

The Effect of Aging on Biaxial Flexural Strength of Lithium Disilicate and Strength Gradient Zirconia

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Abstract

Purpose: The aim of this study was to investigate the effect of thermocycling and cyclic loading on biaxial flexural strength (BFS) of Lithium disilicate and Monolithic strength-gradient zirconia ceramics.

Materials and methods: Forty disc-shaped specimens (2mm thickness, 10mm diameter) were prepared: Twenty discs each were manufactured from Lithium disilicate (e.max CAD) and monolithic strength-gradient zirconia (ZirCAD). Half of the samples in each group (n=10) were subjected to thermocycling (TC) (5 °C to 55 °C for 6000 cycles with 30 seconds dwell time and 7-second transfer time), followed by cyclic loading (CL) (1.2×10^6 load cycles, 50N load, frequency 1.5 cycles per second) and subsequently all samples were subjected to BFS test using a universal testing machine, according to ISO standard 6872 (2024).

Four groups were evaluated (n=10) were: (e.max CAD, e.max CAD+ TCCL, ZirCAD, ZirCAD+ TCCL). Values of biaxial flexural test were calculated and analyzed using the SPSS version 26.0 (SPSS, Chicago, IL, USA). Two-way ANOVA test followed by Bonferroni test were used to detect statistical differences between the two ceramic materials and to detect the effect of TC and CL for each ceramic material, separately.

Results: Mean BFS for ZirCAD groups was 1184.60 MPa $\pm$ 229.6 SD and for e.max CAD groups was 472.76 MPa $\pm$ 39.8 SD. BFS for ZirCAD groups was significantly higher ($p < 0.001$) than that of e.max CAD groups. However, TC and CL had no statistically significant effect on the BFS values of both ceramic groups (p -value= 0.573). The mean BFS values for non-TCCL ZirCAD was 1165.88 MPa, whereas for ZirCAD+ TCCL was 1203.32 MPa. On the other hand, the mean BFS values for non-TCCL e.max CAD was 484.37 MPa and for e.max CAD+ TCCL was 461.145 MPa.

Conclusion: The two tested materials significantly differ in their recorded BFS, however, thermocycling and cyclic loading didn't show any significant influence on the BFS values, calling for revision of their inclusion in future studies.

Introduction

In recent years, there has been a noticeable shift toward the use of ceramic materials in restorative dentistry, driven by their superior esthetics, biocompatibility, and mechanical performance ¹.

Among glass-based ceramics, lithium disilicate stands out as one of the most widely used and clinically studied materials due to its unique combination of esthetics and strength ^{2,3}. Building on the foundation of earlier pressed ceramics like IPS Empress 2, the IPS e.max system was developed. Followed by the introduction of IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein) in 2005 .

Conversely, Zirconia dental restorations have garnered significant popularity as a restorative option recently ^{5,6}. The unique properties of pure zirconia are inherently tied to its crystalline phases, which

manifest at different temperatures: monoclinic, tetragonal, and cubic ⁷.

Consequently, multilayered zirconia was introduced to overcome the inherent trade-off between mechanical integrity and optical appeal. These zirconia blocks feature a functional gradation, both in material composition and esthetic appearance, across the layers of the restoration ⁸.

To access the mechanical merits of ceramic materials through flexural strength, there are different standard methods available including uniaxial and biaxial flexural tests ⁹. Biaxial flexural strength (BFS) tests offer various advantages over uniaxial tests ^{10,11, 12}. Furthermore, to assess performance consistency and structural uniformity of such ceramics, the Weibull modulus (m) is often used, since it provides a statistical measure directly linked to the distribution of these flaws ^{13,14}.

Various systems have been developed to mimic the oral environment, with notable contributions from DeLong and Douglas, Krejci et al., and other researchers ^{15,16}. Thermocycling exposes materials to repeated temperature changes, typically between 5°C and 55°C, in an attempt to replicate the intraorally induced thermal stresses ^{15,17}.

Simultaneously, cyclic loading mimics the functional stresses of mastication by applying repetitive, low-intensity forces over time ^{18,19}. When used in combination, thermocycling and cyclic loading offer a more comprehensive evaluation of a material's long-term durability ¹.

Therefore, the aim of the present study was to investigate the effect of thermocycling and cyclic loading on biaxial flexural strength of lithium disilicate and monolithic strength gradient zirconia.

Materials and methods

Forty disc-shaped specimens fabricated from monolithic strength-gradient zirconia material (IPS e.max® ZirCAD® Prime, Ivoclar Vivadent, Schaan, Liechtenstein), and Lithium disilicate material (IPS e.max® CAD, Ivoclar Vivadent, Schaan, Liechtenstein) shade BL2, were evaluated in this study. Based on a power analysis which showed that for $\alpha = 0.05$, power = 0.8, 40 disc-shaped specimens from both groups were fabricated.

Twenty discs ($n = 20$) were prepared of ZirCAD group and 20 discs of e.max CAD group. Each disc was 10 mm in diameter and 2 mm in thickness. Table 1 shows the materials used in this study, their composition, and manufacturers.

Table .1 Materials used in this study, as provided by the manufacturer.

Brand	Material	Chemical composition	Manufacturer
IPS e.max® ZirCAD® Prime	incisal: 5Y-TZP dentin: 3Y-TZP	Zirconium oxide (ZrO ₂) 88.0–95.5%, Yttrium oxide (Y ₂ O ₃) 4.5% – 7.0%, Hafnium oxide (HfO ₂) ≤ 5.0%, Aluminium oxide (Al ₂ O ₃), ≤ 1.0%, Other oxides ≤ 1.5%	Ivoclar Vivadent AG (Schaan, Liechtenstein)
IPS e.max® CAD	lithium metasilicate crystals (Li ₂ SiO ₃) embedded in a glassy phase	SiO ₂ 57.0–80.0%, Li ₂ O 11–19%, K ₂ O ≤ 13.0, P ₂ O ₅ ≤ 11%, ZrO ₂ ≤ 8%, ZnO ≤ 8%, Al ₂ O ₃ ≤ 5%, MgO ≤ 5%, Coloring oxides ≤ 8%	Ivoclar Vivadent AG (Schaan, Liechtenstein)

Utilizing CAD software (3Shape Dental Software), 20 disc-shaped ZirCAD specimens were fabricated utilizing the digital data. A five-axes milling machine was used to fabricate the specimens from partially sintered monolithic zirconia (CORiTEC 350i imes-icore, Hessen, Germany). Cuts were directed towards the middle part of zirconia blocks^{20,21}, then samples were removed from the blank to be finished (Zolid Sinter-State Finishing Kit; Amann Girrbach AG). Samples were then fully sintered in a special furnace (Programat S1 1600, Ivoclar Vivadent AG, Schaan, Liechtenstein) for approximately 4.5 hours at 1,500°C in accordance with the manufacturer's recommendations and no further finishing was provided.

Whereas for lithium disilicate specimens, 20 CAD discs were milled using the same method as zirconia discs (3Shape Dental Software). Samples were milled from IPS e.max CAD blocks in the blue stage using the same milling machine. After 30 minutes of wet milling, the specimens were in the blue stage and, following the manufacturer's recommendations, were crystallized for 20 minutes at 840 °C in a ceramic furnace (Programat P300; Ivoclar Vivadent).

Half of the samples of each material were subjected to thermocycling (TC) in distilled water bath at temperature ranging from 5–55 °C for 6000 cycles over the period of 6 days^{22,23}. TC was performed using a self-assembled machine constructed by the team of prosthodontics in Jordan University of science and technology.

In preparation for the cyclic loading (CL) process, a detailed design was done using AutoCAD software. This design aimed to precisely replicate the layout of the machine's base within the working area to ensure optimal seating of the samples. Subsequently, to print out this design, “Formlabs Form 4B” 3D printer (Somerville, Massachusetts, United states of America) was employed to produce each disc's individual resin base using IBT Flex Resin. Figure 1. shows AutoCAD design, 3D printed resin holder.

The same samples which went through TC, were subsequently subjected to 1.2×10^6 cycles of 50 Newton CL in a chewing simulator at a frequency of 1.5 Hz over ≈ 9 days. Under the experimental conditions, the circumstances were deliberately designed to simulate the effects and outcomes that would typically arise from 5 years of clinical use²⁴. After completion, none of the specimens failed under the TC-CL phase.

In this study, all specimens were eventually subjected to the BFS static loading until fracture. This testing configuration is recommended by the International Organization for Standardization (ISO) due to its ability to standardize specimen parameters such as thickness, diameter, surface finish, and geometry ²⁵. To ensure adequate statistical power, a sample size calculation was performed using an 80% power threshold, resulting in the inclusion of 20 specimens per material ⁶.

The load at fracture (Newton) of the ceramic discs was measured using a computer-controlled electromechanical universal testing machine (Jinan Testing Equipment, WDW-20, China), and the test setup being in compliance with ISO standard 6872 (2024) for dental ceramics ^{26,27}. For the piston-on-three-ball test, the sample holder was made of 3 steel balls each measuring 3.2 mm in diameter and spaced 120 degrees apart to form a triangle on a supporting circle of Ø 10 mm diameter (ISO Standard 6872, 2024), to make up the disc support area. The load was applied centrally through a flat end tungsten piston with a diameter of (Ø = 1.4 mm) (ISO Standard 6872, 2024) ¹¹ at a crosshead speed of 1 mm/min until catastrophic failure. Figure 2. shows the universal testing machine with a specimen.

The BFS values were subsequently calculated using the following formula according to ISO

6872 standards ^{11,28}:

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

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

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

Where σ is the maximum tensile stress in megapascals (MPa), P is the failure load in

Newtons(N), ν is the Poisson's ratio, r_1 is the radius of the support circle(mm), r_2 is the radius

of the loaded area (mm) and r_3 is the radius of the specimen (mm). Given that Poisson's ratio

for ZirCAD = 0.3, Poisson ratio for CAD = 0.26, $r_1 = 5\text{mm}$, $r_2 = 0.7\text{mm}$ and $r_3 = 5\text{mm}$.

Subsequently, strength variation among each group was evaluated by calculating the Weibull

modulus(m) using the following equation: ^{14,29}

$$P(\sigma) = 1 - \exp(-\sigma / \sigma_0)^m$$

Where $P(\sigma)$ is the fracture probability; σ is the fracture strength at a given $P(\sigma)$; σ_0 is the

characteristic strength; and m is the Weibull modulus, which is the slope of the $\ln(\ln 1/(1-P))$

versus $\ln \sigma$ plots ²⁹.

Analytical data calculations were carried out using the SPSS version 26.0 (SPSS, Chicago, IL, USA) statistical program. All data were subjected to Levene’s test of homogeneity of variance ($\alpha = 0.05$) following the assumption of equal variance. The test result was non-significant above 0.05, indicating normal distribution of the data. Parametric descriptive statistics were calculated including the mean, standard deviation, and the 95% confidence interval.

A statistical significance was determined given a p-value less than 0.05. Mean values for BFS were analyzed by two-way ANOVA test, followed by Bonferroni post hoc with flexural strength as the dependent variable, to examine the effects of Groups (ZirCAD and CAD) and the effect of TCCL on the dependent variable BFS. Furthermore, the BFS values for each ZirCAD group (ZirCAD, ZirCAD+TCCL) and the BFS values for each e.max CAD group (e.max CAD, e.max CAD+TCCL) were also analyzed using Weibull statistics. The analysis was performed using the maximum likelihood estimation (MLE) method in R (R Core Team, 2024) within the RStudio environment (Posit Software, PBC, Version 2024.12.1).

Results

Visual inspection of the fractured specimens in all groups revealed a consistent failure pattern, characterized by complete fracture through both ZirCAD and e.max CAD specimens, with all having at least one fracture line passing through the center at the piston contact area.

The assumption of normality was assessed using the Kolmogorov–Smirnov test. In all groups, the p-values were greater than 0.05, indicating no significant deviation from normality. Therefore, parametric tests were used for subsequent analyses.

The results of the two-way ANOVA test showed a statistically significant main effect of material type on BFS values. Mean BFS for ZirCAD groups (ZirCAD and ZirCAD+TCCL) was 1184.60 MPa $\pm$ 229.6 SD while, for e.max CAD groups (e.max CAD and e.max CAD+TCCL), it was 472.76 MPa $\pm$ 39.8 SD ($p < 0.001$). This indicates that ZirCAD group demonstrated significantly greater BFS compared to e.max CAD group, regardless of the TCCL conditions. As shown in Table 2 and Fig. 3.

Table 2
The effect of material type (group) as a factor on the dependent variable (BFS).

Gr	Observed Mean $\pm$ SD	95% CI of Estimated marginal mean (lower-upper)
ZirCAD	1184.602 $\pm$ 229.552	1108.207-1260.997
e.max CAD	472.758 $\pm$ 39.820	396.363-549.153

*The 95% confidence intervals correspond to the estimated marginal means obtained from the two-way ANOVA and were calculated using the pooled residual standard error of the model.

Two-way ANOVA test also showed that the effect of TCCL as a factor was not statistically significant ($p = 0.895$). To assess the effect of TCCL on each material group separately, pairwise comparisons were made using the Bonferroni adjustment. The BFS of TCCL and non-TCCL samples within either material group was not statistically significant. The mean BFS values for non-TCCL specimens was 1165.88 MPa for ZirCAD and 484.37 MPa for e.max CAD, whereas, for the TCCL specimens, BFS values for ZirCAD⁺ TCCL was 1203.32 MPa and for e.max CAD⁺ TCCL was 461.145 MPa. (p - value = 0.573) As shown in Table 3 and Fig. 4.

Table 3
The interaction of TCCL factor on each group.

Group	TCCL	Mean $\pm$ SD	95% Confidence Interval (lower- upper)
ZirCAD	Yes	1203.322 $\pm$ 264.858	1095.284-1311.361
	No	1165.882 $\pm$ 200.785	1057.843-1273.921
e.max CAD	Yes	461.145 $\pm$ 40.390	353.107-569.184
	No	484.370 $\pm$ 37.636	376.331-592.409

*The estimated marginal confidence interval.

Weibull analysis was conducted to assess the reliability and failure probability of the two ceramic materials, in terms of their BFS. The Weibull modulus (m), was found to be higher for e.max CAD group ($m = 10.98$) compared to ZirCAD group ($m = 5.37$). The Weibull statistical analysis of the BFS data is summarized in Table 4.

Table 4
The Weibull statistical analysis of the BFS data.

Parameter	Weibull modulus (m)	Characteristic strength (MPa) σ_0	Strength for 10% probability of failure (MPa) $\sigma_{0.1}$	Strength for 5% probability of failure (MPa) $\sigma_{0.05}$	Strength for 1% probability of failure (MPa) $\sigma_{0.01}$
ZirCAD	5,37	1302,71	856,75	749,28	553,13
CAD	10,98	479,79	390,91	366,11	315,625

For ZirCAD group, the characteristic strength (σ_0), was noticeably greater (1302.71 MPa) than for e.max CAD group (479.79 MPa). The difference between the two materials was further highlighted by the estimated BFS values for various failure probabilities, at 10% probability of failure ($\sigma_{0.1}$), at 5% probability of failure ($\sigma_{0.05}$) and at 1% probability of failure ($\sigma_{0.01}$).

Discussion

The present study investigated how TC and CL, as means of artificial aging, influence the BFS of two ceramic materials in restorative dentistry; monolithic strength-gradient zirconia (IPS e.max ZirCAD Prime) and monolithic lithium disilicate (IPS e.max CAD). The results showed no significant effect of TC and CL on BFS of both ceramic materials ($p = 0.895$). Based on the results of this in vitro study, the first null hypothesis, that TC and CL have no statistically significant effect on BFS of both ceramic materials, was accepted. However, the IPS e.max ZirCAD Prime group exhibited significantly higher BFS values compared to IPS e.max CAD group ($p < 0.001$), regardless of TCCL conditions. Hence, the second null hypothesis, that there is no statistically significant difference in BFS between ZirCAD group and e.max CAD group, was rejected.

The findings of this study, not only contribute to the existing body of knowledge on ceramic performance under TCCL but also offer comparative data between two distinct material classes; glass ceramics and polycrystalline ceramics. The inclusion of Weibull analysis further strengthens the reliability assessment, which is not always incorporated in similar studies³⁰.

This difference reflects the intrinsic structural differences between the two materials and confirms ZirCAD group's superior mechanical performance, which is consistent with findings in existing literature²¹. This consistency is often quantified using the Weibull modulus (m). A higher Weibull modulus indicates a more uniform flaw distribution and, thus, a more predictable failure behavior, an essential characteristic in the assessment of ceramic materials intended for clinical use³¹.

Disc-shaped specimens measuring 10 mm in diameter and 2 mm in thickness were used to provide a standardized and reproducible testing format which allows consistent control over mechanical variables such as thickness and surface uniformity. Similar dimensions have been employed in previous studies^{20,32–34}. Moreover, to facilitate CL, specimens were secured in customized Stereolithography 3D-printed resin holders, ensuring stable positioning and uniform stress distribution throughout testing.

The significant main effect of material type on BFS ($p < 0.001$) supports previous findings in the literature as Pjetursson et. al (2015) and Yan et. al (2018), that zirconia has superior mechanical properties compared to lithium disilicate. The high mean BFS values of zirconia (> 1160 MPa) versus lithium disilicate (470 MPa) can be attributed to its yttria-stabilized tetragonal crystal structure, which enhances fracture resistance through transformation toughening^{35,36}. In contrast, the glass-

ceramic microstructure of lithium disilicate, despite its favorable esthetics, provides lower mechanical strength and is more susceptible to stress- induced crack propagation ^{20,21}.

Contrary to the initial assumption that TC would degrade BFS due to hydrothermal aging and microcrack formation, our results showed no statistically significant effect of 6,000 thermal cycles on either zirconia or lithium disilicate. These findings are consistent with previous studies reporting that short- to moderate-term TC (e.g., 5,000–6,000 cycles) may not significantly impact the mechanical performance of dental ceramics ³⁷.

Weibull statistical analysis provided additional insight into each material's reliability. Despite being stronger, zirconia had a lower Weibull modulus ($m = 5.37$), which suggests that its mechanical behavior is more variable. On the other hand, despite its lower BFS, Lithium disilicate showed a higher Weibull modulus ($m = 10.98$), indicating more consistent strength values and greater predictability in performance ³⁸.

This variance between strength and reliability can be clinically significant. While zirconia can tolerate greater occlusal stresses, its strength distribution may lead to unexpected failures under some circumstances this influence should also be substantiated by the difference in clinical applications of both materials ^{39,40}.

The negligible impact of TCCL on BFS under the study conditions suggests that both materials maintain mechanical integrity in the face of thermal aging, reinforcing their suitability for long term clinical applications ^{41,42}.

One of the limitations of this study is that it was conducted under controlled in-vitro conditions, which do not fully replicate the complex oral environment. The number of thermocycles, temperature range of the water bath applied, and the number of cycles in mechanical loading may limit the generalization of the findings. Furthermore, the small sample size ($n = 20/\text{material}$) may influence the sensitivity of statistical analysis despite normality assumptions being met.

An additional limitation was the absence of natural tooth substrates, as materials were made into

disc specimens and not bonded to teeth to be tested afterward, which may influence the material behavior compared to clinical scenarios. Nonetheless, the method used allows uniformity regarding sample geometry, by eliminating possible variables introduced by tooth anatomy^{21,32,34,43}.

Future studies should focus on using long-term aging protocols, such as increased thermocycling and dynamic fatigue tests as well as investigating the effects of bonding, surface treatments, and phase transformation in zirconia on long-term clinical performance.

Conclusions

Within the limitations of this in vitro study, the following conclusions can be withdrawn:

IPS e.max ZirCAD Prime exhibits significantly higher BFS compared to IPS e.max CAD, regardless of thermocycling and cyclic loading exposure.

BFS of IPS ZirCAD Prime and IPS e.max CAD are not significantly affected by thermocycling and cyclic loading as performed in this study, suggesting that both ceramic systems possess sufficient mechanical strength under moderate artificial aging methods.

Abbreviations

3 Y-TZP 3 Mol % Ytria-Stabilized Tetragonal Zirconia Polycrystals

4 Y-TZP 4 Mol % Ytria-Stabilized Tetragonal Zirconia Polycrystals

5 Y-TZP 5 Mol % Ytria-Stabilized Tetragonal Zirconia Polycrystals

ISO The International Organization for Standardization

E.max CAD IPS e.max CAD

ZirCAD IPS e.max ZirCAD Prime

TC Thermocycling

CL Cyclic loading

Declarations

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Author Contribution

Conceptualization: DA, NE, Experimental work: DA, Interpretation of data: DA, NE, AM Writing original draft: DA, NE, Prepared tables: DA, Review and editing: NE, AM All authors have read and reviewed the manuscript.

Data Availability

This article has all the data that were collected or analyzed during this study

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Figures

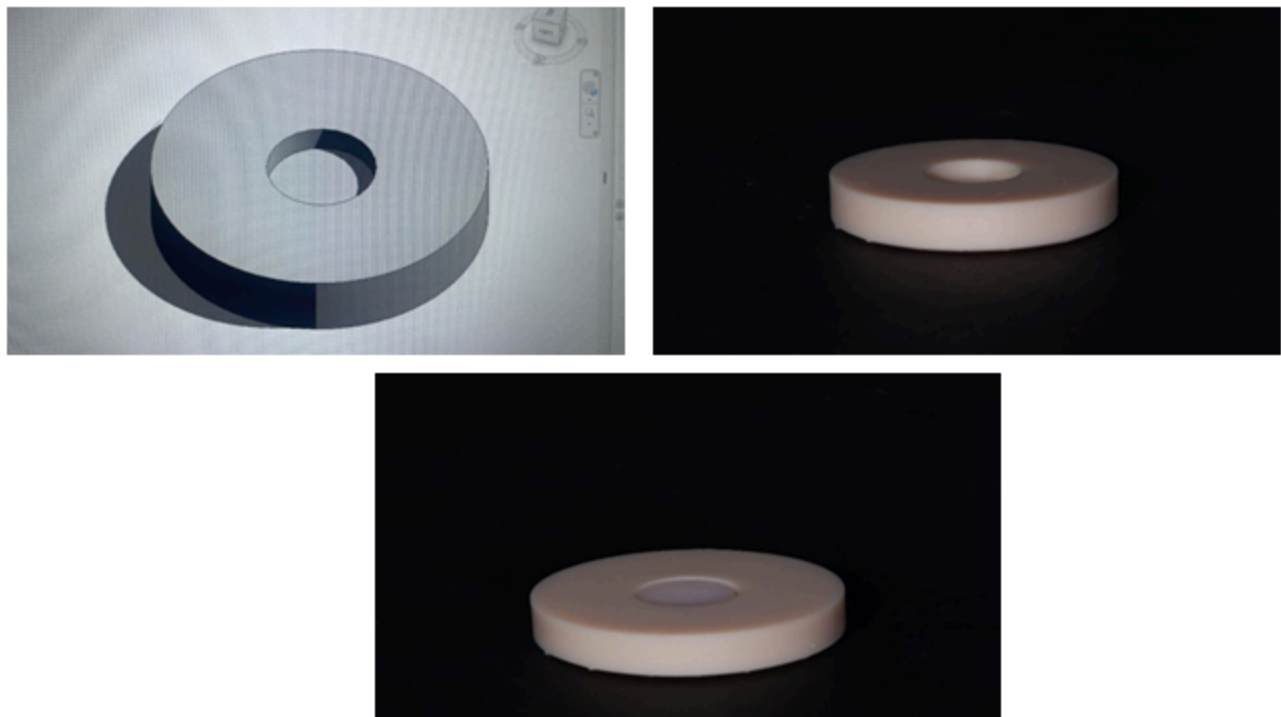


Figure 1

(a) AutoCAD design for the base, (b) The 3D printed resin base, (c) The prepared monolithic zirconia sample from IPS e.max® ZirCAD® Prime, shade BL2.



Figure 2

Load-to-failure test of samples using universal testing machine showing the piston touching the tested specimen.

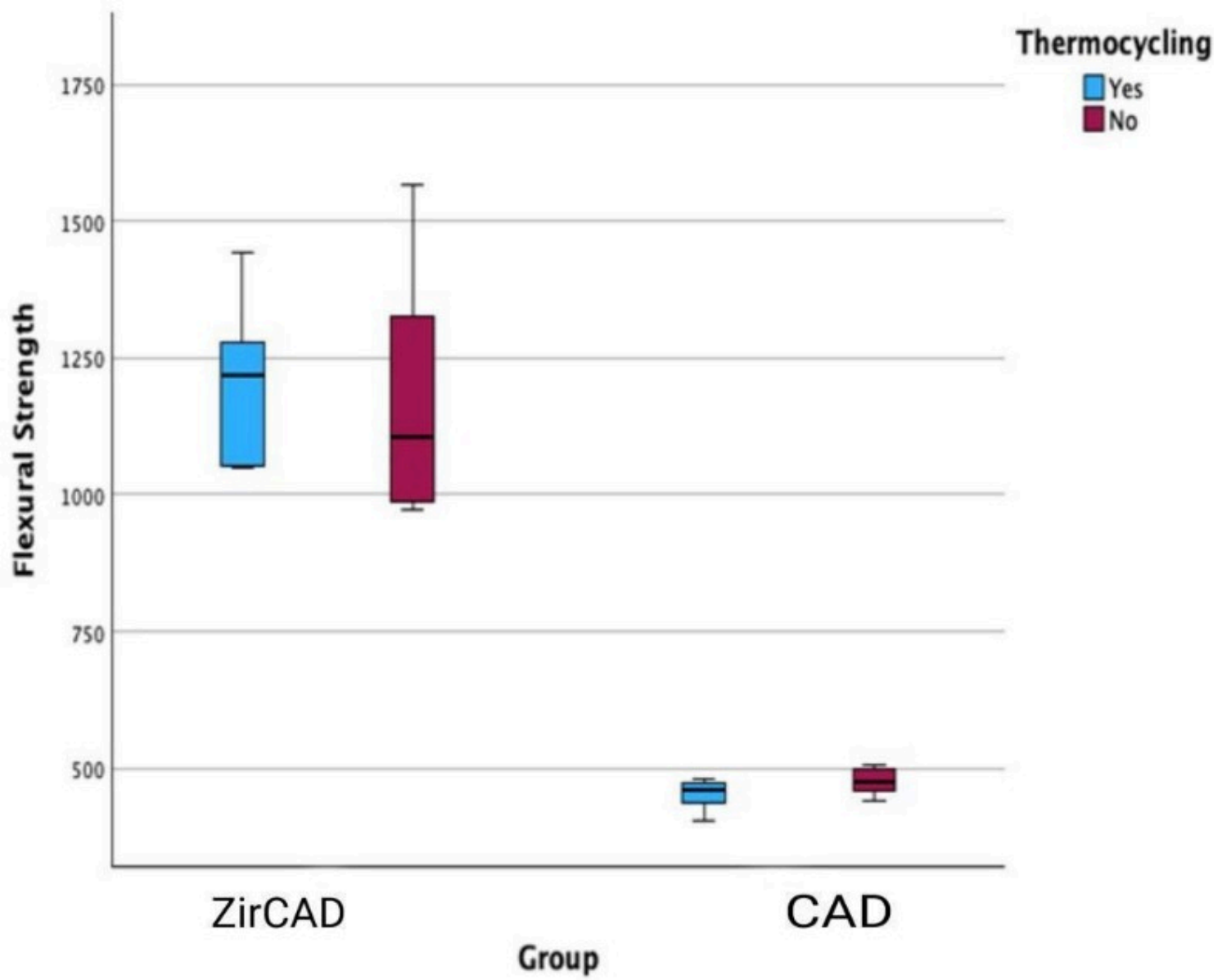


Figure 4

The effect of TCCL on both groups.