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Original Article

Interplay between resin cements and surface-treated Poly-Ether-Ether-Ketone (PEEK): effect of aging

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Aim: This study assessed the effect of thermal aging on the interfacial strength of resin cements to surface-treated PEEK. **Methods:** Ninety-six PEEK blocks were allocated into 4 groups (n=24), according to following surface treatments: SB - sandblasting with aluminum oxide; SA - acid etched with 98% sulfuric acid; CA – coupling agent (Visio.link, Bredent) and CO - control group (untreated). Surface roughness (Ra) was measured and one cylinder (1-mm diameter and height) of Rely-X Ultimate - ULT (3M/ESPE) and another one of Panavia V5 - PAN (Kuraray) were constructed on the treated or untreated PEEK surfaces. Half of the samples of each group were thermal aged (1,000 cycles). Samples were tested at a crosshead speed of 1 mm/min in shear mode (μSBS). Ra and μSBS data were compared using one- and three-way ANOVA, respectively, and Tukey's tests. **Results:** SA and SB samples had the roughest surfaces, while CA the smoother ($p<0.001$). Thermal aging reduced μSBS regardless the surface treatment and resin cement used. There was interaction between surface treatment and resin cement ($p <0.001$), with ULT showing higher μSBS values than PAN. SA provided higher μSBS than SB for both resin cements, while for CA μSBS was higher (PAN) or lower than SB (ULT). **Conclusion:** Aging inadvertently reduces interfacial strength between PEEK and the resin cements. If ULT is the resin cement of choice, reliable interfacial strength is reached after any PEEK surface treatment. However, if PAN is going to be used only SA and CA are recommended as PEEK treatment.

Keywords: Polymers. Resin cements. Shear strength. Aging.

Introduction

Poly-ether-ether-ketone (PEEK) is a thermoplastic polymer with attractive properties such as low allergenic potential, non-metallic color, high polishing, wear resistance, lightness and reduced biofilm formation make it as an alternative to prosthetic and restorative materials[1,2]. In Dentistry, the clinical applications of PEEK include framework for fixed and removable prostheses, crowns, abutments, dental implants, occlusal guards, orthodontic wires, and posts[2-4].

Despite its versatility, PEEK has low free energy and inert hydrophobic surface which pose challenges to bonding procedures to dental materials[5-7]. In order to increase surface energy and provide functional groups for improved bond strength with resin materials, as a previous step to bonding, PEEK surface has been subjected to physical or chemical treatments, including sulfuric acid etching, sandblasting, silica coating, coupling agent, laser and plasma[4,8-11]. However, the bonding result depends not only on the PEEK surface treatment, but also on the adhesive or resin cement itself and on the interplay between surface-treated PEEK surface and adhesive/resin cement[7,11]. These two later aspects are especially important if one considers the myriad of available adhesives and resin cements and their compositions, which can affect bonding to PEEK. One example are resin cements containing 10-methacryloxydecyl dihydrogen phosphate (10-MDP). Although such component contributes to the overall polymerization process of some resin cements, such as in Panavia V5, there are speculations that 10-MDP negatively affect bonding to PEEK due to its phosphate group, which does not react with PEEK[12].

The understanding of the interaction of surface-treated PEEK-resin cement is even more important if one considers that such materials face biochemical and physicomechanical degradation processes in the oral cavity. Factors including saliva, acidic conditions, temperature oscillations, and masticatory stresses may hinder the properties of resin cements over time. Aging by simulating oral conditions, such as thermocycling, has been used to anticipate the impact of degradation processes[13]. However, to the best authors' knowledge, to date, the effect of thermal aging has been investigated between surface-treated PEEK and resin cement has only been investigated plasma-treated PEEK[14], which is less tangible to the clinicians. As for the combination surface-treated PEEK/adhesive/composite system, chances are that the repetitive temperature changes could strain the interface between surface-treated PEEK and resin cement, and affect the bonding stability, which would have the influence of the composition of the resin cement.

Based on the aforementioned rationales, this study aimed to assess the effect of thermal aging on the interfacial strength between surface-treated PEEK and resin cements. We tested the null hypothesis that there would be no effect of surface treatment of PEEK, resin cement and thermal aging, neither alone nor interacting, on micro-shear bond strength (μSBS) between PEEK-resin cement.

Material and methods

Experimental design

This study had two parts. In Part One, the samples were 24 PEEK blocks whose surface was subjected to four different surface treatments as follows: 98% sulfuric acid etching (SA); sandblasting (SB); pentaerythritol triacrylate (PETIA)-containing coupling agent (CA, Visio.link, Bredent, Germany) and untreated control surface (CO). The dependent variable was surface roughness. In Part Two of this study samples of Part One were bonded to two dual-cure resin cements (RelyX Ultimate – ULT and Panavia V5 - PAN, Table 1) and unaged or aged using thermocycling. The dependent variable was μSBS.

Table 1. Description of the resin cements.

Characteristics	RelyX Ultimate (ULT)	Panavia V5 (PAN)
Monomers	Base paste: methacrylate monomers catalyst paste: methacrylate monomers	Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate
Inorganic fillers	43% by volume silanized filler particles, alkaline filler particles size: 13 μm	38% by volume silanized barium glass particles, silanized fluoralminosilicate glass particles, colloidal silica, silanized aluminum oxide particles size: 0.01-12 μm
Initiators	Sodium p-toluenesulfonate, sodium persulfate, terc.butil 3,5,5-trimethylperoxyhexanoate	dl-camphorquinone
Shade	A1	Clear
Manufacturer, batch #	3M/ESPE; 4471448	Kuraray; 000001

Bis-GMA: bisphenol A-glycidyl methacrylate; TEGDMA: triethylene glycol dimethacrylate.

Based on a pilot study, in which the effect size was 0.183, a total of 21 samples per group would be required to detect significant difference, at 5% significance level and 80% of power. Three samples were added in each group in order to compensate for eventual sample loss due to premature failure during thermocycling. Each group had therefore 24 samples.

Part one – sample preparation, surface treatment, surface roughness evaluation and AFM imaging

Using a milling system (CNC Discovery D600, Indústrias Romi SA, Brazil), 96 PEEK blocks (MGM Plásticos de Engenharia, Brazil) were machined to 10x10x5.5 mm. PEEK blocks were then randomly allocated into four groups (n = 24) to receive one of the following surface treatments:

SA: 200 µl of 98% sulfuric acid etching (ECIBRA/CETUS, Brazil) for 60 s[11], followed by immersion in distilled water for 15 s to stop the chemical reaction and rinsing with distilled water for 15 s;

SB: sandblasting with aluminum oxide particles[11] (average particle size: 125 µm) for 20 s under 3 Bar (pressure), at an angle of 45 degrees and 10 mm-distance between the surface and the nozzle (Sandblaster Basic Master and Cobra, Renfert, Germany), and rinsing with distilled water for 15 s;

CA: application of PETIA-containing coupling agent[11] (Visio.link, Bredent, Germany), using a Microbrush® applicator and light-curing for 90 s (Valo, Ultradent Products, USA);

CO: Control (untreated surface).

After the surface treatments, PEEK blocks were measured for average surface roughness (Ra) using a profilometer (Mitutoyo SJ210, Mitutoyo Sul Americana Ltda, Brazil). The cut-off was set at 0.25 mm and total transverse length was 1.25 mm. Measurements were made in three different directions (0, 45 and 90º) of the sample.

Representative images of surface-treated-samples were obtained under atomic force microscopy (Dimension® Icon AFM System with ScanAsyst®, Bruker Nano Surfaces Division, USA), operating in intermittent mode, with a scanning area of 2x2 µm.

Part two – fabrication and bonding of resin cement cylinders, thermal aging, µSBS testing and failure mode examination

Directly on the surface of each sample, two translucent Tygon tubes with an internal diameter of 1.0 mm[15] and a height of 1.0 mm were used as matrices. One trained operator using magnifying loupes (Galilean HD 3.3, ExamVision, Denmark) positioned the matrices on the PEEK surface. The resin cements ULT e PAN were mixed according manufacturers' direction. Each matrix carefully received one of each resin cement. A Mylar strip was positioned over the filled tube and gently pressed. The resin cements were light-cured through the Mylar strip, according to the recommendations of each manufacturer: 20 s for ULT and 10 s for PAN, with the Valo curing light (Ultradent Products, USA) at standard power (1000 mW/cm^2). Matrices were then carefully removed using a sharp blade to expose the resin cement cylinders. Each cylinder was examined using magnifying loupes to identify possible defects (bubbles and flow of resin cement beyond the limits of the bonding area). All the samples, formed by the PEEK surface and one cylinder of each resin cement, were stored in distilled water at 37ºC for 24 h and randomly allocated to be either thermal aged or remain unaged.

The samples thermal aged underwent 1,000 hydrothermal cycles in water between 5ºC and 55ºC, with 30 s dwell time (MCT, Elquip, Brazil).

The samples were mounted into a jig attached to a universal testing machine (DL 200, EMIC, Brazil). A 0.2-mm diameter orthodontic wire was looped around the base of the resin cement cylinder as close as possible to the PEEK-cylinder interface and a shear force was applied to cylinder (Figure 1) at a crosshead speed of 1 mm/min until failure occurred[16]. The µSBS values was calculated in megapas-

cals (MPa) by dividing the load at failure point (newtons) by the surface area of the PEEK-resin cement bonding.

Fractured μSBS samples were then examined for their failure modes with a stereomicroscopic loupe (EK3ST, Eikonal Equip, Brazil) at 10X magnification and classified into: adhesive failure (between PEEK and resin cement), cohesive failure in PEEK, cohesive failure in the resin cement and mixed failure (Figure 1).

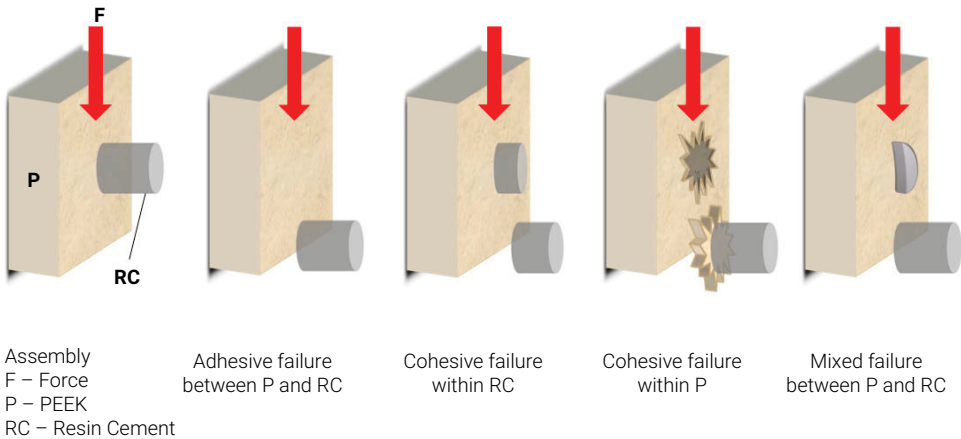

Figure 1. Schematic drawing of the sample tested for micro-shear bond strength (on the left) and the four different possible failure modes.

Statistical analysis

Due to the lack of normality, data were square-root transformed. One-way analysis of variance compared surface roughness data (Part One), while the effect of surface treatment, resin cement, thermocycling and their interactions (Part Two) were tested using three-way analysis of variance. All multiple comparisons were performed with Tukey's test. The calculations were run on SPSS (SPSS Inc., USA), at a significance level of 5%.

Results

Surface pre-treatments significantly affected roughness ($p < 0.001$), with both SB and SA groups significantly rougher than CO, whereas CA presented the smoothest surface (Table 2). Figure 2 shows AFM images and revealed that CO samples (Figure 2D) had a primary texture featuring some grooves caused by the extrusion process after casting, whereas samples that received CA (Figure 2C) expressed a flat surface with a micellar aspect. The samples of SB group (Figure 2B), on the other hand, exhibited an irregular surface, with few and sparse pits, while those etched by SA (Figure 2A) had the surface changed to a spongy pattern with marked and wider depressions.

Table 2. Means and standard deviations of surface roughness (Ra, µm) of PEEK after different physical or chemical treatments.

Surface treatment	Surface roughness
SA	1.412 (0.546) C
SB	1.528 (0.140) C
CA	0.127 (0.073) A
CO	0.465 (0.107) B

SA (98% sulfuric acid etching); SB (sandblasting with aluminum oxide particles);CA (PETIA-based coating agent - Visio.link). Groups followed by dissimilar capital letters differ from each other.

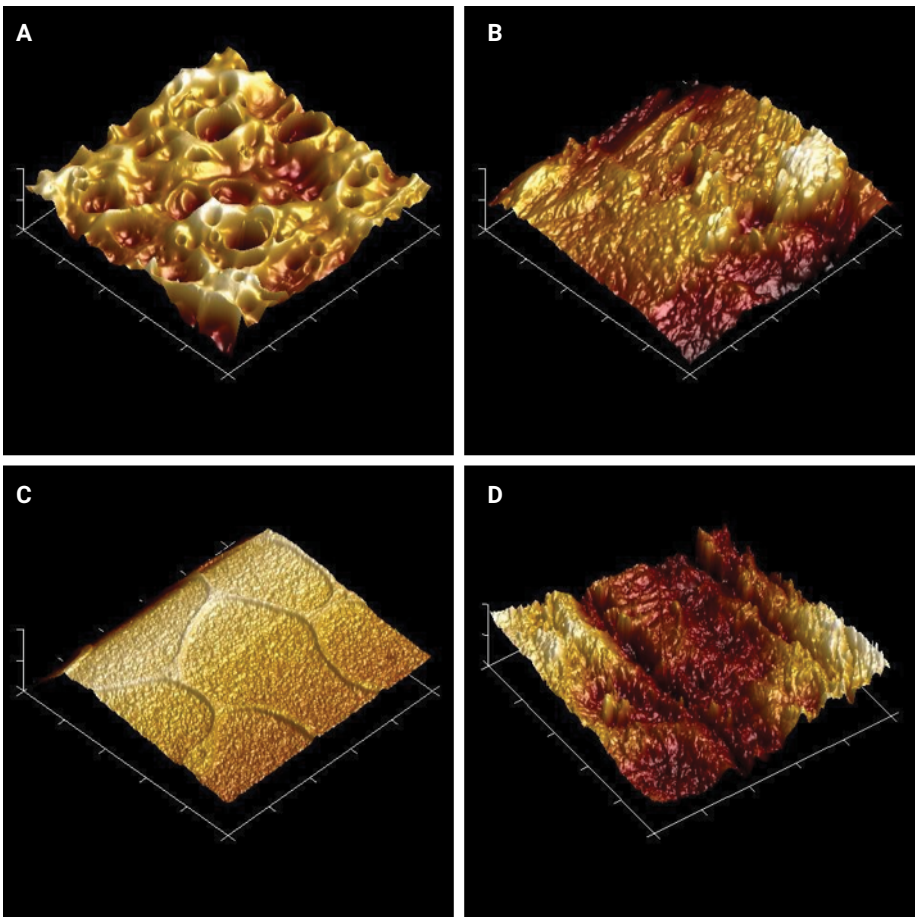

Figure 2. AFM images of PEEK etched with 98% sulfuric acid (A), sandblasted (B), subjected to coupling agent (C) and untreated (control, D).

Table 3 presents µSBS data which demonstrated no significant interaction among surface treatment, resin cement and thermal aging ($p = 0.575$), but a significant interaction was noticed between surface treatment and resin cement ($p < 0.001$). This interaction was explored using Tukeys' test and showed that compared to SB, SA provided higher µSBS to both PAN and ULT resin cements. However, while for PAN no

difference existed between the μSBS when PEEK surface received SA or CA, for ULT, CA resulted in lower μSBS values. Regardless of the surface pretreatment performed, ULT resulted in higher values of μSBS to PEEK (Table 4). As no other significant interaction was detected (surface treatment x thermal aging: p = 0.182; resin cement x thermal aging: p =0.458), then it was checked the effect of the main variable, which was shown to be statistically significant. Specifically, regardless of the surface treatment and resin cement used, thermal aging significantly reduced μSBS between resin cements and PEEK surface by 15.6%, [thermal unaged: 18.46 MPa (11.85 MPa); aged: 15.57 MPa (12.64 MPa)].

Table 3. Means and standard deviations (MPa) of bond strength between resin cements and surface-treated PEEK, unaged and thermal aged.

Surface treatment	Panavia V5 (PAN)		Rely-X Ultimate (ULT)	
	Unaged	Thermal aged	Unaged	Thermal aged
SA	16.05 (11.91)	11.69 (10.33)	30.44 (8.83)	33.22 (11.62)
SB	3.43 (2.76)	5.28 (10.07)	27.52 (7.97)	23.23 (5.77)
CA	14.44 (7.91)	7.18 (4.04)	18.89 (6.00)	12.51 (3.43)

SB (sandblasting with aluminum oxide particles); SA (98% sulfuric acid etching); CA (PETIA-based coating agent - Visio.link). According to three-way analysis of variance: interaction among surface treatment x resin cement x thermal cycling (p = 0.575); interaction between surface treatment x resin cement: p < 0.001; interaction between surface treatment x thermal cycling: p = 0.182; interaction resin cement x thermal cycling: p = 0.458; main variable thermal cycling: p = 0.013).

Table 4. Bond strength means and standard deviations (MPa) between resin cements and PEEK subjected to different surface treatments, regardless whether thermal aged.

Surface treatment	Panavia V5 (PAN)	Rely-X Ultimate (ULT)
SA	13.96 Aa (11.16)	31.83 Ab (10.19)
SB	4.35 Ba (7.28)	25.38 Bb (7.15)
CA	10.81 Aa (7.17)	15.70 Cb (5.79)

SA (98% sulfuric acid etching); SB (sandblasting with aluminum oxide particles); CA (PETIA-based coating agent - Visio.link). Means followed by different capital letters indicate difference among surface treatments within each column. Means followed by different lower-case letters indicate difference between resin cements within each row.

Adhesive failure was predominant in all groups. Mixed failures occurred in samples bonded with ULT but not with PAN. In samples that received CA pre-treatment, those thermal aged had adhesive failures only, while 8.33% of unaged samples had mixed failures. The same proportion of mixed failures was seem in the SA pre-treated group that was unaged. Still in unaged samples, 16.6% of SB group samples had mixed failures. When thermal aged, SA and SB groups mixed failures occurred in 50,0% of 33.3% of the samples. Cohesive failure within PEEK occurred in a single sample (8.33%) pertaining to SA group (unaged).

Discussion

The findings of this study demand rejection of the null hypotheses as thermal aging and the interplay between surface treatment of PEEK and resin cement significantly affected μSBS values. The reasons why thermal aging reduced the μSBS values are twofold: a) causing water sorption and hydrolytic degradation at bonding interfaces and, b) causing thermal stress due to differences in the coefficient of thermal expansion and condutivity between PEEK and resin cement[17,18].

Water sorption can plasticize, break hydrogen bonds within the resin matrix, cause polymer swelling and ultimately hinder the properties of resin cements[17]. Water sorption can also cause hydrolytic degradation of the resin matrix, the filler/matrix interface, or the filler. In effect, there are reports showing that both ULT and PAN present water sorption. ULT contains phosphoric acid modified methacrylate monomers, which have the capability to bind water at hydroxyl groups[18]. In addition, ULT has alkaline fillers, which bind water by starting an acid-base reaction[18]. PAN, on the other hand, presents water sorption because it contains hydrophilic aliphatic dimethacrylate, but as there are no phosphate/hydroxyl groups or alkaline fillers, water sorption has been shown to be reduced[18]. As a result, for both resin cements (ULT e PAN) thermocycling increases water sorption and solubility[18].

Still with respect to the explanations why thermocycling reduced μSBS values in the current study, cyclic temperature changes can generate expansion and contraction stresses, leading to microcracks within the resin cement[18]. Such events can cause microcracks and thereby increase water sortion and solubility of resin cements[18]. However, stress can concurrently occur at the PEEK-resin cement interface, as the coefficient of thermal expansion of pure PEEK has been described to be half of resin cements such as ULT[19,20].

One can argue that a higher number of thermal cycles could better represent the long-term aging, especially because 10,000 cycles have been described to correspond to approximately one year of clinical service[21] and higher numbers of thermal cycles have been described in PEEK experiments[5]. However, it is worth mentioning that in these publications the samples were prepared for shear bond testing not for μSBS, as used in the current paper[5]. Preliminary experiments of our group showed that 10,000 thermal cycles caused debonding of 92% of the samples during thermocycling. Even during 5,000 thermal cycles an extensive proportion of samples prematurely failed (67%). The explanation for debonding may be probably found in the aggravated action of temperature oscillations in the PEEK-resin cement interface, because of a lower bonding area in μSBS testing in comparison to the shear bond method. Thus, in order to have minimal premature failure and make it feasible to mearure μSBS values, we run 1,000 cycles.

Interesting to notice is that previous literature data in which the authors thermocycled ULT 10,000x the bond strength of this resin cement was reduced in 14.7%[22], an amount equivalent to that observed in our study (15.6%) using 1,000 thermal cycles. This similar reduction despite the different number of thermal cycles may be ascribed to the fact that in the cited paper the bonding area was increased and samples were tested in tensile rather than microtensile mode.

Besides the effect of thermal aging, surface treatment also played a role on μSBS values. Regardless of the resin cement, SA provided higher μSBS than SB. Figures 1A and 1B substantiate this finding showing, respectively, marked versus sparse pits on the PEEK surface. The effect of SA stems from the cleavage of benzene rings by attacking PEEK carbonyl and ether groups and the introduction of sulfonic acid groups in the PEEK polymer chains[23,24]. A micromorphological change is generated, but probably in a range not significantly different from SB in terms of Ra values, in accordance with a previous study[25]. However, other papers have indicated that SB promotes smoother[26] or rougher surface than SA[27-29]. Such differences may be attributed to variation in the size of aluminum oxide particles, the pressure and duration of blasting[30,31]. In effect, in the present study, the pressure used during blasting was higher than that used in some previous studies[7,32]. The pressure of 3 Bar was chosen in an attempt to achieve greater bond strength, since it has been reported that PEEK bond strength is enhanced by increasing blasting pressure[28,33]. However, bonding to sandblasted or any pretreated surface proved to be dependent on the resin cement used, as PAN systematically provided lower μSBS than ULT. This result substantiates the speculation that 10-MDP present in PAN can negatively affect bonding to PEEK is correct.

In this regard, however, it is relevant to verify whether the μSBS values reached the 10 MPa threshold, considered as a clinically acceptable value in a number of published papers as cited elsewhere[10]. Our data showed that in only one combination of surface treatment (SB) and resin cement (PAN) the μSBS was below the 10 MPa threshold. Despite the proximity between the average μSBS and the 10-MPa threshold, the combination between CA as a pretreatment for PEEK and PAN as the resin cement is electable. CA (Figure 2C) created a surface with micellar aspect promoted by the chemical interaction between PEEK and methylmethacrylate (MMA) and PETIA[12] that constitutes the coupling agent (Visio.link). However, the efficiency of such interaction has been significantly higher following air abrasion and sulfuric acid etching[11].

It is noteworthy noting that in a previous study that tested PEEK bonded to titanium bases showed that the weaker interface was between the PEEK and a resin cement[34]. This finding validates the importance of the present paper in further explores the interfacial strength between PEEK and different resin cements, especially under aging. However, one should bear in mind that in continuation to this study, it would be valuable to test whether or not the bonding capacity of resin cements to PEEK and its longevity would hold when resin cements are sandwiched between PEEK and dental substrates (or composite resins). This set up would be feasible through micro-tensile testing. If possible obtaining micro-tensile samples using resin cements sandwiched between PEEK and other substrates, the results would allow gaining additional insights into the predictability of the interfacial strength under clinical circumstances involving PEEK usage.

Based on the current findings, thermal aging reduced the interfacial strength between PEEK and resin cements, but if ULT is the resin cement of choice, reliable interfacial strength is reached after any PEEK surface treatment. However, if PAN is going to be used only SA and CA are recommended as PEEK treatment.

Data availability

Datasets related to this article will be available upon request to the corresponding author.

Conflict of interest

None.

Authors contribution

Joly AM: collected data, drafted the manuscript; Ramos GG: interpreted the data, revised the manuscript; Turssi CP: conceived and designed the study, data analysis, revised the manuscript. All the authors actively participated in discussing the manuscript's findings and have revised and approved the final version of the manuscript.

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