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预酸蚀乳牙牙本质对自酸蚀粘接系统粘接强度的影响*

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摘要 目的:探讨预酸蚀乳牙牙本质对自酸蚀粘接系统粘接强度的影响。**方法:**随机选取 28 颗健康乳磨牙,磨除颊舌面釉质,暴露牙本质粘接面,沿近远中向劈开形成 56 个样本,随机分为 7 组($n=8$)。直接涂布组(A1 组,A2 组和 A3 组)分别涂布 Adper™ Easy One(AEO),Xeno-V(XV)和 OptiBond All In One(AIO)三种自酸蚀粘接剂,预酸蚀组(B1 组,B2 组和 B3 组)在涂布三种自酸蚀粘接剂前先使用 35%磷酸酸蚀乳牙牙本质 15 s,对照组(C 组)使用 Prime & Bond NTTM(NT)全酸蚀粘接剂,每个样本用 Z350 复合树脂堆砌成直径为 3 mm 的树脂小柱,通过剪切试验测试剪切粘接强度,并通过扫描电子显微镜观察断裂表面形态。**结果:**B1 组,B2 组的剪切粘接强度值明显高于 A1 组,A2 组($P<0.001$);B3 组与 A3 组的剪切粘接强度值比较无明显差别($P=0.94$)。A2 组的剪切粘接强度值低于 C 组($P<0.05$);B1 组的剪切粘接强度值明显高于 C 组($P<0.001$)。扫描电镜观察结果显示各组试件断裂面形态多为牙本质和复合树脂界面破坏。直接涂布组(A1 组,A2 组和 A3 组)断裂多发生在混合层的底部,树脂突较少且低于小管口。B1 组和 B2 组试件断裂面可见多数牙本质小管被树脂突填满,断裂多发生在混合层的中上部。B3 组试件断裂面可见牙本质小管空虚,树脂突较少。**结论:**预酸蚀乳牙牙本质可以提高 AEO,XV 两种自酸蚀粘接剂的剪切粘接强度。自酸蚀粘接剂处理乳牙牙本质可以达到全酸蚀粘接剂处理的粘接强度,但应用自酸蚀粘接剂前预酸蚀乳牙牙本质可以获得更高的粘接强度。

关键词:预酸蚀;乳牙牙本质;剪切粘接强度

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Effect of Additional Acid Etching on the Shear Bond Strength of Self-etching Adhesive Applied to Primary Teeth Dentin*

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ABSTRACT Objective: To investigate the effect of additional acid etching on the shear bond strength of Self-etching adhesive applied to primary teeth dentin. **Methods:** 28 healthy deciduous teeth were randomly selected, the lip and cheek enamel were removed, the dentin bonding surface was exposed, and 56 samples along the proximal and distal directions were formed. They were randomly divided into 7 groups ($n=8$), nonetching groups(A1,A2,A3 group)was coated with Adper™ Easy One (AEO), Xeno-V (XV) and OptiBond All In One (AIO) self-etching adhesive, and additional etching groups(B1, B2, B3 group)were etched with 35% phosphoric acid for 15 s before application of the Self-etching adhesive. Control group(group C)was primed with Bond & Bond NTTM (NT), and then use Z350 composite resin to build a resin column with a diameter of 3 mm. Shear test was conducted to test the shear bond strength and the morphology of the fracture surface was observed by scanning electron microscopy. **Results:** The shear bond strength values of group B1 and group B2 were significantly higher than those of group A1 and group A2 ($P<0.001$). There was no significant difference in the shear bond strength between group B3 and group A3($P=0.94$). The shear bond strength value of group A2 was lower than that of group C ($P<0.05$). The shear bond strength of group B1 was significantly higher than that of group C ($P<0.001$). The result of TEM observation indicates that the fracture surface morphology of each group was mostly dentin and composite resin interface damage. Nonetching groups (group A1, A2, and A3) mostly occurred at the bottom of the mixed layer, and the resin protrusions were less and lower than the small nozzles. In the fracture surface of the specimens of group B1 and group B2, most of the dentinal tubules were filled with resin protrusions, and the fracture occurred mostly in the upper middle part of the mixed layer. In the fracture surface of the B3 group, the dentinal tubules were emptied and the resin was less. **Conclusions:** Additional acid etching improve the shear bond strength of AEO and XV self-etching adhesives applied to primary teeth dentin. The self-etching adhesive agent of the deciduous dentin can achieve the bond strength of the total-etching adhesive agent, but the use of a one-step self-etch adhesive with prior acid etching can result in a greater bond strength in primary tooth dentin.

Key words: Additional acid etching; Primary teeth dentin; Shear bond strength

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前言

树脂充填术是乳牙龋齿治疗的主要手段^[1],近年来自酸蚀粘接剂因操作简便,具有较高的临床诊疗效率而备受儿童口腔科医生的青睐。但乳牙与成熟恒牙的化学结构和微观形态均存在差别^[2],研究表明自酸蚀粘接系统应用于乳牙的粘接强度明显低于恒牙^[3-4],且自酸蚀粘接系统对乳牙的粘接强度低于酸蚀-冲洗粘接系统^[5-7]。有学者提出使用自酸蚀粘接剂前预酸蚀乳牙釉质可以提高粘接强度^[8-11],但关于使用自酸蚀粘接剂前预酸蚀乳牙牙本质的实验研究报道较少。本实验以乳牙牙本质为研究对象,探讨了预酸蚀乳牙牙本质对自酸蚀粘接系统粘接强度的影响,以期自酸蚀粘接系统应用于乳牙提供参考。

1 材料与方法

1.1 材料

Gluma Etch 35 Gel 型酸蚀剂购自德国贺利氏公司;Adper™ Easy One 粘接剂购于美国 3M 公司;Xeno-V 粘接剂购于美国 Densply 公司;OptiBond All In One 粘接剂购于美国 Kerr 公司;Prime & Bond NTTM 粘接剂购于美国 Densply 公司;通用型树脂 Filtek Z350 购于美国 3M 公司;光敏固化灯(3M, ESPE, 美国);微机控制电子万能材料试验机(Zwick, 德国);扫描电子显微镜(Hitachi, 日本)。

1.2 方法

1.2.1 实验牙收集 收集哈尔滨医科大学口腔医学院儿童口腔科因乳牙滞留而拔除的乳磨牙 28 颗,要求无龋坏,无裂纹,无釉质缺损等。去除表面软组织及牙石后储存于 4℃ 生理盐水中(3 个月内使用)。

1.2.2 实验样本的制备及分组 选取乳牙牙冠颊舌面牙本质为实验区,流水冷却下高速涡轮手机磨除颊舌面釉质,体视显微镜下观察牙本质完全暴露。使用低速牙钻截除牙根,并沿近远中方向将牙冠劈成两部分,共 56 个样本,用自凝塑料将样本包埋成 9 mm×9 mm×9 mm 的规则模型,确保颊舌面实验开窗区暴露并高于自凝塑料平面 0.2 mm。使用 600 目 SiC 砂纸在流水状态下打磨牙本质表面,形成牙本质粘接面。56 个实验样本随机分为 7 组,每组 8 个样本。直接涂布组(A1 组,A2 组和 A3 组)根据粘接剂使用说明(表 1)分别涂布 Adper™ Easy One(AEO),Xeno-V(XV)和 OptiBond All In One(AIO)三种自酸蚀粘接剂;预酸蚀组(B1 组,B2 组和 B3 组)先使用 35%磷酸酸蚀乳牙牙本质 15 s,加压冲洗 15 s,气枪轻吹牙面,然后按照粘接剂使用说明分别涂布以上 3 种自酸蚀粘接剂;对照组 C 组按照使用说明使用 Prime & Bond NTTM(NT)全酸蚀粘接剂。将直径为 3 mm,高 3 mm 的圆柱形塑料模具固定在实验牙面上,模具与牙本质粘接面垂直,然后用 Z350 复合树脂充填于内,光照。取下模具,每个样本上形成一个直径为 3 mm 的树脂小柱,小柱的长轴与牙本质粘接面垂直。将制备好的实验样本在蒸馏水中保存 24 h,然后测定剪切粘接强度。

表 1 本实验涉及的粘接剂的使用方法

Table 1 Application procedures for the adhesives used in this study

Adhesives	Application procedures
Adper™ Easy One(AEO)	Apply the adhesive to all surfaces of the cavity and rub it in for 20 s, air thin the liquid for 5 s, cure the adhesive with a commonly used curing light for 10 s
Xeno-V(XV)	The same as above
OptiBond All In One (AIO)	Scrub the surface with a brushing motion for 20 s, apply a second application of this adhesive with a brushing motion for 20 s, dry the adhesive for 5 s, and light cure for 10 s
Prime & Bond NTTM(NT)	Acid etching of enamel and dentin for 15 s, pressure washing for 10 s, swabbing with cotton balls, applying adhesive 20 s under wet condition, and light cure for 10 s

1.2.3 剪切粘接强度测定 将制备好的实验样本置于电子万能材料试验机上测试剪切粘接强度,剪切力的方向平行于粘接界面,加速度为 0.5 mm/min,直到树脂与牙面断裂,记录实验数据,带入计算公式,计算剪切粘接强度(shear bond strength, SBS)。

$$\text{剪切粘接强度(SBS)} = \frac{\text{树脂小柱断裂时的最大载荷(N)}}{\text{粘接面积(mm}^2\text{)}}$$

1.2.4 扫描电子显微镜观察 将测试结束后 24 h 的样本固定于铝片上,暴露断裂面,真空喷镀金膜,扫描电子显微镜观察断裂面形态。断裂面的类型分为 4 种:① 牙本质内聚破坏;② 复合树脂内聚破坏;③ 牙本质和复合树脂界面破坏;④ 混合破坏:内聚破坏和界面破坏都存在。

1.3 统计学分析

使用 SPSS20.0 软件分析实验数据,各组数据均用 $\bar{x} \pm s$ 表示,两组间正态分布计量资料均数比较采用独立样本 t 检验,多

组间正态分布计量资料均数比较采用单因素方差分析, $P < 0.05$ 表示有统计学意义。

2 结果

2.1 剪切粘接强度的测定

3 种自酸蚀粘接剂直接涂布组与预酸蚀组的剪切粘接强度值见表 2。B1 组,B2 组的剪切粘接强度值明显高于 A1 组,A2 组($P < 0.001$),B3 组与 A3 组的剪切粘接强度值无明显差别($P = 0.94$)。

3 种自酸蚀粘接剂直接涂布组(A 组)与全酸蚀粘接剂 C 组组间比较显示:A2 组的剪切粘接强度值低于 C 组($P < 0.05$),其他组间比较无明显统计学差异,见表 3。

3 种自酸蚀粘接剂预酸蚀组(B 组)与全酸蚀粘接剂 C 组组间比较显示:B1 组的剪切粘接强度值明显高于 C 组($P < 0.001$),其他组间比较无明显统计学差异,见表 4。

表 2 预酸蚀乳牙牙本质对自酸蚀粘接剂剪切粘接强度值(SBS)的影响($\bar{x} \pm s$, MPa)Table 2 Effects of additional etching on shear bond strengths(SBS) of one-step self-etch adhesives applied to primary teeth($\bar{x} \pm s$, MPa)

	A groups	B groups	P value
AEO	21.15 $\pm$ 2.90(A1)	29.77 $\pm$ 2.12(B1)	<0.001
XV	18.43 $\pm$ 0.74(A2)	26.38 $\pm$ 2.57(B2)	<0.001
AIO	22.66 $\pm$ 5.74(A3)	22.86 $\pm$ 4.46(B3)	0.94

Note: AEO: Adper™ Easy One adhesive; XV: Xeno-V adhesive; AIO: OptiBond All In One adhesive.

表 3 直接涂布组(A 组)与对照组 C 组剪切粘接强度值(SBS)比较($\bar{x} \pm s$, MPa)Table 3 Comparison of shear bond strength(SBS) values between noneching groups(A groups) and control group(C group)($\bar{x} \pm s$, MPa)

Groups	Amount (n)	SBS
Group A1	8	21.15 $\pm$ 2.90
Group A2	8	18.43 $\pm$ 0.74*
Group A3	8	22.66 $\pm$ 5.74
Group C	8	24.10 $\pm$ 3.53

Note: compared with the control group, * $P < 0.05$.

表 4 预酸蚀组(B 组)与对照组 C 组剪切粘接强度值(SBS)比较($\bar{x} \pm s$, MPa)Table 4 Comparison of shear bond strength(SBS) values between additional etching groups(B groups) and control group(C group)($\bar{x} \pm s$, MPa)

Groups	Amount (n)	SBS
Group B1	8	29.77 $\pm$ 2.12 [△]
Group B2	8	26.38 $\pm$ 2.57
Group B3	8	22.86 $\pm$ 4.46
Group C	8	24.10 $\pm$ 3.53

Note: compared with the control group, [△] $P < 0.05$.

2.2 扫描电子显微镜观察结果

扫描电子显微镜成像见图 1, 各组试件断裂面形态多为牙本质和复合树脂界面破坏。直接涂布组(A1 组、A2 组和 A3 组)断裂多发生在混合层的底部, 树脂突较少且低于小管口, 管间牙本质脱矿不明显, 甚至可见较明显的玷污层。B1 组和 B2 组试件断裂多发生在混合层的中上部, 可见大部分牙本质小管被树脂突填满, 树脂突突出于牙本质小管口, 管间牙本质明显脱矿, 胶原纤维网暴露。B3 组试件断裂面可见牙本质小管口开放, 管周牙本质及管间牙本质界限明显, 牙本质小管空虚, 树脂突较少。

3 讨论

第四次全国口腔健康流行病学调查显示 12 岁儿童恒牙患龋率为 34.5%, 5 岁儿童乳牙患龋率高达 70.9%^[12]。乳牙龋损进展速度极快, 患儿通常没有明显的自觉症状, 容易被患儿及家长忽视, 短时间内就可能发展成严重的牙髓病及根尖周疾病, 乳牙根尖周炎症会波及继承恒牙胚, 导致继承恒牙釉质发育不全或萌出方向异常。严重的乳牙根尖周炎症还会继发颌面部间隙感染, 甚至危及患儿生命。因此, 乳牙龋齿的治疗意义重大。树脂充填技术是乳牙龋齿治疗的主要手段^[1], Degrange 等人研究发现粘接失败是导致复合树脂修复失败的主要原因^[13]。近年来, 国内外学者针对如何有效提高牙体硬组织和树脂之间的粘接强度展开了大量的实验研究。

窝洞预备过程中, 机械切削牙本质形成的碎屑中有机物发生变性, 与唾液、牙本质小管溢出液和其他污染物相互混合粘附于洞壁牙本质的表面, 形成厚度约为 1~5 μm 的牙本质玷污层, 有效去除玷污层可以提高牙本质的粘接强度^[14-17]。自酸蚀粘接系统含有酸性单体, 酸性单体可以溶解玷污层^[18,19], 使表层牙本质发生脱矿, 同时单体可渗入到脱矿层的底部形成混合层, 实现树脂本质间的粘接^[20,21]。Nakabayashi 等发现树脂本质间的粘接力主要是混合层和树脂突相互嵌合所形成的微机械力^[22]。

近年来, 自酸蚀粘接系统因其技术敏感性较低, 临床操作时间短, 具有较高的临床诊疗效率而备受儿童口腔科医生的青睐, 但自酸蚀粘接系统广泛应用于临床首先要确保有足够的粘接强度。乳恒牙牙本质在组织学和形态学上有较大差异, 乳牙牙本质的有机质含量较高, 牙本质小管的密度较高, 直径较大, 管周牙本质较厚^[23]。有学者研究发现自酸蚀粘接系统应用于乳牙的粘接强度明显低于恒牙^[3,4]。原因可能是自酸蚀粘接系统的酸性单体无法彻底溶解玷污层^[24]。既往研究显示 35% 的磷酸可有效溶解玷污层而不会导致牙本质过度脱矿^[25,26]。Erickson 等人研究发现预酸蚀恒牙牙釉质可以显著提高自酸蚀粘接系统的粘接强度^[8]。Lenzi 等人实验发现预酸蚀乳牙牙釉质也可以提高自酸蚀粘接系统的粘接强度^[10]。目前, 国内关于使用自酸蚀粘接系统前预酸蚀乳牙牙本质的研究鲜有报道, 本实验则以乳牙牙本质为研究对象, 探究了预酸蚀乳牙牙本质对自酸蚀粘接系统粘接强度的影响。

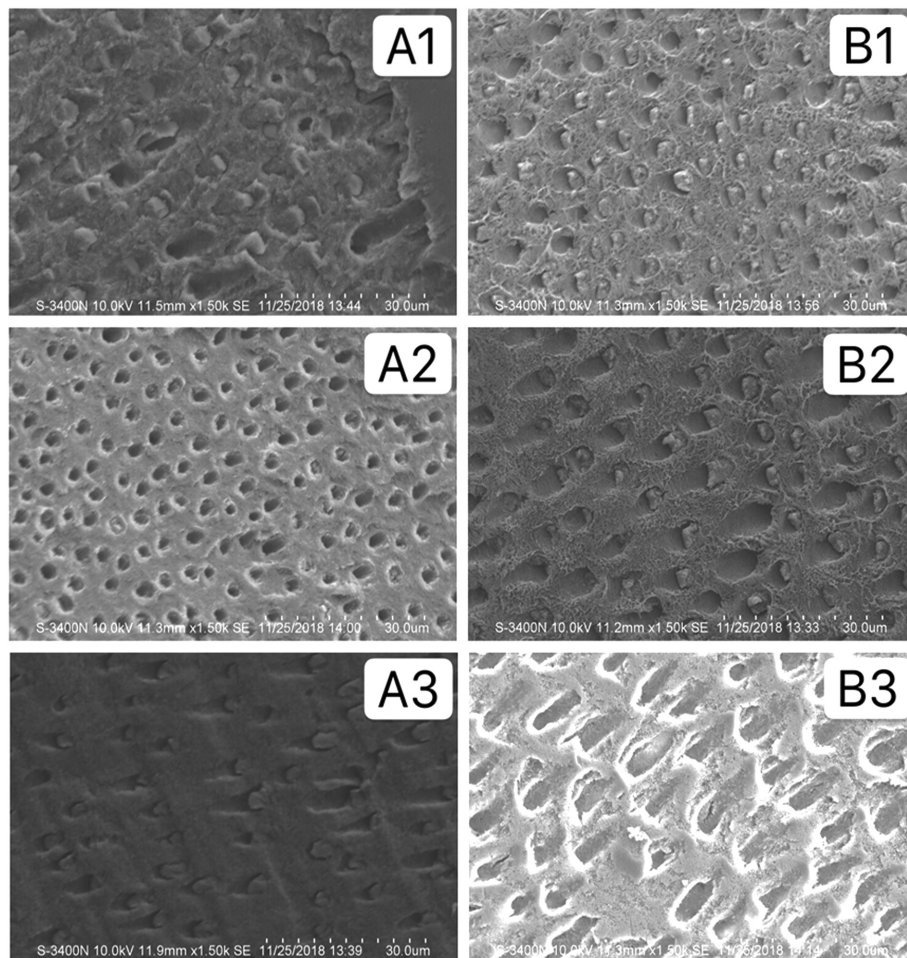


图1 扫描电镜成像

Fig.1 Scanning electron micrograph

本实验研究结果显示预酸蚀乳牙牙本质可以提高 AEO 和 XV 两种自酸蚀粘接剂的牙本质粘接强度,这与 Kim 等^[27]人的研究结果一致。扫描电镜观察断裂面形态显示 A1 组和 A2 组试件断裂主要发生在混合层底部,树脂突较少且低于小管口,管间牙本质脱矿不明显,甚至可见较明显的玷污层;B1 组和 B2 组试件断裂面可见多数牙本质小管被树脂突填满,断裂主要发生在混合层的中上部,树脂突出于牙本质小管口,管间牙本质明显脱矿,胶原纤维网暴露。原因可能是自酸蚀粘接系统内含有的酸性单体酸性较低,无法去除牙本质表面的玷污层,35%的磷酸预处理可以有效的溶解玷污层而提高了牙本质的粘接强度。B3 组与 A3 组的剪切粘接强度值无显著差异,可能是因为 AIO 自酸蚀粘接剂需要连续涂布两次,每次涂布 20 s,酸性单体可以有效溶解玷污层,形成均匀一致的混合层,35%的磷酸预处理反而会导致牙本质过度脱矿,混合层增厚,粘接单体无法充分渗入到混合层底部,在混合层底部形成影响粘接强度的孔隙结构^[28]。Oliveira 等人也发现无论是自酸蚀粘接系统还是全酸蚀粘接系统,较薄的混合层有利于提高树脂本质间的粘接强度^[29]。另外,酸蚀后的冲洗和干燥可能导致暴露的胶原纤维网坍塌,也会影响树脂本质间的粘接强度。这与扫描电镜结果相一致,图 1 可见 A3 组试件多数牙本质小管被树脂突填满,大部分树脂突与牙本质小管口平齐或略低于牙本质小管口,B3 组试件管周及管间牙本质脱矿明显,牙本质小管口空

虚,树脂突较少。Van 等人研究发现自酸蚀粘接系统中的酸性单体酸性较弱,仅使表层牙本质发生脱矿,单体渗入深度不够,自酸蚀粘接系统处理牙体硬组织的粘接强度低于酸蚀 - 冲洗粘接系统^[6]。因此,本实验选择三种临床上常用的自酸蚀粘接剂为实验组,全酸蚀粘接剂为对照组,探究自酸蚀粘接系统和酸蚀 - 冲洗粘接系统应用于乳牙的粘接强度是否存在差异。

3 种自酸蚀粘接系统直接涂布组(A 组)与对照组 C 组进行比较,结果显示 A2 组的剪切粘接强度明显低于 C 组,其他组间比较无差异,原因可能是 XV 自酸蚀粘接系统所含酸性单体的酸性较弱,无法有效去除牙本质表面的玷污层,无法形成均匀一致的混合层,影响了树脂与牙本质之间的粘接强度。另外, XV 自酸蚀粘接系统中的溶剂为乙醇基溶剂,以往研究表明乙醇 / 水基粘接剂的粘接强度低于丙酮基粘接剂的粘接强度^[30]。3 种自酸蚀粘接系统预酸蚀组(B 组)与对照组 C 组进行比较,结果显示 B1 组的剪切粘接强度明显高于 C 组,其他组间比较无差异。AEO 自酸蚀粘接系统含有纳米颗粒的丙烯酸单体, Luiz 等认为丙烯酸单体固化时体积收缩不明显,挥发性较低,可以形成高质量的聚合物,从而增强了粘接系统的粘接强度^[31]。35%的磷酸预处理则有助于清除牙本质表面的玷污层,充分暴露胶原纤维网,提高了牙本质粘接强度。

综上所述,本实验结果表明预酸蚀乳牙牙本质可以提高 AEO、XV 两种自酸蚀粘接系统的剪切粘接强度。自酸蚀粘接系

统处理乳牙牙本质可以达到酸蚀 - 冲洗粘接系统处理的粘接强度,而应用自酸蚀粘接系统前预酸蚀乳牙牙本质可以获得更高的粘接强度。而关于预酸蚀乳牙牙本质是否存在术后敏感及远期临床效果则需要进一步的实验研究。

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