithic restorative applications, with consistent yttrium and cubic content across layers, the distinction between layers lies in the pigmentation, creating shade variations rather than differences in translucency.[25, 26]

While conventional fabrication techniques of FPDs are still valid and reliable, FPDs constructed using CAD-CAM technology stand at the forefront of dental prosthetics for their precision and potentially superior marginal accuracy by minimizing human error through digital precision.[27, 28] Numerous studies have rigorously evaluated the long-term prognosis of long-span fixed dental prostheses, which showed that zirconia-based long-span FPDs possess superior strength and can withstand cyclic loading conditions.[29]

These findings consistently indicate that such FPDs provide the requisite durability for an extended period of clinical service.[30] New generations of Zirconia have undergone many studies under different sintering protocols.[20, 31, 32] It has not yet undergone a sufficient evaluation of its clinical performance against veneered Zirconia 3Y-TZP, as veneered zirconia is the clinically used form of zirconia-based full coverage restoration, with the scientific data on them being scarce.[33-35]

Therefore, the objective of this study is to evaluate the mechanical behavior and fracture resistance of these materials to ascertain their clinical strength under varying occlusal forces. The null hypothesis was that no difference would be found in the fracture resistance between monolithic gradient zirconia 5Y-TZP and 3Y-TZP and bilayered zirconia 3Y-TZP.

MATERIAL AND METHODS

The materials used in this study are listed in **Table (1).** Ten zirconia four-unit fixed dental prostheses were fabricated from two zirconia-based materials and split into two groups (n=5): 3Y-TZP bilayered zirconia and gradient 5Y-TZP/3Y-TZP monolithic zirconia according to the power analysis and based upon the results of Hamza TA and Sherif RM.[36] The mean and standard deviation for fracture resistance values were 1742.9 (102.7) and 1267.8 (86.1) Newtons, respectively, and the effect size (d) was 5.01. Using an alpha (α) level of (5%) and Beta (β) level of (20%) i.e. power = 80%; the minimum estimated sample size will be three samples per group. The sample size will be increased to five specimens per group. Sample size calculation was performed using G*Power version 3.1.9.2.

One stainless-steel master model was designed to simulate a mandibular first premolar and second molar to replace a missing second premolar and first molar, prepared to receive zirconia fully contoured four-unit FPD. The model was constructed with a 1.0-mm-deep chamfer finish line width,[37] and 4.5-mm occlusal-gingival height to simulate a four-unit FPD from a mandibular right first premolar to a second molar.[38]

The stainless-steel model was screwed in a custom-designed stainless-steel holder with a 23 mm distance between the centers of the

Table (1): Materials used in the study.

Serial	Brand Name	Description	Manufacturer	Composition	Lot Number
1	HT White Zirconia	High translucency biological nano zirconia	UPCERA	ZrO2 + HfO2 + Y2O3 Y2O3 Al2O3 Others oxides >99% 4.5%-6% <0.5%	T-06856
2	IPS e.max ZirCAD Prime	Strength gradient monolithic zirconia	Ivoclar Vivadent AG	ZrO2 88.0%-95.5%, Y2O3>4.5%- ≤7.0%, AL2O3≤1.0%, HfO2≤5.0%, other oxides≤1.5%	Y51110
3	Ketac Cem	Glass ionomer cement	3M ESPE Dental Products	Glass powder, pigments, polycarboxylic acid, tartaric acid, water, conservation agents	37235
4	Acrylic resin	Acrylic material – cold cure	Acrostone	Powder: PMMA prepolymer or copolymer, Di- benzoyl peroxide (DBP)(initiator) (1-2%), Pigment (1%). Liquid: methacrylate, hydroquinone (inhibitor)	F694587

master dies to correspond to the average inter-abutment clearance between a first premolar and a second molar.[34] The model was digitized with a 3D light scanner (Freedom HD; DOFlab), and a virtual model was created by a CAD software program (exocad v2.3 DentalCAD; exocad GmbH). Two designs were created, a fully contoured design for the monolithic zirconia 5Y-TZP and 3Y-TZP, and a framework design of 0.6 mm thickness that was veneered using conventional layering technique for the bilayered zirconia 3Y-TZP **(Figure 1)**.

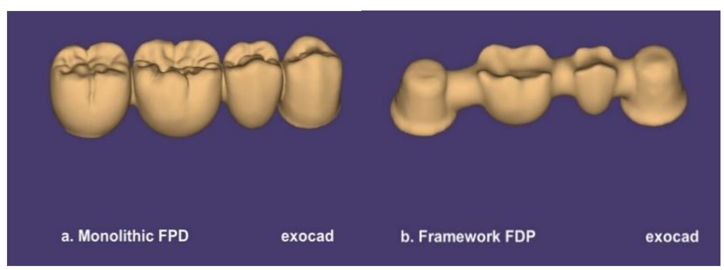

Figure (1): CADCAM design for the monolithic gradient zirconia Group (A) and framework design for the bilayered zirconia Group (B).

The wall thickness was set at 1 mm for both groups with 0.6 mm of zirconia framework (3Y-TZP) and 0.4 mm of ceramic veneer layer in the bilayered zirconia 3Y-TZP group, and 1 mm of wall thickness in the monolithic zirconia group 5Y-TZP and 3Y-TZP. The virtual cement spacer was set at 30 µm and applied 1.0 mm above the margin **(Figure 2)**. The data obtained were sent to a

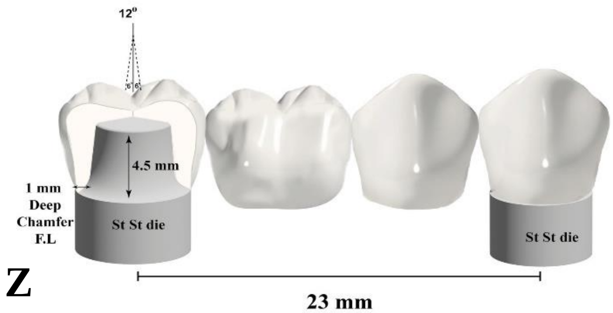

Figure (2): Monolithic gradient zirconia bridge design (Z) and bilayered zirconia 3Y-TZP bridge design (BL).

milling machine (DWX-51D; Roland DG Corp), where zirconia FPDs were milled from a pre-sintered 3Y-TZP blank and 5Y-TZP/3Y-TZP gradient zirconia blank.

After dry milling, the frameworks of both groups were sintered to the full density at 1500 C for 10 hours in a sintering furnace (5 motions furnace; bredent GmbH). The zirconia framework in the control group was hand-veneered with a 0.28g to 0.12 ml powder-to-liquid ratio of ceramic (BAOT ZCG, Dental zirconia porcelain PFZ). Sections of a silicone imprint acquired from the monolithic zirconia group were utilized to standardize and replicate the contour of the monolithic zirconia group 5Y-TZP and 3Y-TZP.[39]

Specimens were stored in distilled water for 24 hours at 37 C before the cyclic loading test. Cyclic loading was performed by a chewing simulator (CS-4.4, SD Mechatronik, GmbH) **(Figure 3).** A load of 50 N, lateral movement of 0.7 mm, and vertical movement

Figure (3): Specimen under cyclic loading.

of 2.5 mm were applied at a frequency of 1.5 Hz for 150.000 cycles to simulate six months of clinical use.[5] All specimens were examined after cyclic loading for cracks, and all were intact. Then the fracture resistance test was carried out using a universal testing machine and applied vertically with a Ø6-mm stainless-steel rod attached to the upper movable compartment of the testing machine at the connector area between the two pontics at a crosshead speed of 1 mm/ min until fracture occurred.[40, 41] A 0.5-mm-thick tin foil was placed between the loading device and the pontic to distribute homogenous force **(Figure 4).**

Figure (4): Gradient zirconia 5Y-TZP and 3Y-TZP, and bilayered zirconia 3Y-TZP fracture resistance testing.

The fracture load in newtons (N) was recorded when the fracture load decreased by 1% of the peak load. Subsequent fractographic analysis by Scanning Electron Microscope (JCM-7000 NeoScope Benchtop SEM) at various magnifications identified the points of fracture on the specimens at magnifications X30, X100, and X400 to identify the fracture behavior (**Figure 5**).

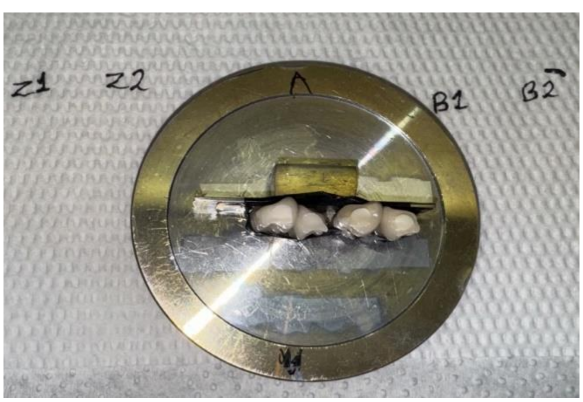

Figure (5): Samples preparation for Scanning Electron Microscope.

Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov and Shapiro-Wilk tests). Data were presented as mean, standard deviation (SD), median, minimum and maximum values. For parametric data, an independent t-test was used to compare the two groups. Mann-Whitney U test was used for non-parametric data. The significance level was set at $P \leq 0.05$. Statistical analysis was performed using a statistical software program (SPSS 20, Graph Pad Prism, IBM Corp).

RESULTS

As listed in **Table (2)**, it was revealed that the significant level (P-value) was shown to be insignificant as P-value > 0.05, which indicated that data originated from normal distribution.

Accordingly, an independent t-test was used to compare both groups. All descriptive results of both groups are presented in **Table**

(3).

Table (2): Normality exploration of both groups.

	P value	Indication
Test Group	> 0.05	Normal data
Control Group	> 0.05	Normal data

an independent t-test, which revealed that monolithic zirconia 5Y-TZP and 3Y-TZP (845 ± 157) was significantly higher than bilayered zirconia 3Y-TZP (601 ± 89) as P=0.02, with 245 mean difference. Fracture of all specimens occurred distally in the connector region; the veneer was chipped before complete fracture in the control group, and then the zirconia framework fractured.

Table (3): Descriptive statistics of fracture resistance in both groups.

		Monolithic zirconia 5Y-TZP and 3Y-TZP	Bilayered zirconia 3Y-TZP
Mean		845	601
95% Confidence Interval for Mean	Lower arm	650	491
	Upper arm	1040	712
Median		845	616
Std. Deviation		157	89
Minimum		681	501
Maximum		1041	690
Range		360	189
Interquartile Range		308	177

In the monolithic zirconia 5Y-TZP and 3Y-TZP: The mean ± standard deviation was (845 ± 157) with minimum (681), maximum (1041), range (359), median (845), and interquartile range (308) In the bilayered zirconia 3Y-TZP: The mean ± standard deviation was (601 ± 89) with minimum (501), maximum (690), range (189), median (616), and interquartile range (177). Analytical results showed the mean difference between both groups and the standard deviation of fracture resistance, which is presented in **Table (4)**. Comparison between both groups was performed by using

Fractographic analysis was done on specimens from each group using SEM. **(Figure 6)** represents the fractured gradient zirconia group under X30, X100, and X400 magnification of SEM. The crack origin is shown by the red circle in the gradient zirconia group in Figure 6A. Radiating lines, characteristic of wake hackles, are also prominent. The overall direction of crack propagation is shown by the green arrow in Figures 6A and 6B.

Table (4): Comparison between fracture resistance of test/control groups by independent t-test.

Group	Mean	Standard Deviation	Difference (Independent t-test)				
			Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference		P value
					Lower	Upper	
Monolithic Zirconia	845	157	244	81	58	430	0.02*
Bilayered Zirconia	601	89					

**Significant difference at P<0.05.*

Figure (6): Gradient zirconia 5Y-TZP and 3Y-TZP under X30,100 and 400 magnifications.

In the SEM image (**Figure 7**), the fracture morphology of bilayered zirconia is depicted across magnifications of x30, x100, and x400. All fractographic features were presented mostly in the porcelain veneer layer.

In Figure 7A, numerous hackle lines radiate from the main crack line within the ceramic layer are shown by the red circle. Figure 7B shows the main crack line, marked by green arrows. The direction of crack propagation is demarcated around the main

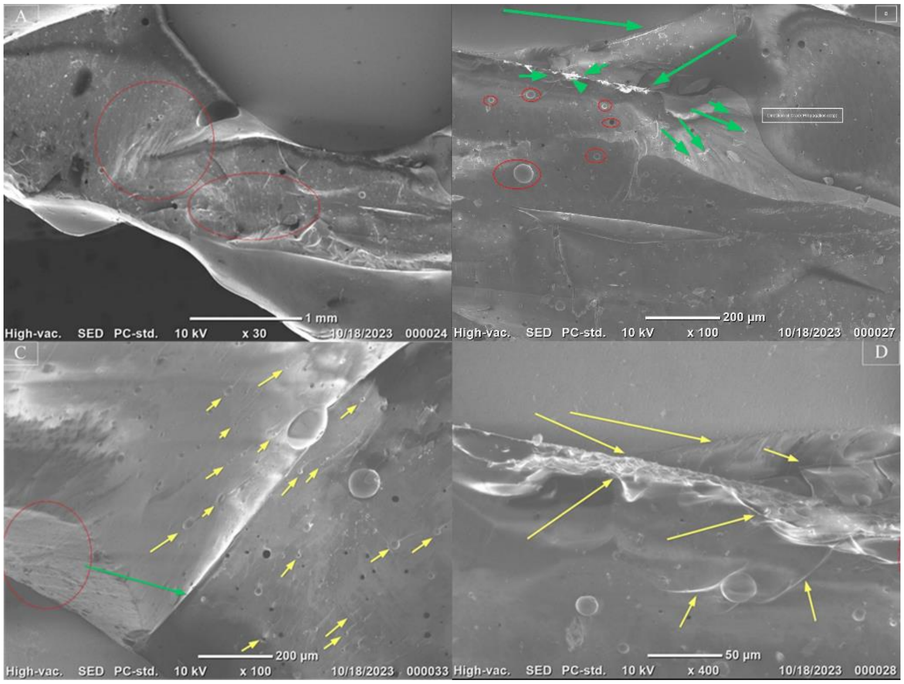

Figure (7): 3Y-TZP zirconia under X30,100 and 400 magnifications.

crack. Figure 7C exhibits an array of wake hackles, indicated by yellow arrows, with the green arrow pinpointing the main crack line, which denotes the direction of crack propagation (dcp). A red circle encapsulates a 'mist' area characterized by delicate hackle lines emanating from the primary fracture. Lastly, Figure 7D reveals the twist hackles around the main fracture line, identified by yellow arrows.

DISCUSSION

This in vitro study examined the effect of cyclic loading on the fracture resistance of 3Y-TZP and monolithic gradient 5Y-TZP/ 3Y-TZP zirconia four-unit FPD. The null hypothesis that no difference in fracture resistance would be found between 3Y-TZP bilayered zirconia and gradient 5Y-TZP/3Y-TZP four-unit full-contour FPD were rejected.

In the present study, the mean fracture load values were higher for the monolithic zirconia 5Y-TZP/3Y-TZP full-contoured four-unit FPD than bilayered zirconia 3Y-TZP. Both results are higher than normal masticatory forces that vary from 200N to 540N.[31] The higher fracture resistance observed in the full-contoured 5Y-TZP/3Y-TZP fixed partial dentures (FPD) might stem from the greater zirconia thickness—1 mm in the monolithic zirconia restorations, compared to a 0.6 mm zirconia thickness in the bilayered zirconia's group, which is layered by an 0.4 mm ceramic veneer.

Furthermore, the initial fracture of ceramic veneer might attribute to the cause.

This suggests that the increased thickness of zirconia is likely a key factor contributing to the higher fracture resistance. The obtained results agree with previous studies testing the fracture strength of aged monolithic and bilayer zirconia-based crowns.[42, 43] The findings of this study differ from previous research done by Attia MA and Shokry TE that examined how dynamic loading impacts the fracture resistance of gradient zirconia 5Y-TZP/3Y-TZP and 3Y-TZP zirconia frameworks.[38] The discrepancy could arise from the fact that the 3Y-TZP zirconia framework and monolithic zirconia 5Y-TZP/3Y-TZP had the same thickness. Thus, 3Y-TZP would show higher fracture resistance than 5Y-TZP/3Y-TZP gradient zirconia. This could be why the zirconia 3Y-TZP group showed greater strength than the monolithic zirconia combining 5Y-TZP and 3Y-TZP.

Further study was conducted using a fractographic analysis of specimens from both groups, gradient zirconia 5Y-TZP/3Y-TZP and 3Y-TZP bilayered zirconia using SEM. **Figure (6)** represents Z1 under X30, X100, and X400 magnification of SEM. The fracture in the monolithic zirconia FPD seems to have originated from the area pointed by the arrow in Figure 6A, where the smooth surface transitions into a rough fracture pattern, indicative of the compression curl. Radiating lines, characteristic of wake hackles, are prominent around this area, extending from specific singularities. The overall direction of crack propagation is shown in Figures 6A, and 6B, moving slightly towards the upper right, as deduced from the wake hackle patterns and the positioning of the compression curl.

The fracture morphology of bilayered zirconia is portrayed across magnifications of x30, x100, and x400 in **(Figure 7).** Figure 7A contrasts the smoothness of the zirconia substrate with the disrupted ceramic veneer. It is evident that the manual layering technique of ceramic build-up has inherently led to the formation of air voids throughout its matrix. These voids represent potential focal points for stress accumulation, precipitating expedited crack propagation. Encircled in red, numerous hackle lines radiate from the main crack line within the ceramic veneer layer, yet the zirconia core remains unaffected by such fracture propagation.

Figure 7B delineates the main crack line, marked by green arrows, and a finer hackle line branching from the primary crack. The direction of crack propagation is demarcated around the main crack, with a conspicuous presence of voids, again highlighted in red. Subsequently, Figure 7C exhibits an array of wake hackles, indicated by yellow arrows, with the green arrow pinpointing the main crack line, which denotes the direction of crack propagation (dcp). A red circle encapsulates a 'mist' area characterized by delicate hackle lines emanating from the primary fracture. Lastly, Figure 7D reveals the twist hackles near the main fracture line, identified by yellow arrows. The upper segment of the image reveals the zirconia core's section, where no trace of crack progression is visible, attributing to the inherent resilience conferred by its polycrystalline structure.

Evidently from the conducted fractography of the two groups, the test group consisting of 5Y-TZP/3Y-TZP monolithic gradient zirconia and the control group comprising bilayered zirconia 3Y-TZP, it was observed that the initial fracture points consistently occurred at the connector, same as most of the literature.[38] Based on these findings, it is recommended that the connector region in fixed partial denture designs be structurally reinforced. This reinforcement can be achieved by increasing the thickness of this area beyond the standard 9 mm cross-section, as commonly referenced in the dental literature.[30] Additionally, for the gradient technology employed in Zircad Prime, which utilizes a combination of 5Y-TZP and 3Y-TZP, it is recommended that the composition of the connector area be enhanced. This could be effectively implemented by incorporating a more detailed mapping within the zirconia disc, clearly indicating the anticipated position of the connector.

Such a strategic approach would allow for a higher concentration of 3Y-TZP and a reduced presence of 5Y-TZP in the connector region, thereby potentially increasing the dental restoration's overall structural integrity and fracture resistance. This tailored material distribution aligns to optimize the mechanical properties of zirconia-based dental prosthetics, ensuring their durability and reliability in clinical applications.

A potential limitation of this study is that it did not test the differences in marginal gaps between the two groups following cyclic loading. Additionally, the use of cyclic loading as a simulation of clinical loading has its constraints, and the application of higher horizontal loading could have potentially altered the outcomes of the fracture resistance tests.

CONCLUSIONS: Based on the findings of this in vitro study, the conclusion was that the fracture resistance of monolithic 5Y-TZP/3Y-TZP zirconia four-unit fixed partial dentures was significantly higher than that of bilayered zirconia 3Y-TZP fixed partial dentures. Furthermore, both four-unit bilayered zirconia 3Y-TZP and monolithic zirconia 5Y-TZP/3Y-TZP FPDs demonstrated fracture load values that are higher than normal masticatory forces.

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