

INVESTIGATING THE EFFECT OF THE FRAMEWORK MATERIAL, VENEERING TECHNIQUE AND AGING ON FLEXURAL STRENGTH OF CORE/VENEERED RESTORATIONS

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ABSTRACT

Objective: To evaluate the flexural strength of different combinations of modified PEEK and 3-YTZP veneered restorations as well as the effect of aging on those combinations.

Materials and Methods: Two core materials; partially sintered tetragonal monolithic zirconia and modified PEEK, and three veneering materials; lithium disilicate glass-ceramic, packable composite, and veneering porcelain were used. A total of sixty specimens were randomly divided into 3 groups (n=20). The specimens were constructed in the form of bilayered core/veneer disks of diameter 12 mm and total thickness 1.5 mm. Half of the specimens of each group were tested for biaxial flexural strength using the piston-on-three-balls test. The other half of the specimens were subjected to artificial aging and then tested for biaxial flexural strength.

Results: Two-way ANOVA showed there was a significant effect for the material of the bilayered restoration, aging, and the interaction between them on biaxial flexural strength. Unaged veneered Zirconia and Crea.lign veneered PEEK had significantly higher values (398.85 ± 29.58) and (391.31 ± 15.59) respectively than IPS e.maxCAD veneered PEEK (222.13 ± 18.77). For the aged subgroups, veneered Zirconia (303.51 ± 35.64) and Crea.lign veneered PEEK (277.10 ± 13.37) also showed significantly higher values than IPS e.maxCAD veneered PEEK (198.10 ± 6.57). For IPS e.max CAD veneered PEEK, there was no significant difference between unaged and aged sub-groups ($p=0.100$). However, for other materials, aging caused significant decrease in biaxial flexural strength values ($p<0.001$).

Conclusions: Both Crea.lign veneered PEEK and porcelain veneered 3-YTZP could be used with the same efficiency, regarding flexural strength, as bi-layered restorations. Aging has a negative effect on both materials' combinations

KEYWORDS Aging, BioHPP, Flexural strength, Bilayered restorations, PEEK

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INTRODUCTION

For the past couple of decades and with the progressive development of dental materials and advancement of CAD/CAM technology, prosthetic treatment for the replacement of multiple missing teeth has greatly improved⁽¹⁾. However, the proper choice of the prosthetic material depends not only on its ability to withstand the masticatory forces, but on its esthetic outcome as well⁽²⁾. Since the prosthetic material influences the transmission mechanism of stresses generated during function and transferred to the underlying supporting structures, the elastic modulus of the material is regarded as a key player affecting the success of the restoration. In literature, it has been documented that materials having more compatible elastic modulus tend to bend under load and distribute stresses more evenly^(1,3-5).

For many years, metal-ceramic restorations have been deemed as the gold standard fixed dental prosthesis owing to their good elastic moduli, fracture strength, and high porcelain-to-metal bond strength⁽⁶⁾. However, the esthetic complications of such restorations rooting from their metal grayish color, the possibility of corrosion and degradation as well as the risk of metal allergy increased the demand for metal-free dental prostheses and led to the development of esthetically biocompatible metal-free materials⁽⁷⁾.

Yttria-stabilized tetragonal zirconia (3Y-TZP) was developed and became introduced in the market as an esthetic alternative for metal-ceramic restorations. It represents a versatile material with its excellent mechanical properties, biocompatibility, and relatively good esthetic properties compared to metal. With the improvements in nano-technology, came the evolution of nano-structured polycrystalline zirconia in an attempt to improve its esthetic value. It's characterized by its high flexural strength (900-1100 MPa) and fracture toughness (3.5-4.5 MPa-m^{1/2}), yet very high modulus of elasticity (200-210 GPa), and less than optimum

optical properties for use in the esthetic zone. A drawback that made veneering essential in highly esthetic areas^(8,9).

Nevertheless, the inertness and surface stability of Y-TZP, caused a challenge in trying to establish a durable chemical or mechanical bond between zirconia and veneering porcelain, and chipping of the veneering ceramic was reported to be the main drawback of these restorations^(3,10,11).

Recently, Polyetheretherketone (PEEK) which is a high-performance polymer, grabbed the attention of dental researchers because of its low plaque affinity, light weight, resistance to water absorption, high biocompatibility and, most interestingly, low modulus of elasticity (4 GPa). All of which made PEEK a favorable dental material. It has thus been introduced as an alternative to metal alloys and zirconia for prosthetic frameworks⁽¹²⁾.

Continuous attempts to improve the mechanical and physical properties of pure PEEK, led to the development of BioHPP. It contains ceramic micro-particles incorporating 20% nano-ceramic fillers such as aluminum oxide and zirconium oxide. It is claimed to be more resistant than PEEK, and can be used with implants due to the consistent homogeneity of the structure and modulus of elasticity (4.6 GPa) that is close as possible to the bone (4GPa), thus the chewing pressure is transmitted as gently as possible, and the risk of failure is reduced⁽¹³⁾.

However, both 3Y-TZP and BioHPP are considered opaque and display less than favorable optical properties. Thus, veneering is always indispensable specifically when esthetics is of prime importance^(10,14). Different veneering techniques based on layering, pressing or Cad-on technique have been reported and documented for 3Y-TZP frameworks, yet results have been contradicting^(15,16). On the other hand, BioHPP frameworks can be veneered with composite resin, polymethyl methacrylate resin (PMMA), high-impact PMMA veneers, or bonded to lithium disilicate. Multiple

studies have previously shown that PEEK veneering with light-cured composites made the chipping clinically repairable and helped prevent the wear of opposing teeth. However, the surface inertness of PEEK still represents a challenge when bonding with a veneering material is considered. Bearing in mind the aforementioned facts together with the scarce data about the long-term clinical performance and the limited studies available on PEEK reliability to replace 3-YTZP in core-veneered restorations, a question is raised about which veneering method provides the best mechanical performance and effective load bearing capacity ^(12,14,17-21).

One additional fact not to be overlooked when evaluating the mechanical performance of such bi-layered restorations is their continuous exposure in the oral environment to various damaging stimuli including humidity, temperature changes and mastication. In-vitro simulation of such conditions could be helpful when evaluating the mechanical behavior and longevity of different restorations. Thermocycling is considered one of the most widely employed methods to simulate the effect of clinical oral conditions on different restorative materials. Currently, there is limited data concerning the effect of aging on the strength properties of bi-layered PEEK restorations ^(20,22-24).

Likewise, it is well documented that 3Y-TZP-based restorations are subjected to low-temperature degradation (LTD) when exposed to humidity at low temperatures. LTD occurs in temperatures as low as room temperature and up to 400°C leading to a spontaneous transformation of zirconia from its metastable tetragonal phase into the monoclinic one without the application of external loads. Artificial accelerated aging is a laboratory method claimed to simulate the effects of intraoral conditions by initiating a cascade of material changes within biomedical zirconia similar to that caused by LTD. Different studies have suggested that steam sterilization of zirconia in an autoclave at 134°C and

2 bar steam pressure for 5 hours simulates 15-20 years at 37°C ^(25,26).

In the light of aforementioned data, that study aimed to evaluate the flexural strength of different combinations of BioHPP and 3-YTZP veneered composites as well as the effect of aging on those combinations. The null hypothesis is that neither the material combination nor aging would affect flexural strength.

MATERIALS AND METHODS

Two core materials; partially sintered tetragonal monolithic zirconia (3 mol% Y-TZP) (BruxZir-FullStrength Solid, Glidewell Laboratories, USA) and modified PEEK: BreCAM BioHPP (bre.CAM. BioHPP; bredent GmbH & Co KG), and three veneering materials; lithium disilicate glass-ceramic (IPS e.max CAD Ivoclar Vivadent AG), light-cured direct veneering composite resin (Crea.lign Bredent GmbH & Co KG) and veneering porcelain: VI-TAVM 9 ENL (VITA, Zahnfabrik H. Rauter GmbH & Co. KG) were used in this study as seen in table (1). A total of sixty core/veneered specimens (12mm X 1.5 mm) divided randomly into 3 groups (n=20) were employed in the study. A sample size calculation using a significance level of a=.05 and a power of b=.80 estimated a sample size of 20 using pilot data. Similar previous studies have used a similar sample size ^(27,28).

TABLE (1): Sample Grouping

Material	Unaged	Aged	Total
Porcelain veneered Zirconia (Zr) (n=20)	n=10	n=10	n=20
IPS e.max veneered PEEK (EM) (n=20)	n=10	n=10	n=20
Crea.lign veneered PEEK (CL) (n=20)	n=10	n=10	n=20
Grand Total	n=60		

All specimens were designed to have a core thickness (0.7 mm) and a veneer thickness (0.8 mm). First, a standard tessellation language (STL) file of a Ø15×1mm disk was designed for the zirconia core specimens to allow for the 20% shrinkage during sintering, another one of a Ø12×0.7 mm disc for the PEEK and finally a third one of Ø12×0.8 mm disc for IPS e.max CAD were designed with a software program (Exocad GmbH, Germany). The files were then sent to Arum version 5x-400 milling machine (Daejaeon, Republic of Korea) for milling of the discs. The final thickness was checked with a digital caliper.

Zirconia discs were then sintered in a high-temperature zirconia furnace (in fire HTC speed, Sirona Dental Systems) according to the manufacturer's specifications and IPS e.max CAD discs were crystallized in a special ceramic furnace; Programat P300/G2 (Ivoclar Vivadent, Schaan, Liechtenstein) as recommended by the manufacturer.

All zirconia and modified PEEK discs were then sandblasted on one surface, which is the one planned for veneering, with 110 µm alumina powder at a distance of 10 mm and 3.5 psi using a special holder for standardization. All discs were ultrasonically cleaned in an ultrasonic water bath (L&R Transistor Ultrasonic T14) and left to dry.

For group (Zr) veneering, VM9 Enamel Light (ENL) powder and liquid were mixed according to the manufacturer's instructions and veneered to the zirconia cores with layering technique using a split Teflon mold (12 mm diameter and 1.5 mm height). Veneering was carried out in two firing cycles in a porcelain furnace (Programat P300/G2) to compensate for firing shrinkage. Specimens were then finished and polished (Microdont, São Paulo, Brazil). After polishing, samples were glazed with VITA AKZENT glaze and fired according to the manufacturer's recommendations.

For group (EM), all IPS.e.max CAD discs were acid etched according to the manufacturer's guidelines for 20 seconds with 5% hydrofluoric acid (BISCO, USA) before a layer of silane coupling agent (BISCO, USA) was applied. The BioHPP core and the veneering IPS e.max discs were assembled and cemented together in a custom-made metal mold using dual-cured resin cement (Dua-Link, BISCO, USA). The assembled discs, within the mold, were pressed between two glass plates under a five-kilogram load to assure having a uniform thickness of the luting cement. To ensure complete cement polymerization, the load was removed and polymerization was continued for 3 minutes with bre.Lux Power Unit (Bredent GmbH, Germany). Finally, each specimen was polished (OptraFine, Ivoclar Vivadent, Schaan, Liechtenstein) following the manufacturer's recommendations.

For (CL) group, a Co-Cr mold was used to control the veneering layer thickness, where each BioHPP disc was secured in the mold and a thin uniform layer of Crea.lign opaquer (Bredent GmbH, Germany) was applied to the BioHPP sandblasted surface and light polymerized (bre.Lux Power Unit, Bredent GmbH, Germany). Afterward, the mold was filled with Crea.lign dentin paste (Bredent GmbH, Germany), pressed between two glass plates to obtain a smooth uniform veneering layer, and light cured with bre.Lux Power Unit. All specimens were finally polished (Visio.lign Toolkit, Bredent GmbH, Germany) according to the manufacturer's instructions.

Before verifying the final thickness of all the veneered specimens, an air/water spray was used to remove any polishing residues.

Afterward, half of the specimens of each group were tested for biaxial flexural strength using the piston-on-three-balls test. Specimens were placed concentrically on the hardened stainless steel supporting balls to ensure the load was centralized.

With a universal testing machine (Instron Co., Canton, Mass.), load was applied using a punch of tip diameter 2 mm at the center of the specimen at cross-head speed 1 mm/min. The core surface

was placed on the tensile side for all specimens. The load at the point of fracture was recorded and biaxial flexural strength was calculated through the following equation;

$$\sigma = \frac{6M}{t_a^2 K_{2p}} \left[\frac{E_b t_b (1 - \nu_a^2)}{E_b t_b (1 - \nu_a^2)} + \frac{t_a (1 - \nu_a^2) \left(1 + \frac{t_b}{t_a}\right) \left(1 + \frac{E_a t_a}{E_b t_b}\right)}{t_b \left(1 + \frac{E_a t_a}{E_b t_b}\right)^2 - \left(\nu_a \frac{\nu_b E_a t_a}{E_b t_b}\right)^2} \right]$$

$$K_{2p} = 1 + \frac{E_b t_b^3 (1 - \nu_a^2)}{E_a t_a^3 (1 - \nu_b^2)} + \frac{3(1 - \nu_a^2) \left(1 + \frac{t_b}{t_a}\right)^2 \left(1 + \frac{E_a t_a}{E_b t_b}\right)}{\left(1 + \frac{E_a t_a}{E_b t_b}\right)^2 - \left(\nu_a + \frac{\nu_b E_a t_a}{E_b t_b}\right)^2}$$

Where; (σ): Bi-axial flexural stress, (t_a) and (t_b): Thicknesses of the two material layers where (a) is the material on top and (b) is the material at the bottom, (E_a) and (E_b): Young's modulus of the core and veneering layers, respectively, (ν): Poisson's ratio that is calculated as the mean value between Poisson's ratios of the core and the veneering material, (M): Maximum bending moment calculated from the equation;

$$M = \frac{W}{4\pi} [(1 + \nu) \log \frac{A}{R} + 1]$$

Where; (W): Load, (R): Equivalent radius of loading, and (A): Radius of the circle of the support points (5 mm), and;

$$R = \sqrt{1.6B^2 + d^2 - 0.675d}$$

Where; (B): Radius of the piston tip (1 mm), and (d): Thickness of the specimens (1.5 mm).

The other half of the specimens were subjected to artificial aging. Group (Zr) specimens were subjected to accelerated aging in a steam autoclave (Sturdy SA-260MA- Class B) (Sturdy Industrial Co.LTD, New Taipei City, Taiwan) at 134°C with 2 bars pressure for 1 hour which was clinically representative of approximately 3-4 years of intraoral service. On the other hand, group (EM) and (CL) specimens were placed in a thermal cycling furnace (julabo.THE-1100) for artificial aging. The temperature range for the water baths was set between 5–55°C, with an immersion time of 30 seconds. 30,000 cycles simulating three years of intra-oral service were employed. Following artificial aging of all groups' specimens, biaxial flexural strength was tested in the same previous way.

Data was collected and tabulated. Numerical data were presented as mean and standard deviation (SD) values. Statistical analysis was performed with R statistical analysis software version 4.1.3 for Windows

RESULTS

Shapiro-Wilk's test was used to test for normality and showed no violation of normality of data distribution. The homogeneity of variances was tested using Levene's test.

Results of two-way ANOVA presented in table (2) showed there was a significant effect for the material of the bi-layered restoration, aging, and the interaction between them on biaxial flexural strength ($p < 0.001$).

Comparison of simple main effects presented in table (3) showed that for both unaged and aged sub-groups, there was a significant difference between different materials. Unaged (Zr) and (CL) groups had significantly higher values (398.85 ± 29.58) and (391.31 ± 15.59) respectively than (EM) group (222.13 ± 18.77). For the aged subgroups, (Zr) and (CL) also showed significantly higher values

(303.51 ± 35.64) and (277.10 ± 13.37) respectively than the (EM) group that showed flexural strength value (198.10 ± 6.57).

For (EM) group, there was no significant difference between the unaged and aged sub-groups ($p=0.100$). However, for the other groups, aging caused a significant decrease in biaxial flexural strength values ($p<0.001$).

TABLE (2): Two-way ANOVA test results

Parameter	Sum of squares	df	Mean square	f-value	p-value
Material	118621.01	2	59310.50	120.11	<0.001*
Aging	45469.90	1	45469.90	92.08	<0.001*
Material*Aging	11311.33	2	5655.67	11.45	<0.001*
Error	11851.11	24	493.80		

*significant ($p<0.05$)

TABLE (3): Comparisons of Simple Main Effects for Biaxial Flexural Strength (MPa)

Aging	Biaxial flexural strength (MPa) (Mean \pm SD)			p-value
	Porcelain veneered Zirconia (Zr)	IPS e.max veneered PEEK (EM)	Crea.lign veneered PEEK (CL)	
Unaged	398.85 ± 29.58^A	222.13 ± 18.77^B	391.85 ± 15.59^A	<0.001*
Aged	303.51 ± 35.64^A	198.10 ± 6.57^B	277.10 ± 13.37^A	<0.001*
p-value	<0.001*	0.100	<0.001*	

*: Significant at $P \leq 0.05$,

Different superscripts in the same row indicate statistically significant difference between materials

DISCUSSION

It was reported that the ultimate strength of all-ceramic systems could only be assessed by evaluating the mechanical behavior of the core-veneer composite. Bearing in mind that the stress distribution in core-veneer restorations is more complex than in single component structures, different tests were developed for evaluating the strength of these systems^(29,30).

Multiple tests are available for testing flexural strength among which are; three-point flexural test, four-point flexural test, and bi-axial flexural test⁽³¹⁾.

However biaxial flexural strength test was chosen for our study since force is applied to the center of the specimen sparing the effect of edge defects that usually leads to early failure.³¹ The biaxial flexural test also allows meaningful comparisons of strength values among different studies when comparable specimen preparation techniques and test parameters are used⁽³²⁾.

This in-vitro study evaluated the effect of different core and veneering materials combinations, as well as the effect of aging on the biaxial flexural strength of core veneered restorations. The null hypothesis was rejected as there was a significant effect (P -value <0.001) for both tested factors.

Based on data obtained in our study, both porcelain veneered zirconia and Crea.lign veneered PEEK showed comparable flexural strength values. This finding is probably due to the almost equally high inherent flexural strength of both 3-YTZP and modified PEEK ranging from 900-1200 MPa. It was stated that strength, reliability, and mode of fracture of bi-layered composites is determined by the material on the bottom surface under biaxial tensile stress^(32,33).

However, IPS e.max CAD veneered PEEK, on the other hand, displayed significantly lower flexural strength values than both porcelain veneered zirconia and Crea.lign veneered PEEK.

Multiple factors are known to affect the whole strength of the bi-layered restorations such as residual stress, interfacial bonding strength, transformation toughening of zirconia, thickness of the core and veneering material, the direction of loading, as well as the modulus of elasticity and fracture resistance of each layer^(5,32,33).

Considering the effect of the material's modulus of elasticity and the fact that materials having a more compatible modulus of elasticity are more likely to bend under load and distribute stresses more evenly, we can explain the significantly lower values of IPS e.max CAD veneered groups in our study⁽²⁷⁾. IPS e.max CAD is a highly rigid material with an elastic modulus (95GPa). Consequently, when used for veneering of the more elastic BreCAM BioHPP (4 GPa), areas of stress concentration occurred causing failure⁽⁴⁾. On the contrary, the group veneered with Crea.lign composite having an elastic modulus of 4.4 GPa showed significantly high flexural strength values, which agrees with the results of Beleidy et al⁽²²⁾. Additionally, the presence of 50 % nano-ceramic fillers in the Crea.lign composite resin matrix has definitely played a role in improving its mechanical properties and contributing to the high flexural strength values⁽³⁵⁾.

Bearing in mind that interfacial bonding is considered a major factor affecting the flexural strength, the significantly low values of the IPS e.max CAD veneered PEEK are quite explainable. Sloan et al.⁽¹⁸⁾ in a recent study, showed that lithium disilicate bonded to BioHPP displayed a low flexural strength. They explained this through the inert nature of PEEK rendering bonding to it to be almost absolutely of micro-mechanical nature. In a different research, Taufall et al.⁽²⁰⁾ concluded that surface pretreatment had a significant role in the adhesive failure of bi-layered restorations regardless of the veneering material strength. They suggested that the bonding between the PEEK surface and the adhesive is only and solely of mechanical nature despite the use of air abrasion that has the capability to enhance the micro-roughness thus permitting

better infiltration of the adhesive material. On the other hand, they stated that the use of Visio.link containing an active ingredient is capable of modifying the PEEK surface and thereby creating a chemical bond between the veneering material and the adhesive⁽³⁶⁾.

Regardless of the core-veneer combination used in our study, exposing the different combinations to artificial aging trying to simulate the oral conditions, resulted in a significant reduction in biaxial flexural strength values for both porcelain veneered zirconia and Crea.lign veneered PEEK specimens.

Shedding the light on the impact of thermocycling on Crea.lign veneered PEEK, our results are contradicting a couple of previous studies^(20,24) claiming that thermal cycling had no effect on the flexural strength or load-bearing capacity of PEEK. However, they are consistent with other studies in the literature that showed a significantly lower fracture resistance for Crea.lign veneered PEEK cores after aging⁽²²⁾.

Such deleterious effect of thermocycling could be attributed to the impact of thermal cycling on the composite resin. Previous studies have documented that when a resinous material is exposed to thermocycling, interactions take place between water and the epoxy network causing hydrolytic break-down. Additionally, it was documented that thermocycling could lead to water sorption and uptake by the epoxy network leading to swelling of the matrix and debonding at the filler-matrix interface. Consequently, more channels are created allowing further water penetration and leading to resin softening^(37,38).

Likewise, porcelain veneered zirconia specimens were also shown to display a significant reduction in flexural strength values after accelerated autoclave aging which agrees with Flin B et al.⁽³⁹⁾. They showed, through fractography, that the depth of LTD reached up to 60 μ m for 3-YTZP specimens from the surface. It's postulated that microcracks in the transformed surface layer decreased the

fracture strength since they act as crack-like flaws. It has been also suggested that a superficial layer of monoclinic phase results in tensile stresses in the veneering porcelain layer and thus adversely affects the bond strength since the coefficient of thermal expansion (CTE) of monoclinic zirconia ($7.5 \times 10^{-6}/k$) is significantly lower than that of tetragonal zirconia ($10.8 \times 10^{-6}/k$)⁽⁴⁰⁾.

Our results clearly show that Crea.lign veneered PEEK could serve equally to porcelain veneered zirconia bi-layered restorations in terms of flexural strength. However, clinicians must bear in mind the negative effect of aging on the performance of both materials' combinations. For better simulation of the in-vivo conditions, fracture resistance of complex anatomical restorations that mimic crowns and bridges similar in geometry to the main prosthesis needs to be examined.

CONCLUSIONS

Considering the limitations of this study and the future need for further in vivo studies, it's seen that both Crea.lign veneered PEEK and porcelain veneered 3-YTZP could be used with the same efficiency, regarding flexural strength, as bi-layered restorations. However, a point not to be missed is the negative effect of aging on both materials' combinations.

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