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· 综述 ·

瓷表面处理用氢氟酸替代物四丁基双氟氢氟化铵研究进展

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【摘要】 氢氟酸是常见的玻璃陶瓷修复体粘接前的表面处理剂,但使用氢氟酸存在较高的安全风险,因此寻找氢氟酸替代物一直是研究热点。四丁基双氟氢氟化铵是一种氟化物,化学活性低于氢氟酸,所以安全性更高,对玻璃陶瓷表面形态改变也更小、更表浅。目前,大多数实验室研究和临床病例报告表明,四丁基双氟氢氟化铵表面处理后的玻璃陶瓷的机械强度、粘接强度能够满足临床需求,并且四丁基双氟氢氟化铵在部分国家已经作为氢氟酸替代物用于临床。将来,还可根据研究结果进一步改进含四丁基双氟氢氟化铵的瓷表面处理剂的性能,或者研究出更多的氢氟酸替代物。本文从对玻璃陶瓷的表面形态、机械强度、粘接强度的影响方面,对四丁基双氟氢氟化铵作为氢氟酸替代物的研究进展进行综述。

【关键词】 玻璃陶瓷; 表面处理; 氢氟酸; 四丁基双氟氢氟化铵; 自酸蚀表面处理剂; 表面形态; 机械强度; 粘接强度

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Research progress on tetrabutylammonium dihydrogen trifluoride as a substitute for hydrofluoric acid used for porcelain surface treatment CHEN Jing, CHEN Wenchuan. State Key Laboratory of Oral Diseases & National Clinical Research Center for Oral Diseases & Department of Prosthodontics, West China Hospital of Stomatology, Sichuan University, Chengdu 610041, China

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【Abstract】 Hydrofluoric acid is a common surface treatment agent for glass ceramic restorations before bonding. However, the use of hydrofluoric acid has a high safety risk, so the search for hydrofluoric acid substitutes has been a research hotspot. Tetrabutylammonium dihydrogen trifluoride is a kind of fluoride, whose chemical activity is lower than that of hydrofluoric acid, so it's safer, and the surface morphology changes on glass ceramics caused by it are smaller and more superficial. At present, the vast majority of laboratory studies and clinical case reports indicate that the mechanical strength and bonding strength of glass ceramics treated with tetrabutylammonium dihydrogen trifluoride can meet the clinical requirements. In the future, according to the research results, the performance of porcelain surface treatment agents containing tetrabutylammonium dihydrogen trifluoride can be further improved, and more hydrofluoric acid substitutes may be developed. In this review, the research progress of tetrabutylammonium dihydrogen trifluoride as a substitute for hydrofluoric acid is reviewed in terms of the influence of the surface morphology, mechanical strength, and bonding strength of glass ceramics.

【Key words】 glass ceramics; surface treatment; hydrofluoric acid; tetrabutylammonium dihydrogen trifluoride; self-etching surface treatment agent; surface morphology; mechanical strength; bonding strength

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嵌体、高嵌体、贴面、髓腔固位冠等通过树脂粘接固位的玻璃陶瓷修复体在口腔修复领域的应用越来越广泛。玻璃陶瓷粘接前的表面处理对形成稳固粘接至关重要。氢氟酸(hydrofluoric acid, HF)和硅烷偶联剂处理是玻璃陶瓷表面处理的金标准。但是,氢氟酸对皮肤、眼睛、呼吸道等具有强腐蚀性,还可通过皮肤进入血液和骨组织造成损害^[1]。因此,氢氟酸替代物的研究成为热点。其中,四丁基双氟氢氟化铵(tetrabutylammonium dihydrogen trifluoride, TDTF)在部分国家已经作为氢氟酸替代物应用于临床。TDTF是一种氟化物,化学活性低于氢氟酸,安全性更高,对玻璃陶瓷表面形态改变也更小、更表浅。目前,绝大多数实验室研究和临床病例报告表明,TDTF处理后的玻璃陶瓷能够满足临床需求。然而,关于TDTF和氢氟酸表面处理效果对比的研究结果不一致,并且缺乏随机对照临床试验。本文在归纳总结相关文献的基础上,比较氢氟酸及其替代物TDTF的作用原理、对玻璃陶瓷的表面形态、机械强度以及粘接强度和耐久性的影响,从而对TDTF这一氢氟酸替代物的研究进展进行综述。

1 氢氟酸和四丁基双氟氢氟化铵的作用原理

玻璃陶瓷的主要成分是二氧化硅玻璃基质。氢氟酸能够选择性溶解玻璃基质,增加粘接面粗糙度、粘接面积和表面活性。氢氟酸是一种弱酸,它对玻璃基质的溶解并非通过酸蚀作用,而是通过活性氟离子与玻璃基质生成可被水冲去的氟硅酸。

氢氟酸与玻璃陶瓷发生反应的是活性氟离子,因此氢氟酸替代物的研究对象主要是既有活性氟离子,又具有更高的生物安全性的氟化物。TDTF是一种氟化物,其化学活性低于氢氟酸,安全性更高,对玻璃陶瓷的作用也更温和。另外,因为TDTF的化学性质不同于氢氟酸,所以制造商可以将TDTF和硅烷偶联剂合为一瓶,简化操作步骤,因此含有TDTF的成品表面处理剂又被称作自酸蚀陶瓷表面处理剂。TDTF与玻璃基质的具体化学反应目前尚不清楚。能谱分析发现,TDTF处

理后,玻璃陶瓷表面有氟残留,其临床意义尚不明确^[2]。

2 氢氟酸和四丁基双氟氢氟化铵对玻璃陶瓷粘接效果的影响

2.1 对玻璃陶瓷表面形态的影响

玻璃陶瓷表面形态改变,粗糙度增加,是其与树脂粘接剂形成微机械嵌合,从而稳固粘接的必要条件。氢氟酸处理会明显改变玻璃陶瓷表面形态^[3];并且,形态改变与氢氟酸浓度和作用时间正相关。氢氟酸处理强度较低时在表面形成微孔,而处理强度较高时则形成大而深的不规则沟纹^[4]。另外,氢氟酸处理强度过高会造成玻璃陶瓷表面的过度溶解,如10%氢氟酸处理白榴石玻璃陶瓷60 s不仅会溶解大量的玻璃基质,也会溶解部分白榴石晶体,这与材料的成分和结构特点有关^[4]。

TDTF处理也能够改变玻璃陶瓷表面形态,但较氢氟酸处理后的表面形态改变更小、更表浅,且主要发生在材料表面原本的沟纹周围^[2]。将TDTF处理时间从40 s延长到2 min,虽然玻璃陶瓷表面的形态改变增加,但是仍然小于5%氢氟酸处理60 s的形态改变^[5]。在处理深度方面,Murillo-Gómez等^[4]通过扫描电镜比较氢氟酸和TDTF处理玻璃陶瓷的深度,发现玻璃陶瓷的处理深度与氢氟酸的作用强度呈线性增长关系,10%氢氟酸处理60 s会溶解0.3~0.6 mm深度的玻璃基质,而5%氢氟酸或TDTF的作用则较表浅。总体而言,TDTF处理玻璃陶瓷较氢氟酸处理后的表面形态改变更小、更表浅,并且随作用时间延长玻璃陶瓷发生的形态改变也更少,也表明TDTF临床使用的技术敏感性更低。

2.2 对玻璃陶瓷机械强度的影响

玻璃陶瓷修复体的机械强度至关重要,因为修复体的破裂意味着修复失败。玻璃陶瓷修复体表面粗糙度越大,其本身的机械强度越小。这是因为玻璃陶瓷表面的形态改变可能引入结构缺陷,导致应力集中、裂纹扩展^[6]。Murillo-Gómez

等^[4]建议,处理贴面这样菲薄的修复体时,可使用更温和的方法(5%氢氟酸或TDTF)处理玻璃陶瓷,以保护材料结构完整性。

Luo等^[7]的研究显示,使用9.5%氢氟酸处理二硅酸锂玻璃陶瓷20、40、60、120 s,玻璃陶瓷挠曲强度随着处理时间增加而降低。但是,在进行树脂粘接以后,各组玻璃陶瓷的挠曲强度均较粘接前明显增加,并且不同处理组之间无明显差异。这是因为,树脂粘接剂充填入玻璃陶瓷表面粗糙形态后,瓷修复体和树脂粘接剂之间的紧密接触能形成持续均匀的界面,强化修复体^[8]。所以,表面粗糙度的增加对修复体机械强度形成积极的或者消极的影响,既取决于是否引入表面结构缺陷,也取决于树脂粘接剂对粗糙形态的充填程度。因此,在要求范围内使用氢氟酸对玻璃陶瓷修复体进行表面处理并树脂粘接后,通常不会因为增加表面粗糙度而明显降低机械强度^[9]。

有研究比较了TDTF和氢氟酸处理后的玻璃陶瓷树脂粘接后的机械强度,结果显示,TDTF处理组能获得和氢氟酸处理组相似甚至更佳的抗疲劳强度,并观察到导致修复体破裂的裂纹来源于粘接界面的基础裂纹^[10]。也有研究显示,TDTF处理可获得更好的机械强度。Schestatsky等^[11]研究发现,TDTF处理后的玻璃陶瓷有更好的机械强度可靠性。Dapieve等^[12]研究发现,人工老化不影响TDTF和5%氢氟酸处理后的玻璃陶瓷粘接后的抗疲劳强度;将TDTF的反应时间从40 s增加至2 min也不影响抗疲劳表现。但是,也有研究显示,TDTF处理后的玻璃陶瓷机械强度并不优于氢氟酸处理组。Tribst等^[13]检测二硅酸锂玻璃陶瓷粘接后的双轴挠曲强度发现,虽然TDTF处理组的即刻双轴挠曲强度略高于氢氟酸组,但是在人工老化以后,TDTF组的双轴挠曲强度发生了明显的降低(10.7%)。另外,Scherer等^[14]发现,对于粘接后的二硅酸锂玻璃陶瓷,TDTF处理组在人工老化前后的挠曲强度均低于5%氢氟酸处理组。总体而言,尽管这些研究针对TDTF和氢氟酸相比较的结果不一致,但这些研究均显示TDTF组老化后的机械强度足够承受除夜磨牙以外的正常咬合力,展示出良好的使用前景。

2.3 对玻璃陶瓷粘接强度的影响

玻璃陶瓷修复体与树脂粘接剂获得较高的粘接强度是修复成功的必要条件。研究表明,氢氟酸浓度和处理时间都会影响玻璃陶瓷的粘接强

度^[15-17]。但过度延长氢氟酸处理时间或增加氢氟酸浓度并不能提高粘接强度^[18],处理时间延长至2 min甚至会降低粘接强度^[19]。但是,使用更温和的TDTF处理二硅酸锂玻璃陶瓷时,完成涂布后,将处理时间从40 s延长到80 s和2 min对粘接强度无影响^[20],表明其临床使用的技术敏感性更小。

修复体粘接界面同时存在抗拉伸强度和抗剪切强度,抗拉伸强度相比抗剪切强度更能反映实际粘接强度。对于抗剪切强度,许多研究显示TDTF处理与氢氟酸处理相似甚至更好^[5,21-22];但也有研究显示,TDTF处理玻璃陶瓷粘接后获得的抗剪切强度明显低于氢氟酸处理的结果^[2,23]。对于抗拉伸强度,大部分研究显示TDTF处理玻璃陶瓷粘接后获得的抗拉伸强度与氢氟酸处理相似甚至更好^[18,24-25]。

2.4 对玻璃陶瓷粘接强度耐久性的影响

修复体的粘接耐久性对修复的长期成功至关重要。老化会使粘接界面的化学结合发生水解,并且形成切变应力,使裂纹扩展,降低粘接强度。许多研究显示,TDTF处理和氢氟酸处理多种玻璃陶瓷获得的粘接强度在人工老化以后明显下降,并且二者下降后的粘接强度类似^[24,26-30]。Prado等^[31-32]的研究结果显示,在老化试验以后,TDTF处理组的抗拉伸强度降低更少,表现出更高的粘接稳定性。TDTF组处理后的玻璃陶瓷表面有更少的碳,意味着硅烷偶联剂层更加均匀,更不易发生水解,这可能是粘接耐久性更好的原因之一^[2]。但是,Dimitriadi等^[33]的实验结果显示,TDTF处理后的玻璃陶瓷在老化前后的粘接强度都比氢氟酸处理组更低。大部分实验中,TDTF和氢氟酸人工老化处理后的样本都是以附着性或者混合性脱粘接为主^[2,22-28,30],表明TDTF处理和氢氟酸处理后的玻璃陶瓷都有较好的粘接强度耐久性。不同的研究结果可能是因为不同的实验设计,比如有的研究表面处理前对玻璃陶瓷表面进行了抛光^[23],有的研究在氢氟酸处理后使用了磷酸去除沉淀物^[21]。

临床病例报告显示,使用TDTF处理后进行粘接的玻璃陶瓷修复体,在6个月^[22]和2年^[34]后,美学和功能表现良好,未出现裂纹、边缘渗漏或脱粘接。但TDTF应用于临床时间较短,因此临床病例报告较少,也缺乏随机对照临床试验。

3 总结与展望

氢氟酸能够方便有效地对玻璃陶瓷进行表面

处理,获得理想的表面形态、粘接强度和耐久性;并且因为树脂粘接对玻璃陶瓷的加强作用,使得氢氟酸对玻璃基质的过度溶解问题不再突出,按要求操作后能够获得理想的机械强度;使用氢氟酸主要的问题还是它的高安全风险。TDTF作为氢

氟酸替代物,安全性更高,临床的技术敏感性更低。并且,TDTF对玻璃陶瓷表面形态改变更小、更表浅,处理后的玻璃陶瓷获得的机械强度和粘接强度能够满足临床需求(表1)。

然而,TDTF作为氢氟酸替代物还需要被进一

表1 氢氟酸和四丁基双氟氢氟化铵对玻璃陶瓷表面处理的对比

Table 1 Comparison of hydrofluoric acid and tetrabutylammonium dihydrogen trifluoride in surface treatment of glass ceramics

Items	Hydrofluoric acid	Tetrabutylammonium dihydrogen trifluoride
Security risks	High	Low
Action principle	Selective dissolution of silica glass substrate	Selective dissolution of silica glass substrate
Surface morphology change of glass ceramics	Big and deep	Small and superficial
Mechanical strength of glass ceramics	Reinforced by resin adhesive filling, it can meet clinical needs	The results are inconsistent with HF comparison. It can meet the clinical needs
Glass ceramic bonding strength and durability	Clinical gold standard	The results are inconsistent with HF comparison. It can meet the clinical needs
Technical sensitivity	Higher	Lower

步研究,因为尽管处理玻璃陶瓷在目前的实验室研究和临床病例报告中有较好的结果,但目前还缺乏随机对照临床试验,并且它与玻璃陶瓷的具体化学反应尚不明确。将来,研究者们还可能通过改进目前研究的氟化物,或者发现新的能够安全有效地处理玻璃陶瓷的化学物质,从而研究出更多的氢氟酸替代物。

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