The Effect of Surface Pretreatment and Water Storage on the Bonding Strength of a Resin Composite Cement to Modified PEEK

Keywords

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ABSTRACT

The bonding quality of bonding to polyether ether ketone (PEEK) after different surface treatments and adhesive regimens was assessed through shear bond strength. Materials and Methods: Sixty modified PEEK disks were cut out of BioHPP round blanks using CAD-CAM procedures. Disks were subjected to the following surface pretreatments: (A) Sandblasting with alumina (Rocatec) and application of adhesive bonding agent visiolink (control group) (B) Sandblasting with silica-modified alumina (Cojet), application of silane agent Espe Sil, followed by application of adhesive bonding agent Visio-Bond (C) Sandblasting with silica-modified alumina (Cojet), application of silane containing primer-adhesive Clearfil Ceramic Primer, followed by application of adhesive bonding agent visiolink. A dual-curing resin composite cement (combolign) was luted to all treated surfaces. Each group was further divided to subgroups of 10 specimens which were stored in distilled water at 37°C for 150 days without further thermocycling. Specimens were then submitted to shear bond strength testing. Results: Group (B) was statistically significant different from group (A) (control group). Water storage condition had no significant influence on final bond strength. Conclusions: Use of different conditioning protocols had a significant effect on the final bond strength of composite resin cement to PEEK surface. Water storage did not significantly influence bonding.

INTRODUCTION

Polyether ether ketone (PEEK) is a high-melting thermoplastic polymer material derived from the vast family of polyaryletherketones.

The exceptional mechanical properties of PEEK, especially its modulus of elasticity (similar to cortical bone and dentin) have recently made it popular for dental applications. PEEK has initially been used as a dental implant material or for the fabrication of provisional abutments. [1-4] It has also been used for other various prosthodontics applications, fixed or removable.[5-11]

PEEK can be either pressed or milled. During heat pressing, pellets or granules are pressed using a special vacuum-pressing device in a dental laboratory.[12,13] With milling, prefabricated PEEK blanks are milled using CAD-CAM systems.[12,13]

Modified PEEK forms have been created with the addition of various reinforcing agents, mostly in inorganic form, to make this material more suitable for dental use. [5,13-19] Moreover, PEEK can be easily shaped with the use of standard dental burs for resin materials or special laboratory burs.[13,19, 20]

Low elastic modulus and biocompatibility have rendered PEEK into a suitable framework material.[19,21] However, PEEK's high opacity and grey hue require the use of veneering composites to provide for esthetics.[13,19,22-26] One major problem is the difficulty of PEEK to create an adequate bond with these veneering materials, because of its low surface energy and chemical inertness, making chemical processing almost impossible.[27] Industry has been bonding elastomers to PEEK by surface treating with conventional abrasive treatments, acid etching, plasma or laser techniques followed by the application of epoxy adhesives.[19,20,24] However, these techniques require the use of special equipment and, sometimes, additional space. As a result, their application in dentistry is difficult.[20,21]

In dental practice, knowledge regarding the potential and limitations of the material's adhesion with resin composites is limited.[20,24] As a result, more research in the field is required to establish safe and easy methods for material treatment before adhesion.[24]

The first shear strength testing regarding PEEK was conducted in 2010 by Schmidlin *et al.*[20] This study tested bonding efficiency of a self-adhesive composite resin cement (RelyX Unicem) or a combination of liquid adhesive/hybrid resin (Heliobond/Tetric) on air-abraded or acid-etched PEEK. The use of a hydrophobic bonding agent (Heliobond) in combination with a composite resin (Tetric) led to adequate bonding to PEEK, with shear strength values that ranged between 11,5±3,2 MPa (for air-abraded group) and 18,2±5,4 MPa (for acid-etched group). However, no statistically significant differences were recorded among the groups.

A lot of studies have been conducted in order to determine the best treatment surface and the best primer/bond combination that would enhance composite resin bond to PEEK.[28-30]

The purpose of this *in-vitro* study was to evaluate the effect of surface treatment and water storage on composite resin bond strength to PEEK surface.

The formulated null hypothesis was that the three surface conditioning protocols would have no effect on the bonding strength of the composite cement. Moreover, a hypothesis for water storage was formulated, speculating that water storage of the material for 150 days would result in reduction of bonding strength compared to initial reference water storage for 3 days.

MATERIALS AND METHODS

Table 1. Materials used in this study

Materials	Brand name	Maker	Composition
PEEK	breCAM. BioHPP	Bredent GmbH & Co KG	Poly-ether-ether-ketone, titanium oxides 20% wt
Al_2O_3 particles	Rocatec Pre		Aluminum oxide powder with 110μm mean particle size
Silica-coated Al_2O_3 particles	Cojet System	3M ESPE	Silica-coated aluminum oxide powder with 30μm mean particle size
Bonding Agent	visio.link	Bredent GmbH & Co KG	Methylmethacrylate monomers, pentaerythrol tryacrylate, photo-initiators
Silane Agent	ESPE Sil	3M ESPE	3-Methacryloxypropyl-trimethoxysilane, ethanol
Bonding Agent	Visio-Bond	3M ESPE	Disacrylate monomers (bis-GMA), aminodiol methacrylate, camphor quinone, benzyl-dimethyl-ketale, stabilizers
Primer/ Bonding Agent	Clearfil Ceramic Primer	Kuraray Noritake Dental Inc	3-Methacryloxypropyl-trimethoxysilane, 10-methacryloyloxydecyl-dihydrogen phosphate, ethanol
Resin Composite Cement	combo.lign	Bredent GmbH & Co KG	Dibenzoyl-hyperoxide, 1, 3 –butanodiole dimethacrylate, bis-phenole bis-glycidilic methacrylate monomers, tricyclodekane bisacrylate monomers, camphor quinone

A modified PEEK material (BioHPP; Bredent GmbH & Co.KG) reinforced with 20% ceramic particles was tested.

Sixty modified PEEK disks of 10mm in diameter and 5mm thick were cut out of BioHPP round blanks (breCAM.BioHPP, Bredent GmbH & Co.KG) using CAD-CAM technology. Each PEEK disk was embedded into a Plexiglas hollow cylinder (12mm diameter and 50mm height) and epoxy resin (Struers Epofix, Struers ApS, Ballerup, Denmark) was pured in the other end to fill the cylinder and to fixate disk position. (*Figures 1*, and *2*).

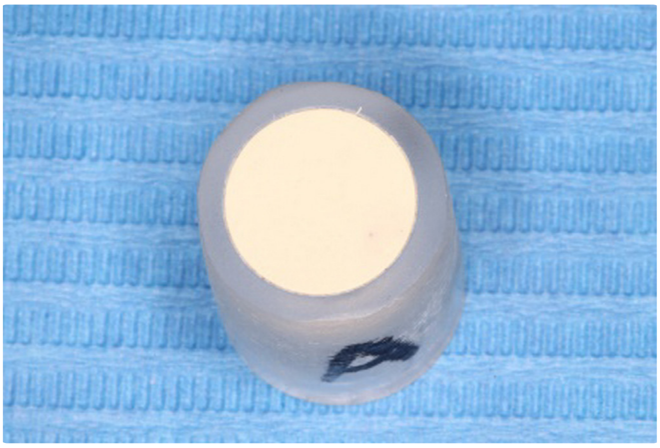

Figure 1: PEEK disk embedded in plexiglass tube

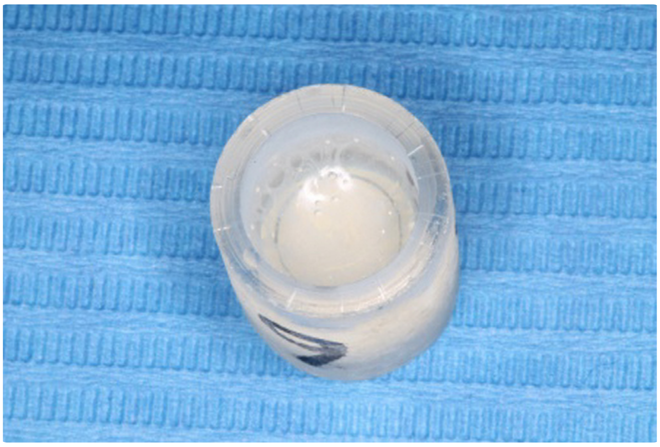

Figure 2: PEEK disks embedded in epoxy resin

Twenty four hours later, the free end of each embedded PEEK disk was polished using SiC disks of 600grit (Dap V, Struers ApS, Ballerup, Denmark) under constant water sprinkling. Following polishing, all specimens were cleaned in ultrasonic bath using 99% isopropanol solution for 10 minutes and dried with oil-free air spray for 15s before surface treatment. Specimens were then divided in 3 groups of 20 each and received the following surface treatments:

Group A (control group): Air-particle abrasion with alumina with a mean particle size of 110μm (Rocatec Pre; 3M ESPE) for 15s under air pressure of 2Mpa and a distance of 10mm between the nozzle and the treated surface. Drying with oil-free air for 15s, followed by bonding agent application (visio.link; Bredent GmbH & Co.KG) and light-curing for 90s.

Group B: Air-particle abrasion with silica-modified alumina with a mean particle size of 30μm (CoJet™Sand, Rocatec® Plus; 3M ESPE) for 15s under air pressure of 0,28MPa and a distance of 10mm between the nozzle and the treated surface. Drying with oil-free air for 15s. Silane agent application (Espe Sil; 3M ESPE) for 30s without drying, followed by bonding agent application (Visio-bond; 3M ESPE) and light-curing for 20s.

Group C: Air-particle abrasion with silica-modified alumina with a mean particle size of 30μm (CoJet™Sand, Rocatec® Plus; 3M ESPE) for 15s under air pressure of 0,28MPa and a distance of 10mm between the nozzle and the treated surface. Drying with oil-free air for 15s. Application of a silane containing primer-bond (Clearfil Ceramic Primer; Kuraray Noritake Dental Inc.) for 180s without drying, followed by bonding agent application (visio.link; Bredent GmbH & Co.KG) and light-curing for 90s.

A special apparatus made out of Teflon was designed to accurately and repeatedly fit over the Plexiglas cylinders at the exposed PEEK surface. An opening of 2mm in diameter and 4mm in height was designed at the center of this apparatus (nest) to host the tested composite resin cement (combo.lign; Bredent GmbH & Co.KG) *(Figures 3* and *4*). The resin cement was light-cured for 90s.

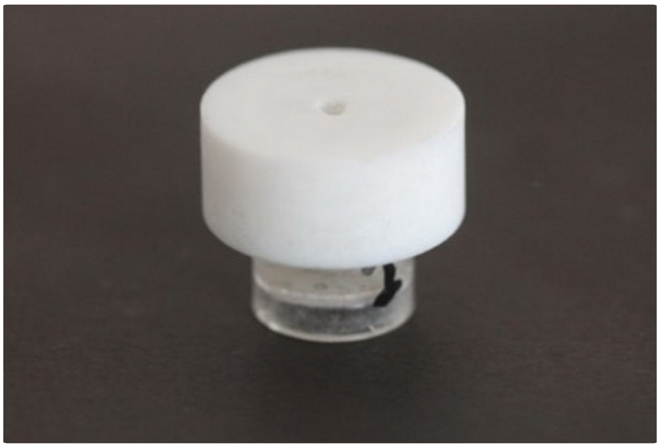

Figure 3: Specimen in Teflon apparatus

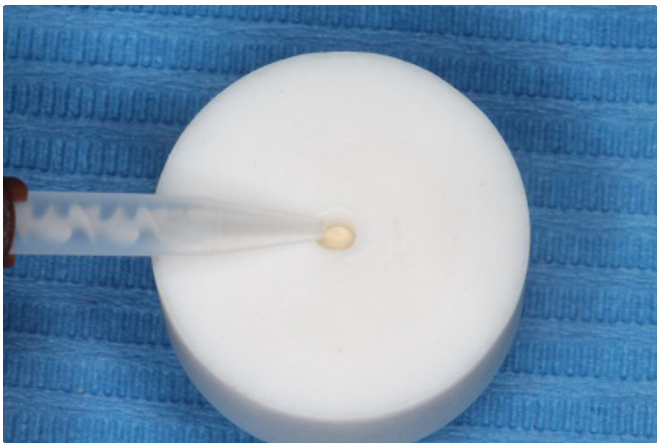

Figure 4: Nest for resin cement placement

Each group was further divided into subgroups of 10 specimens. Storage in distilled water at 37°C for either 3 days or for 150 days without any further thermocycling was applied accordingly.

All specimens were submitted to shear bond strength testing using a universal shear strength testing machine (Aegis 10 Tensometer, Monsanto). Constant force of 2Kn was applied near the PEEK-resin composite interface with a speed of

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1mm/min until failure. For every test the load at failure was recorded. Shear bond strength was calculated using the equation σ=F/a, where σ is the shear bond strength, F the load at failure (N) and a the area of adhesion interface (mm^2). All values were calculated in MPa (N/mm^2) *(Figure 5)*.

After de-bonding, the failing surface was examined with a stereomicroscope at 10x magnification and the type of failure was considered. Failure was considered adhesive if fracture was located in the interface, cohesive when the failure was located in the resin composite or mixed when both types of failure were located in the de-bonded area *(Figures 6* and *7*).

Figure 5: Shear bond testing apparatus (Universal testing machine)

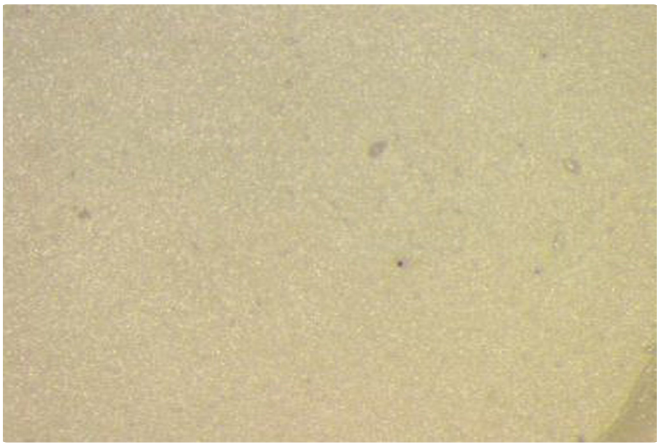

Figure 6: Adhesive failure mode

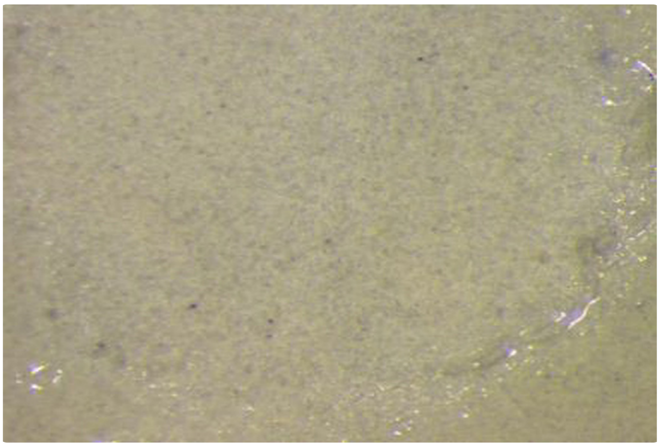

Figure 7: Cohesive failure mode

STATISTICAL ANALYSIS

Data processing software (IBM-SPSS 25.0) was used for the statistical analysis. A two-way Anova test was used for the analysis of variance. Also, a Levene's test and post-hoc analysis with multiple comparison Tukey's test were conducted. The minimum desired level of statistical significance was set to p=0.05.

RESULTS

Two-way ANOVA confirmed the existence of statistical difference between the three treatment groups, whereas there was no statistical significance found for aging variable (storage conditions) or for interaction between the examined variables. As a result, both initial hypotheses were rejected.

Tukey's test revealed that Group B (Air-particle abrasion Cojet 30μm + Espe-Sil - Visio-Bond) showed statistically significant difference with control Group A (Air abrasion 110μm + visio.link). Moreover, there were no statistical differences observed between control Group A (Air abrasion 110μm + visio.link) and Group C (Air abrasion 30μm + Clearfil Ceramic Primer - visio.link), or between Group B (Air abrasion 30μm + Espe-Sil - Visio-Bond) and Group C (Air abrasion 30μm + Clearfil Ceramic Primer - visio.link) *(Table 2)*.

Table 2. Mean values recorded for each tested group.

	Dependent Variable: Shear Strength (MPa)		
Test Group	**Mean**	**Std. Deviation**	**N**
A-CG	15.2	6.8	20
B	24.1	13	20
C	16.9	8.1	20

Finally, as mentioned above, regarding influence of storage conditions in the final bond strength, there were no statistically significant differences between all subgroups. The mean values observed for each subgroup are shown analytically in Table 3.

Most specimens showed an adhesive type of failure *(Table 4)*. A cohesive type of failure was recorded only for two specimens of Group A (control group) after water storage for 150 days (20%), while the rest of the two subgroups showed adhesive failure type in percentage of 70%. All Group B specimens showed adhesive failure type (100%) regardless of storage conditions. Finally, Group C specimens showed adhesive type of failure in percentages of 80% and 90% for 3 and 150 days water storage subgroups respectively.

Table 3. Mean values observed for each subgroup

	Dependent variable: Shear Strength (MPa)			
Test Group	**Storage Conditions**	**Mean**	**Std. Deviation**	**N**
A-CG	3 days	15.9	5.6	10
	150 days	14.5	8	10
B	3 days	20.6	9.5	10
	150 days	27.7	15.4	10
C	3 days	19.1	9.3	10
	150 days	14.8	6.4	10

Table 4. Type of bond failure among the groups

Group	Storage Time (days)	Adhesive Type (%)	Cohesive Type (%)	Mixed Type (%)
A	3	70	-	30
A	150	70	20	10
B	3	100	-	-
B	150	100	-	-
C	3	80	-	20
C	150	90	-	10

DISCUSSION

Bonding of composite resin cements to modified PEEK surface is essential to the application of the material in clinical dentistry.

Air-particle abrasion has been considered one of the simplest surface treatments for metal-ceramics and implant restorations.[23,26] Air-particle abrasion increases surface roughness and has a positive contribution to bond strength by promoting micro-mechanical interlocking with dental adhesives.[23,26] Moreover, it cleans and activates the surface by thoroughly removing organic contaminants.[23,26] Air-particle abrasion with silica-coated alumina particles, also known as tribochemical, combines advantages of conventional sandblasting (micromechanical connection) with chemical connection.

In the present study, 30μm particle size silica-coated alumina (Cojet) was used in order to determine if this particle size could actually modify PEEK surface morphology effectively. The high mean values recorded using this system, even after prolonged water storage showed that the system could potentially be used for effective surface pretreatment of bonded PEEK. Moreover, the small particle size and the low air pressure used could make intraoral repair of PEEK restorations an easy and simple process in case of chipping of veneering resins.

According to the results, the combination of tribochemical treatment (Cojet) with silane agent (ESPE Sil) and a methacrylate-containing bonding agent (Visio-Bond) led to the highest bond strength (24,18 MPa) (Group B). This result is in agreement with the findings of Fuhrmann *et al.*,[22] which reported a mean value between 12,5-23,6 MPa for tribochemical treatment combined with a silane agent (Monobond Plus) and a methacrylate-containing primer (Luxatemp Glaze & Bond). However, this was a tensile test study and no direct comparisons can be done.

Rosentritt *et al.*[13] tested the influence of 18 different combinations of surface treatment and conditioning on the shear bond strength between PEEK and composites, and reported values that in some cases reached 27,1±3,1 MPa.

Stawarczyk *et al.*[19] examined the influence of acid-etching and air-particle abrasion on shear bond strength of PEEK with two different veneering resin composites (Gradia, Sinfony). Acid-etching led to the highest mean values (14,30 MPa and 18,56 MPa respectively), whereas air-particle abrasion did not give satisfactory results. Mean values recorded were much lower than the ones found in our study. Those low values are attributed to the lack of bonding systems used prior to veneering resin application.

Silthampitag *et al.*[26] tested the effect of surface treatment (acid-etching or air abrasion) on bond strength between PEEK and a flowable composite resin (Filtek Z350XT), with or without use of adhesive bonding agent (Heliobond). Authors mentioned that acid-etching with 98% sulfuric acid solution and bonding with Heliobond led to the highest bond strength values (27,36±4,44 MPa). Moreover, authors concluded that surface topography seems to affect the adhesion more than surface roughness.

Silane containing primer-bonding agents (Clearfil Ceramic Primer) have been used in previous studies without satisfactory results.[21,28,29]

In the present study, Clearfil Ceramic Primer application was followed by adhesive bonding agent (visio.link) in combination with silica-modified air-particle abrasion (Cojet) on PEEK surface (Group C). However, this combination did not lead to a durable bonding, with a mean value of 16,98 MPa while control Group A had a mean value of 15,27 MPa (no statistically significant difference). Moreover, this group was the only one that was influenced (non significantly) by the aging process, as the mean value was reduced after 150 days of water storage (14,82 MPa compared to 19,13 MPa initially). This result could be related to the ethanol solvent contained into the bonding agent. Ethanol can infiltrate abraded PEEK surface in a greater depth. However, this could have a negative effect, as the bonding agent 'disappears' inside the abraded PEEK, resulting in less chemical interaction with the hydrophobic adhesive bonding agent visio.link. The values mentioned above are similar to those reported in the studies of Kern & Lehmann and Hallmann *et al.*[21,28] However, these studies used a tensile bond test to assess bond strength. Consequently, results can't be directly compared.

In the present study, water storage was used as the aging process. Presence of water molecules in the bonding interface may cause splitting of the covalent bonds, leading to a decrease in bond strength.[30] For this reason, it was supposed that water storage for 150 days would lead to a decrease in bond strength. However, this was not the case for any of the studied groups.

There is no consensus in the literature regarding the effect of aging on bond strength of PEEK biomaterials. In general, studies report a statistically significant influence of aging process to final bond strength. [13,24,25,30] However, most of these studies refer to tensile tests and this influence was not statistically significant for all studied groups.

To summarize, mean values of the examined surface treatment protocols ranged at the same level or, in some cases, surpassed those of similar previous studies. However, since there are no literature guidelines regarding the lowest acceptable bond strength to a PEEK surface, no safe conclusions about their efficiency could be reported. Finally, the increased percentage of adhesive failure type dictates the necessity in finding ways of further improving the material's bonding potential.

To this direction, further research including clinical trials of the suggested protocols is required to verify the results above.

CONCLUSIONS

With the limitations of this *in-vitro* study, the following conclusions can be drawn:

1. Use of different conditioning protocols had a significant effect on final bond strength.
2. Water storage conditions had no significant influence on final bond strength.
3. Combination of tribochemical coating, silane containing primer-bonding agent and methacrylate-containing bonding agent (Air abrasion 30μm + Clearfil Ceramic Primer - visio.link) did not lead to any improvement in bond strength.
4. Combination of tribochemical coating, silane agent and methacrylate-containing bonding agent (Air abrasion 30μm + Espe-Sil - Visio-Bond) led to satisfactory values and could be suggested as an alternative pretreatment method of bonded PEEK biomaterials.

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