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Posted Date: 28 September 2023

doi: 10.20944/preprints202309.1985.v1

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Article

Relation of Crown Failure Load To Flexural Strength for Dental Polymers and a Fiber Reinforced Composite Resin

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Abstract: Dental polymers are often advertised and selected for use in restorations based upon the flexural strength of prefabricated discs, which may not accurately reflect their performance in dental applications. The objectives of this study were, therefore, to determine whether the three-point bending (3PB) test for flexural strength would be predictive of the "crunch-the-crown" CTC test for the failure load. Three brands of polymers (Trilor, Juvora, and Pekkton) were fabricated into rectangular bars and fully contoured crowns (10 specimens of each polymer brand, 30 specimens of each shape). Bars were tested in 3PB to determine flexural strength, and crowns were CTC tested to determine failure load after luting to resin abutments. Statistical significance was evaluated by one-way ANOVA ($\alpha = 0.05$) and Pearson's correlation coefficient. The fracture mechanisms were characterized by scanning electron microscopy (SEM). There were significant differences ($p < 0.05$) in the mean crown failure loads (Trilor [7,033 N] > Juvora [5,217 N] > Pekkton [3,023 N]) and mean flexural strengths of the bars (Trilor [468 MPa] > Juvora [197 MPa] = Pekkton [192 MPa]). There were no correlations between flexural strength and failure load. The mode of crown fracture was different between the polymers, and included deformation (Juvora), ductile-to-brittle fracture (Pekkton), and a combination of cracks and deformation (Trilor). The flexural strength was not correlated with the corresponding crown failure load. Dental practitioners should not rely on the value of flexural strength obtained from the three-point bending test, as advertised from the manufacturer, to predict the performance of polymeric crowns.

Keywords: polymer; flexural strength; composite resin; fiber; crown; bar

1. Introduction

Polymer-based materials are an ideal choice for dental applications that require bending, low density, and/or high adhesion [1,2]. A variety of monolithic materials are available, some of which can be reinforced with fibers or particles. These reinforcements generally increase the elastic modulus, strength, and wear resistance of the matrix [3]. Reinforcement of resin materials with fibers can increase mechanical properties and improve the clinical performance of dental restorations during their clinical function [4–6]. The improvement of composite resin depends on the fiber type (glass [7,8], polyethylene [7], carbon [8]) and its orientation [7].

Polyaryletherketones (PAEKs) are a family of high-performance semicrystalline (crystalline as well as amorphous) materials. Two commercially available PAEKs for dental applications are PolyEtherEtherKetone (PEEK) and PolyEtherKetoneKetone (PEKK). PEEK is manufactured through CAD/CAM technology, while PEKK may also be heat pressed [9]. PEEK was initially applied to frameworks for fixed [10–12], and removable partial dentures [13], implant abutments [14,15], surgical membranes [16], postcores [17], and implant bodies [18].

PEKK was introduced in dentistry more recently and has higher compressive strength and ductility and better long-term fatigue properties than unreinforced PEEK. PEKK has served as a framework for implant-supported completely fixed dental prostheses because of its light weight [19]. Klur et al. [20] reported that PEKK can be used as a stabile framework material for provisional fixed partial dentures; however, fractures of PEKK-made cantilever bridges occurred after a short time.

 doi:10.20944/preprints202309.1985.v1

Shams et al. [21] reported that PEKK coping veneered with cemented IPS e.max CAD can be an alternative to monolithic IPS e.max CAD endocrowns in terms of fracture resistance.

Rohr et al. [22] reported that Pekkton molar crowns on zirconia implants exhibited fracture load values similar to or higher than lithium disilicate crowns. Prechtel et al. [23] reported that 3D printed and milled indirect PEEK molar inlays presented a higher fracture load than the expected physiological and maximum chewing forces. Mangoush et al. [24] reported that CAD/CAM fabricated upper central incisor crowns made of single-structure short fiber-reinforced composites demonstrated encouraging performance related to their fracture behavior. Zimmermann et al. [25] reported a higher fracture load of particle-filled composite resin CAD/CAM crowns compared to ceramic CAD/CAM crowns for 0.5 mm thicknesses.

It has been shown that the crunch the crown (CTC) tests do not replicate clinical stresses generated during loading (which are tensile on the intaglio surface, not compressive on the outer surface) and provide little, if any, information regarding the mechanical failure of materials during in-service usage [10]. Manufacturers depend on flexural strength information to differentiate between materials because they have more standardized specimen dimensions and loading conditions, as illustrated in the ISO and ASTM standards. It is not clear, however, if such data are predictive of the performance of dental crowns, which motivated the present efforts.

Rodrigues et al. [26] reported that flexural strength as measured by 3-point bending was higher than that measured by 4-point bending for microhybrids and nanofill composites. Miura et al. [27] reported that the biaxial flexural strength exceeded the 3-point bending strength, which exceeded the 4-point bending strength for dental hard resins. Pick et al. [28] reported that piston-on-ball (POB) tests detected more differences and displayed less data scattering for resin composites compared with three-point bending tests. Additionally, the results from the POB test were closer to the strength of the material, as estimated by finite element analysis. Winter et al. [29] reported that the fracture resistance and flexural strength of CAD/CAM polymer-based material were lower than those of glass ceramic but still sufficient for use in the first molar region, without studying the correlation between the two mechanical properties. Rohr et al. [22] reported that a linear trend was found between the fracture load and the fracture toughness of Pekkton material.

The objectives of this study were, therefore, to determine whether the three-point bending (3PB) test for flexural strength would be predictive of the "crunch-the-crown" CTC test for the failure load. We hypothesized that the 3PB flexure strength would be predictive of CTC failure load for the different polymeric materials.

2. Material and Methods

Three polymeric materials (Trilor; TRI, Juvora; JUV, Pekkton, PEK) were provided, and the specimens were fabricated as follows: crown-shaped specimens were used to test the failure load using CTC, and rectangular shape bars were used to test the flexural strength using 3PB. The specifications of the test materials, including the identifier, manufacturer, class, Young's modulus, and Poisson's ratio, are shown in Table 1 [30]. The classification of mechanical properties according to the geometry and dimensions is detailed in Table 2. The sample size was determined from past publications (n =10) [10,12,31].

Table 1. The abbreviation, manufacturer, color, class, Young's modulus and Poisson's ratio of the test materials.

Test Materials	Abbreviation	Manufacturer	Young's Modulus (GPa)	Poisson's Ratio
Trilor	TRI	Bioloren S.r.l., Saronno (Varese), Italy	26	0.25
Juvora	JUV	JUVORA Dental, Lancashire, UK	4	0.36
Pekkton	PEK	Cendres+Métaux SA, Biel-Bienne, Switzerland	5	0.38

Epoxy Resin	Epoxy	American Dental Supply, Inc. Allentown, PA	4	0.3

Table 2. Classification of structural/mechanical properties according to the specimen geometry and dimensions.

Structural/Mechanical Property	Specimen Geometry	Type of Test	Description	Number of Specimens per Material	Abbreviation
Failure Load (N)	Crown on resin Abutment	Crunch-the-Crown (CTC)	Maxillary Right 1st Molar with the thickness of 0.8 mm at the central fossa	10	CTC - Crown
Flexural Strength (MPa)	Bar	Three-Point Bending (3PB)	width = 2.0 mm ± 0.2 mm, thickness = 2.0 mm ± 0.2 mm, length = 25.0 mm	10	3PB - Bar

Burn-off tests were conducted to measure the fiber weight percentage of the fiber-reinforced polymeric materials. The tests were carried out based on the procedure specified in ASTM D3171-2015 [32] to determine the constituent content of composite materials. The mass before burn off (initial mass M_i) and after burn off (M_f) was measured, and the following equation was used to calculate the fiber weight percentage W_r:

$$W_r = (M_f / M_i) \times 100. \quad (1)$$

An ivorine maxillary 1st molar tooth (Model #R861; Columbia Dentoform Corp, Long Island City, NY) was prepared and then duplicated and poured with a resin material (Die Epoxy Type 8000, American Dental Supply, Inc. Allentown, PA) to fabricate resin abutments (10 abutments per brand, height = 8 mm from the highest point of the occlusal surface to the base) [33]. The epoxy resin abutments were digitized using a 3D scanner (D2000, 3Shape A/S, Copenhagen, Denmark) to fabricate the crown shape specimens using a Wieland Mini milling machine (Wieland Dental + Technik GmbH & Co. KG, Germany). The crown specimens were fabricated with the following specifications: cement gap =.075 mm, extra cement gap =.120 mm, and polymer thickness at the central fossa = 0.8 mm according to manufacturer recommendations.

The cementation technique followed previous publications [34,35], and Panavia V5 (Kuraray Medical Inc, Japan) was used to attach the crowns to the corresponding resin abutments. A load of 50 N was applied on the monobloc of the crown-resin abutment after removal of excess cement [34,35]. All cemented crown-on-resin abutments were stored for 24 hours in water at 37 °C.

A steel ball (diameter of 11.37 mm) was used in the CTC test to crush the cemented crown-on-abutment specimens using a mechanical testing machine (MTS 858 Mini-Bionix, MTS Systems, Eden Prairie, MN) at a speed of 0.5 mm/min until failure [33]. A polyethylene sheet (0.9 mm thickness) was placed between the ball and the crown to distribute the loaded occlusal region [33]. The failure load was recorded as the maximum force for each test.

The bars were fabricated, and their edges were bevelled using a Wieland Mini milling machine. The bar specimens were fabricated with the following dimensions: 2.0 ± 0.1 mm wide, 2.0 ± 0.1 mm thick, and 25.0 ± 2.0 mm long.

A mechanical testing machine was used to load the bar specimens at 0.5 mm/min until the specimens failed. The test span was 20.0 mm. The three-point flexural strength (MPa) was calculated according to the ISO 20795-1:2013 standard [36]:

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$$\sigma = \frac{3Fl}{2bh^2} \quad (2)$$

where F is the maximum load in Newtons; l is the test span in mm; and h and b are the bar thickness and width, respectively, in mm.

Scanning electron micrographs (Quanta FEG 650 scanning electron microscope, FEI, Hillsboro, Oregon) were obtained after experimental failure of the specimens, which were sputtered with gold palladium. A high-vacuum mode (accelerating voltage = 30 kV) was used. Energy dispersive spectroscopy (EDS) was conducted at different points to determine the polymer/fiber composition.

Statistical differences comparing mechanical properties were determined from one-way ANOVA with Tukey post hoc tests. Data transformation using rankings was used if needed to provide for equality of variances (Levene test) and to better normalize the distribution (Shapiro–Wilk test). The coefficient of Pearson's correlation was used to determine the correlation between the flexural strength and failure load. Regression of mean values correlating various data points was performed using the data analysis tool in Excel. The results returned a correlation coefficient and p value.

3. Results

The epoxy matrix of the TRI material was completely burned off in air, and the residue was calculated to be 63 wt% glass fibers.

The mean and standard deviation for the flexural strength and failure load for each polymer are listed in Table 3. TRI had a higher flexural strength and failure load than PEK and JUV ($p < 0.05$). There was no significant difference ($p > 0.05$) in the flexural strength between JUV and PEK, as listed in Table 3. There was no correlation between flexural strength and failure load, as shown in Figure 1. The failure load (TRI > JUV > PEK) was not proportional to flexural strength, as indicated by $R^2 = 0.31$ and $p = 0.62$.

Table 3. Mean values of measured flexural strength (MPa) and crown failure load (N) of TRI, JUV, PEK with different specimen geometries (3PB-Bar, CTC-Crown).

		Mean of Strength/Load	SD
3PB-Bar	TRI	468 MPa[a*]	97
	JUV	197 MPa [b*]	10
	PEK	192 MPa [b*]	15
CTC-Crown	TRI	7,033 N[a*]	794
	JUV	5,217 N[b*]	169
	PEK	3,023 N[c*]	418

* Different letters within the test (3PB, CTC) indicate that there is a significant difference between the materials. Letter a means the highest value, and c means the lowest value. The same order of the letters within the test with different tests confirms the correlation. There was no correlation between the 3PB and CTC tests because they have different orders of materials. TRI = Trilor, JUV = Juvora, PEK = Pekkton, 3PB = Three-point bending, CTC = Crunch-the-Crown.

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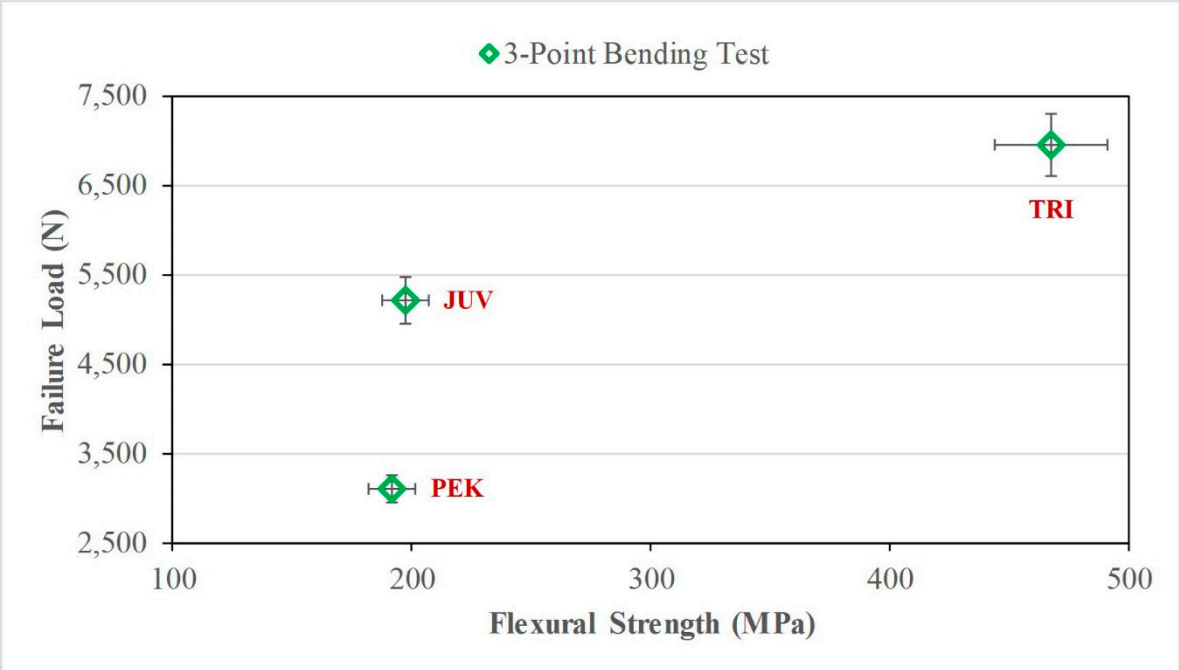

Figure 1. Flexural strength (bar specimens) with failure load (crown specimens). There was no correlation (R^2 = 0.31 and P =.062) between flexural strength and failure load.

The SEM images illustrated in Figure 2 on the cracked surface of a TRI bar showed that the material had woven fabric in a 0/90 weave pattern. The fiber diameter ranged from 12 to 14 μm. In addition, the fiber contained Si (24 wt%), Al (8 wt%), Ca (15 wt%), and O (50 wt%) elements (based on the EDS spectrum), as illustrated in Figure 2, which indicates that the fiber in TRI is glass. TRI specimens (bars and crowns) failed by a combination of cracks and deformation at the occlusal surface of the crowns as well as the center of the bars.

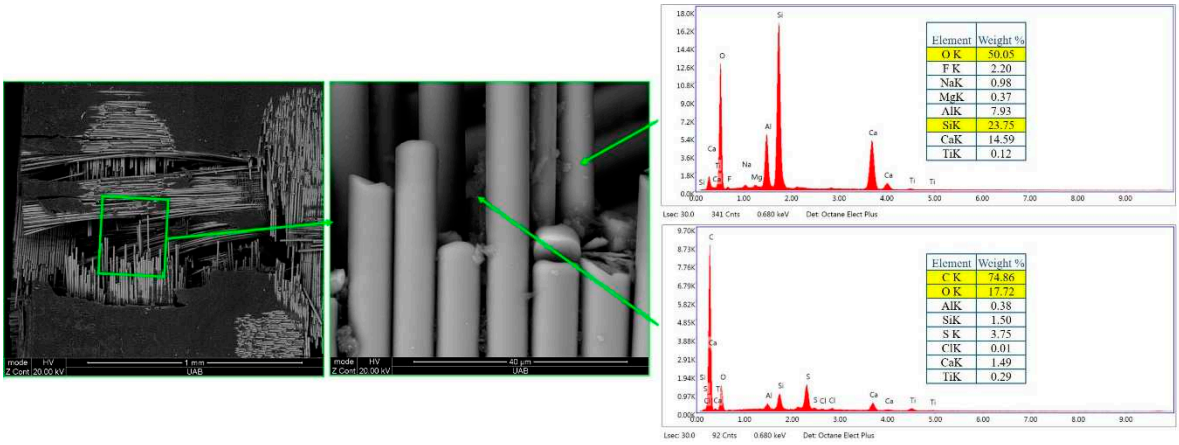

Figure 2. The BSE microphotographs of fractured surfaces after the three-point bending test for TRI at different magnifications (top: 50x = bottom. Left =100x, bottom right = 2500x). The microstructure presented fibers with concentrations of Si and O, which confirmed that the fibers were glass (right upper panel) compared to the EDS for the resin matrix (right lower panel). BSE = backscattered (reflected) electrons, TRI = Trilor, Si = silicone, O = Oxygen, EDS = Energy dispersive spectroscopy.

The mode of fracture was different for JUV and PEK materials (Figure 3). The JUV crowns (Figure 3, left) failed with surface cracking that did not progress, leaving the crowns relatively intact. In contrast, the PEK crowns (Figure 3, right) failed by ductile-to-brittle fracture, as evidenced by

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dimple formation (micro void coalescence) underneath the occlusal surface, surrounded by brittle fracture surfaces and fractured completely into two pieces.

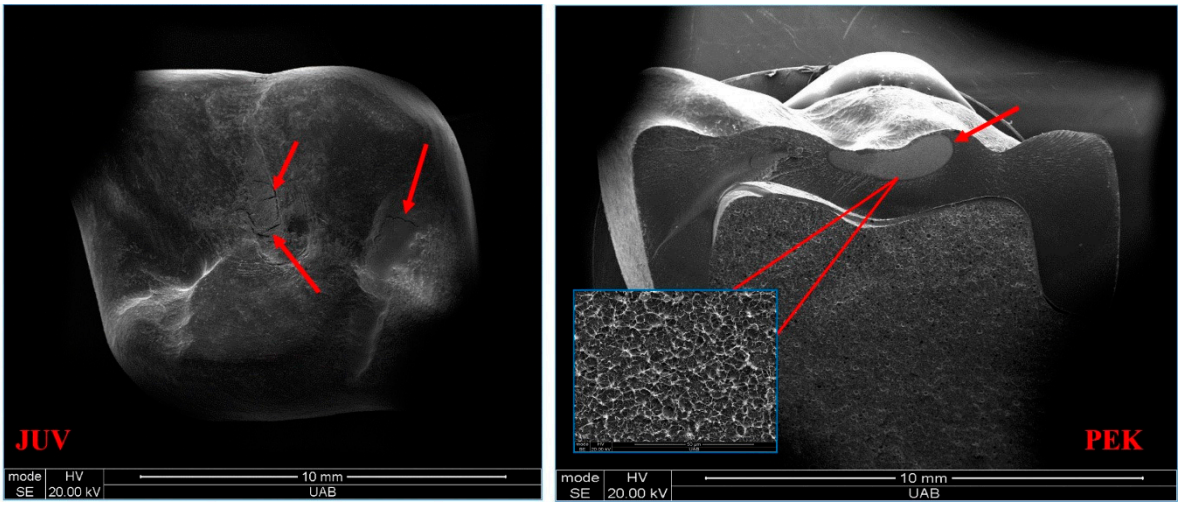

Figure 3. SEM micrographs of fracture surfaces of a JUV (PEEK) at 20x and PEK (PEKK) at 21x crowns. The JUV crowns (left) failed by crushing of the occlusal surface, with the remaining crown still intact. Surface cracking was observed on JUV irradiation. Dimple features characteristic of ductile fracture were evident for PEK crowns (right), which broke in half into two pieces as a result of occlusal surface pressure from the ball indenter of the CTC test. SEM = Scanning electron micrographs, JUV = Juvora, PEK = Pekkton, CTC = Crunch-the-Crown.

4. Discussion

The composition of the three polymeric materials was confirmed by the use of EDS, in which JUV and PEK had carbon and oxygen as the main elements (hydrogen cannot be captured by EDS). The TRI material presented fibers characterized in the SEM images, where the 12 to 14 μm diameters confirm that the fibers are glass (carbon fiber diameters are typically less than 10 μm), which was further confirmed by the burnout procedure. In the present study, the fibers were not totally oxidized, and there were residuals after burnout, which confirms that the fibers were not carbon. Furthermore, the TRI displayed tooth color, which further confirms that the fibers are glass, as carbon fibers produce dark specimens that are not suitable for dental applications.

In the present study, the flexural strengths obtained from the 3PB test for bar specimens created from three commercial dental polymers were investigated for their correlation with failure loads for a complicated crown shape. The goal of the study was to "bridge the gap" between a mechanical property determined from a controlled test and how the material performs as a crown. Flexural strength testing is a standardized approach with a defined size, shape, and loading conditions. CTC testing is intended to provide a reasonable indication of how a material will behave when it is fabricated into a crown, where the geometry is not simple and the stress state is complex. Furthermore, epoxy resin abutments were used instead of human molar teeth, because it would be difficult to standardize the dimensions of the molars. Our use of resin abutments simulates human hydrated dentin (18 GPa) [37], since epoxy presents a similar elastic modulus (4 GPa) [38]. The 3PB test was used as recommended by ISO 20795-1 for testing the flexural strength of polymers [36].

The specimens were not polished in the present study because it is difficult to standardize polishing of the irregular occlusal surfaces of the crowns with the same consistency as bar specimens with standardized flattened surfaces. Furthermore, the crowns were not exposed to artificial aging (thermocycling) to make the study more clinically relevant because it will have a different treatment from the bar specimens in which thermocycling is not recommended by the ISO recommendation for testing flexural strength; therefore, thermocycling was not done intentionally. We understand that the materials used in the present study are applied clinically as a core design and must be covered by composite resin for esthetic reasons [12]; however, the purpose of the present study is not to

 doi:10.20944/preprints202309.1985.v1

simulate the clinical oral environment since the CTC test does not result in clinically observed failures when the load is applied eccentrically but to see if the two different specimen geometries (bar and crown) with the exact surface treatments after machining from raw prefabricated discs will correlate to determine if the 3PB test will be predictive of the CTC test.

Presently, the measured failure loads for the crowns did not correlate with the flexural strengths found through 3PB of corresponding bar specimens; therefore, our hypothesis that the 3PB would be predictive of failure load of the different polymeric materials was rejected. These results were consistent with our previous findings for zirconia, where piston on 3-ball (biaxial flexion) and 4-point bending test results did not correlate with corresponding CTC test results [39]. In contrast, 4-point flexural strengths correlated positively with CTC failure loads in glass ceramic materials; however, there was no correlation of CTC failure load with biaxial flexural strength [40].

The three polymeric materials exceeded the range of human biting forces[41] which makes them useful for dental applications. However, the TRI material may be more useful for frameworks of monolithic crowns and fixed partial dentures in posterior regions because it showed higher failure load and flexural strength than JUV and PEK, which do not have such reinforcement with fibers. The fibers bear most of the externally applied load and contribute to most of the strength of composite materials as dictated by different fiber types, percentages, and orientations [7,8].

The failure loads for crowns composed of JUV and PEK were significantly different, and the mode of failure was different for each. The JUV specimens failed by plastic deformation with cracks visible by SEM. The crowns and bars were intact and not separated. PEK specimens failed by ductile-to-brittle fracture, whereby both the crowns and the bars were broken into two pieces. This result is due to JUV having much higher ductility (up to 150% strain to failure) compared to PEK (13% strain to failure).

The prepared TRI specimens had exposed matrix and fibers after machining from the raw TRI discs. After loading the TRI specimens (crowns and bars), cracks appeared, and they failed by presenting both plastic deformation and cracks. This likely resulted from cracking caused by matrix fracture, fiber fracture, and/or delamination between the glass fiber layers, which was beyond the scope of this study. Any surface cracks exposing underlying fibers would have deleterious effects in the oral environment and may contribute to hygroscopic expansion [37]. Presently, the TRI specimens did not fracture like the PEK specimens because of the fibers that held the specimen together.

In our study, the polymeric crowns were machined from solid discs with full contour design (0.8 mm) and cemented on resin abutments. They yielded fracture loads of 3023±418 N for PEK and 5217±169 N for JUV, which were higher than the recorded maximum biting forces [41]. Rohr et al. [22] reported that the fracture load of milled molar PEK crowns on zirconia implants (ceramic implant, 4.0 mm) was 2921±300 N. Shams et al. [21] reported that the fracture load for PEK crowns was 1831.37±240.69 N. The reduction in failure loads compared to our study was likely due to their veneering and thermocycling processes, as well as their endocrown designs. Elmougy et al. [37] reported that PEK crowns failed by fracture with slight deformation at the occlusal surface at loads of 2037±49 N, which is lower than the value obtained presently. This discrepancy is likely due to differences in crown thickness and the nature of the supporting resin material. Rodríguez et al. [10] reported that the fracture load for three-unit posterior fixed partial denture frameworks (0.7 mm occlusal thickness) with an intermediate pontic of PEEK material was 3132±307 N. Prechtel et al. [23] reported that the fracture load for milled indirect PEEK inlays is 2981 N.

Elmougy et al. [37] reported a biaxial flexural strength of 227 MPa for Pekkton bars, whereas in the present study, the flexural strength from 3PB was 192 MPa. This discrepancy is likely a result of the different loading conditions, as well as the fact that our specimens were not polished (we could not standardize the polishing procedures between the bars and crowns). Suzaki et al. [7] reported that the flexural strength conducted by 3PB was between 254.2±22.3 MPa for fiber-reinforced composite resin (TRINIA, SHOFU) compared with 468±97 N (TRI) in our study. The difference may be attributed to the fiber type and orientation. Shrivastava et al. [13] reported that the flexural strength of PEEK by 3PB was 183 MPa, which is close to 197 MPa in our study.

It is evident that further investigation is necessary to consolidate and establish the method as widely accepted in the scientific community for the investigation of dental materials. Additionally, considering the increasing use of PEEK in dentistry, the authors recommend evaluation through clinical trials as a more suitable approach for comparing the reliability of flexural strength. The results of this study suggest that dental practitioners should not rely on the values of flexural strength obtained from three-point bending tests, as advertised by the manufacturer, to predict the performance of polymeric crowns. Although the CTC does not duplicate in-vivo loading, it is likely a better representative than flexural tests. The present study is limited in that only 10 specimens were used for each material. Future research should consider these shortcomings and expand the investigation to include measures of fracture toughness.

5. Conclusions

Based on the findings of this in vitro study, the following conclusion was drawn:

1. The flexural strength of the bars did not correlate with the corresponding failure load of the crowns.
2. The TRI had a higher failure load and flexural strength than JUV and PEK, likely due to the presence of woven glass fiber (63 wt%) reinforced composite resin.
3. The JUV specimens failed by deformation only, while the TRI specimens failed by deformation and cracks. The PEK specimens failed by fracture.

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