

ANALYSIS OF THE BIAxIAL FLEXURE STRENGTH OF BILAYERED POLYETHERETHERKETONE (PEEK) USING VARIOUS VENEERING MATERIALS

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ABSTRACT

Purpose: is to evaluate the flexure strength of various veneering materials connected to PEEK.

Methods: 24 PEEK discs were made from PEEK cylinders. Based on the kind of veneering materials employed, they were categorized into three groups: Group 1: (C) is made up of PEEK discs veneered with nanohybrid composite (n=8), Group 2:(RC) of reinforced composite (BRILLIANT Crios) (n=8), and Group 3: (LD) of PEEK discs veneered with lithium disilicate ceramics IPS e.max CAD (Ivoclar Vivadent) (n=8). According to the manufacturer's instructions, the surfaces of the PEEK and reinforced composite specimens were sandblasted prior to priming, while LD was treated with hydrofluoric acid. Following the surface treatment process, various primers were applied to each treated specimen. Group C composite was applied to the treated PEEK specimens' surfaces as a veneer. A light-curing adhesive resin cement, was utilized to connect LD and RC. Following that, the samples were kept in water for 24 hours at 37 °F. Biaxial flexure strength (BFS) was measured using a universal testing device, and scanning electron microscope analysis was done to determine the failure modes. The information was gathered, processed, and statistically examined.

Results: Results showed that there was no significant difference between tested groups ($p=0.334$). The highest strength value was found with LD group (136.89 ± 27.5 MPa), while the lowest value was found with RC group (117.59 ± 30.35 MPa).

Conclusion: Considering the distinct protocols for resin cementation of the core and various veneering materials, all of the veneers under investigation are comparable in terms of mechanical properties.

KEYWORDS: Hybridization, polymer, Resin, Glass Ceramic, Milled

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INTRODUCTION

Around the world, practitioners' interest in creating restorations with CAD/CAM technologies is increasing. Additionally, the demand for nonmetallic restorations by patients and dentists has prompted researchers to look for alternate materials and tweak the existing materials to improve their aesthetic and functional qualities. In an effort to improve the mechanical qualities of resin-based materials, the polymer chains of those materials were modified. As a result, high-performance polymers began to attract attention in the dental industry due to their superior mechanical capabilities compared to other polymers. ^(1,2)

PEEK has a wide range of qualities, including high biocompatibility, good mechanical properties, high-temperature resistance, chemical stability, polishability, good wear resistance and low plaque affinity. In comparison to stiff framework materials like zirconium oxide and metal alloys, PEEK is as elastic as bone and has a low modulus of elasticity of 4 GPa, providing a cushioning effect and dampens loads exerted on the abutment teeth. ⁽³⁻⁶⁾

PEEK lacks good aesthetics and is opaque. The combination of a strong core and an aesthetically pleasing veneer has been described in literature as the cornerstone of prosthetic dentistry for core veneered restorations. For many years, veneer has been effective. A core substance is needed to reinforce the integrity and durability of the restoration because veneering materials only work to reconstruct the restoration's surface layer. ^(7,8) When preparing a PEEK prosthesis, it is therefore essential to use it in conjunction with extra veneering. High aesthetic standards are often met by manually veneering with resin composites onto machine-milled or heat-pressed PEEK material, or by veneering it with prefabricated CAD/CAM ceramic, composite veneers or ceramic crowns. ⁽⁹⁾

With CAD/CAM composite veneering,

there is less chance of discoloration, consistent polymerization, and wear resistance than with manual veneering. ^(10,11) As there is a chance of discoloration with resin composites veneer, a design consisting of a single monolithic lithium disilicate veneer cemented on top of the PEEK frameworks was introduced ⁽¹²⁻¹⁴⁾. This design eliminates such veneering problems and improves the mechanical, biological, and aesthetic performance of implant-supported fixed prostheses. Since the individually cemented lithium disilicate ceramic veneer is made up of about 70% lithium disilicate crystals ($\text{Li}_2\text{Si}_2\text{O}_5$) embedded in a glassy matrix, it not only eliminates the veneering step and problems associated with it but also improves aesthetics. ^(15,13)

The fracture load of PEEK veneered dental prosthesis using various materials and techniques was assessed by Taufall et al. Digital veneers had the highest fracture load values, showing that this method is more dependable than traditional ones. ⁽¹⁰⁾ Adhesive failures have been attributed to inadequate bonding between PEEK and the veneering shell when the PEEK substrate was not etched or given any surface treatment. ⁽¹²⁾ In order to achieve a promising and durable bonding on PEEK surfaces, it was discovered and clinically advised that a resin varnish containing a methacrylate group must be applied after air abrasion ⁽¹⁶⁻¹⁸⁾.

Since aesthetic considerations continue to be a significant clinical reality and benchmark, veneered PEEK with various esthetic materials needs to be assessed. Many studies have looked into how various PEEK and veneering material combinations affect the mechanical performance of such bi-layered restorations ^(14,19), yet there is still a dearth of information. This study's objective is to assess the biaxial flexure strength of three veneering materials bonded to PEEK. According to the null hypothesis, there would be no difference in the flexure strength of different examined veneering materials.

MATERIALS AND METHODS

This research was accepted by the members of the Research Ethics Committee, Faculty of Dentistry, Ain Shams University, Cairo, Egypt (FDASU-REC) with approval number FDASU-Rec PC072336 with exemption.

A power analysis was created to have sufficient power to apply a statistical test of the null hypothesis, which states that there is no difference in flexural strength between the tested groups. It was calculated that 24 samples (8 samples each group) would be the minimum required sample size (n). The sample size was determined using G*Power version 3.1.9.7⁽²⁰⁾ and the statistical analysis of the findings from earlier studies.⁽¹⁹⁾

Samples preparation

Four cylinders of PEEK (Cupra, Whitepeaks Dental Solutions, Germany), one cylinder of reinforced composite (BRILLIANT Crios, Coltène, Germany), and two cylinders of partially crystallized low translucency lithium disilicate ceramic IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein) were produced with final dimensions of 14x14 mm. The CAD software 3D builder (3D Builder, Microsoft, WA, USA) was used to develop the cylinders for PEEK and reinforced composite. Then the STL file was imported with the Dental CAM software, the design was nested in the blank, and the milling machine (VHF camfacture, Ammerbuch, Germany) was given instructions to mill the cylinders. The cylinders were still attached to the primary blank when the process of milling was completed; a diamond disc was used to separate the cylinders from the main blank. For the preparation of lithium disilicate (Ivoclar, Vivadent) veneered specimens, partially crystallized IPS e-max CAD blocks were first shaped into a cylindrical form using a lathe machine to produce a 14mm-diameter cylinder. A digital caliper was used to confirm the diameter of each cylinder.

Forty disc-shaped specimens with a 0.8mm thickness were produced after the designed cylinders were cut; there were 24 PEEK, 8 lithium disilicate, and 8 reinforced composite discs. The discs were cut using a low speed diamond blade on a slicing machine (Isomet 4000 precision cut, Buehler, USA) under water coolant. After sectioning, the samples were visually inspected for any surface flaws. An electronic caliper was used to measure the thickness of each specimen. Lithium disilicate samples were crystallized in a ceramic furnace (Ivoclar Programmat P310) and the firing cycle was adjusted according to the manufacturer's guidelines. On the basis of the kind of veneering material used, 24 PEEK discs were then randomly divided into three groups (n=8). Group 1: (C) PEEK discs veneered with nanohybrid composite (n=8), Group 2:(RC) PEEK discs veneered with reinforced composite (n=8), and Group 3: (LD) PEEK discs veneered with lithium disilicate ceramics IPS e.max CAD (n=8).

Conditioning of bonded surfaces:

According to the manufacturer's instructions, PEEK and reinforced composite discs were air abraded on one side using 110 microns and 50 microns aluminum oxide particles (Renfert, Hilzingen, Germany), respectively, at 2.8 bar for 15 seconds at a fixed distance of 10 mm by the same operator. Any surface residue was then removed by washing the discs in an ultrasonic bath of distilled water for three minutes. In order to remove any last traces of moisture from the specimens' surfaces, they were then air-dried for 15 seconds. Sandblasted surface of PEEK specimens was conditioned with a single layer of Z-Prime Plus (Bisco, Schaumburg, Illinois, U.S.A), a methacrylate-based universal primer that was rubbed on the air-abraded surfaces of the 24 specimens before bonding using a brush for 10 seconds. The specimen's surface was then air dried for 5s to thin the primer layer. For reinforced composite discs one coat of single bond universal was applied, air dried with oil-free air for 10 s, to

generate a good bond to the resin matrix. Then it was light cured for 20 seconds using a light-emitting diode curing unit (Radii plus, SDI limited dental, Australia), with an output of 3000 mW/cm² before cementation.

Lithium disilicate discs were etched using hydrofluoric acid (BISCO Inc. Schaumburg, U.S.A) as recommended by the manufacturer, ultrasonically cleaned in an ultrasonic bath filled with deionized water for 3 minutes, then air dried with oil-free air, and salinized with one coat of Bisco Porcelain Primer (BISCO, USA) for 60 s.

Veneering of PEEK discs:

The PEEK disc was placed, with its treated surface facing upward in a specially designed round-shaped split teflon mold with dimensions of 14 mm in diameter and 1.65mm in thickness, designed to accommodate the total specimen thickness of 1.6 mm and leave an additional 0.05mm for cement thickness, in order to obtain a bi-layered specimen for groups (LD) and (RC). The treated PEEK surface was covered with a layer of light-cured resin cement (Choice2, BISCO Inc. Schaumburg, U.S.A), and discs of the veneering materials were placed on the corresponding PEEK substrate with their treated surfaces facing the cement surface. The mold with the bi-layered specimen inside it was placed between two glass plates, and a loading device with a weight of two kilograms was applied in order to produce a uniform thickness of the luting cement. After weight removal, further polymerization was carried out for 180 seconds.

The direct veneering composite (group C) was made using a circular split teflon mold. The mold has an interior dimension of (14mm x 1.65mm). The constructed Teflon mold was set on a glass slab with the PEEK disc inside with its treated surface facing up, then the Nanohybrid Composite (Tetric N-Ceram, Ivoclar Vivadent, Liechtenstein) was packed inside. To create an even layer after

packing, a second glass slab was placed with finger pressure onto the top of the composite. Then, it was exposed to light for 360 seconds using a light-curing device with a 3000 mW/cm² output (Radii plus, SDI limited dental, Australia). The final thickness of the bi-layered discs was verified using an electronic digital caliper. Specimens were stored after complete polymerization in distilled water for 24 hrs before biaxial flexure strength testing using the universal testing machine.

Biaxial flexural strength test:

Using a piston-on-three-ball approach in accordance with ISO 6872, the biaxial flexural test was selected for the experiment. Data were collected using computer software (Instron® Bluehill Lite Software), and testing was carried out at a cross-head speed of 1 mm/min using a computer controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA). At room temperature and relative humidity of 60% ±5%, the test was carried out. The disk-shaped specimens were centered on top of three 2.1 mm diameter steel spheres. Then, using a universal testing machine, a steel piston with a flat-end tip of 2.58 mm in diameter applied a load perpendicular to the center of the specimens until breakage occurred. The tension side of the PEEK disc was its polished surface, whereas the loaded side was its veneered surface. To ensure that the load was distributed evenly, a thin sheet of tin was placed between each sample and the tip of the load applicator. The biaxial flexure strength was calculated according to the following equation :

$$S = -0.2387P (X - Y)/d^2,$$

where S is the biaxial flexural strength at fracture (in MPa), P is the load at fracture (in N), and d is the specimen thickness at fracture origin (in mm).

The X { $X = (1 + \nu) \ln(B/C)^2 + [(1 - \nu)/2](B/C)^2$ } and Y { $Y = (1 + \nu) [1 + \ln(A/C)^2] + (1 - \nu) (A/C)^2$ }, where ν is the Poisson's ratio, A is the support sphere

radius (mm), B is the radius of the tip of the piston (mm), and C is the specimen radius (mm). The load-deflection curves were recorded with computer software (Instron® Bluehill Lite Software).

Failure mode analysis

After loading to fracture, visual and Digital photography was used for the fracture investigation (Taiwan: Canon EOS 800D Digital SLR). Two observers evaluated the specimens' modes of failure and classified them into the following groups based on their patterns of failure: a) adhesive (failure with veneer detachment and no composite cement remnants left on the PEEK surface), b) cohesive failure in PEEK, c) cohesive failure in veneering material and d) mixed cohesive/adhesive failure. One representative sample from each subgroup was examined using an environmental scanning electron microscope (Inspect S, FEI business, USA) to examine the veneer-PEEK interface after a fracture. Images at 500 X magnification were captured and interpreted.

RESULTS

Biaxial flexure strength test:

The mean and standard deviation (SD) values were used to represent numerical data. To check for normality, the Shapiro-Wilk test was applied. Using Levene's test, the homogeneity of variances was examined. The homogeneity condition was not broken, and the data were normally distributed. One-way ANOVA was used to analyse intergroup comparisons, and then Tukey's post hoc analysis was performed. For all tests, the significance level was set at $p < 0.05$. R statistical analysis software for Windows3, version 4.3.1, was used to conduct the statistical analysis⁽²¹⁾.

According to the findings, there was no significant difference between the tested groups ($p = 0.334$). The LD group had the greatest strength value (136.89 ± 27.51 MPa), followed by the C group (133.13 ± 22.25 MPa), and the RC group

(117.59 ± 30.35 MPa). In figure (1), the overall statistics and the findings of intergroup comparisons of various veneering materials are shown.

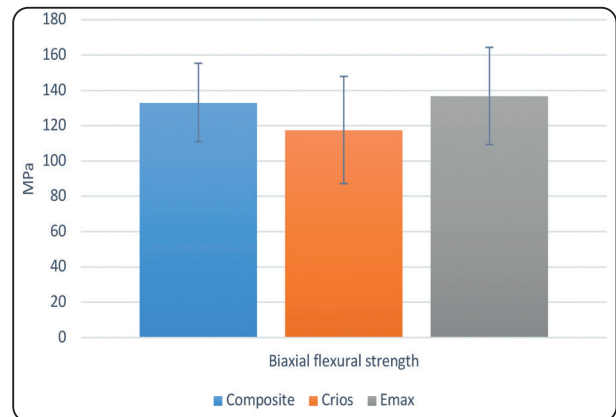


Fig. (1): Bar chart showing mean and standard deviation values (error bars) of biaxial flexural strength (MPa)

Mode of failure:

For the three groups, All the PEEK substrates were intact and none of them underwent any type of failure. Failure types "veneer cracking" and "mixed cohesive/adhesive failure" were both observed. Veneer cohesive and mixed cohesive/adhesive failure rates of 40% and 60%, respectively, were seen for groups C and RC, whereas 60% and 40%, respectively, were seen for group LD. Different modes of failure are shown in Fig (2).

The scanning electron microscope photos of Group C showed that fractured composite had a smoother fracture line in comparison to two other materials and was debonded from the micro irregularities in the PEEK bonding surface creating a gap as represented in Fig (3A). For RC a surface crack was noticed that extended downwards to the PEEK core in some areas with a sharp fracture line while in other areas no cracks were observed, and the reinforced composite was fused with the PEEK demonstrating adequate bonding (Fig 3B). Group LD revealed an "integration" at the interface with PEEK as no gaps or cracks were noticed after fracture (fig.3C).

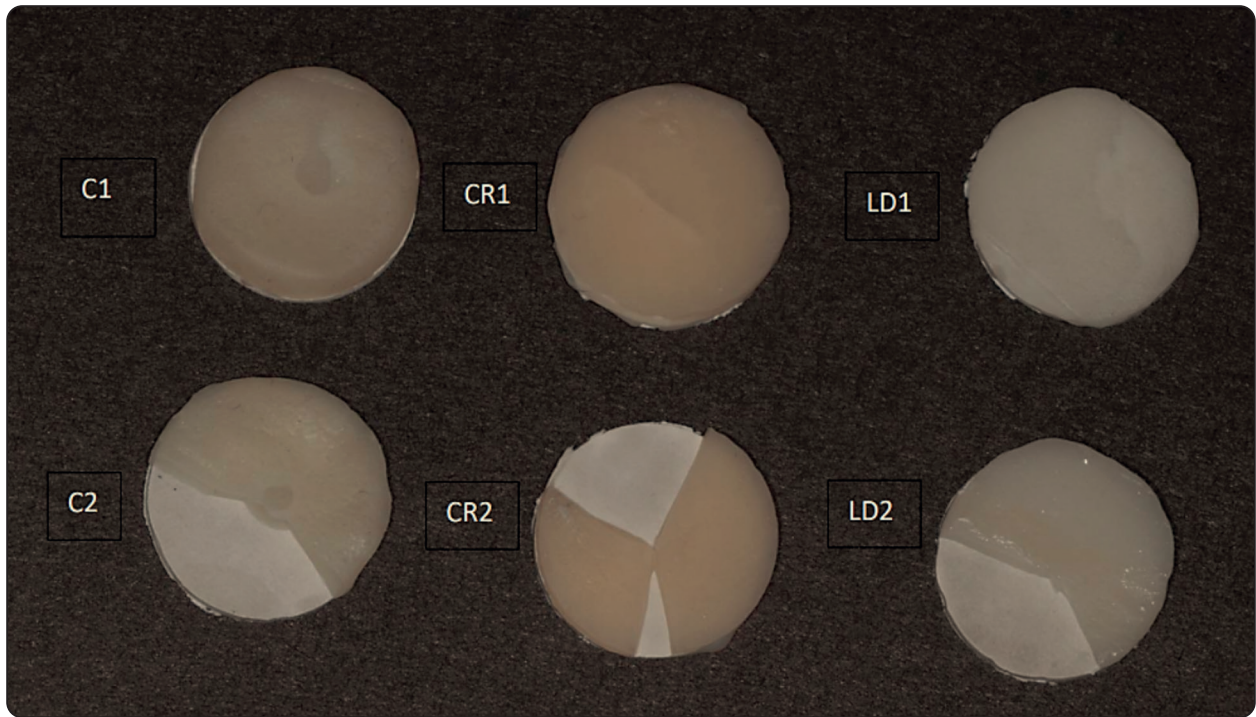


Fig (2): Digital photo represent different failure modes: C1, CR1 and LD1 for Cohesive failures in veneering Composite, Reinforced Composite and Lithium disilicate respectively. C2, CR2 and LD2 for mixed cohesive/adhesive failure in PEEK veneered with Composite, Reinforced and Lithium disilicate respectively.

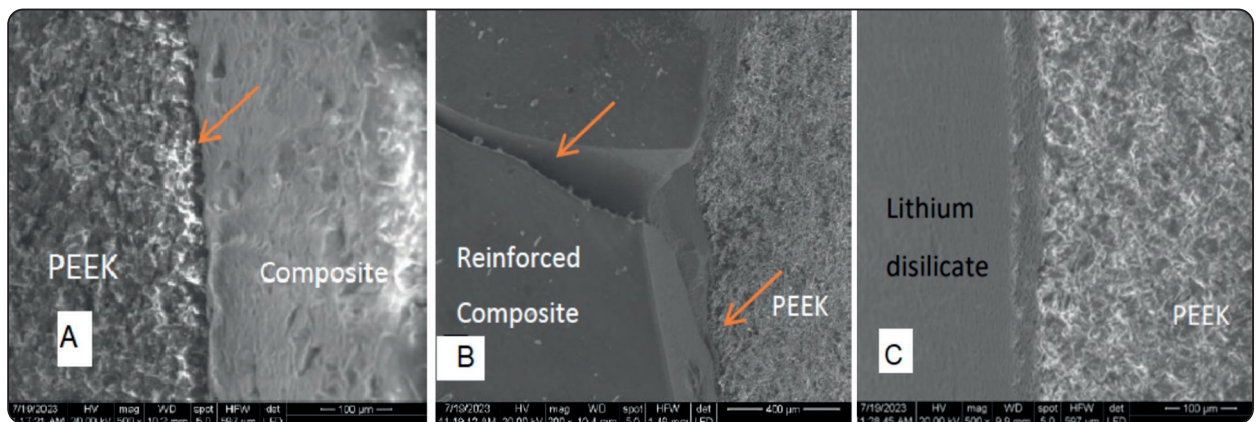


Fig (3): SEM photos of PEEK and different veneers interfaces after flexural strength test.

DISCUSSION

For an extended period now, composite veneered PEEK has been used clinically as a rigid material for implant abutments and FPD frames. (2, 22) The primary drawback is that it ages and becomes discolored. (11) With the development of new computer-aided design (CAD)/computer-aided

manufacturing systems and novel surface treatment protocols (16-18), veneering with lithium disilicate and milled reinforced composite on PEEK appears to be a practical treatment option.

Compared to resin composites, entirely ceramic materials, like IPS Empress CAD, exhibit a far better degree of resistant to wear , translucency and

long-term color stability.⁽²³⁾ In contrast to materials with a higher E-modulus like lithium disilicate, composites offer better shock absorption of the masticatory pressure due to their lower modulus of elasticity when compared to ceramics. In particular, for crowns on implant abutments, CAD/CAM resin composites are gaining popularity since they demonstrate great fatigue resistance, speed up “chewing comfort” for the patient, and have no catastrophic breakdowns.^(24,25)

Systematic reviews show that posterior teeth restored with resin composite are subject to destructive stresses, such as masticating hard food and unobservant attrition and bruxism, which cause their wear.⁽²⁶⁾ Although lithium disilicate veneered PEEK has been used clinically for some time, there isn't much information available about it. Therefore, it was the goal of this study to evaluate the biaxial flexure strength and compare the failure modes of PEEK discs veneered with direct composite with those veneered using milled lithium disilicate and milled reinforced composite.

According to the manufacturer, a single layer of Z-Prime Plus, specially formulated with MDP (Bisco, Schaumburg, Illinois, U.S.A.), was applied to the sandblasted surface of PEEK specimens using Al₂O₃ 110 um at 2 bar to generate an acceptable adhesive potential of the veneering material to PEEK. Since it has been demonstrated that combining light-cured adhesives with dual-cured resin cements results in noticeably weaker bonding because of the incompatibility between the peroxide amine catalyst in the dual-cured resin and the acidic resin monomer in the adhesive, therefore, PEEK samples were bonded to the veneering Lithium disilicate and milled composite of 0.8mm thicknesses using light cure resin cement (Bisco Choice 2) in this study.⁽²⁷⁾

In comparison to manually layered traditional composite resin groups, we found that groups of CAD-milled reinforced composite groups had mean biaxial flexural strength values that were comparable. A milled one could eliminate some

of the intricate manual stages involved in the traditional layering of composite. The only manual process involved adhering the veneer to the substrate with the adhesive. It is a promising substitute for traditional veneering since pre-manufactured digital veneering has a better level of curing than traditional hand veneering using composite resin.⁽¹⁰⁾ This result appears to be in agreement with the earlier research.⁽¹⁴⁾ It may be assumed that the use of light cure resin cement was a factor in the study's increased bond strength between milled composite treated with light cure adhesive before bonding.⁽²⁷⁾ A solid bond was created between the light-cured resin cement and the light-cured adhesive applied to the treated surface of reinforced composite disc.

According to Tartuk et al.,⁽²⁸⁾ the composite veneered PEEK experienced adhesive failures, which close to our results that showed 60% mixed cohesive/adhesive failure for C and CR groups. (figure 2) Our SEM analysis, which showed a demarcation line suggesting areas of debonding at the interface between PEEK and composites, could also support such a contention. (figure 3A)

By acid etching lithium disilicate-reinforced glass ceramic surfaces with (5–10%) HF acid, a typical rough surface was created that increased the retentive area to silane agents and resin-matrix cements.⁽²⁹⁾ The most crucial element in improving adhesion between two different materials appears to be the micromechanical locking produced by resin penetration in the pits and grooves. Consequently, it has been revealed that surface topography affects load-bearing capacity.⁽³⁰⁾ This could account for the LD group's higher BFS than the C and RC groups. The highest BFS of lithium disilicate in addition to the lower fractural strength of composite may also be attributed to this. This was corroborated by the SEM, which showed sufficient resin penetration into the lithium disilicate-treated surface as compared to bonded composite surfaces. (figure 3C)

Since there was no statistically significant difference between direct composite and veneers

created with milled lithium disilicate and milled reinforced composite ($P > 0.05$), the null hypothesis was not rejected in the current investigation. This outcome is in line with that of Gupta et al., who found no statistically significant difference in the load-bearing capacity between composite veneered PEEK and lithium disilicate veneered PEEK.⁽¹²⁾ Our results are seen to be against the previously mentioned work by Gouda et al.⁽¹⁴⁾ where results revealed that groups manually layered with conventional composite resin showed the highest mean biaxial flexural strength values while CAD milled lithium disilicate veneered groups showed the lowest mean biaxial flexural strength. Regarding mechanical perspective, all tested veneering are accepted taking into account the resin cementation process for both the core and veneer material.

CONCLUSIONS

- Within the confines of this in vitro study, it could be speculated that digitally veneered PEEK frameworks made of either CAD reinforced composite or lithium disilicate might be successful and comparable to traditional composite veneering PEEK.
- Because of occlusal forces, pH changes, and other unfavourable elements, it is believed that the actual oral environment is harsher than that which is reproduced by the current in vitro circumstances. Therefore, additional clinical research is necessary.

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