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# 浓硫酸酸蚀聚醚酮酮的时间对其与牙本质剪切粘接强度的影响

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【摘要】 **目的** 研究浓硫酸酸蚀聚醚酮酮(polyether-ketone-ketone，PEKK)不同时间对其与牙本质剪切粘接强度的影响，为临床使用PEKK修复体的粘接操作提供科学依据。**方法** 制备PEKK试件44个，随机平均分为A、B、C、D 4组：A组为对照组，仅用水磨砂纸打磨；B组、C组、D组为实验组，分别用98%浓硫酸酸蚀经打磨试件表面5 s、30 s、60 s。另外，每组随机抽取1个试件用慢速切割机制备出剖面，在扫描电镜下观察其剖面的表面形貌。4组试件与牙本质通过树脂粘接后在37 ℃蒸馏水浸泡24 h，测量剪切粘接强度后统计分析，通过扫描电子显微镜与体视显微镜检查试件断裂界面，统计粘接失败类型。**结果** B组PEKK试件酸蚀处理后剖面呈海绵状且孔隙大小均匀，C组、D组可见试件剖面有破坏状孔蚀样结构；剪切粘接强度B组(16.84 ± 1.84)MPa、C组(12.33 ± 1.22)MPa和D组(6.44 ± 1.18)MPa均大于A组(3.99 ± 1.06)MPa($P < 0.05$)，其中B组与牙本质的剪切粘接强度最高(16.84 ± 1.84)MPa。**结论** 采用98%浓硫酸酸蚀PEKK表面5 s处理方法可以使PEKK与牙本质获得较好的剪切粘接强度。

【关键词】 聚醚酮酮； 表面处理； 牙本质； 粘接； 底涂剂； RelyX™ Ultimate； 剪切粘接强度； 扫描电镜

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**Effects of concentrated sulfuric acid etching durations on the shear bond strength between polyether-ketone-ketone and dentin**

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【Abstract】 **Objective** To study the effects of different concentrated sulfuric acid etching durations on the shear bond strength between polyether-ketone-ketone (PEKK) and dentin, providing a scientific basis for the clinical bonding procedures of PEKK prosthesis. **Methods** Forty-four PEKK specimens were prepared and randomly divided into four groups: group A was the control group, which was only polished with abrasive papers, group B, group C and group D were experimental groups, which were etched by 98% concentrated sulfuric acid for 5 s, 30 s and 60 s, respectively. In addition, one sample was randomly selected from each group, and the profile was prepared by a slow cutting machine. The surface morphology of the profile was observed under SEM. After the four groups of specimens and dentin were bonded by resin, they were soaked in distilled water at 37 ℃ for 24 h. After the shear bonding strengths were measured,

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the fracture interfaces of the specimens were examined by the scanning electron microscopy and stereomicroscopy, and failure models of bonding were analyzed. **Results** After acid etching treatments, the cross-sectional images in group B presented uniform spongy shapes, while the cross-sectional images in group C and group D showed destructive pore structures. The shear bond strengths of group B (16.84 ± 1.84) MPa, group C (12.33 ± 1.22) MPa and group D (6.44 ± 1.18) MPa were higher than that of group A (3.99 ± 1.06) MPa ($P$ < 0.05). The highest shear bond strength was observed in group B (16.84 ± 1.84) MPa. **Conclusion** The surface treatment of 98% sulfuric acid etching for 5 s manifested the best bond strength between PEKK and dentin.

**【Key words】** polyether-ketone-ketone; surface treatment; dentin; bonding; primer; RelyX™ Ultimate; shear bond strength; scanning electron microscope

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聚芳醚酮（polyarylether-ether-ketone，PAEK）作为新型人体植入材料在临床上应用广泛，其良好的理化性能和生物安全性已被证实[1-2]。近年来PAEK在口腔医学领域的应用逐渐受到关注，目前研究较多是PAEK家族的聚醚酮酮（polyether-ketone-ketone，PEKK）和聚醚醚酮（polyether-ether-ketone，PEEK）[3]。其中，PEEK为PAEK家族早期产品材料[4]，已经在口腔医学领域被应用于牙种植体[5]、临时基台[6]、固定义齿[7-8]以及活动义齿[9]等方面。PEKK是新一代研发的PAEK家族材料，其力学性能和生物相容性显著优于PEEK材料[10-11]，已有研究报道将其应用于口腔修复体制作[12]。

由于PEKK外观为不透明的灰白色，作为固定义齿修复材料需要考察其与饰面树脂、牙本质两个界面的粘接效果。现有文献报道了通过浓硫酸酸蚀PEKK后与饰面树脂粘接可获得良好的粘接效果[13]，本课题组前期研究中也系统探讨了PEKK与饰面树脂的粘接效果[14]，但关于PEKK表面处理后与牙本质的剪切粘接强度尚无研究报道。本实验将探讨不同浓硫酸酸蚀时间对PEKK与牙本质剪切粘接强度的影响，为临床PEKK修复体粘接操作提供科学的实验依据。

## 1 材料与方法

### 1.1 实验材料及仪器

材料：新鲜人牙齿牙本质，PEKK材料（Pekkton，Cendres+MétauxS，瑞士），底涂剂Visiolink（Bredent，德国），粘接剂Single Bond Universal（3M，德国），复合树脂RelyX™ Ultimate（3M，美国）。

仪器：扫描电子显微镜（Hitachi S-4800，日本），全自动抛光机（Mecatech234，法国），KQ-250DE型数控超声波清洗器（昆山市超声仪器有限公司，中国），慢速切割机（Isomet 1000，美国），LED光固化灯（义获嘉伟瓦登特公司，列支敦士登），体式显微镜（尼康SMZ1500，日本），万能力学试验机（MTS，美国）。

### 1.2 实验方法

1.2.1 人牙本质试件制备 经南京市口腔医院伦理委员会批准后［2019NL-031（KS）］，选择40颗离体完整无龋第三磨牙。根据ISO 29022:2013标准，第三磨牙拔出后在流动水中彻底清除血液及附着组织，然后保存在4 ℃下的0.5%的氯胺溶液中，牙拔出后2周内使用。将聚甲基丙烯酸树脂倒入硅橡胶模具（直径18 mm，高度15 mm）中，然后将处理后的第三磨牙放入模具内，颊面暴露于高出硅橡胶模具上缘2 mm的位置。待固化后第三磨牙分别用120 #、400 #的碳化硅水磨砂纸带水研磨（压力30 N）直至暴露牙本质面，粘接前始终保持牙本质湿润状态并在1 h内完成粘接。

1.2.2 PEKK试件制备 将Pekkton圆盘进行数控切削，制备44个（直径4 mm，高度5 mm）PEKK试件。所有试件经电子游标卡尺测量，均为直径4 mm，高度5 mm。试件在30 N压力下，依次在全自动抛光机上用280 #、400 #、800 #、1 200 #、1 500 #、2 000 #、3 000 #砂纸湿磨（每个试件研磨6 min），75%乙醇超声震荡洗涤10 min后气枪吹干待用。

1.2.3 实验分组与PEKK试件表面处理 44个PEKK试件随机分为4组，每组11个，进行表面处理操作，A组：对照组，用水磨砂纸打磨；B组：用98%

浓硫酸酸蚀经打磨试件表面5 s;C组:用98%浓硫酸酸蚀经打磨试件表面30 s;D组:用98%浓硫酸酸蚀经打磨试件表面60 s。然后用去离子水仔细冲洗1 min,气枪吹干试件表面。

1.2.4 扫描电镜观察 在4组不同表面处理后的PEKK试件中,每组随机抽取1个试件,使用慢速切割机制备出剖面,超声震荡涤洗后然后采用扫描电镜观察4组试件剖面的表面形貌。

1.2.5 剪切实验试件的准备 每个预处理后的PEKK试件表面用底涂剂(visiolink)处理后光固化90 s。每个牙本质试件气枪轻吹5 s后,在其表面粘贴带有4 mm直径圆孔的不透明胶带以确定粘接区域,胶带厚度为50 μm。先在圆孔内均匀涂布粘接剂Single Bond Universal 20 s后气枪轻吹5 s,直至其不再流动后光固化10 s。然后在圆孔内填满树脂RelyX™ Ultimate,PEKK试件沿胶带定位于圆孔后施加750 g恒定载荷,挤出多余的树脂,并使用小毛刷将其清除,再用光固化灯从PEKK试件四个方向各照射10 s,光固化后的剪切实验试件置于37 ℃恒温蒸馏水中避光储存24 h。

### 1.3 剪切强度测量

将剪切实验试件固定于万能力学实验机上,调整加载头使其与试件粘接界面贴合。加载头以0.5 mm/min的加载速度对PEKK与树脂粘接界面进行加载,加载方向为垂直方向,粘接界面断裂后记录最大剪切力值F(N),测试精度为0.01 MPa。剪切粘接强度(MPa)=剪切压力(N)/粘接面积($mm^2$)。

### 1.4 观察试件粘接失败类型

断裂试件表面的粘接失败类型在体式显微镜以及扫描电镜下观察,可归类为5种:①牙本质和树脂界面断裂,表现为牙本质表面没有树脂残留;②PEKK和树脂界面断裂,表现为PEKK表面没有树脂残留;③牙本质内聚破坏,表现为牙本质发生断裂,破坏发生于牙本质内部;④PEKK内聚破坏,表现为PEKK发生断裂,破坏发生于PEKK内部;⑤混合破坏,即同时有界面破坏和内聚破坏,表现为PEKK和牙本质表面残留部分树脂,剩余区域未见附着物。

### 1.5 统计学方法

采用统计软件SPSS 17.0对数据进行统计学分析,各组数据用均数 ± 标准差表示,对各组剪切粘结强度值进行正态性检验与方差齐性检验后行方差分析,并对组间进行SNK-$q$检验,$P < 0.05$认为差异具有统计学意义。

## 2 结 果

### 2.1 扫描电镜结果

扫描电镜结果如图1所示,A组剖面未见有孔隙状结构,切割纹路清晰规则,整体较为平坦;B组剖面类似海绵状结构,且孔隙大小均匀;C组剖面孔隙大小不均匀且孔隙数量逐渐减少,出现少量孔状塌陷;D组剖面孔隙存在大量崩解并失去其海绵状稳定结构。

### 2.2 剪切粘接强度测试结果

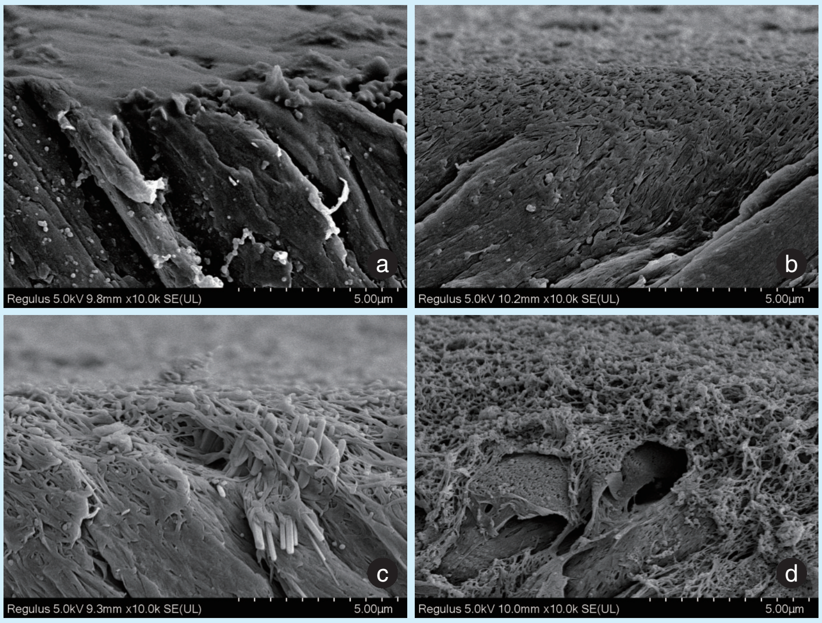
剪切强度测量统计结果如图2所示,B组(16.84 ± 1.84)MPa、C组(12.33 ± 1.22)MPa和D组(6.44 ± 1.18)MPa剪切粘接强度均大于A组(3.99 ± 1.06)MPa($P < 0.05$),实验组中B组的剪切粘接强度最高。

### 2.3 粘接失败类型观察结果

试件与牙本质的粘接断裂界面在体式显微镜下进行观察,统计观察结果如表1。A组和D组粘接失败主要是因为PEKK与树脂界面发生破坏;B组和C组粘接失败主要是因为发生混合破坏。电子显微镜下观察每组PEKK粘接断裂界面,图3所示,A组PEKK断裂表面较为平坦,无残留附着物;B组断裂表面较凸出,有大量残留附着物;C组与D组PEKK断裂表面稍凹陷且有大量蚀孔样结构,C组残留附着物多于D组。

## 3 讨 论

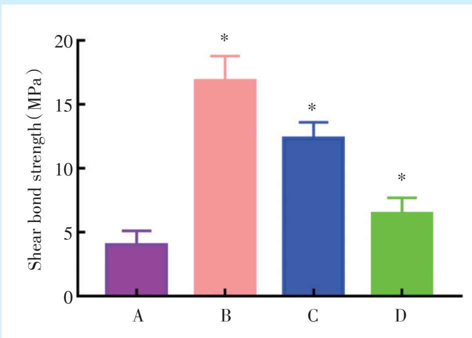
PEKK表面处理的方法包括酸蚀、喷砂等离子处理等[13,15-16]。浓硫酸能打开PEKK苯环之间的功能性羰基与醚基,有利于粘接材料与酸蚀后的PEKK结合[17-18]。有研究表明浓硫酸处理除了能够增加表面粗糙度,其磺化反应生成的基团还可以与甲基丙烯酸甲酯发生化学反应以增加粘接强度[17,19]。本实验在预处理后的PEKK表面采用了底涂剂(visiolink)进行处理,这种底涂剂的主要成分是甲基丙烯酸甲酯,因此,浓硫酸处理后的PEKK能进一步与visiolink中的甲基丙烯酸甲酯反应以提高剪切粘接强度。而喷砂只是单纯通过增加表面粗糙度来提高修复材料与粘接材料之间的粘接强度,等离子处理虽然可以提高表面能,但不能提高与粘接材料的粘接强度[15]。此外,有文献报道浓硫酸酸蚀时间对PEKK与饰面树脂粘接强度有影响[13],因此本实验选择了三种不同酸蚀时间(5 s、30 s、60 s),以比较研究不同酸蚀时间对PEKK与牙本质剪切粘接强度的影响。




a: A group, there is no pore structure in the section, the cutting lines are clear and regular, and the whole is relatively flat; b: B group, the section has a spongy structure, and pore size is uniform; c: C group, pore size is not uniform, the number of pores decreases gradually, and a small amount of porous collapse occurs; d: D group, large amount of disintegration exists in the pore; A: control group, no etching treatment with concentrated sulfuric acid; B: etched by 98% concentrated sulfuric acid for 5 s; C: etched by 98% concentrated sulfuric acid for 30 s; D: etched by 98% concentrated sulfuric acid for 60 s; PEKK: polyether-ketone-ketone

Figure 1 Cross-sectional scanning electron microscopy images of PEKK after different concentrated sulfuric acid etching durations

图1 浓硫酸表面处理不同酸蚀时间后PEKK剖面的扫描电镜图




*: *vs*. A group, $P < 0.05$; A: control group, no etching treatment with concentrated sulfuric acid; B: etched by 98% concentrated sulfuric acid for 5 s; C: etched by 98% concentrated sulfuric acid for 30 s; D: etched by 98% concentrated sulfuric acid for 60 s; PEKK: polyether-ketone-ketone

Figure 2 Comparison of shear bond strengths between PEKK and dentin after different concentrated sulfuric acid etching durations

图2 浓硫酸表面处理不同酸蚀时间后PEKK与牙本质剪切粘接强度的比较

本实验结果表明浓硫酸表面处理能够有效提高PEKK与牙本质的剪切粘接强度，其中浓硫酸酸蚀5 s剪切粘接强度最高，高于氧化锆或钴铬合金与牙本质之间的剪切粘接强度[20-22]。PEKK被浓硫酸处理5 s后剖面呈海绵状粗糙，且孔隙大小均匀，能有效提高剪切粘接强度。此外，PEKK分子式中的苯环被浓硫酸磺化后生成的基团能与visio.link反应，从而提高了剪切粘接强度[17,19]。

本实验结果还显示在3种不同酸蚀时间中，浓硫酸酸蚀时间越长，PEKK与牙本质之间的剪切粘接强度越低。浓硫酸处理5 s可见其剖面类似海绵状结构，且孔隙大小均匀；随着酸蚀时间增加，剖面孔隙大小变得不均匀，且孔隙数量逐渐减少，出现少量孔状塌陷；酸蚀时间60 s后，材料剖面孔隙已经崩解塌陷失去其海绵状稳定结构。这一现象提示随着浓硫酸酸蚀时间增加，PEKK剖面形貌可能由相对稳定的海绵状结构逐渐向一个孔隙减少甚至塌陷的结构转变。其次，浓硫酸酸蚀处理5 s

**表1** 浓硫酸表面处理不同酸蚀时间后粘接失败类型

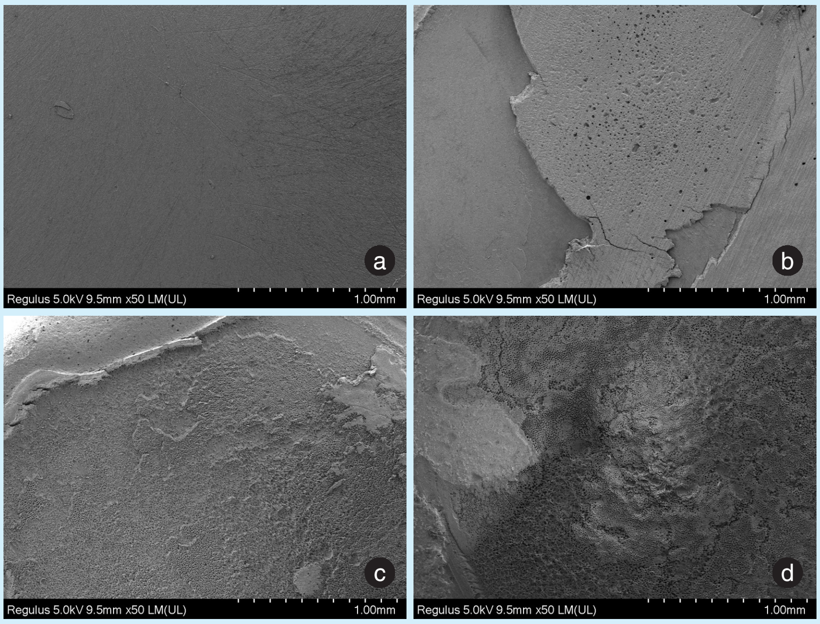
Table 1 The bonding failure types after different concentrated sulfuric acid etching durations

| Groups | Adhesive cement/dentin | Adhesive cement/PEKK | Cohesive in dentin | Cohesive in PEKK | Mixed |
|---|---|---|---|---|---|
| A | 0 | 10 | 0 | 0 | 0 |
| B | 0 | 0 | 0 | 1 | 9 |
| C | 0 | 4 | 0 | 0 | 6 |
| D | 0 | 8 | 0 | 0 | 2 |

A: control group, no etching treatment with concentrated sulfuric acid; B: etched by 98% concentrated sulfuric acid for 5 s; C: etched by 98% concentrated sulfuric acid for 30 s; D: etched by 98% concentrated sulfuric acid for 60 s; PEKK: polyether-ketone-ketone

后，PEKK断裂表面较凸出，有大量粘接树脂残留；随着酸蚀时间增加，PEKK断裂表面稍凹陷，粘接树脂残留减少并可见部分微孔状结构暴露。因此，本实验结果显示浓硫酸酸蚀30 s以上，剖面形成的孔状塌陷结构可能不利于粘接材料与PEKK表面形成有效且稳定的粘接。关于粘接失败类型的结果也提示，酸蚀30 s以上的PEKK与树脂粘接界面破坏明显多于酸蚀5 s组，而酸蚀5 s组大部分表现为混合破坏。

根据上述分析结果，PEKK经浓硫酸酸蚀5 s合并visiolink处理，与牙本质剪切粘接强度最高，该结果为临床PEKK修复体牙本质粘接操作提供了理论依据。另外，本实验缺乏对表面处理和粘接树脂成分对PEKK与牙本质的粘接性能影响及远期效果的研究，后续将计划补充考察PEKK与牙本质粘接界面模拟受口腔环境冷热循环与应力作用下的变化情况。




a: A group, the fracture surface is relatively flat without residual attachment; b: B group, the fracture surface is protruding with a large amount of residual attachments; c: C group, the fracture surface is slightly depressed and has a large number of corrosion-like structures; d: D group, the fracture surface is slightly depressed and has a large number of corrosion-like structures, the surface has less residual attachment than that of group C; A: control group, no etching treatment with concentrated sulfuric acid; B: etched by 98% concentrated sulfuric acid for 5 s; C: etched by 98% concentrated sulfuric acid for 30 s; D: etched by 98% concentrated sulfuric acid for 60 s; PEKK: polyether-ketone-ketone

Figure 3 Scanning electron microscopy images of bonding failure types after different concentrated sulfuric acid etching durations

**图3** 浓硫酸表面处理不同酸蚀时间后粘接失败类型的扫描电镜图

综上，浓硫酸表面酸蚀处理能够有效提高PEKK材料表面性能，提高其与牙本质的粘接效果，经浓硫酸酸蚀5 s结合底涂剂处理的PEKK与牙本质粘接效果较好。

**【Author contributions】** Wang CW performed the experiments, analyzed the data and wrote the article. Sun FF, Yang CC, Ding L, Chen X and Zhang JQ performed the experiments. Wu GF designed the study and reviewed the article. All authors read and approved the final manuscript as submitted.

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