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Article

Flexural strength of polyether ether ketone high-performance polyether in comparison with base metal alloy and Zirconia ceramic.

Resistencia a la flexión del poliéter éter cetona poliéter de alto rendimiento en comparación con la aleación de metal base y la cerámica de circonio.

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Abstract: Optimal flexural strength is a critical prerequisite for prosthetic frameworks. This study aimed to assess the flexural strength of polyether ether ketone (PEEK) polymer compared to a base metal alloy and high-strength Zirconia ceramic commonly used in prosthodontic treatments. **Materials and Methods:** In this *in vitro*, experimental study, 10 bar-shaped samples measuring 18×5×2mm were fabricated of each the PEEK polymer, nickel-chromium base metal alloy and zirconia ceramic. Half of the samples in each group were subjected to 5000 thermal cycles between 5°C - 55°C with 20 seconds of dwell time and 20 seconds of transfer time to simulate oral conditions. All samples then underwent three-point bending test. Two-way ANOVA followed by Tukey's test were applied to compare the mean flexural strength of the groups with and without thermocycling at 0.05 level of significance. **Results:** The flexural strength of base metal alloy, Zirconia and PEEK was 1387.70±45.50 MPa, 895.13±13.99 MPa and 192.10±5.37 MPa, respectively. The difference was significant among the groups (p<0.001). Thermocycling had no significant effect on the flexural strength of samples in any group (p=0.306). **Conclusion:** PEEK high-performance polymer had a lower flexural strength than base metal alloy and Zirconia ceramic, and its flexural strength was not affected by thermocycling. PEEK seems to be able to resist masticatory forces in the oral cavity pending further *in vitro* and clinical studies.

Keywords: *Polyetheretherketone; Zirconia; polymers; flexural strength; dental materials; biocompatible materials.*

Abstract: La resistencia a la flexión óptima es un requisito previo crítico para los marcos protésicos. Este estudio tuvo como objetivo evaluar la resistencia a la flexión del polímero de poliéter éter cetona (PEEK) en comparación con una aleación de metal base y cerámica de Zirconia de alta resistencia comúnmente utilizada en tratamientos de prostodoncia. **Materiales and Métodos:** En este estudio experimental *in vitro*, se fabricaron 10 muestras en forma de barra de 18 × 5 × 2mm de cada polímero PEEK, aleación de metal base de níquel-cromo y cerámica de circonio. La mitad de las muestras en cada grupo fueron sometidas a 5000 ciclos térmicos entre 5°C - 55°C con 20 segundos de tiempo de permanencia y 20 segundos de tiempo de transferencia para simular condiciones orales. Todas las muestras se sometieron a una prueba de flexión de tres puntos. Se aplicó ANOVA bidireccional seguido de la prueba de Tukey para comparar la resistencia a la flexión media de los grupos con y sin termociclado a un nivel

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de significancia de 0.05. **Resultados:** La resistencia a la flexión de la aleación de metal base, Zirconia y PEEK fue de 1387,70 ± 45,50 MPa; 895,13 ± 13,99 MPa y 192.10 ± 5,37 MPa, respectivamente. La diferencia fue significativa entre los grupos (p<0,001). El termociclado no tuvo un efecto significativo sobre la resistencia a la flexión de las muestras en ningún grupo (p=0,306).**Conclusión:** El polímero de alto rendimiento PEEK tiene una resistencia a la flexión más baja que la aleación de metal base y la cerámica de circonio, y su resistencia a la flexión no se vio afectada por el termociclado. PEEK parece ser capaz de resistir las fuerzas masticatorias en la cavidad oral, con la necesidad de más estudios *in vitro* y clínicos.

Palabra Clave: *Poliéter éter cetona; circonio; polímeros; resistencia flexional; materiales dentales; materiales bio-compatibles.*

INTRODUCTION.

The increasing price of gold in the recent years has minimized its application in prosthetic dentistry. Base metal alloys are commonly used as an alternative to gold for the fabrication of prosthetic frameworks. However, they have drawbacks such as causing hypersensitivity and allergic reactions, unpleasant sense and taste of metal in the mouth, increased electromagnetic waves and environmental contamination, which warrants finding a more suitable substitute.[1] Pressed alumina and Zirconia ceramics have been suggested as possible alternatives for the fabrication of prosthetic frameworks.[2]

However, the physical, chemical and biomechanical properties of these materials including their elasticity, tolerance, load transfer, water sorption and polishability should be evaluated prior to their use for the fabrication of frameworks.

For instance, ceramic is 20 times harder and gold, titanium and other alloys are 10 times harder than bone and their long-term use can cause wear and destruction of the opposing teeth and alveolar bone.[1,2] The closer the physical properties of these materials to bone, the lower the pressure directly transferred to the abutments and bone would be. Although ceramics are suitable materials for use in patients allergic to base metal alloys, they have drawbacks such as significant difference with bone in terms of their physical and mechanical properties, direct transfer of pressure to abutments, wear of the opposing teeth, relatively high weight, high fabrication cost, low application in fabrication of custom abutments and inability to undergo direct repair in the oral cavity.[3] Therefore, search is still ongoing for an ideal material with physical properties highly resembling those of bone and teeth.

Advances in science and technology resulted in production of advanced polymers, which are used in many fields of science. Bio-high performance polymers available on the market have many applications in medicine and dentistry. They have a long history for the fabrication of prostheses. At present, advanced polymers are used for the fabrication of around 3 million medical, orthopedic and dental products.[4-6]

The polyether ether ketone (PEEK) is a circular and semi-crystalline polymer with a compressive strength of about 6.3 GPa. It has unique physical and mechanical properties and is used in many fields of science. This polymer is a commonly used type of high-performance polymer. It has been used as a biomaterial for the fabrication of different types of dental prostheses. PEEK has ceramic fillers measuring 3.0 to 5.0 µm, which are dispersed homogenously.

Thus, it has excellent polishability and minimal color change and plaque accumulation.[7] Moreover, it can be used in different types of pressed and computer-aided design/computer-aided manufacturing (CAD/CAM) systems due to its optimal mechanical properties. Due to optimal wear resistance, excellent mechanical properties and adequate bond strength of PEEK to composite and tooth structure, fixed partial dentures (FPDs) made of PEEK may show satisfactory survival in the oral environment.[8] However, further studies are required regarding its applications in dentistry.

The important mechanical and physical properties of materials used for the fabrication of dental prostheses include adequate flexural and tensile strength and modulus of elasticity, maximum fracture resistance, optimal bond strength and adequate polishability.[9]

The bond strength of PEEK to composite resins has been previously evaluated.[7-9] However, there is a gap of information regarding the flexural strength of PEEK in comparison to other materials. Since flexural strength is a critical property for dental prosthetic frameworks, this study aimed to assess the flexural strength of PEEK high-performance polymer in comparison with a base metal alloy and Zirconia ceramic commonly used in prosthodontic treatments.

MATERIALS AND METHODS.

This *in vitro*, experimental study evaluated bar-shaped samples (n=10) made of PEEK, base-metal alloy and Zirconia ceramic. Sample size was calculated to be 5 in each of the six groups (PEEK, base metal and ceramic with and without thermocycling). Thus, a total of 30 samples were evaluated, Table 1.

Fabrication of samples

Bar-shaped samples with a rectangular cross-section measuring 18mm in length, 5mm in width and 2mm in thickness were fabricated of PEEK (breCAM BioHPP, Bredent GmbH, Senden, Germany) synthetic polymer, yttrium stabilized tetragonal Zirconia ceramic (Ceramill Zolid FX ML Zirconia, Amann Girrbach, Koblach, Austria) and nickel-chromium base metal alloy containing 73% to 78% nickel and 12% to 14% chromium (Supremcast, American Dent-All Inc, NY, USA).

Ten samples were fabricated of each material. Selection of the size and shape of samples and method of load application were determined according to ISO 6872,[11] ISO 14704,[12] ISO 178[13] and ASTM D790.[14]

Samples with defects were excluded and replaced with sound samples. For the PEEK and Zirconia ceramic samples, first blocks of these materials were obtained. Next, the sample with the desired size was designed using Ceramill Mind design software (AmannGirrbach).

Eventually, the samples were milled by a milling machine (ZENO 4030 M1; Wieland + Dental, Pforzheim, Germany). For the fabrication of base metal alloys, first wax models with desired dimensions were prepared. Investing was performed and the samples were then cast by induction casting method.

After fabrication of samples, their surface was polished with 600 and 800-grit silicon carbide abrasive papers (3M ESPE) with light pressure to obtain a smooth surface. The duration of polishing of each sample with each abrasive paper was about 2 minutes.

Thermocycling

Samples in each group were randomly divided into two equal groups (n=5). Half of the samples in each group were subjected to thermocycling for 5000 cycles between 5°C and 55°C with 20 seconds of dwell time and 20 seconds of transfer time using a thermocycler (Dorsa Co., Tehran, Iran). All samples were incubated at 37°C for 24 hours prior to flexural strength testing.

Flexural strength testing

The flexural strength of all samples was measured using three-point bending test.[15,16] Load was applied by a cylindrical rod to the center of bar-shaped samples positioned on two supports until fracture. Due to stress accumulation at the center of rod prior to fracture of the sample, first, a V-shaped deformation occurs at the site of load application.

Thus, a high load needs to be applied to break the sample. Thus, the flexural strength value may be overestimated.[17] In three-point bending test, samples with 18mm length were positioned on two cylindrical supports with 2mm diameter. Next, using a cylinder with 2mm diameter, load was applied to the center of the upper surface of samples until their fracture.[18]

A universal testing machine (Santam Co., Tehran, Iran) was used for this purpose with a crosshead speed of 1mm/minute. The load was applied uniformly and gradually increased until fracture. The load at failure was converted to flexural strength value using the formula below:

Where Qf is the flexural strength in megapascals (MPa), F is load in Newtons, L is length in millimeters (mm), h is the sample thickness in millimeters and b is the sample width in millimeters (mm).

Data were analyzed using SPSS version 18 (SPSS Inc., IL, USA). The mean and standard deviation of flexural strength in the groups were calculated and tabulated. The Shapiro-Wilk test was applied to assess the distribution of data, which showed that data were normally distributed in all groups.

Thus, two-way ANOVA followed by Tukey's post hoc test were applied to compare the mean flexural strength of the groups with and without thermocycling; $p<0.05$ was considered statistically significant.

RESULTS.

Table 2 shows the mean flexural strength of the groups. Two-way ANOVA showed a significant difference in flexural strength of the groups (Figure 1, $p<0.001$). However, the flexural strength of thermocycled and non-thermocycled samples was not significantly different ($p=0.306$).

In other words, the effect of group on flexural strength was significant ($p=0.001$). The effect of thermocycling on flexural strength was not significant ($p=0.306$). But the interaction effect of group and thermocycling on flexural strength was statistically significant ($p=0.011$).

Table 3 shows pairwise comparison of the groups. In general, the flexural strength of PEEK was significantly

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Figure 1. Comparison of the mean flexural strength of the groups with/without thermocycling.

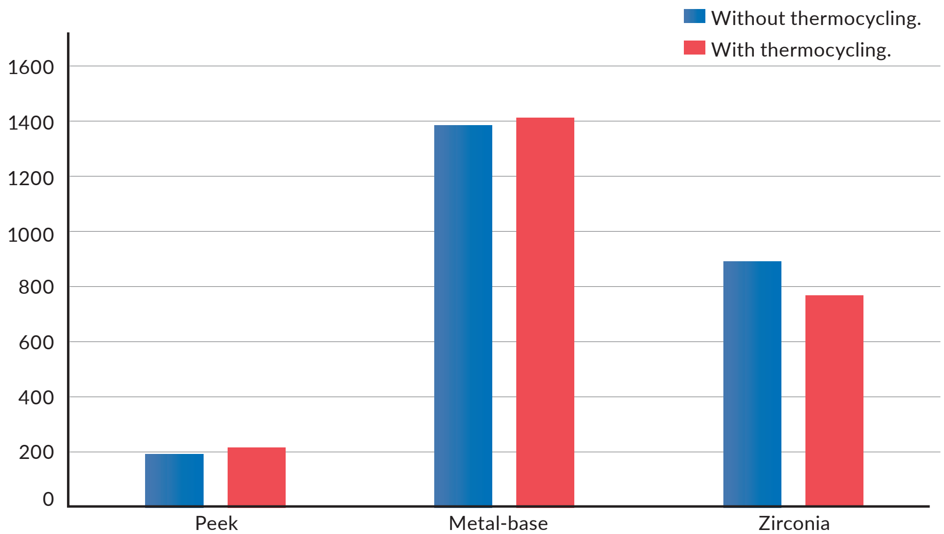
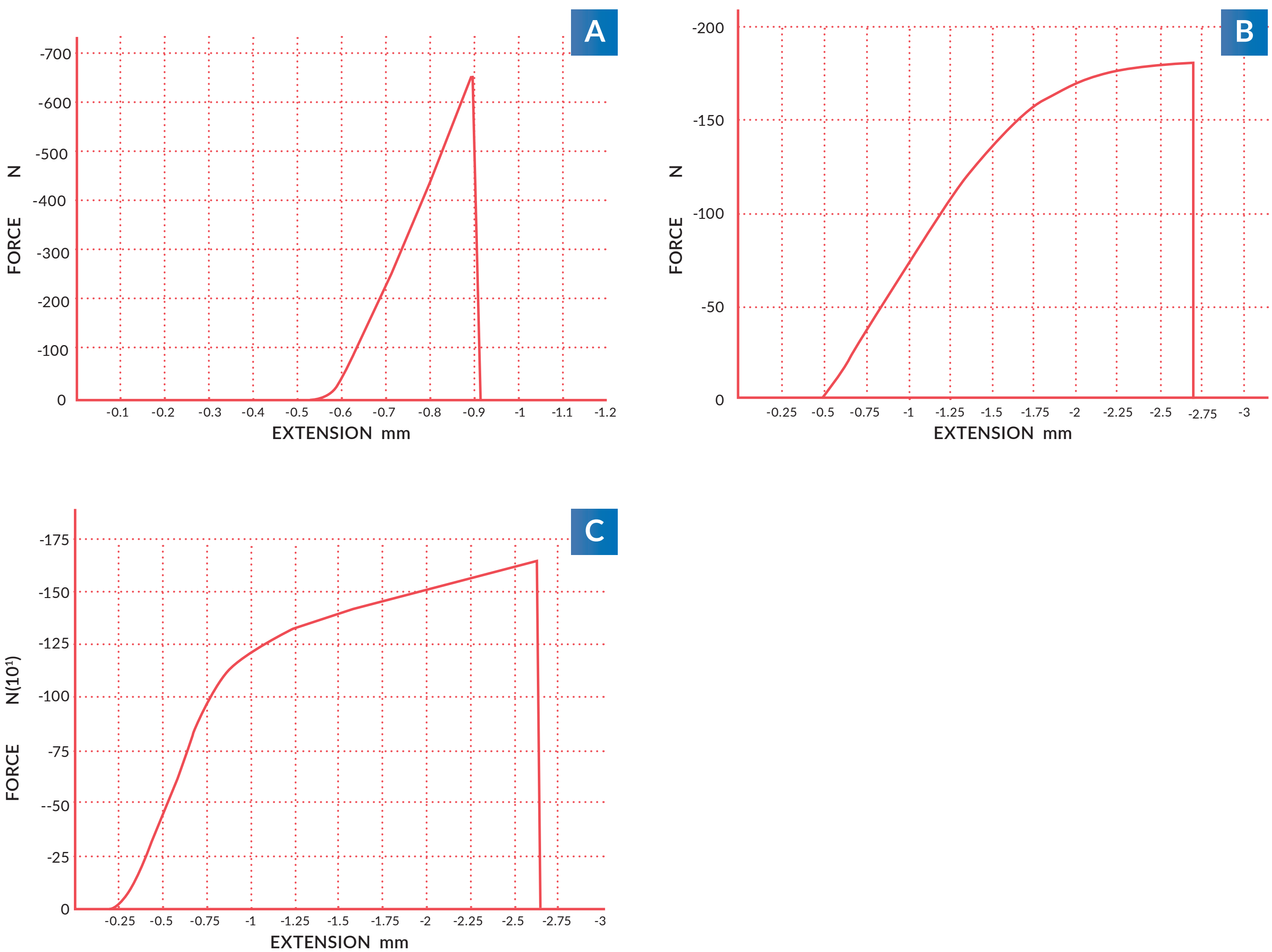


Figure 2. Force/extension curve of three materials.



A: zirconia sample. **B:** PEEK sample. **C:** base metal sample. Note the fragile behavior of Zirconia compared to the plastic nature of base metal extending the fracture limit to its most. The PEEK samples illustrated an intermediary behavior.

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Table 1. Materials used in this study and their characteristics.

Material	Commercial brand	Manufacturer	Batch number of Lot number
PEEK	breCAM BioHPP	Bredent GmbH, Senden, Germany	540 0203 1
Base metal alloy	Supremcast	American Dent-All Inc., NY, USA	----
Zirconia ceramic	Ceramill Zolid FX ML Zirconia	Amann Girrbach, Koblach, Austria	761739

Table 2. Mean flexural strength (MPa) of the groups (n=5).

Group	Thermocycling	Mean	Std. deviation	Minimum	Maximum
PEEK	No	192.1006	5.37458	184.28	196.53
	Yes	216.9936	39.52101	152.63	244.81
Base metal	No	1387.7039	45.49852	1342.34	1453.59
	Yes	1416.3137	105.84987	1279.91	1538.38
Zirconia	No	895.1265	13.99460	884.33	921.35
	Yes	772.8480	85.45137	680.25	861.88

Table 3. Pairwise comparisons of the groups.

Group (I)	Group (J)	Mean difference (I-J)	Std. error	*p*-value
Base metal	PEEK	1197.462	27.697	0.0011
	Zirconia	568.022	26.383	0.0023
PEEK	Base metal	-1197.462	27.697	0.0011
	Zirconia	-629.440	26.383	0.0046
Zirconia	Base metal	-568.022	26.383	0.0023
	PEEK	629.440	26.383	0.0046

lower than that of other two materials ($p<0.001$) while the flexural strength of base metal was significantly higher than that of other groups ($p<0.001$).

The force/extension curve of the majority of base-metal samples indicated relatively high deformation in these samples. However, almost all Zirconia samples showed a fragile nature and no plastic deformation. Some of the PEEK samples showed plastic deformation before fracture (Figure 2).

DISCUSSION.

This study assessed the flexural strength of PEEK in comparison to Zirconia and base metal alloy, which are commonly used in dental restorations. The effect of thermal cycles on flexural strength of these materials was also evaluated.

The results showed that the flexural strength of PEEK was significantly lower than that of Zirconia and base metal alloy and it was not affected by thermocycling. However, no similar previous study is available on this topic to compare our results with.

PEEK is believed to be suitable for the fabrication of restorations using the CAD/CAM system. This polymer has optimal mechanical properties, which indicate its ability for use as a core material in FPDs.

Clinical studies with a short follow-up period have shown that PEEK can be successfully used for the post and core restorations[19] and FPD frameworks.[20] Han *et al.*,[21] used PEEK for the fabrication of framework of implant-supported and tooth-supported restorations in a case report and reported successful short-term (one-year) clinical results. However, there is no long-term clinical study on the efficacy of this polymer for the fabrication of FPDs.

The majority of studies on the applications of PEEK in prosthodontics had an *in vitro* design and focused on the mechanical properties and bond strength of this material.[3-7] In the present study, the flexural strength of PEEK samples was 192.1±5.37 MPa. A previous study with a similar methodology reported that the flexural strength of this polymer (manufactured by different companies) ranged from 170.37 to 1009.63 MPa.[22]

It should be noted that many factors such as the details of polymer composition according to the protocol adopted by the manufacturer and the manufacturing process of PEEK can affect many of its properties such as its flexural strength. Evidence shows that FPDs made of PEEK by the CAD/CAM system have higher fracture strength than those manufactured by the pressed technique.[23]

The manufacturing company can also affect the strength of PEEK such that Schwitalla *et al*.,[22]showed that the modulus of elasticity of PEEK ranged from 2.73 GPa for an infilled brand to 47.27 GPa for carbon fiber reinforced PEEK. The flexural strength of PEEK polymer is variable from 170.37 to 1009.63 MPa. In the present study, only one type of PEEK polymer was evaluated; no previous study was found on the properties of PEEK produced by this manufacturer.

The mechanical properties of Zirconia ceramic are highly superior to those of other dental ceramics.[24,25] The flexural strength of Zirconia ranges from 800 to 1200 MPa.[26,27] On the other hand, its fracture toughness is 6 to 10 MPa, its compressive strength is 2000 MPa and its failure load is 706 N.[28] It seems that the fracture strength of Zirconia is higher than the masticatory forces. Thus, such frameworks probably have optimal clinical efficacy for use in the oral cavity.[29]

In the present study, the mean flexural strength of Zirconia was 895.13±13.99 MPa. The stress-strain curve of Zirconia also showed its fragile nature. Previous studies on flexural strength of Zirconia have reported both lower[18] and higher.[30,31] values. A previous study with similar methodology showed the flexural strength of Zirconia to be 1143 MPa. Another study reported that the flexural strength of polished Zirconia rods was 1200 MPa. A more recent study reported that the flexural strength of partially stabilized monolithic Zirconia, fully stabilized Zirconia, and core Zirconia was 1008, 582 and 1034 MPa, respectively.[15]

On the other hand, Siarampi *et al*.,[32] evaluated the flexural strength of two commercial Zirconia brands used in core and reported that the flexural strength of IPS e.max zirCAD was 463 MPa while that of Weiland ZENO was 546 MPa. Three-point bending test was used for assessment of flexural strength of Zirconia in three other studies.[33] The mean flexural strength of Zirconia was reported to be 952 MPa and 752 MPa.[34] in two of them. The third study conducted by Mohammadi-Bassir *et al*.,[35] reported the mean flexural strength of Zirconia to be 935.58 MPa. Similarly, Ceramill core Zirconia was used in our study and its flexural strength was found to be 895.13±13.99 MPa.

Despite the extensive use of base metals in dental prostheses, studies on their flexural strength are limited. A previous study measured the flexural strength of chromium-cobalt alloy to be 1945 MPa.[36] This value was 1640 MPa for nickel chromium alloy in another study.[37] Rocha *et al*.,[38] reported the flexural strength of nickel chromium alloy to be 1488 MPa. In our study, the flexural strength of base metal alloy was 1387.7±45.50 MPa. Although none of the previous studies available on this topic used the same commercial brand of base metal alloy as ours, our findings were in agreement with theirs.[37,38] Also, the stress-strain curve of this alloy indicated that it had higher plastic behavior than the other two materials and was less fragile.

The main components of the nickel chromium alloy used in FPDs include nickel (70% to 80%) and chromium (13% to 20%).[39] Any change in the percentage of each component can alter the yield strength, hardness and modulus of elasticity of the alloy. Thus, the production protocol determined by each manufacturer plays an important role in its mechanical properties. For instance, the yield strength of these alloys varies from 260 to 807 MPa and their hardness varies from 175 to 335 depending on their commercial brand.[40]

In our study, PEEK showed the lowest and base metal alloy showed the highest flexural strength. The difference in this regard was significant among the groups. Search of the literature yielded only one relatively similar study on the fracture strength of three-unit implant supported fixed partial dentures made of Zirconia, nickel chromium and PEEK, which showed that the mean fracture strength of Zirconia, nickel chromium alloy and PEEK was 2086, 5591 and 1430 N, respectively.[41]

In this study, the samples underwent 5000 thermal cycles, which corresponds to 6 months of clinical service in the oral cavity according to some.[42-46] and 1.5 years according to some others. Evidence shows that thermal cycles decrease the strength of metal ceramic restorations.

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Nonetheless, thermal cycles had no significant effect on base metal alloy or PEEK in our study. However, they decreased the flexural strength of Zirconia. Thermal changes create stresses in materials made of different components with different coefficients of thermal expansion and moduli of thermal conductivity.[47]

Since the materials tested in our study were mono-phase, no change in their flexural strength by thermocycling was somehow expected. Beuer *et al*.,[48] evaluated the fracture strength of three-unit FPDs made of glass-infiltrated alumina, glass-infiltrated alumina strengthened with Zirconia and yttria-stabilized polycrystalline Zirconia and showed that the latter group had higher fracture strength.

The fracture strength of none of the ceramics was affected by mechanical and thermal cycles performed for aging.[48] Another study assessed the effect of 5000 thermal cycles on flexural strength of Zirconia ceramic crowns with different designs and showed no significant effect of thermocycling on their flexural strength.[49] Taufall *et al*.,[50] measured the fracture strength of three-unit fixed partial dentures made of PEEK before and after 10,000 thermal cycles and showed no change in fracture strength of these restorations.

However, in cases where the PEEK core was digitally veneered, the fracture strength increased from 1882 N before thermocycling to 2021 N after thermocycling.[50] An *in vitro* study showed that PEEK has insignificant water sorption and solubility, and storage in water and artificial saliva had no significant effect on its mechanical properties.[51]

Schwitalla *et al*.,[22] measured the flexural strength of PEEK polymers manufactured by different companies and showed that storage in 37°C water for up to 84 days increased their flexural strength but not significantly. All these data, in accordance with the present study introduce PEEK as a durable material that could be used more frequently in future as a material that would not be significantly affected by thermal fluctuations in oral cavity.

This study had an *in vitro* design. *In vitro* studies are commonly performed to predict the behavior of materials in the clinical setting.[52] However, *in vitro* studies evaluating only one mechanical property cannot perfectly reflect what happens in reality in the clinical setting.[53] For instance, the effect of masticatory forces, veneering and chemical constituents of food and saliva could not be evaluated in our study. Thus, the results of *in vitro* studies cannot be completely generalized to the clinical setting.

However, *in vitro* studies can serve as a basis for further clinical investigations. Future studies are required to assess other properties of these materials such as their bond strength to porcelain and resin and their shear strength. Other mechanical properties of these materials should be evaluated in conditions simulating the clinical setting. Last but not least, clinical studies are required to cast a final judgment regarding the performance of these materials in the clinical setting.

CONCLUSION.

Within the limitations of this *in vitro* study, the results showed that PEEK high-performance polymer had a flexural strength lower than that of base metal alloy and Zirconia ceramic.

The flexural strength of base metal alloy evaluated in this study was higher than that of Zirconia. The flexural strength of PEEK, base metal alloy and Zirconia ceramic was not affected by thermocycling.

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