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Research Article

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Assessment of the Binding Force of Different Peek Binding Agents

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Abstract

Objective: *The purpose of the study was to compare the bonding strength of three bonding agents on polyetheretherketone (PEEK) surfaces against lithium disilicate (LS2).*

Material and methods: *40 PEEK specimens (Techno Med Mineral, Zirkonzahn) and 40 LS2 specimens (IPS e.max Press MT, Ivoclar Vivadent) with a rectangular active area of a 9 mm x 8 mm were used. They were treated by sandblasting with Al2O3, 110 µm, 3 bars, and divided into 4 groups (i) control group with no treatment or subsequently combined with the following conditioning agents: (ii) Zirkonzahn Composite Bonding Liquid for Tecno Med Mineral (Zirkonzahn), (iii) Visio. link (Bredent), (iv) Monobond Plus (Ivoclar Vivadent). All specimens were cemented with Panavia V5, and the two surfaces GFR-PEEK and LS2 were stored in bidi stilled water at 37ºC for 24 hours. Pressure tensile bond strength tests (TBS) were performed. Statistical analysis was performed with a 1-factor ANOVA test and Bonferroni post hoc analysis.*

Results: *The use of Zirkonzahn and Visio link bonding agents resulted in significantly higher TBS than all other methods. Between Zirkonzahn and Visio. link, there was no statistically significant difference.*

Conclusion: *The use of bonding agents containing methyl methacrylate (MMA), pen- taerythritol triacrylate (PETIA) and dimethacrylates, offers the best adhesive performance for PEEK.*

Keywords: PEEK, GFR-PEEK, CAD/CAM, Adhesion, Tensile Strength.

Introduction

Polyetheretherketone (PEEK) is a high-temperature polymer of the polyaryletherketone family, consisting of an aromatic molecular chain, interconnected by ketone and ether functional groups [1, 2]. The choice of materials used in the mouth must be biocompatible or biotolerable. In the case of PEEK, it's a biocompatible material with low adherence affinity for dentobacterial plaque, with good aesthetics, with some degree of reduce in discoloration and characteristics similar to dental structure and bone [3]. Currently, medicine is oriented towards body-like and metal-free materials, to avoid the risk of oral galvanism, among others [4]. It has been shown that the "metal ions released in the mouth can affect cellular structure, alter cell function (membrane permeability and enzymatic activity), promote immune and inflammatory alterations, cause allergic effects, and alter genetic material" [5]. This can also occur when doing dental work such as: fixed prosthesis, removable prosthesis, prosthesis on implants, as well as orthodontic devices [6].

In medicine, PEEK has proven to be an excellent substitute for titanium (Ti) in orthopedic applications, due to its excellent mechanical, chemical and thermal properties, as determined by its high strength combined with adequate milling and grinding properties [1, 7]. Although pure (unfilled) polyaromatic polymers have a low Young's modulus of 3.6 GPa, modifications were made by reinforcing with carbon fiber (CFR-PEEK) increasing it to 18 GPa and glass fiber (GFR-PEEK) increasing to 12 GPa, resembling the me- chemical properties of bone (cortical bone 18 GPa), causing less stress to the material [1, 8]. PEEK could represent a viable biomaterial, not only capable of replacing conventional polymers, but also metals, alloys, and ceramics in the field of dentistry. It's starting to be used for implant bars, temporary abutments, healing abutments, and bracket material in the field of removable dental prostheses [9, 10].

In general, block materials for computer-aided design (CAD)/computer-aided manufacturing (CAM) are being used more commonly in daily clinical practice, they have better mechanical and even optical properties than conventional materials and are easier to use [10]. Today the vast majority of dental materials are found in monolithic disc presentations for CAD/CAM use, increasing fracture resistance; one of these materials is PEEK.

Being a monolithic material, it doesn't have aesthetic properties since it's a monochromatic material. It requires a coating due to its low translucency and grayish pigmentation. However, "PEEK has an inert hydrophobic surface and low surface free energy", which results in better adhesion properties [11, 12, 20-22]. The adhesive properties are important for stability of the prosthetic and implant areas. There is more documentation on resin-based adhesive systems for PEEK [12, 20-22].

Micromechanical retention can be achieved by altering the surface texture using me- chemical or chemical means, resulting in an enlarged surface area with microscopically small cuts in the altered surface. The cements or adhesives applied can flow into these cuts and, when hardened, interlock to provide stable retention [13].

Keeping this concept in mind, we had the idea of pre-treating the PEEK surface, chemically and/or mechanically, and applying a bonding agent, to increase retention levels on the substrate.

Industrially, elastomers are normally joined to PEEK by means of mechanical or chemical treatments (conventional abrasives, acid etching, engraving with laser or plasma techniques) to create roughness on the surface of the material before using an epoxy adhesive [14].

Currently, there is no standard protocol for the PEEK surface. Although there have been several reports evaluating the bond strength between PEEK and resin-based fixation materials.

Two things must be considered: (1) the PEEK surface must be modified, which is considered as a pre-treatment and (2) application of an adhesive system for conditioning, which will allow chemical interactions with the cement material [11, 15, 20-22].

The mechanical pre-treatments on the PEEK surface can be: sandblasting with Al2O3, tribochemical silica coating, etching with sulfuric acid, different plasma treatments and even the use of laser (Er: YAG, is the most common for dental use). The best results are obtained by sandblasting with Al2O3. Notably, the particle size and the pressure used play an important role in the roughness of the substrate, and therefore, an average of 110 µm and 2 to 3 bars has been established, using an inclination of 45º at a distance of 10 mm [2]. Conversely, use chemical treatment with 98% sulfuric acid for 60 seconds to help resin penetration [10, 16], or piranha solution consisting of sulfuric acid (H2SO4) and hydrogen peroxide (H2O2), for 30 seconds. The use of sulfuric acid at a concentration of 98% is extremely corrosive to health, and therefore is not clinically feasible [12].

Chemical treatment is known to further enhance bonding when applying coupling agents, which are bifunctional molecules that allow a chemical reaction between the inorganic ceramic and the organic resin cement. The most commonly used coupling agents are organosilanes, such as γ-methacryloxypropyltrimethoxysilane. Its methoxy group (-OCH3) hydrolyzes in the presence of water to a silanol group (-Si-OH), which can subsequently join with the hydroxyl groups on the ceramic surface, forming a siloxane bond (-Si-O-Si-). The other functional group, a methacrylate group, is capable of polymerizing with organic resin, forming a covalent bond [16].

Silane also increases the hydrophobicity and wettability of the treated surface, thus improving its interaction with hydrophobic resin cements [16, 17]. The use of a specific adhesive containing methyl methacrylate (MMA), pentaerythritol triacrylate (PETIA) and dimethacrylates has been shown to offer the best adhesive performance when binding to PEEK [18].

The role of photopolymerization is important in terms of dental bonding because the photoinitiators have to be activated so that the adhesives or cements can show the most optimal result in terms of bonding. Acrylphosphinoxide photoinitiator is a system commonly used for dental materials and shows maximum absorption at a wavelength of 380 nm. The results and technical details show that this wavelength range is only pro- vided by halogen LCUs, but not by LED-LCUs [19].

Camphorquinone is another well-established photo initiator often used in dentistry. Com- pared to acrylphosphinoxide, camphorquinone shows maximum absorption at higher wavelengths (468 nm). This wavelength range, in turn, is provided by LED-LCUs [19]. Due to the different activation wavelengths of the photo initiators, the photo-curing lamps must have a wide range of wavelengths, continuous and/or ascending.

Conditioners for adhesive systems all contain MMA. Composite Bonding Liquid (Zirkonzahn), visio. link (Bredent group) and Monobond Plus (Ivoclar Vi-vadent) are also report- ed to have other components that increase bonding. Therefore, the purpose of this in vitro study was to compare the capacity of the different bonding agents, and determine which one favors adhesion to GFR-PEEK, as assessed by pressure shear tests.

The researcher's hypothesis was that GFR-PEEK surfaces conditioned with adhesive systems containing of one or more monomers from the PETIA, MMA and dimethacrylate groups have higher tensile strength compared to simply treated surfaces.

Justification

With this in vitro study, the aim was to identify the most optimal bonding agent for PEEK structures, which would increase adhesion to a resinous cement, favoring, not only me- chemical, but also chemical adhesion. Resulting in a longer lasting coating of the PEEK structure.

Objectives

General

To compare the adhesion efficiency of three bonding agents on PEEK surfaces.

Specific

Prepare the experimentation models

1. Apply the different binding agents to each group.
2. Prepare the adhesion phase.
3. Compare the adhesive effectiveness of the groups by assessing tensile strength.
4. Observe surfaces after strength testing, using scanning electron microscope.

Hypothesis

Alternative

The use of pre-treatment plus a chemical conditioner for resinous bon- ding systems containing at least one or two of the following monomers (MMA, PETIA and dimethacrylates) will favor the highest micro tensile strength in the PEEK substrate. NULL: Simple mechanical pre-treatment (sandblasting with Al2O3) is sufficient to gene- rate good adhesion.

Material and Methods

Study Type and Design

EXPERIMENTAL, PROSPECTIVE, TRANSVERSAL, ANALYTIC, COMPARATIVE.

Specimen Preparation and Treatment

40 GFR-PEEK trapezoid shaped specimens were constructed using 1 disc of Tecno Med Mineral, (tooth color, Zikonzahn, Bruneck, Italy) measuring 13 mm x 9 mm at the base, with a 45º slope, active surface of 9 mm x 8 mm, 4.5 mm thick. Plus 40 LS2 specimens (IPS e. max Press MT, Ivoclar Vivadent) with the same measurements.

They were divided into 4 groups, for each conditioning treatment:

- Group A (n= 10): Pre-treatment with Al2O3, 110 µm, 3 bars (control group)
- Group B (n= 10): Pre-treatment with Al2O3, 110 µm, 3 bars + bonding agent
  Composite Bonding Liquid (Zirkonzahn)
- Group C (n= 10): Pre-treatment with Al2O3, 110 µm, 3 bars + bonding agent Visio. link (Bredent)
- Group D (n= 10): Pre-treatment with Al2O3, 110 µm, 3 bars + bonding agent Monobond Plus (Ivoclar Vivadent)

Material	Product name	Manufacturer	Composition	Lot number
Treatment	Aire abrasivo (abrasive air)		110 µm Al2O3	
Bonding agent	Composite Bonding Liquid Visio.link	Zirkonzanh; Brunico, Italy Bredent; Seden, Germany	110 µm Al2O3 3 bars MMA 50-75% MMA, PETIA, dimethacrylates, pho- toinitiators	PC0002 VLPMMA10
	Monobond Plus	voclar Vivadent; Schaan, Lichtenstein	Silane methacrylate, Phosphoric Acid methacrylate, Sulfur methacrylate	Z02FZ2
GFR - PEEK	Tecno Med Mineral	Zirkonzanh; Brunico, Italy		
Lithium disilicate	IPS e.max Press	Ivoclar Vivadent; Schaan, Lichtenstein	Lithium disilicate glass ceramic ingot	

Material	**Product name**	**Manufacturer**	**Composition**	**Lot number**
Cement	Panavia V5	Kuraray Noritake Dental Inc; Sakazu, Japan	(1) Paste A	
			•Diglycidylmethacrylate bisphenol A • Triethylene glycol dimethacrylate • Hydrophobic aromatic dimethacrylate • Hydrophilic aliphatic dimethacryla • Initiators • Accelerator • Silanated barium glass filling • Silanized fluoroaluminosilicate glass filler • Colloidal silicon	

Each PEEK sample, after having been sandblasted from a distance of 10 mm for 10 seconds, was cleaned in alcohol with ultrasound for 5 min and dried with oil-free pressurized air. Then the conditioners were added, following the manufacturer's instructions. Once the surface was pre-conditioned, the bonding agent (Composite Bonding Liquid, Visio. link, or Monobond Plus) was added, with the exception of the control group. Finally, Panavia V5 cement was added following the manufacturer's instructions.

Each LS2 sample was washed with pressurized water, treated with 37% phosphoric acid by rubbing for 60 seconds, washed in an ultrasonic bath with alcohol for 5 minutes and treated with silane by rubbing for 1 min and letting it volatilize for 5 minutes. Finally, the samples were conditioned using chemical etching with 5% hydrofluoric acid for 20 seconds.

Both surfaces, PEEK and LS2 with a PANAVIA V5 interface, were photopolymerized with a VALO Grand lamp at extra power, equivalent to 3,200 mW/cm2.

Dislodgement tests were performed with a tensile testing machine; the resistance to dislodgement was expressed in MPa. Ten samples were taken from each group after dislodgement and analyzed with a scanning electron microscope to observe cement penetration and adhesive or cohesive failure.

Tests were carried out on the Instron Model 4465 Pressure Shear Tester.

Characterization Variable	Indicator (dimension)	Final Value	Type of variable
Protocols	Protocol type	Al2O3 Protocols Protocol type Al2O3 Qualitative, Nominal	Qualitative, Nominal
Adhesion	Dislodgement force	MPa	Quantitative, Ratio / Numerical, Continuous
Etching	Scanning electron microscope	µm	Quantitative, Ratio / Numerical, Continuous
Adhesive failure	Cement on PEEK	Yes	Qualitative
	Surface	No	

Statistical Analysis

Numerical variables were summarized as means, standard deviation and 95% confidence interval. The qualitative variable, failure or no failure, was summarized in absolute frequency or relative frequency. The differences in the numerical variables between the four groups were carried out by one-factor ANOVA followed by a Bonferroni post hoc test. The differences in the percentage of adhesive failure between the groups, was determine using a Chi square test. The level of statistical significance was $p< .05$.

The analyzes were done with the statistical package SPSS version 21.0.

Ethical Aspects of the Study

The protocol was submitted for authorization by the graduate committee, which reviewed the methodology used.

Results

Table 1 compares the adhesion strength of the 40 specimens arranged into four groups. When comparing the groups, there was a statistically significant difference (ANOVA= 14.1; $p < .0001$). Bonferroni's post hoc analysis showed that Composite Bonding Liquid had higher bond strength than the control group and monobond plus ($p< .001$). Similarly, Visio. link also showed higher bond strength than the control group and Monobond plus ($p= .001$). There was no statistically significant difference between Composite Bonding Liquid and Visio. link.

Table 1: Comparison of adhesion strength to GFR-PEEK.

	Average MPa	Mean	Confidence interval 95%
Control group	.87	1.01	.14 – 1.60
Monobond plus	.93	.82	.35 – 1.52
Composite Bonding Liquid	3.97	1.26	3.06 – 4.87
Visio.link	2.34	1.98	1.70 – 2.98

Table 2: Frequency of adhesive failure

	Adhesive failure Number
Grupo control (10)	10
Monobond plus (10)	10
Composite Bonding Liquid (10)	3
Visio.link (10)	3

Discussion

PEEK is one of the most widely used high-performance polymers in the dental field [20]. Currently, the use of materials similar to the human body is more common, due to their biocompatibility [1]. PEEK is used due to its advantageous mechanical properties: resistance to heat and solvents, excellent electrical insulation and robust resistance to wear and fatigue [1, 21]. Although it has good mechanical properties, its inert surface requires mechanical and/or chemical surface conditioning due to poor adherence to coating resin materials [3, 21]. Air abrasion increases surface roughness while removing organic contaminants from the surface, creating a fresh and active surface layer [22]. It also pro- motes micromechanical embedding of polymer-based dental materials and allows the bonding agent to better penetrate them, resulting in micromechanical retention and, presumably, greater surface bonding capacity [10, 12].

The combination of mechanical and chemical pretreatments (eg, air abrasion, piranha solution, or sulfuric acid associated with Visio. link®) improved TBS even after thermo- cycling. Similar results have been reported in previous studies [21].

Our results reinforce the idea that performing micro-retentions on the PEEK surface will help the penetration and binding of bonding agents containing MMA, PETIA and/or dimethacrylates (eg. Visio. link and Composite Bonding Liquid) to improve the bonding strength to PEEK [18, 21, 23].

Conclusion

The bond strength between PEEK and a resin-based cement is significantly increased by performing micro-retentions and using bonding agents that contain at least one or two of the following monomers: MMA, PETIA and dimethacrylates.

More studies are required.

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