

Failure Analysis of Zirconia Versus PolyEtherEtherKetone Custom Made Posts in Endodontically Treated Anterior Teeth: IN VITRO STUDY

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ARTICLE INFO.

Keywords:

Custom post, Endodontically Treated, Failure mode, PEEK, Zirconia

Abstract

Background: PolyEtherEtherKetone and zirconia are two recent esthetic post materials used in treatment of endodontically treated teeth. Although both materials share similar esthetic properties but have different mechanical properties which could affect their mode of failures. Therefore, the aim of the study was to analyze the failure modes of zirconia versus PolyEtherEtherKetone custom made posts in anterior endodontically treated teeth.

Materials and Methods: Thirty-six anterior maxillary incisors with similar dimensions were collected, decoronated, endodontically treated, and post space prepared. The teeth were divided according to material of post into two groups: group (1) PolyEtherEtherKetone post and group (2) zirconia post. PolyEtherEtherKetone posts were surface treated via sulfuric acid while zirconia posts were sandblasted by 50um aluminum oxide particles. All posts were cemented using dual cure self-adhesive cement. Thermocycling at 5,000 cycles was done to all samples. Three slices of 2mm thickness were obtained from each sample. The samples were loaded to failure via universal testing machine at a crosshead speed of 0.5 mm/min. Failure modes were evaluated using a scanning electron microscope under magnifications of 52x, 250x and 400x.

Results: There was no statistically significant difference between failure modes of the two post material types at the cervical, middle as well as apical root levels (P-value = 0.070), (P-value = 0.580) and (P-value = 1), respectively.

Conclusions: The failure modes between both zirconia and PolyEtherEtherKetone posts were comparable, making them viable options as custom-made post materials. However, within the PolyEtherEtherKetone posts adhesive and adhesive-cohesive failures were more common while in zirconia posts cohesive failure was more dominant.

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1. Introduction

During the service of teeth in the oral cavity, they are vulnerable to be exposed to one or multiple destructive factors as caries, erosion, abrasion, and trauma ¹. Whenever these destructive factors proceed enough to violate the pulp, endodontic treatment is done to stop the progression of microbes and bacteria that may cause infections ². Tooth structure may be lost or weakened because of the damage caused by the destructive factors mentioned previously, in addition to the endodontic procedure itself as in gaining access cavity, caries excavation, and unsupported tooth structure removal ². Partial and full coverage crowns are treatment modalities done for preservation of the weakened tooth structure ²⁻⁴.

Post and core restorations improve the bonding of full coverage restorations; however multiple factors determine the necessity of its placement. These factors include the remaining tooth structure, tooth position, direction and amount of force directed towards the tooth in occlusion⁵.

Because of the increased esthetic demand by patients, esthetic posts are commonly used in the anterior region due to the better esthetical properties compared to metal posts⁶. Prefabricated posts such as fiber posts are often used in teeth with moderate destruction, while custom-made posts are mainly used in cases of severely destructed teeth⁷. The introduction of CAD/CAM provided a more precise and an easier method in fabricating restorations from a wide variety of esthetic materials; thus providing alternatives to the traditional materials previously used, such as metal⁸.

Due to the esthetic properties of zirconia, it has been used as a prefabricated and custom-made post material. However its high elastic modulus an issue with its usage^{9,10}. Polyetheretherketone (PEEK) is a polycyclic, aromatic, thermoplastic polymer that is semi-crystalline and has a linear structure; furthermore it provides esthetical compatibility while offering an elastic modulus close to that of the dentin¹¹.

Thermocycling is a procedure that mimics the stresses induced by thermal variations of the oral cavity which the restorations and teeth are subjected to during service¹². The thermal stresses induce aging to the restorations and teeth due the difference in coefficients of thermal expansion between restorative material and tooth structure, thus increasing microleakage and bond failure^{13,14}.

The elastic modulus is a determinant factor of stress distribution¹⁵. Materials with high elastic modulus like zirconia, distributes the forces towards the more flexible surrounding structure; thus, increasing the incidence of irreparable fractures¹⁶. However PEEK's low elastic modulus tends to concentrate the stresses on the post itself rather than adjacent structure¹¹. Due to zirconia's lack of glassy phase and PEEK's bioinert properties^{10,11}, this makes their bonding a challenge; therefore, this factor could affect their mode of failure. Our null hypothesis in this study is that there will be no difference in failure modes between zirconia and PEEK custom made posts. The aim of the study was to analyze the failure modes of zirconia versus PEEK custom made posts in anterior endodontically treated teeth.

2. Material & Methods:

2.1 Sample Size Calculation

A power analysis was designed to have adequate power to apply a two-sided statistical test. By adopting an alpha level of (0.05) a beta of (0.2) i.e. power=80% and an effect size (d) of (0.98) calculated based on the results of Türker, Sevinç¹⁷ the predicted sample size (n) was a total of (36) samples. Sample size calculation was performed using G*Power version 3.1.9.7

2.2 Samples collection

Thirty-Six upper central incisors teeth were collected from MSA University Oral Surgery Clinic. This research was approved by the research ethical committee with approval number of (ETH33). The study followed a study design that was illustrated in Figure (1). Scaling of the teeth was done using an ultrasonic scaler (Cavitron Select SPS; Dentsply Sirona, York, Pennsylvania), after that they were polished using a polishing paste and a brush attached to a low-speed motor. The teeth were disinfected with 5.25% sodium hypochlorite then kept in distilled water at room temperature. Teeth selected for the conduction of the study were selected according to an inclusion criteria which necessitated the teeth to be: sound teeth, free from cracks or fractures, free from restorations, with single root canal, similar in length and width of buccolingual as well as mesiodistal dimensions with 0.5 to 1mm variation checked by using digital caliper (Digital Caliper, Adoric, CHINA), and accessible for root canal treatment. The teeth were put in envelopes then numbered from one to thirty-six. Sequence generator option was selected in (random.org) with the largest value inserted was thirty-six, the smallest value selected was one, and the columns were set at two. A colleague reordered the teeth into their new assigned numbers and groups (I and II); furthermore, the assigned numbers with their original numbering were written by and kept with the colleague.

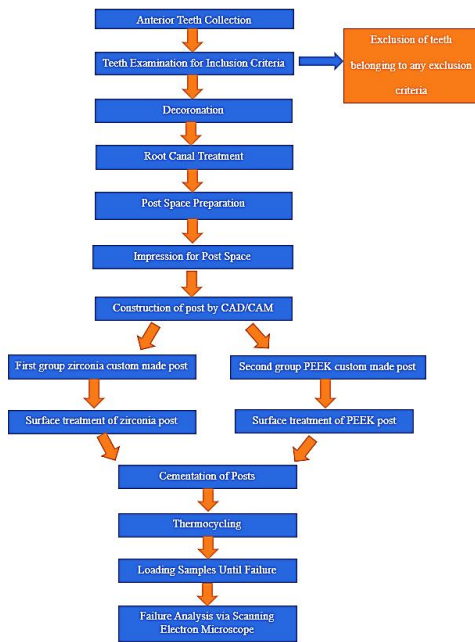


Figure 1. A flow chart visualizing the study design used in this study.

2.3 Samples preparation:

The teeth were decoronated 2mm above the cemento enamel junction using a diamond saw (ISO Met 4000 Buehler Germany precision cutting, Germany) under water coolant. The decoronation process resulted in a standardized teeth length of 15±1mm. The samples were immersed and kept in distilled water. Rotary files (ProTaper Gold; Dentsply, Tulsa Dental Specialties, Tulsa, OK, USA) were used sequentially until the size F3 to achieve mechanical shaping of the canals. The canals were irrigated with 5.25% sodium hypochlorite (NaOCl) in between each mechanical step, and the final irrigation protocol was made in the following order: distilled water, 17% ethylenediaminetetraacetate (EDTA), distilled water, 5.25% NaOCl solution and finally by distilled water to flush the sodium hypochlorite solution. The obturation process was done using lateral condensation technique with the aid of resin sealer (AD Seal; Meta, Biomed, Cheongju, South Korea). After covering the orifices with temporary cement, the teeth were stored in distilled water for 72 hours at room temperature. Blinding wasn't applicable for the operator due to different manufacturing techniques of the custom made posts were employed as the zirconia posts needed sintering after milling while PEEK posts were milled to the original size of the resin pattern; additionally, each of the post materials has distinct visual characteristics that is easily distinguishable. The assessor was blinded during the trial as the samples

were coded with three letters as the following: the first letter symbolled the region (C= cervical, M= middle, A= apical), the second letter ciphered the materials used (P= PEEK and Z= zirconia), finally it ended with a number denoting which sample the slice was acquired from. The statistician was also blinded as the raw data delivered with groups written as I and II with no further details mentioned about identification. A mold former was constructed using duplicating material for a Lego cylinder. Each mold former was used to create epoxy resin mold in which endodontically treated teeth were centralized using surveyor. The mold was left for 24 hours till complete hardening. Gates glidden drills sized (1,2,3,4,5) were attached to a low-speed contra angled handpiece then used to prepare the post space¹⁸; moreover this process was done under copious water irrigation. The gates glidden drills were changed periodically with a new set after usage on every 5 samples. After maintaining the 5mm apical seal, a standardized post space length of 10±1mm was achieved. After preparation, rinsing of the post space was done using 5.25% sodium hypochlorite followed by distilled water then finally dried with paper points according to fit.

2.4 Post fabrication:

The direct impression technique was utilized to construct custom-made posts. Separating medium (Separating Medium, Acrostone Manufacturing and Import Company, Egypt) was agitated in the canal using micro-brush followed by the application of slight air pressure via air syringe. The resin pattern (Duralay, Reliance Dental Mfg. Co., Worth, Illinois) was constructed incrementally using a plastic post until complete build-up of the post pattern was achieved. Resin pattern with 2mm height and flat incisal core was built. The resin patterns were kept in distilled water until the scanning procedure and access was sealed with non-eugenol temporary cement. To ensure that the resin patterns were defect free, each pattern was checked with dental microscope (Lecia M320 F12, Lecia microsystems, Wetslar, Germany) under 10x magnification. Antireflection spray (Scanspray, Renfert, Hilzingen, Germany) was used to coat the resin patterns, which were subsequently scanned utilizing a 3-axis, non-contact blue light dental scanner (Identica Hybrid, Medit, Seoul, Korea). The scanning data was uploaded to the computer by an installed scanning software (COILab scan v2.0.0.4, Medit, Seoul, Korea) to create a 3D- virtual model of the post and core. The final design of the post and core was created using the computer aided design (CAD) program Exocad (Exocad GmbH, Darmstadt, Germany) (version 6.136). The Dental CNC machine (Vhf, K5, Ammerbuch, Deutschland) used

dry milling process with five axis movements to fabricate the posts. The PEEK blank (Bredent, GmbH Senden, Germany) was used to fabricate PEEK posts, while the zirconia blank (Katana HT, Noritake, Kuraray, Japan) was utilized to produce zirconia posts. PEEK posts came in a fully sintered state were milled in 1:1 ratio to the resin pattern, while the zirconia posts were subjected to a firing cycle in a furnace (Tegra Speed, Yenadent, Istanbul, Turkey) according to manufacturer's instructions. After fabrication process, both PEEK and zirconia posts were visualized with dental microscope under 10x magnification to ensure that they were defect and crack free.

2.5 Cementation:

Posts were cleansed with an alcohol swap, then immersed in distilled water for 10 minutes using an ultrasonic cleaner (Ultrasonic Cleaner 4820, Codyson, Shenzhen, China) finally dried with air. PEEK posts were surface treated using 98% sulfuric acid for 60 seconds then were washed in water for another 60 seconds. The posts were dried with air and PEEK primer was applied for 60 seconds, then gently air dried with air spray until glossiness of the primer faded then cured with 400 mW/s for 90 seconds. As for the surface treatment of zirconia posts, a custom-made wooden device was constructed to maintain a distance of 10 mm between the sandblasting tip and the post. The zirconia posts were sandblasted by 50 μ m aluminum oxide particles (Al₂O₃) at 0.2-MPa distance of 10 mm for 10 sec. The samples were then cleaned with an alcoholic swap, dried, treated with zirconia primer (Z-Prime Plus, Bisco Inc., Schaumburg Illinois, U.S.A.) for 60 seconds, and finally dried under a gentle air flow. To standardize the pressure applied during cementation, A special load system was constructed. A dual cured self-adhesive resin cement (RelyX u200 3M Deutschland GmbH, Germany) was applied in the post space preparation with the aid of microtips according to manufacturer's instruction, then the post was inserted into the tooth with a constant load of 1kg. Initial curing (Light cure LED-F; WOODPECKER; Guilin National High-Tech Zone, Guangxi, China) was done for 2-3 seconds then excess cement was removed using a scalpel, then full polymerization was achieved by light curing the head of the post at an output power of 1600 mW/s for 20 seconds in each direction of the tooth. Teeth samples were kept in distilled water at room temperature for 24 hours before thermocycling.

2.6 Thermocycling:

All specimens were subjected to thermocycling using thermocycler (Thermocycler THE-1200, SD Mechatronik, Feldkirchen-Westerham, Germany) with distilled water for 5,000 cycles at 5°C and 55°C with 30 seconds of dwell time and 5 seconds transferring time, which is equivalent to six months in oral cavity^{19,20}.

2.7 Failure Analysis:

Using a precision saw with water cooling system (ISO Met 4000 Buehler precision cutting, Germany), each sample was transversely cut perpendicular to the long axis of the tooth root. Each tooth was cut into three sections of 2 \pm 0.1 mm (cervical, middle, apical)^{17,21}. Load was applied on samples via universal testing machine (Model 3345, Instron Industrial Products, Norwood, MA, USA) at speed 0.5mm/min until failure in an apico-coronal direction^{21,22}.

Environmental scanning electron microscope (SEM Model Quanta 250 FEG, Thermo Fisher Scientific, Netherlands) was used to observe the samples with three magnifications of 52x, 250x and 400x²³. Low vacuum mode was chosen to examine the nonconductive slices of zirconia and PEEK. Three slices apical, middle, coronal of fracture of selected samples from each group were attached to the scanning plate inside of vacuum chamber. The failure modes were classified according to Bansod et al. (2020)²⁴ as the following: adhesive failure between dentin and resin cement, adhesive failure between post and resin cement, cohesive failure of cement, cohesive failure of dentin, cohesive failure of post, or adhesive cohesive failure (mixed failure). The results were recorded photographically.

2.8 Statistical Analysis:

Failure mode data were presented as frequencies and percentages. Chi-square test and Fisher's Exact test were used to compare between failure modes of the two post material types. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY: IBM Corp.

3. Results:

There was no statistically significant difference between failure modes of the two post material types at the cervical, middle as well as apical root levels (P -value = 0.070, Effect size = 0.377), (P -value = 0.580, Effect size = 0.174) and (P -value = 1, Effect size = 0.061), respectively Table (1) Figures (2). Even though the failure modes of the two groups did not differ significantly, both the material

type and the root region influenced the mode of failure. The highest number of PEEK samples exhibited mixed failure, while the highest number of zirconia samples showed cohesive failure. Failure modes were presented in **Figures (3 - 8)**

Table (1): Frequencies (n), percentages (%) and results of Chi-square and Fisher's Exact tests for comparison between failure modes of the

Root level	Failure mode	PEEK (n = 18)		Zirconia (n = 18)		P-value	Effect size (v)
		n	%	n	%		
Cervical	Adhesive	7	38.9	2	11.1	0.070	0.377
	Cohesive	5	27.8	11	61.1		
	Mixed	6	33.3	5	27.8		
Middle	Adhesive	6	33.3	5	27.8	0.580	0.174
	Cohesive	6	33.3	9	50		
	Mixed	6	33.3	4	22.2		
Apical	Adhesive	7	38.9	8	44.4	1	0.061
	Cohesive	3	16.7	3	16.7		
	Mixed	8	44.4	7	38.9		

two post material types

*: Significant at $P \leq 0.05$

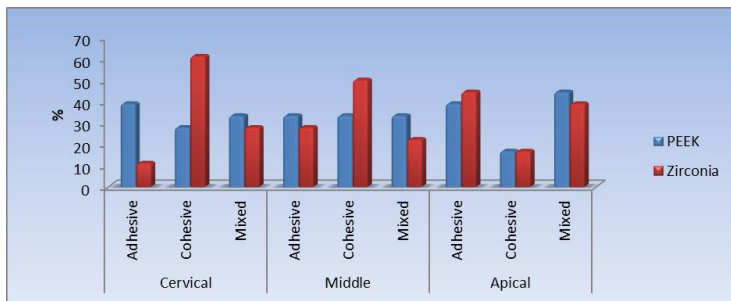


Figure 2. Bar chart representing failure modes of the two post material types

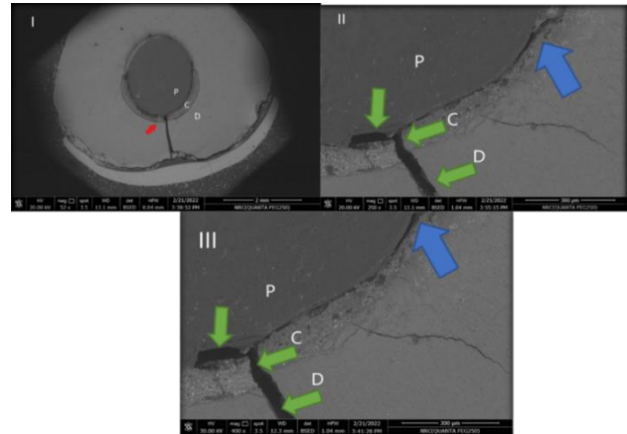


Figure 3. SEM photo depicting adhesive-cohesive failure in a PEEK sample.

The blue arrow in picture III shows adhesive failure as the separation happened between the post and cement, while the green arrows show the crack that is in the post, cement, and the dentin.

The photos of the sample were taken under three magnifications while also the red arrow showing the area of magnification.

I: 52x

II:250x

III: 400x

The above letters P, C, D stand for post, cement, and dentin respectively

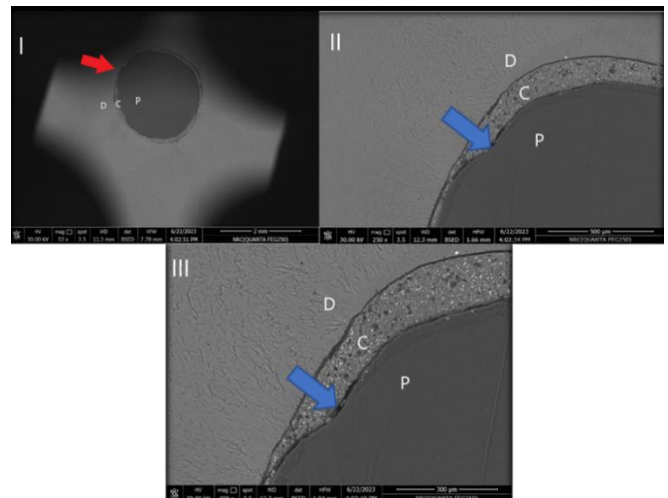


Figure 4. SEM photo depicting adhesive failure between post and cement of a PEEK sample.

The blue arrow shown in pictures II and III shows the area of separation between the cement and post.

The photos of the sample under three magnifications while also the red arrow showing the area of magnification.

I: 52x

II:250x

III: 400x

The above letters P, C, D stand for post, cement, and dentin respectively

4. Discussion

Endodontically treated teeth are grossly destructed teeth that are known to be weaker and more prone to fracture than vital teeth²⁵. The scarcity of tooth structure leads to problems in bonding of restorations; therefore post and core considered as solution to enhance the bonding and restoring endodontically treated teeth with more than 50 % loss in structure^{5,7}. Custom-made metal post and core had been used as a treatment option in endodontically treated teeth. They presented esthetical drawbacks and exhibited a higher frequency of tooth fracture upon failures due to its high elastic modulus⁶. This study was oriented to examine the failure modes of two custom made esthetic posts of zirconia and PEEK in anterior teeth following push out bond strength. Our null hypothesis which stated that there will be no difference in failure modes between zirconia and PEEK custom made posts was accepted.

The endodontic treatment's mechanical preparation was done by Protaper Gold files in a standardized manner for every sample used in accordance to various authors^{22,26,27}. Hussien and Al-Gharrawi (2019)²⁸ conducted a study that compared various Nickel Titanium rotary instruments effects on dentinal root defects incidence, found that flexible files like Protaper Gold, Protaper NEXT, and RECOPRIC Blue produced the least amount of cracks compared to RECIPROC files which were more stiff; thus the flexible files of Protaper Gold was chosen reduce the incidence of cracks that could affect the failure analysis of this study.

As for the chemical preparation, EDTA was first used to dissolve the smear layer and increase the dentin permeability which allowed the NaOCl to properly dissolve the organic substances and eliminate microbes in deeper areas of the root canal^{27,29}. However, distilled water irrigation was done between the irrigation of EDTA and NaOCl to prevent the chemical interaction between the two solutions which may lead to decreased dentin microhardness, change its flexural strength and modulus of elasticity cause irreversible damage of the dentin microstructure³⁰. According to Leal et al. (2015)³¹ who compared several final irrigation protocols effects on push out bond strength of epoxy resin root canal sealer to dentin, found out that the usage of EDTA followed by NaOCl provided more exposure of dentinal tubules which improved the sealer penetration.

Each tooth was embedded into an epoxy block in a centralized manner with a technique using

surveyor and a diamond disc³². The centralization was done to ensure that the stress should be applied uniformly and uniaxially³³.

Scanning Electron Microscope was used as a failure mode analysis method due to its ability to show high details and acquire higher magnification in order to have a more detailed assessment of fracture site and fracture type³⁴.

Although there was no statistical significance between Zirconia and PEEK, within the zirconia group the failure modes were mostly cohesive in cervical and middle regions, followed by adhesive failure in the apical region and the least was adhesive cohesive failure also in the apical region. A possible explanation for the dominance of cohesive failure was stated by Habib et al. (2022)¹⁵ who mentioned that the type of cement, elastic modulus of post material, nature of prepared dentin and regions of stress concentration may increase chances of cohesive or mixed failures. This statement may explain the abundance of cohesive failures in zirconia's cervical and middle; furthermore, the high elastic modulus of zirconia transfers stress to regions with less modulus of elasticity as in cement and dentin hence increases the likelihood of these regions to fracture. Also, the previous statement may also explain the adhesive or adhesive cohesive failure that happened apically.

The adhesive cohesive failure occurrence in the apical part maybe due to not only the difference in elastic modulus between the three components of the slices, but also maybe due to the cement layer was thinner in the apical third when compared to cervical and middle parts³⁵. According to Anaraki et al. (2014)³⁶ the better adaptation of the post in the apical region and subsequently lower thickness of cement in this region, which would decrease the polymerization shrinkage and enhance bond strength. The nature of dentin at this apical area may cause more cohesive and adhesive cohesive fractures^{37,38}. The thin cement layer at the apical part and high elastic modulus of zirconia may contribute to the adhesive-cohesive failure. These factors would contribute to a higher frequency of irreparable failure that would occur at this distant location, thus affecting the prognosis of the restored tooth.

Li et al. (2015)³⁹ was in agreement with our study as it was found the cohesive failure was most common after push out bond strength of zirconia posts. Due to similarity in the usage of sandblasting as it was explained that sandblasting offered better adhesion resulting in more cohesive failures. According to Abd El Aziz et al. (2022)⁴⁰, who evaluated different surface treatment effects on the push out bond strength of zirconia posts and its failure modes. It was observed that zirconia posts treated

with tribochemical silica coating showed a 100% adhesive cohesive failure. The difference in failure mode between our and their study could be due to the different surface treatment methods used.

PEEK had mostly adhesive cohesive failure, followed by adhesive failure, and cohesive failure had the least percentage. The adhesive-cohesive failure was seen mainly in apical regions. A possible reason is that the post treated with sulfuric acid and Visio.link application had resulted in better bond strength that increased the percentage of bond between post and cement⁴¹. Another possible explanations are that the highly adapted custom-made PEEK post decreased film thickness in apical region, nature of dentin and smear layer that maybe present lead to mixed failure at cement-dentin interface⁴¹. The well fitted custom made post had an increased of contact area of both post and dentin⁴². This resulted in a better frictional retention while also providing a more homogenous bonding. The retention enhancement could be beneficial in restoring severely destructed non-vital teeth.

The adhesive failure, which was prominent in the cervical section, was found between cement and post. This may be due to the increased film thickness affecting bond strength adversely by inducing shrinkage stresses that result in subsequent failure in bonding⁴². According to Aleisa et al. (2021)⁴³ stated that the increased thickness of cements increases the probabilities of micro defects, crack initiation and propagation. This phenomenon would cause a decrease in the resistance of thicker cement layers and may affect the longevity of the restoration's service in the oral cavity. Our failure analysis was in agreement with Attia et al. (2021)²² in regard to the adhesive-cohesive failure prominence. This agreement is maybe due the usage of sulfuric acid treatment as PEEK's surface treatment.

The failure mode examined from our study was contradicting with Badimela et al. (2019)²⁶; as prominence of adhesive failure was observed. This difference could be due to the dependance on mechanical retention means of PEEK rather than chemical retention means. Monteiro et al. (2022)⁴¹ observed that most failure after push out bond strength test was adhesive failure, which disagreed with our study. However, the difference could be due to the unsimilar aging methods used.

Limitations of the present study, the study setting was done in an in vitro setting, so simulation of oral conditions was attempted. However, it could not

accurately depict real life oral conditions like mechanical forces, pH changes, and the presence of oral microbes. Although thermocycling was done to mimic the temperature changes in oral conditions, chewing simulation would have provided an analysis closer to real life conditions. Different surface treatments of the custom-made post as well as different luting agents should be also considered.

5. Conclusion:

Because of the shift towards ceramic restorations in recent dentistry, custom-made posts fabricated from zirconia and PEEK materials are emerging as suitable substitutes to cast metal posts due to their esthetic appeal. The lack of concrete knowledge and research about the failure modes occurring in both posts promoted this comprehensive study; furthermore, this study showed that the two posts were comparable in their failure modes following pushout-bond strength test. The failure modes are not only affected by the material type and elastic modulus but are also influenced by several other factors such as the surface treatment used, thickness of cement layer, type of dentin, aging of the restoration and type of forces directed towards the restoration as well as the amount forces applied.

In conclusion, the failure modes between both zirconia and PEEK posts were comparable making them viable options as custom-made post materials. However, within the PEEK posts adhesive and adhesive-cohesive failures were more common while in zirconia posts cohesive failure was more dominant.

Informed consent:

The patients signed an informed consent

Conflict of interest

Declaration of interest: The study was self-funded, and we hereby declare that there was no conflict of interest.

Funding

The research study was self- funded by the authors.

Acknowledgment

The authors would like to thank Dr. Khaled Kera for his efforts in the statistical analysis of the study results.

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