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Fracture load and failure types of different veneered polyetheretherketone fixed dental prostheses

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Abstract: **OBJECTIVE** The aim of this study is to investigate the fracture load of different veneered PEEK 3-unit fixed dental prosthesis (FDPs) after different aging regimens. **METHODS** Congruently anatomically shaped 3-unit FDPs were milled using a master stl-data set and randomly divided into four groups (N = 120, n = 30 per veneering group), which were veneered using different veneering methods: (i) digital veneering with breCAM.HIPC, (ii) conventional veneering with crea.lign, (iii) conventional with crea.lign paste, and (iv) using pre-manufactured veneers visio.lign. The FDPs were then adhesively cemented on a metal abutment and fracture loads were measured in a universal testing machine (1 mm/min) before and after aging (10,000 thermal cycles, 5/55 °C). Two- and one-way ANOVA followed by post hoc Scheffé tests were used for data analysis (p < 0.05). **RESULTS** This investigation showed an influence of the veneering method on the fracture load results independent of the aging level. The highest fracture load was measured for the FDPs with digital veneering (1882 ± 152 N at baseline, 2021 ± 184 N after thermocycling). The remaining groups showed comparable results, and no impact of thermal aging was observed. Digital and conventional veneers showed cracks in the pontic region starting from the connector area as a main failure type after loading, while the pre-manufactured veneers showed predominantly adhesive failures. **CONCLUSIONS** The digital veneering method showed the highest fracture load resistance. Thermal aging showed no impact on the fracture load of all tested veneered PEEK 3-unit FDPs. **CLINICAL RELEVANCE** According to this study results, reliable veneering of PEEK FDPs can be achieved with digital veneering.

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Fracture load and failure types of different veneered polyetheretherketone fixed dental prostheses

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Keyword: fracture load, PEEK, digital veneering, veneering resin composite

ABSTRACT

Objective To investigate the fracture load of different veneered PEEK 3-unit fixed dental prosthesis (FDPs) after different aging regimens. *Methods* Congruently anatomically shaped 3-unit FDPs were milled using a master stl-data set and randomly divided into 4 groups (N=120, n=30 per veneering group), which were veneered using different veneering methods: i. digital veneering with breCAM,HIPC, ii. conventional veneering with crea.lign, iii. conventional with crea.lign paste and iv. using pre-manufactured veneers visio.lign. The FDPs were then adhesively cemented on a metal abutment and fracture loads were measured in a universal testing machine (1mm/min) before and after aging (10.000 thermal cycles, 5/55°C). Two- and one-way ANOVA followed by post-hoc Scheffé tests were used for data analysis ($p < 0.05$). *Results* This investigation showed an influence of the veneering method on the fracture load results independent of the aging level. The highest fracture load was measured for the FDPs with digital veneering (1882 ± 152 N at baseline, 2021 ± 184 N after thermocycling). The remaining groups showed comparable results and no impact of thermal aging was observed. Digital and conventional veneers showed cracks in the pontic region starting from the connector area as a main failure type after loading, while the pre-manufactured veneers showed predominantly adhesive failures. *Conclusions* The digital veneering method showed the highest fracture load resistance. Thermal aging showed no impact on the fracture load of all tested veneered PEEK 3-unit FDPs.

Clinical relevance According to this study results, reliable veneering of PEEK FDPs can be achieved with digital veneering.

INTRODUCTION

The search for biocompatible bone replacement materials in medicine with mechanical characteristics comparable to human bone as an alternative to metals led to plastics, which are used in industrial applications and have a high stability. Polyaryletherketones (PAEK), due to their high mass-based stability and resistance against temperatures, stress and corrosion, were the first promising candidates [1]. Mainly Polyaryletheretherketone (PEEK), Polyetherketoneketone (PEKK) and Polyaryletherketoneetherketoneketone (PEKEKK) found their way into the medical field [2]. In addition to their high biocompatibility, these materials are characterized by

high thermal, chemical and radiological stability. Polyetheretherketone (PEEK) is a high-temperature polymer selected from the family of the aforementioned Polyaryletherketones (PAEK) with outstanding mechanical characteristics [3, 4]. It consists of an aromatic basic structure interconnected by ketones and ether functional groups, which can be classified as a semi-crystalline thermoplastic [5]. Because of the above-mentioned characteristics, in particular the good milling and grinding properties combined with high stability [6] similar to the stability of human bone [3], it is already used in various medical applications such as spine implant or bone substitute technology for large bone defects in traumatology [7, 8].

PEEK is also being used in dentistry as abutment, removable partial denture frameworks and fixed dental prosthetic framework (FDP - fixed dental prosthesis) [9]. In general, there are two production methods for PEEK-FDPs which are press technology or computer aided design/computer aided manufacturing (CAD/CAM). The latter shows lower deformations pattern and higher fracture load values [10, 11].

PEEK is opaque and has generally a white to grey color, however first tooth-colored materials were already introduced to the market. As it is not esthetic, the material cannot be used for monolithic prosthetic solutions in the visible area, making an additional veneering indispensable. The fracture load of a 3-unit PEEK framework without veneering was reported to be 1385 N, which corresponds to be about 2.5 times the average bite force [12] in the posterior area [5].

A variety of studies of bond strength between veneering resin composite and PEEK-framework have been performed already, in which different pre-treatments of the airborne-particle abraded surface with piranha-etching [13, 14, 15, 16], sulfuric acid [5, 9, 12, 17] and cold plasma treatment [18, 19] were tested, providing, however, some conflicting results. Some of these studies examined the influence of the adhesive on the bond strength to PEEK and the vast majority showed nevertheless adequate bonding results with MMA-based adhesive materials comparable to those of conventional framework materials like ceramic or metal alloys [14, 15, 16, 20, 21, 22].

A first peer-reviewed study of veneered PEEK FDPs showed no impact of PEEK surface pretreatment and veneering material on the fracture load results [23]. After thermal cycling, however, all veneered FDPs still showed cracks in the veneering material in the pontic region. After loading, no fractures of the PEEK frameworks were evident in any FDPs, but chipping directly between PEEK and

veneering resin composite was observed. This study used two differently filled (86% versus 74% w/w) veneering composite resins based on the same matrix. The FDPs, which were veneered using the lower filled veneering composite material tended to result in higher fracture loads than those veneered with the higher filled material [23]. In that study - in relation to physiologic mastication forces of 400 N - values up to 277 N for the veneered FDPs were observed. In contrast, PEEK FDPs without veneering showed much higher fracture load results (2354 N) [10]. Because esthetic concerns remain an important clinical reality and benchmark, veneered FDPs should always be assessed, especially because they contrast with standard tests with simplified geometric specimens. Using this approach, however, the fracture load represents the internal tensile stresses within the FDPs after veneering and thermal stress, as well as the bond and flexural strength of the framework together with the veneering resin composite, which results in the lower fracture load of the previous study. Therefore, the authors stated that further in-vitro and in-vivo studies and optimization of the veneering process are still warranted. Therefore, this study investigated different veneering methods for PEEK-frameworks on the fracture load results. The null hypothesis of this study was to test that the veneering method had no influence on the fracture load of PEEK FDPs with 4 different veneering methods, i.e. one digital, two conventional and one pre-manufactured veneer technique.

MATERIALS AND METHODS

The 3-unit Co-Cr-Mo master abutment model ranging from a canine to a second premolar (Fig. 1a) and the PEEK framework used in this study are described in more detail elsewhere [23]. The resulting pontics (Fig. 1b) of the PEEK framework had a 1 mm circular edge, a concave base and a sharp chamfer. Each connector amounted for an area of 11.3 mm², a width of 3.8 mm and a height of 3.2 mm. The thickness of the framework was set at 0.6 mm.

Based on this design, a total of 120 congruent frameworks were milled (Zeno Tec 4030 M1; Wieland Dental+Technik, Pforzheim, Germany) from PEEK blanks (breCAM.BioHPP Discs, bredent, Senden, Germany, LOT: 400177). After detaching the frameworks and removing the mill connectors, the FDPs were abraded with airborne-particles with 110 µm Al₂O₃ powder at 0.25 MPa, at an angle of 45° from a distance of 10 mm (basic Quattro IS; Renfert, Hilzingen, Germany), and subsequently put in an ultrasonic bath for 5 min (L&R Transistor Ultrasonic T14, L&R,

Kearny, NY, USA), which was filled with deionised water. Afterwards, the frameworks were conditioned using visio.link (bredent, LOT: 141432; composition: MMA, products of reaction of 2-propenoic acid with pentaerythritol; diphenyl-(2,4,6-trimethylbenzoyl)-phosphineoxide). Because visio.link is a MMA based adhesive system and most of the recent publications demonstrated that an adequate chemical bond to PEEK can be established [14, 15, 16, 20, 21, 24] with this material, conditioning was carried out by wetting the frameworks with a thin film using a microbrush which was immediately polymerized for 90 s (intensity: 220 mW/cm², Brelux Power Unit; bredent) (Fig. 1c), before a thin film of opaquer (Opaquer combo.lign; bredent) was applied and polymerized for 360 s (Fig. 1d).

The specimens were randomly divided into four veneering groups (n=30/group) as described in Table 1: i) digital veneering with breCAM,HIPC (bredent; Lot No. 406700), ii) conventional veneering with veneering composite resin crea.lign (bredent; Lot No. 130513), iii) conventional veneering with veneering composite resin crea.lign paste (bredent: Lot No. 134524, 141207) and iv) veneering using bonding of pre-manufactured veneers visio.lign (bredent; Lot No. Z3304499, Z3843532, Z3849293, Z3303681).

For the first group with the digital veneering a master FDP with the visio.lign veneers and waxing was manufactured. The shape of the pre-manufactured veneers was taken into account, which can only slightly be changed. In the middle of the pontic region of the first premolar, an impression was formed centrally using a ball (d=6 mm) creating a 3-point contact for the load type during the fracture load test. The design described before results in a thickness of the veneering between 0.8 mm and 1.2 mm as visualized in the crosscut of a connector in Fig. 2. Then two scans (3 Shape; strip light scanner; Wieland Dental+Technik) were performed, one from the PEEK-framework on the metal abutment model and another one from the master FDP on the metal abutment model (Fig. 3). These scans were subtracted from each other and led to the design of the digital veneer which was subsequently milled (Zeno Tec 4030 M1; Wieland Dental) from breCAM.BioHPP discs. After detaching of the veneers and removing the mill connectors, the veneers were airborne-particle abraded from inside with 110 µm Al₂O₃ powder at 0.25 MPa at an angle of 45° from a distance of 10 mm, and subsequently put in an ultrasonic bath filled with deionised water for 5 min. Immediately after drying the inner surface, the veneers were conditioned from inside with visio.link (bredent, LOT: 141432) and polymerized for

90 s. The prepared frameworks were put on the alloy models and the veneers were filled with combo.lign (bredent; Lot No. 132420) before pressing them on the frameworks and polymerizing them for 180 s at 220 mW/cm² (brelux Power Unit; bredent). After removing the surplus, the FDPs were polished (Opal L, Renfert; Lot No. 520-0001; Abraso Starglanz; bredent) by a blinded operator (S.T.) (Fig. 3).

The second and third group used a conventional veneering composite resin material. For the second group, a translucent silicone moulding (visio.sil; bredent) of the master bridge was manufactured, but because of the third group's resin exhibited a higher viscosity, a two-piece moulding was used for the latter group, which was also made of a translucent light polymerizing plastic material (Versyo.putty; Heraeus Kulzer, Hanau, Germany). The PEEK-frameworks for the two groups were additionally prepared with a second opaque liner (crea.lign opaquer; bredent; Lot No. 131137) and polymerized for 360 s (brelux Power Unit). Afterwards, the moulding was filled with the composite resin (crea.lign for the second group and crea.lign paste for the third group) and the alloy model with the attached PEEK-framework was pressed into the silicon moulding. Refinement and polishing were carried out as described above.

The fourth group was veneered using pre-manufactured veneers (visio.lign) which covered only the vestibular side. The veneers were ground using a mould of silicon (visio.sil) of the master FDP to fit the shape. Refining was carried out as mentioned before, using a masked operator (S.T.) focusing on the shape, which should fit the master FDP again. Polishing was carried out as described above.

The FDPs were then randomly allocated to two groups per veneering material and aging level. One half of each veneering group was thermocycled (Thermocycler THE 1100; SD Mechatronik, Feldkirchen-Westerham, Germany) from 5 °C to 55 °C with a dwell time of 20 s for 10.000 cycles. Thereafter, all FDPs were adhesively fixed on the airborne-particle abraded and visio.link conditioned CoCrMo alloy models using Multilink Automix (Ivoclar Vivadent, Schaan, Liechtenstein; Lot No. 503821) and a standardized load of 100 N for 15 min. Then the specimens were stored for 48 h in deionized water at 37 °C. Load bearing tests were carried out using a standardized machine (Zwick 1445; Zwick, Ulm, Germany). For that the FDPs were positioned in the machine, a tin foil of 0.5 mm thickness was positioned on the FDP and the stress stamp to avoid force peaks. Subsequently the stress stamp of hemispherical shape (D=6 mm) was positioned in the mould in the occlusal area of

the first premolar. The load was applied from the vertical direction at a crosshead speed of 1 mm per minute (Fig. 4). Failure was defined as the moment at which the measured force of the load dropped by 10% under the maximum point.

The Kolmogorov-Smirnov test was used to verify a normality of data distribution. Descriptive statistics (mean, standard deviation (SD), 95 % confidence intervals (CI)) were computed. Significant differences between the groups were tested with 2-way and 1-way ANOVA, followed by the Scheffé post-hoc test. All statistical tests were calculated using IBM SPSS (Version 23; IBM Corporation, Armonk, New York, USA) ($p < 0.05$).

RESULTS

The descriptive statistics are summarized in Table 2. The Kolmogorov-Smirnov test indicated no evidence for violation of normality assumption regarding the distribution of the data ($p < 0.05$). According to the 2-way ANOVA, the results showed that the veneering method ($p < 0.001$) had a significant effect on the fracture load results of the tested PEEK FDPs. In contrast, the aging level ($p = 0.798$) as well as the interaction between both parameters were not significant ($p = 0.290$). Subsequently, the data set was split based on aging level and the impact factor of the veneering methods were analyzed separately.

Digitally veneered FDPs showed significantly higher fracture load results compared to the remaining veneering groups ($p < 0.001$), regardless of the aging level. The remaining groups were in the same value range.

The fracture type analysis showed two typical modes. For the first three groups, i.e. the digital and conventional veneering, results showed that the fracture type was comparable and showed cracks in the veneering in the pontic region starting from the connector area (Fig. 5). In the fourth group, the failure type of the pre-manufactured veneering could not be visually detected. However, the load-bearing curves showed a failure and also acoustically a distinct crack could be heard. Hence, an adhesive failure between the PEEK-framework and pre-manufactured veneering was assumed. After cutting of the tested FDPs a final failure can be excluded. The failure was caused by an adhesive breakdown between the visio.link layer and the PEEK framework in all cases, as evidenced by the completely exposed PEEK surface (Fig. 6).

DISCUSSION

The investigation of the influence of the different veneering methods on the load-bearing capacity was the main goal of this study. Generally, all tested FDPs showed sufficient fracture resistance compared with the anticipated bite force [6], and therefore both the chosen thickness of the framework and the chosen thickness of the veneering can be recommended. The digitally veneered FDPs showed a higher load-bearing capacity than the three other groups, which were all in the same range. Therefore, the null hypothesis of this study that the veneering method had no influence on the fracture load of PEEK FDPs had to be rejected.

Load deflection curves, one for each group are provided in Fig. 7. One can see that the curves in principle have the same shape. An exception is the curve for HIPC (initial) veneer, showing a slight discontinuity in the gradient between 0.3 and 0.5 mm which is probably attributable to adjustment of the tin foil. Generally, the curves show a non-linear elastic behavior of the probes and the relationship between the vertical deflection and the force is thus a function of higher order. In general, the modulus of elasticity of the veneered FDPs seems, however, to be independent of the veneering and therefore as to be substantially due to the characteristics of PEEK.

The reason for the increased stability of the digital veneering could be, among others, that some complex manual steps in the manufacturing process could be reduced. By this, simply expressed, only the adhesive bonding of the veneer to the framework was the only manual step, whereas in all other veneering methods as a variety of error sources in the manufacturing process were excluded. These errors add up and may in the end have a negative impact on stability. A second reason could be the higher level of curing in the pre-manufactured digital veneering compared with conventional manual veneering with veneering composite resin, since this is associated with higher strengths [25].

The modulus of elasticity of the examined veneering materials could also have an influence on the load-bearing capacity. PEEK as a framework has a modulus of elasticity of 4.0 GPa. The e-modulus of the used veneering material HIPC was 2.8 GPa, which is together with the visio.lign veneering material, which displayed the lowest modulus of elasticity of of all investigated materials. For the veneering materials crea.lign and crea.lign paste, the values accounted for 4.4 GPa and 5.5 GPa, respectively. When veneering with the pre-manufactured visio.lign veneers of lower e-modulus, failure was not within the veneers, but rather in the connector area

as with any other veneering material under investigation. This could explain the lower values of the load bearing capacity with visio.lign veneers as compared to the HIPC veneering. The failure zone was veneered with combo.lign, which had a modulus of elasticity of 8.5 GPa. This has to be considered in upcoming studies and when repairing veneers, this should be carried out with combo.lign whereas the remaining veneer should be replaced with a more elastic material.

In contrast to the challenges mentioned with conventional veneering composite resin [23], no pre-test failures related to thermocycling were observed in this study and the thermocycling had no influence on the load-bearing capacity. The failure type cracking was observed for the first three groups while the fourth group showed an adhesive failure. Nevertheless, the fourth group showed comparable fracture loads to the FDPs with conventional veneering composite resin.

The higher standard deviation of the other three groups compared to the group with digital veneering could be explained by the manual steps in the conventional veneering, since the applied manufacturing process should ensure a high degree of congruence between the outer contour, but slight deviations cannot be entirely excluded, which can be considered as a limitation of this study.

The weak spot in the first three groups of digital and conventional veneering is quite clearly located at the connectors, which is attributed to the smallest thickness of the PEEK-framework at these points. Under axial load, regardless of the veneering material, at this position the distortion values are highest, making the fracture load of the veneering material being reached first. In the fourth group the veneer seems to have a higher strength, so that the adhesive bond fails before the veneer can even break. The reason for the pure adhesive breakdown lies in the pre-treatment. Although the airborne-particle abrasion improves the micro roughness, increases the surface area and allows a better infiltration of the adhesive material the bonding still remains almost only mechanical between the PEEK surface and the adhesive. In contrast to this the veneering material is chemically bonded to the adhesive visio.link layer in addition and therefore creates a stronger bonding in all cases investigated in this study.

In the presented results one must take into account that the model material CoCrMo has a much higher elastic modulus than the hard tooth tissue. Furthermore, the physiological tangential movement of the abutment teeth in the experiment is not

modeled and therefore the fracture test allows comparison between the different veneering materials but has limited clinical relevance.

Before clinical studies are carried out, further studies should model the physiological mobility of natural teeth using a periodontal ligament as well as the E-modulus of the dental hard tooth tissue of the actual abutment teeth, expecting lower values of the fracture load. Also an investigation of the combination of pretreatment using airborne-particle abrasion with different particle size and acids is of major interest. In addition, the fracture load of different digital veneering materials on PEEK frameworks could be subject of a next study.

CONCLUSIONS

PEEK may be a suitable material for removable prostheses when considering the results of this study. However, long-term investigations and advancement of PEEK CAD/CAM processing are still warranted. Apart from the advantages resulting from the industrial production on a large scale as resistance against wear, standardized polymerization and a relatively low discoloration potential, veneering using CAD/CAM method result in a lower monomer content, which implies the biggest advantage over the manual veneering in future clinical use.

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COMPLIANCE WITH ETHICAL STANDARDS

All procedures performed in studies involving human participants were in accordance with the ethical standards of the institutional and/or national research committee and with the 1964 Helsinki Declaration and its later amendments or comparable ethical standards.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

FUNDING

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ETHICAL APPROVAL

This article does not contain any studies with human participants or animals performed by any of the authors.

INFORMED CONSENT

For this type of study, formal consent is not required.

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Framework	breCAM.BioHPP (PEEK) N=120 LOT: 400177							
Veneer	Digital veneers breCAM.HIPC (n=30) LOT: 406700		Conventionell veneers				Premanufactured veneers visio.lign (n=30) LOT: Z3304499 Z3843532 Z3849293 Z3303681	
			crea.lign (n=30) LOT: 130513		crea.lign paste (n=30) LOT: 134524 141207			
Aging	initial	10000 thermo- cycles	initial	10000 thermo- cycles	initial	10000 thermo- cycles	initial	10000 thermo- cycles
Quantity	15	15	15	15	15	15	15	15

Table 1:Study design

Framework	breCAM.BioHPP (PEEK) N=120 LOT: 400177							
Veneer	Digital veneers breCAM.HIPC (n=30) LOT: 406700		Conventionell veneers				Premanufactured veneers visio.lign (n=30) LOT: Z3304499 Z3843532 Z3849293 Z3303681	
			crea.lign (n=30) LOT: 130513		crea.lign paste (n=30) LOT:134524 141207			
Aging	initial	10000 therm o- cycles	initial	1000 0 therm o- cycle s	initial	10000 therm o- cycles	initial	10000 thermo -cycles
Mean [N]	1882 ^b	2021 ^b	1138 ^a	1008 ^a	1226 ^a	1229 ^a	1213 ^a	1149 ^a
Mean- deviation [N]	152	184	278	372	280	239	380	274
95% CI [N]	1797 - 1967	1919 - 2124	984 - 1293	802 - 1215	1070 - 1382	1096 - 1362	1002 - 1425	997 - 1301

Table 2: Statistic results

Figure 1: a) The master 3-unit from canine to second premolar Co-Cr-Mo abutment model (up, left); b) PEEK pontic framework (up, right); c) visio.link conditioned PEEK framework (down, left); d) conditioned PEEK framework with polymerized opaquer combo.lign (down, right)

Figure 2: Crosscut of a HIPC veneered FDP in the connector area

Figure 3: PEEK-framework on the metal abutment model (powder conditioned for 3D-Scan); Master FDP on the metal abutment model (powder conditioned for 3D-Scan); FDP with PEEK framework and digital veneer (from left to right)

Figure 4: FDP positioned in the testing machine with tin foil and stress stamp of hemispherical shape

Figure 5: Example for fracture type of the first three groups, showing cracks in the pontic region starting from the connector

Figure 6: Completely exposed PEEK surface after cutting a tested, HIPC veneered FDP

Figure 7: Load-deflection curves

Table 1: Study design

Table 2: Statistic results