Journal of Oral Biosciences ∎ (∎∎∎∎) ∎∎∎–∎∎∎

Contents lists available at ScienceDirect

Journal of Oral Biosciences

journal homepage: www.elsevier.com/locate/job

Original Article

Evaluation of shear bond strength between PEEK and resin-based luting material

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ARTICLE INFO

Article history:
Received 14 June 2017
Received in revised form
29 August 2017
Accepted 1 September 2017

Keywords:
PEEK
Resin-based luting material
Shear bond strength

ABSTRACT

Objectives: The purpose of this study was to evaluate the bond strength of polyetheretherketone (PEEK) to resin-based luting material.
Methods: Eighty PEEK specimens were randomly divided into two groups ($n = 40$/group): no treatment and sandblasting. Each of the 40 specimens of dental gold-silver-palladium alloy (PALLAZ12-n; Yamamoto Precious Metal Co., Ltd., Osaka, Japan), zirconia (Aadva Zirconia; GC, Tokyo, Japan), and hybrid composite resin (CERASMART; GC, Tokyo, Japan) was used as a control material for PEEK. Each group was divided into four subgroups ($n = 10$) for the different resin-based luting materials: Panavia® V5 (Kuraray Medical, Tokyo, Japan), RelyX™ Ultimate Resin Cement (3M ESPE, St Paul, MN, USA), G-CEM Link Force (GC, Tokyo, Japan), and Super-Bond C&B (Sun Medical, Siga, Japan). The resin-based luting materials were bonded onto the specimens. All specimens were stored in distilled water at 37 °C for 24 h. Bond strength was measured with a shear test, and failure modes were assessed by stereomicroscopy. The surfaces were observed by scanning electron microscopy after the various pretreatments.
Results: Compared with the control group, the PEEK group showed a significantly lower ($p < 0.05$) shear bond strength for most of the specimens. Among PEEK groups, the most frequent failure mode was adhesive failure between the material and the resin-based luting material.
Conclusions: This study found that the bond strength between PEEK and resin-based luting materials was not adequate for clinical use of PEEK.

1. Introduction

Polyetheretherketone (PEEK) has received attention as a new dental material [1]. PEEK has superior mechanical strength, heat resistance, and chemical resistance and can be easily worked. It has a structure in which the benzene ring is linked with an ether or ketone bond [2]. PEEK is used industrially in parts that operate in harsh environments requiring resistance to heat and chemicals, such as in automobiles, aircraft, and semiconductors [3–6]. PEEK is radiolucent and is, therefore, compatible with imaging technologies such as computed tomography, magnetic resonance imaging, and radiography [7,8]. Applications of PEEK extend from the aircraft and automobile industries to biomaterials used in medical implants [9]. Currently, PEEK is being introduced for use in dental applications such as transitional abutments [10], healing abutments [11], and dental clasps [12]. Because of its nonmetallic color, low weight, and high strength, PEEK can also be used as an alternative rigid material in partial dentures and fixed dental prostheses [13,14]. As no plasticizer or flame retardant is added to PEEK in the polymerization process, the polymer is of high purity, and biocompatible prostheses have been developed utilizing this property. When used in crowns and fixed dental prostheses for the posterior teeth, PEEK offers advantages in resisting the strength and attrition of the opposing teeth.

However, PEEK has low adhesion to resin-based luting material [15]. The bond strength of PEEK to composite resin is low because PEEK resists its chemical inertness, low surface energy, and surface modification [16]. Adhesion between resin-based luting materials and materials used in crowns and fixed dental prostheses is an indispensable property of dental restorative materials [17]. Adhesive properties, which are important for the stability of a prosthesis in dental applications, are influenced by the nature of the resin-based luting material [18]. Currently, there is no established protocol for cementing crowns and fixed dental prostheses using PEEK. Although there have been several reports evaluating the bond strength between PEEK and resin-based luting material [18,19], only one or two resin-based luting materials were used in

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http://dx.doi.org/10.1016/j.job.2017.09.003
1349-0079/

these reports. To the best of our knowledge, the bond strengths between PEEK and the newly improved resin-based luting materials used in this study have not previously been evaluated. The purpose of this study was to evaluate the bond strength of PEEK to a variety of resin-based luting materials. The specific aim of the study was to determine the adhesive strength between PEEK and several types of resin-based luting materials, and to compare the PEEK bonding strengths with those of conventional crown and fixed dental prosthesis materials.

2. Materials and methods

2.1. Specimen preparation and surface treatment

The composition and details of the materials used in this study are shown in Table 1. Each of the 80 cylindrical PEEK specimens (diameter, 6 mm; thickness, 4 mm) and disk-like PEEK specimens (diameter, 10 mm; thickness, 3 mm) was sectioned from a PEEK disk (Vestakeep® PEEK, tooth-colored; Daical-Evonik, Tokyo, Japan) using a low-speed precision diamond saw cutting machine under water coolant. Dental gold-silver-palladium alloy (PALLAZ12-n; Yamamoto Precious Metal Co., Ltd., Osaka, Japan), zirconia (Aadva Zirconia; GC, Tokyo, Japan), and hybrid composite resin (CERASMART; GC, Tokyo, Japan) were used as control materials for PEEK. Each of the 40 cylindrical specimens was embedded in an autopolymerizing acrylic resin (Tray Resin II; Shofu, Kyoto, Japan) and polished with a grinding and polishing machine (MetaServ; Buehler, London, UK) under running water at 150 rotations/min using 120-, 400-, and 600-grit rotating silica carbide papers (Abrasive Paper Disc; Sankyo-Rikagaku, Saitama, Japan) for 30 s. Subsequently, all the specimens were ultrasonically cleaned (UT-206; Sharp, Osaka, Japan) in ethanol and distilled water for 5 min and air dried.

Eighty PEEK specimens were randomly divided into two test groups according to the surface treatment protocol. One protocol was no surface pretreatment (no treatment), and the other protocol was airborne-particle abrasion with 50-μm alumina oxide particles at 0.1 MPa at 10-mm distance for 10 s (sandblasting).

2.2. Bonding procedure

Each specimen group was divided into four subgroups ($n = 10$) depending upon the nature of the cement used: Panavia® V5 (Kuraray Medical, Tokyo, Japan), RelyX^{TM} Ultimate Resin Cement (3M ESPE, St Paul, MN, USA), G-CEM Link Force (GC, Tokyo, Japan), and Super-Bond C&B (Sun Medical, Siga, Japan) (Table 2). A piece of double-sided polyethylene adhesive tape with circular holes (diameter, 4.0 mm; thickness, 0.1 mm) was placed on the specimen to define the bonding area.

PEEK was not primed with a primer treating agent, as valid priming has not been elucidated. However, the surfaces of the control materials were primed in accordance with the manufacturer's instructions for each cement. When Panavia®V5 was used, Clearfil® Ceramic Primer Plus (Kuraray Medical) was applied and air dried. When RelyX^{TM} Ultimate Resin Cement was used, Scotchbond Universal (3M ESPE) was applied and air dried. When G-CEM Link Force was used, G-Multi Primer (GC) was applied and air dried. When Super-Bond C&B was used, V-Primer (Sun Medical) was applied to PALLAZ 12-n, and Super-Bond Universal Ceramic Primer (Sun Medical) was applied to Aadva Zirconia and CERASMART, and dried with air. Next, a constant load was applied to the cylindrical specimen to enable its adhesion to the resin-based luting material (Fig. 1). Excess cement was removed from the bonding edge with a disposable brush. Following adhesion to the composite resin-based adhesive cement, the specimens were light-cured under a G-Light Primal Plus light source (GC) (2000 mW/cm^2) for 40 s and then kept at room temperature for 30 min, after which all specimens were stored in distilled water at 37 °C for 24 h.

Table 1
Materials tested in this study.

Materials	Component	Manufacturer
PEEK (VESTAKEEP®, tooth-colored)	Polyetheretherketone, titanium dioxide	Daicel-EVONIK
PALLAZ12-n	Gold (12%), palladium (20%), silver (49.5%), copper (16.9%)	Yamamoto Precious Metal
Aadva Zirconia (ST14)	Zirconium dioxide, yttrium oxide, aluminium oxide	GC
CERASMART (A3LT)	UDMA, Bis-MEPP, methacrylate (Multifunctional), pulverizing silica, barium glass powder	GC

UDMA: Urethane(meth)acrylate, Bis-MEPP: Bisphenol-A-ethoxylate(2)dimethacrylate.

2.3. Measurement of shear bond strength

Shear bond strength was measured with a universal testing machine (AG-X Plus; Shimadzu, Kyoto, Japan). The specimens were fixed with a special fixture, and the loading piston was brought close to the bonding surface. The load was applied with a crosshead speed of 1 mm/min with the bonding surface parallel to the loading piston (Fig. 2). The maximum load before debonding was measured. The bonding strength was calculated using the following formula: force to failure/bonding area (MPa = N/mm^2).

2.4. Analysis of failure mode

Four failure modes were defined as follows: (a) adhesive failure mode between materials and resin-based luting materials, (b) cohesive failure mode within resin-based luting materials, (c) cohesive failure mode within materials, and (d) mixed failure mode with both cohesive and adhesive failures. The debonded surface was examined under a digital camera (MR-14EX II; Canon Production Printing Systems, Tokyo, Japan) to evaluate the failure modes of the specimens.

2.5. Scanning electron microscopy analyses

The fracture surface of a specimen was observed with a scanning electron microscope (SEM) after sputter-coating with gold at 2 kV with a working distance of 5.0–6.0 mm.

2.6. Statistical analysis

Statistical analyses were performed to compare shear bond strength for different materials and resin-based luting materials by two-way analysis of variance, followed by the Tukey's multiple comparison post hoc test. A value of $p < 0.05$ was considered to indicate statistical significance. Statistical analyses were conducted with SPSS Statistics version 19 for Windows (IBM Corporation, Armonk, NY, USA).

3. Results

The means and standard deviations of the shear bond strength of specimens with differently treated surfaces are shown in Table 3. Shear bond strength ranged from 47.9 ± 7.2 MPa (combination of Aadva Zirconia and RelyX^{TM} Ultimate Resin Cement) to 0.6 ± 0.5 MPa (combination of PEEK sandblasted treatment and