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The Effect of Thermocycling on the Shear Bond Strength of Polyether Ether Ketone (PEEK) to Resin Cements: A Comparative Study

Dr. Nishath Ayesha[1]*, Dr. Sunil Kumar. M.V.[2], Dr. Sadiq Mohammad Sabir Ali[3]

[1]*Bds, MDS, PHD Scholar, Department of Prosthodontics, Maharaja Vinayak Global University, Jaipur,
Email: nishathayesha001@gmail.com, Phone number: 8790144486.
[2]Professor, Department of Prosthodontics, Maharaja Vinayak Global University, Jaipur, Email: drsunilmv@hotmail.com, Phone number: 9828960414
[3]BDS, MDS, PhD Scholar, Department of Prosthodontics, Maharaja Vinayak Global University, Jaipur,
Email: Ddentics@gmail.com, Phone number: 7702368197

***Corresponding Author:** Dr. Nishath Ayesha
*Bds, MDS, PHD Scholar, Department of Prosthodontics, Maharaja Vinayak Global University, Jaipur,
Email: nishathayesha001@gmail.com, Phone number: 8790144486.

Abstract
Background: Polyether ether ketone (PEEK) is a promising polymer for dental restorations due to its excellent mechanical properties. Its wide clinical application ranges from prosthetic frameworks to orthodontic appliances. However, understanding the effect of thermocycling on the bond strength between PEEK and resin cements is crucial for its broader clinical utilization. Thermocycling tests offer insights into the durability and reliability of PEEK-based restorations, advancing dental materials and techniques. This study aimed to evaluate and compare the thermocycling effect on the shear bond strength of RelyX Unicem and G-CEM Linkace to PEEK surfaces.
Methods: Forty disk-shaped PEEK specimens (10 × 3 mm) were fabricated and randomly assigned to two groups: Group A, cemented with RelyX Unicem, and Group B, cemented with G-CEM Linkace. Specimens from both groups underwent 500 thermocycles at 5°C and 55°C. Shear bond strengths were determined using a universal testing machine at 1 mm/min. Scanning electron microscopy (SEM) was employed to ascertain failure types, observing fractured interfaces at 5.00 kV x 500 SE at 100 m and 5.00 kV x 150 SE at 300 m magnification.
Results: The mean shear bond strength for RelyX Unicem cement in the control group was significantly higher than in the test group. For G-CEM Linkace cement, the mean shear bond strength was 5.07 ±0.95 MPa in the control group and 4.01±0.68 MPa in the test group, showing a 20.9% reduction. RelyX test group exhibited maximum loss of shear bond strength, while the G-CEM test group showed minimum loss. After thermocycling, G-CEM Linkace demonstrated maximum shear bond strength, and RelyX Unicem showed minimum. An adhesive type of failure was consistently observed for both cementing materials.
Conclusion: Thermocycling, which simulates oral stimuli, can alter certain properties of PEEK. Post-thermocycling, G-CEM LinkAce exhibited the highest shear bond strength among the studied cementing materials. SEM observations confirmed an adhesive failure mode on PEEK surfaces in both groups.

Keywords: poly ether ketone, relyx unicem, thermocycling.

Introduction

A healthy oral cavity is fundamental to overall well-being, with teeth playing a crucial role in mastication, speech, facial aesthetics, and self-confidence. The World Health Organization (WHO) and World Dental Federation (FDI) emphasize oral hygiene as an integral aspect of self-care. Despite declining tooth loss in developed countries, disparities persist globally due to various factors like caries, trauma, periodontal diseases, and orthodontic extractions. 1,2

Restorative dentistry has advanced significantly, offering diverse materials and techniques for non-invasive, long-lasting, and predictable solutions. Dental implants, rooted in the jaw with biomaterials, have become a highly efficient solution for missing teeth. Conventionally, titanium and its alloys are popular due to their low density, high flexural strength, corrosion resistance, mechanical resilience, and biocompatibility. However, concerns exist regarding titanium's association with allergies, granulomatous reactions, and contact dermatitis. Titanium implants can also induce stress-shielding, leading to peri-implant bone loss, and cause image distortions in MRI scans.

Ceramics are emerging as a viable alternative to metal implants, offering properties such as being inorganic, non-metallic, polycrystalline, brittle, wear-resistant, and bio-inert. Ceramics provide superior aesthetics over porcelain-fused-to-metal (PFM) restorations, which often exhibit a blackish hue. Ceramic restorations allow for varying opacity and translucency, facilitating natural-looking results and simpler preparation techniques that reduce trauma to gingival tissue.3,4

Polyether ether ketone (PEEK), a novel polymer, has gained attention for dental restorations due to its excellent mechanical properties. PEEK belongs to the polyaryletherketone (PAEK) family, known for robust mechanical properties, chemical

resistance, and biocompatibility. Its clinical applications are extensive, including prosthetic frameworks, implant abutments, and orthodontic appliances. However, PEEK's inert surface poses challenges for bonding with resin cements.
Thermocycling tests are crucial for evaluating new materials like PEEK as they simulate the thermal stresses of the oral environment in vitro. These tests provide valuable insights into the durability and reliability of PEEK-based restorations, guiding clinicians in selecting appropriate materials and bonding protocols for functional and long-lasting restorations. This study aimed to evaluate and compare the effect of thermocycling on the shear bond strength of PEEK with two resin cements. 5

Methodology
The study involved forty PEEK disk-shaped specimens, each with dimensions of 10 × 3 mm. These specimens were fabricated through milling to ensure uniform size and shape. To improve bonding strength, the surface of each PEEK specimen underwent alumina air blasting using 50 μm alumina particles for 10 seconds.
The specimens were randomly divided into two equal groups, Group A and Group B, with 20 specimens in each.

- **Group A:** PEEK specimens were bonded with RelyX Unicem (3M ESPE). The adhesive was allowed to self-cure for 2 minutes, followed by light curing for 20 seconds.
- **Group B:** PEEK specimens were bonded with G-CEM LinkAce. This adhesive was light-cured for 20 seconds and then allowed to self-cure for 4 minutes.

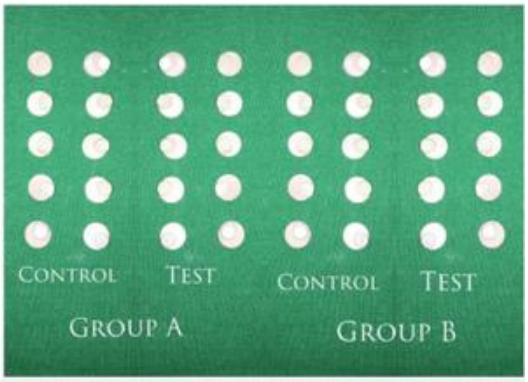

Figure 3: PEEK disc cemented with Rely X Unicem (group A) and Gcem Linkace (group B).

A cylindrical putty mould index (5 × 4 mm) was used to standardize the application of the resin cements.
Thermocycling Procedure: Specimens from both groups underwent thermocycling. The thermocycling process involved 500 cycles at temperatures of 5°C and 55°C. Distilled water was used to maintain these temperatures in the thermal baths.
Shear Bond Strength Measurement: The shear bond strengths for all specimens were calculated using a universal testing machine. Force was applied at a speed of 1 mm/min until failure. The shear bond strength was determined by dividing the maximum load applied by the cross-sectional area of the specimen.
Failure Type Analysis: Scanning electron microscopy (SEM) was conducted to determine the type of failure that occurred at the bond interface. The fractured interfaces were observed under a scanning electron microscope at magnifications of 5.00 kV x 500 SE at 100 m and 5.00 kV x 150 SE at 300 m. Specimens were treated with argon deposition for 30 seconds before SEM analysis. A calibrated examiner, blinded to the group allocation and treatment method, assessed the specimens to determine the failure type. Failure types were classified as:

- **Adhesive failure:** No resin cement remnants on the PEEK surface.
- **Combined adhesive/cohesive failure (mixed failure):** Partial resin cement remnants on PEEK, with exposed PEEK surface.
- **Cohesive failure:** Failure entirely within the resin cement.

Statistical Analysis: The shear bond strength data was statistically analyzed to evaluate the effects of thermocycling and to compare the performance of the two resin cements. A comparative evaluation was performed to assess the impact of thermocycling on bond durability and the relative performance of the adhesive systems.

Results and Observation
The study revealed significant differences in shear bond strength between the control and thermocycled (test) groups for both resin cements.

RelyX Unicem Cement:

- In the control group, the mean shear bond strength for RelyX Unicem cement was 3.91 ± 0.38 MPa.
- In the test group (after thermocycling), the mean shear bond strength was 1.87 ± 0.38 MPa.
- A maximum shear bond strength of 4.33 MPa and a minimum of 3.31 MPa were observed in the control group.
- In the test group, the maximum shear bond strength was 2.342 MPa, and the minimum was 1.018 MPa.
- RelyX Unicem test group showed the maximum loss of shear bond strength after thermocycling.

G-CEM Linkace Cement:

- In the control group, the mean shear bond strength for G-CEM Linkace cement was 5.07 ± 0.95 MPa.
- In the test group (after thermocycling), the mean shear bond strength was 4.01 ± 0.68 MPa.
- A 20.9% reduction in shear bond strength was noted for G-CEM Linkace after thermocycling.
- The maximum shear bond strength for G-CEM Linkace in the control group was 5.98 MPa, and the minimum was 3.65 MPa.
- In the test group, the maximum shear bond strength was 4.78 MPa, and the minimum was 2.648 MPa.
- G-CEM Linkace test group demonstrated the minimum loss of shear bond strength after thermocycling and showed maximum shear bond strength post-thermocycling compared to RelyX Unicem.

Overall Comparison of Cements Post-Thermocycling: After thermocycling, G-CEM Linkage consistently showed higher shear bond strength compared to RelyX Unicem (p<0.005).

Table 1: Comparison between shear bond strength of two cements after thermocycling using paired t test.

Test	Groups	N	Minimum	Maximum	Mean	SD	P-value	53.4 % increased
	RelyX	10	1.02	2.34	1.87	0.43	<0.0001	
	G-CEM	10	2.65	4.78	4.01	0.68		

Scanning Electron Microscope (SEM) Observations: SEM evaluation of the fractured interfaces revealed peeling/separation on the substrates for both types of specimens. The fracture was consistently observed at the interface between the adhesive and the substrate. Minimal to no adhesive residue was observed on the PEEK surface of the specimens, leading to the conclusion that an adhesive type of failure occurred for both RelyX Unicem and G-CEM Linkace cements. This finding aligns with observations from other studies like Scherrer et al (2008) ,Kim et al (2016) and Nikita et al (2022),

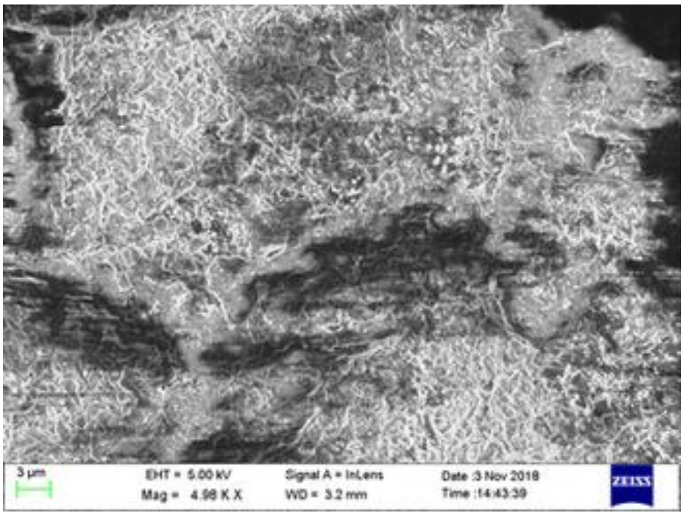

Figure 1 SEM image demonstrating adhesive failure in specimens cemented with using RelyX unicem

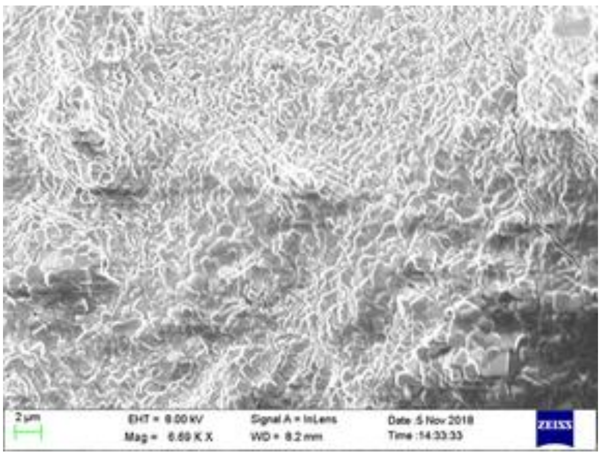

Figure 2 : SEM image demonstrating adhesive failure in specimens cemented with using G-CEM LinkAce resin cement.

Discussion

The integration of PEEK in dentistry represents a significant advancement due to its exceptional mechanical properties, biocompatibility, and chemical resistance. Its applications span various prosthetic solutions, including provisional abutments, implants, and components in removable dental prostheses. The present study focused on the crucial aspect of PEEK's bond strength with resin cements, particularly under thermocycling conditions, which mimic the thermal stresses experienced in the oral environment.6-9

Our findings indicate a significant reduction in shear bond strength for both RelyX Unicem and G-CEM Linkace after thermocycling. This is consistent with previous research highlighting that thermocycling can lead to degradation of bond strength over time, affecting the longevity of dental restorations. Researchers have used thermocycling to evaluate several properties of PEEK. In a recent study conducted by Priya et al (2023) assessed the flexural strength (performance) of PEEK in comparison with auto-polymerised and CAD/CAM milled poly methyl methacrylate resins (PMMA). These researchers used the 'three-point bend test' for determining both the long and the short-term mechanical stability of the dental materials.

Commenting on the process of thermocycling Priya et al, remarked that 10000 cycles of the thermocycling process represents one year of clinical usage of restorative material and hence can indirectly give a clue to researcher about the effect of several innate physical and chemical properties of oral niche on the restorative material.10

Salem et al (2023) evaluated surface hardness and flexure strength of acetyl resin and PEEK using 5000 cycles of thermocycling. They concluded that 5000 cycle represented of six months of clinical use in oral cavity.11

The observed decrease in bond strength underscores the importance of evaluating dental materials under conditions that simulate the dynamic oral environment to predict their long-term clinical performance. Thermocycling tests provide valuable insights into the durability and reliability of PEEK-based restorations, ultimately contributing to the advancement of dental materials and techniques. In accordance to the protocol suggested by Nikitha et al (2022) specimens from both the groups (group A and group B) were subjected to thermocycling for 20 s at 5°C and 55°C with a 5-s interval between each cycle. Each specimen was subjected to 500 cycles. ISO TR 11405 (1994) – Number of cycles: 500 cycles Temperature: 5–55° Dwell time ≥20 s. 12

The study protocol included holding the specimens in a metal holder in a UTM. In this study, the load was applied parallel to the long axis of the specimen through a wedge at the PEEK resin cement interface. The maximum load at failure was recorded by the system's software and the strengths of the shear bond [MPa] were obtained by dividing the failure load [N] with the total area of bonding.12 The ability of G-CEM Linkace to better maintain its bond strength with PEEK under thermal stress is a crucial factor for clinical success, as it implies a more stable and potentially longer-lasting restoration. This outcome is particularly relevant for clinicians selecting appropriate materials and bonding protocols to ensure functional and durable restorations.

A key finding was that G-CEM Linkace exhibited superior shear bond strength after thermocycling compared to RelyX Unicem. This suggests that G-CEM Linkace may offer greater bond durability with PEEK when subjected to thermal fluctuations. This aligns with previous studies that have comparatively evaluated resin cements, showing variations in performance under different environmental conditions.

In spite of several studies have investigated the bond strength of many interfaces, till recent date there is no standardized procedure that can be recommended for the assessing bond strength.13

The failure mode analysis can provide invaluable insights for determining the flaws in various testing techniques and therefore will aid reliably improving and thus providing the reliable results with actual strength of junction of adhesion.15

Wang et al. (2018) classified failure types into 3 i.e. the adhesive type, the cohesive type and the mixed categories type. This classification provides insights into how different bonding mechanisms can affect the overall strength and durability of restorations.14

The SEM analysis consistently revealed an adhesive type of failure for both cements, meaning the fracture occurred predominantly at the interface between the PEEK surface and the resin cement. The absence or minimal presence of resin cement remnants on the PEEK surface supports this observation. This adhesive failure pattern suggests that the weakest link in the bonded assembly was the interface itself, rather than cohesive failure within the resin cement or the PEEK material. This highlights the ongoing challenge of achieving strong and durable adhesion to PEEK's inert surface. While surface treatments like alumina air blasting were employed to enhance roughness and bonding, the adhesive failure mode indicates a need for further optimization of surface modification techniques or bonding agents to achieve a more robust interface. Researchers have explored various surface treatments, such as plasma treatment and sandblasting, to increase surface roughness and enhance bond strength to PEEK. Despite these advancements, the optimal surface treatment for PEEK remains an area for further investigation.

The properties of PEEK, including its mechanical resemblance to human cortical bone and dentin, its lightweight nature, and resistance to corrosion, make it an attractive alternative to conventional metallic dental materials. Its radiolucency is also advantageous for post-surgical imaging. However, its inherent radiolucency can be addressed by adding radio-opacifiers like barium sulfate when visibility in imaging is required. Despite its promising attributes, challenges like its low surface energy, which can hinder cellular adherence and osseointegration, need to be addressed through surface modifications. Many research studies have explored various surface modification techniques to improve wettability and bioactivity, such as oxygen plasma etching, laser treatment, and hydroxyapatite coating.

This study adds to the growing body of literature on PEEK in dentistry, specifically addressing its bond strength under simulated oral conditions. The findings provide valuable information for the selection of resin cements when bonding to PEEK, emphasizing the importance of considering long-term durability influenced by thermocycling. Future research could focus on novel surface treatment strategies and resin cement compositions to further enhance the bond strength and minimize the impact of thermal stresses on PEEK restorations.

Conclusion

Based on the findings of this study, thermocycling significantly impacts the shear bond strength of PEEK to resin cements, mimicking the effects of the oral environment in vitro. Among the two resin cements evaluated, G-CEM LinkAce demonstrated superior shear bond strength after thermocycling compared to RelyX Unicem. Scanning electron microscopic observations consistently indicated an adhesive type of failure on the PEEK surfaces for both groups, suggesting that the interface between the PEEK and the resin cement remains the critical area for bond integrity. These results highlight the importance of careful material selection and continued research into optimizing bonding protocols for PEEK in dental restorative applications to ensure durable and long-lasting restorations.

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