

基于激光烧蚀碳纤维离子化质谱法快速鉴别 化妆品包装材料

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摘要: 本研究采用激光烧蚀碳纤维离子化质谱(LACFI-MS)法对市面上常见品牌的妆前乳、洁面泡沫的包装进行真伪鉴别。通过将碳纤维通入辅助溶剂, 并施加高电压, 无需对化妆品包装进行预处理, 即可直接进行激光烧蚀; 激光解吸的化学成分在空气中被电离, 进而进入质谱仪分析。利用该方法对妆前乳和洁面泡沫包装的不同颜色区域(深蓝、金、灰、乳白、黑等)进行分析, 结果表明: 正品包装在不同批次间具有稳定、可重复的特征质谱峰(如 m/z 320、412、264 等); 而赝品信号峰杂乱, 特征峰差异显著; 部分赝品样品间存在共有特征峰(如 m/z 342、347、663、680), 提示可能源于共同的造假源头。此外, 本研究还通过串联质谱(MS/MS)对特征峰进行结构验证。该方法无需样品预处理, 具有绿色、灵敏度高、操作简单的优点, 可实现化妆品包装真伪的高通量、快速鉴别。

关键词: 化妆品包装; 质谱; 直接分析; 敞开式离子化; 碳纤维离子化

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Rapid Identification of Cosmetic Packaging Materials Based on Laser Ablation Carbon Fiber Ionization Mass Spectrometry

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Abstract: The rapid rise of counterfeit high-quality cosmetic packaging has become a major challenge for regulatory authorities, manufacturers, and forensic identification agencies. Traditional identification strategies—such as visual inspection, print quality assessment, or chromatographic and spectroscopic analysis—have become increasingly ineffective against counterfeit packaging materials that closely mimic the appearance and structural design of genuine products. In this study, a method based on laser ablation carbon fiber ionization mass spectrometry (LACFI-MS) was proposed for the authenticity identification of cosmetic packaging, without the need for sample pretreatment. The cosmetic packaging sample was placed directly on an x - y moving platform without any preprocessing. The laser working voltage was adjusted through a transformer to accurately locate the area to be

tested (including various color areas such as deep blue, gold, gray, etc.) at low energy, and then the voltage was increased to the operating conditions for laser ablation, desorbing components from the packaging surface. The desorbed components were ionized by a charged carbon fiber tip (applying high voltage) in air, assisted by a continuous flow of methanol-water solvent (1:1, *V/V*) at a flow rate of 2 $\mu\text{L}/\text{min}$, combined with mass spectrometric detection, allowing the acquisition of the chemical fingerprint profiles of different colored regions of cosmetic packaging such as concealer and cleansing foam. Experimental results indicated that genuine packaging has stable and repeatable characteristic mass spectral peaks (such as *m/z* 320, 412, 264, etc.) across different batches, while counterfeits showed chaotic signals with significant differences in characteristic peaks. In contrast, the spectral patterns of counterfeit samples show significant heterogeneity and are generally chaotic, characterized by highly fluctuating peak distributions and a large number of irreproducible ion peaks. This variability suggests inconsistent raw material sources, weak formula control, and low production standards. Notably, some counterfeit samples share specific ion clusters (e.g., *m/z* 342, 347, 663, 680), indicating that they may originate from common suppliers or related counterfeiting networks. This demonstrated that LACFI-MS is not only suitable for authenticity identification but also has the potential to trace the origins and circulation routes of counterfeit packaging in illegal supply chains. The study verified the structures of key diagnostic peaks through surface tandem mass spectrometry (MS/MS), confirming the reliability of the assigned chemical structures for the characteristic peaks. Comparative analysis with the literature indicated that the numerous unique differential ions in the counterfeit samples may originate from low-quality photoinitiators, plasticizers, fatty amide lubricants, or low-cost ink/coating additives—substances often associated with nonstandard production processes. These findings further demonstrate the potential safety risks and compositional instability of counterfeit packaging materials. Overall, the LACFI-MS combines multiple advantages, such as rapid analysis, ease of operation, no need for sample pretreatment, high sensitivity, and excellent capability to distinguish subtle chemical differences. This technique not only efficiently and accurately distinguishes between genuine and counterfeit products but also has the potential to trace the source of counterfeiting, thereby providing strong technical support for the regulation of the cosmetics market.

Key words: cosmetic packaging; mass spectrometry; direct analysis; ambient ionization; carbon fiber ionization

近年来,化妆品产业持续发展,包装作为产品制造的终端环节,承担着保障存储安全、维护运输稳定以及辅助市场营销等多重职能^[1]。其中,纸制品包装和塑料类包装占据主导地位,构成了化妆品包材应用的主体^[2-3]。作为主流包材,虽便于生产,但因易复制而成为化妆品造假的主要途径^[4-6]。化妆品包装造假工艺已从粗糙模仿升级为“精仿”,对传统肉眼鉴别方式提出了挑战^[7]。

为应对这一挑战,国内外研究者和产业界致力于发展多元化的真伪检测技术^[8],主要分为2大类:一类是基于包装与标签的宏观物理鉴

别,如使用工具辅助观察印刷精度、包装材质、批次编码、防伪标签(全息图^[9]、二维码^[10]、射频识别 RFID^[11])等,该方法虽简便,但易漏检^[12-13];另一种是基于产品内在成分的微观化学鉴别,也是目前研究的重点,利用光谱学(如近红外光谱(NIR)^[14]、拉曼光谱^[15])、色谱学(如高效液相色谱(HPLC)^[16]、气相色谱-质谱(GC-MS)^[17])以及稳定同位素分析^[18]等手段,通过建立正品的“指纹图谱”数据库,实现对产品化学成分的定性与定量分析,从而为真伪判定提供客观、科学的依据。Andoh等^[19]将傅里叶变换红外光谱(FTIR)与化学计量学相结合,用于区分正品和假冒化妆

品的包装材料(如塑料、玻璃),实现了高精度无损检测。

近年来,质谱(MS)凭借高灵敏度、高特异性等优势,已成为分析物质组成和结构不可或缺的“黄金标准”手段^[20-24]。在中药分析领域,张强等^[25]报道了碳纤维电离质谱(CFI-MS)技术的双重应用价值:其一,无需前处理即可直接表征中药及其复方制剂中多种挥发性成分;其二,能够据此快速鉴别中药材的新鲜程度。在化妆品鉴别领域,刘鸣畅等^[26]采用快速蒸发电离质谱(REIMS)结合化学计量学模型,实现了化妆品真伪的快速鉴别与质量溯源;夏泽敏等^[27]建立了超高效液相色谱-串联质谱(UPLC-MS/MS)法快速测定化妆品中 *N,N*-二甲基-1,3-二氨基丙烷(DMAPA)、*N*-[3-(二甲氨基)丙基]月桂酰胺(LAPDMA)、*N*-[3-(二甲氨基)丙基]肉豆蔻酰胺(MYPDMA)3种有机胺类风险物质;Rubio等^[28]采用GC-MS技术结合三阶校准方法,实现了防晒霜中2,6-二叔丁基-4-甲基苯酚(BHT)、二苯甲酮(BP)、邻苯二甲酸二异丁酯(DiBP)和羟苯甲酮(BP3)4种添加剂的准确识别与定量分析,有效克服了共洗脱干扰和保留时间漂移的问题,成功应用于7款市售防晒霜样品的检测。然而,现有技术多集中于对化妆品的鉴别,忽略了对其包装材料的分析^[29-30]。因此,有必要建立一种简单、快速、直观的检测方法,将“不可见特征”转化为“可量化的检测信号”。

本研究将采用激光烧蚀碳纤维离子化质谱(LACFI-MS)法对化妆品包装进行真伪鉴定,利用激光烧蚀对化妆品包装不同颜色区域的化学成分进行在线、原位提取,样品分子到达施加高

电压的碳纤维离子源附近被电离,随后进入质谱离子传输管完成检测,旨在提升化妆品市场监督与质量管控效率提供技术参考。

1 实验部分

1.1 主要仪器与装置

ZenoTOF 7600 四极杆飞行时间质谱仪:美国 Sciex 公司产品;碳纤维:日本 TORAY Industries 公司产品;Milli-Q 纯水仪:美国 Millipore 公司产品;直流高压电源:宿迁波尔高压电源有限公司产品;激光电压调节模块:青岛欣易云联电子科技有限公司产品。

本实验室开发的 LACFI-MS 装置示意图和整体结构示于图 1。LACFI 离子源由 2 部分组成:1)激光器,通过烧蚀样品表面,实时原位提取样品;2)碳纤维电离源,由 1 根嵌套在金属管的长 15 mm 碳纤维束构成,通过施加高压实现离子化。

激光器参数:激光波长 450 nm,输出功率 2.5 W,脉宽 120 ns,重复频率 20 kHz。

碳纤维规格:3 K(K 代表 1 000 根碳纤维单丝组合而成的束丝),直径 6.9 μm 。

在操作过程中,将辅助溶剂甲醇-水(1:1, *V/V*)以 2 $\mu\text{L}/\text{min}$ 持续通入碳纤维,碳纤维电离源在质谱仪入口斜前方,与质谱仪入口同轴。

向碳纤维施加 3.5 kV 电压,碳纤维尖端与质谱入口水平距离为 5 mm。

1.2 主要材料与试剂

妆前乳、洁面泡沫样品:由上海市食品药品检验研究院提供,包括品牌专柜与官方渠道购买的正品、监管与投诉渠道获得的假冒样本,样本信息列于表 1。甲醇:色谱级,德国 Merck 公司

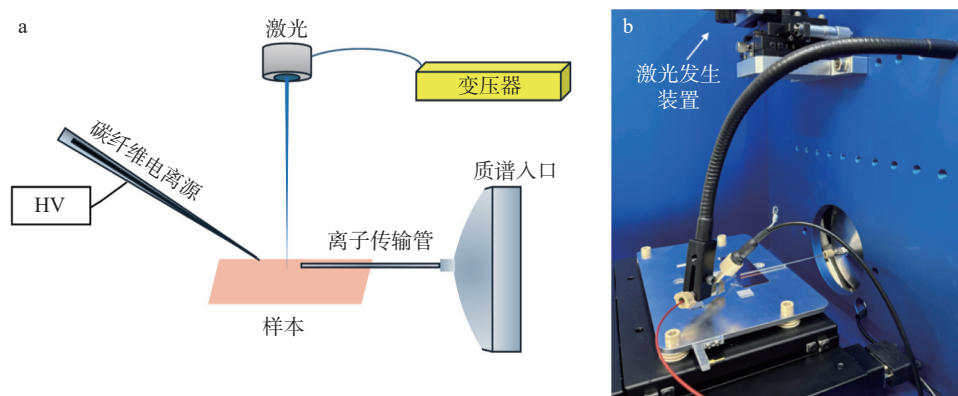


图 1 LACFI 电离源示意图(a)和实物图(b)

Fig. 1 Schematic (a) and physical image (b) of LACFI source

产品。

为确保包装区域具有代表性,本研究依据涂层体系、油墨配方及印刷工艺的差异,选取不同颜色区域(金、黑、蓝、白、灰等)进行分析,尤其关注品牌标识、主色块等关键视觉元素,以便更灵敏地捕捉包装化学成分的差异。

表 1 妆前乳、洁面泡沫的样品信息

Table 1 Information on primer and cleansing foam

样品 Sample	编号 No.	是否正品 Genuine or not
妆前乳	P-1	是
	P-2	是
	P-3	否
	P-4	否
	P-5	否
	P-6	否
洁面泡沫	CF-1	是
	CF-2	是
	CF-3	是
	CF-4	否
	CF-5	否
	CF-6	否

1.3 实验条件

一级质谱质量扫描范围 m/z 50~1 000, 离子传输管温度 75 °C, 检测器压强 1.52×10^{-6} kPa。采用串联飞行时间质谱(TOF MS/MS)进行特征峰结构验证。

1.4 实验方法

将各批次包装样品置于 x - y 轴移动平台上, 控制移动平台可实现单个样品依次检测。通过变压器(图 2)调节激光器工作电压, 首先降低激



图 2 激光电压调节示意图

Fig. 2 Laser voltage regulation schematic

光能量, 通过摄像头观察斑点位置, 以精确定位待测区域(图 3); 定位完成后, 升高电压至工作条件, 利用激光烧蚀解吸样品成分, 并在空气中由带电碳纤维尖端实现样品分子的高效离子化, 最终导入质谱仪进行分析。

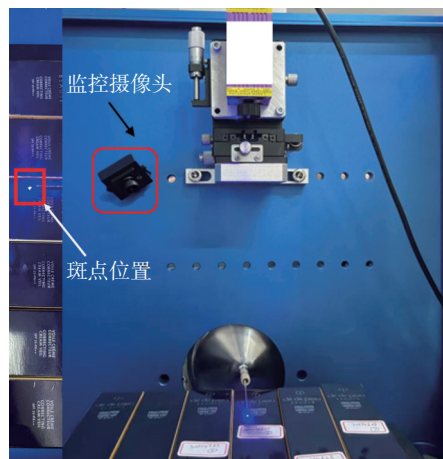


图 3 采样区定位示意图

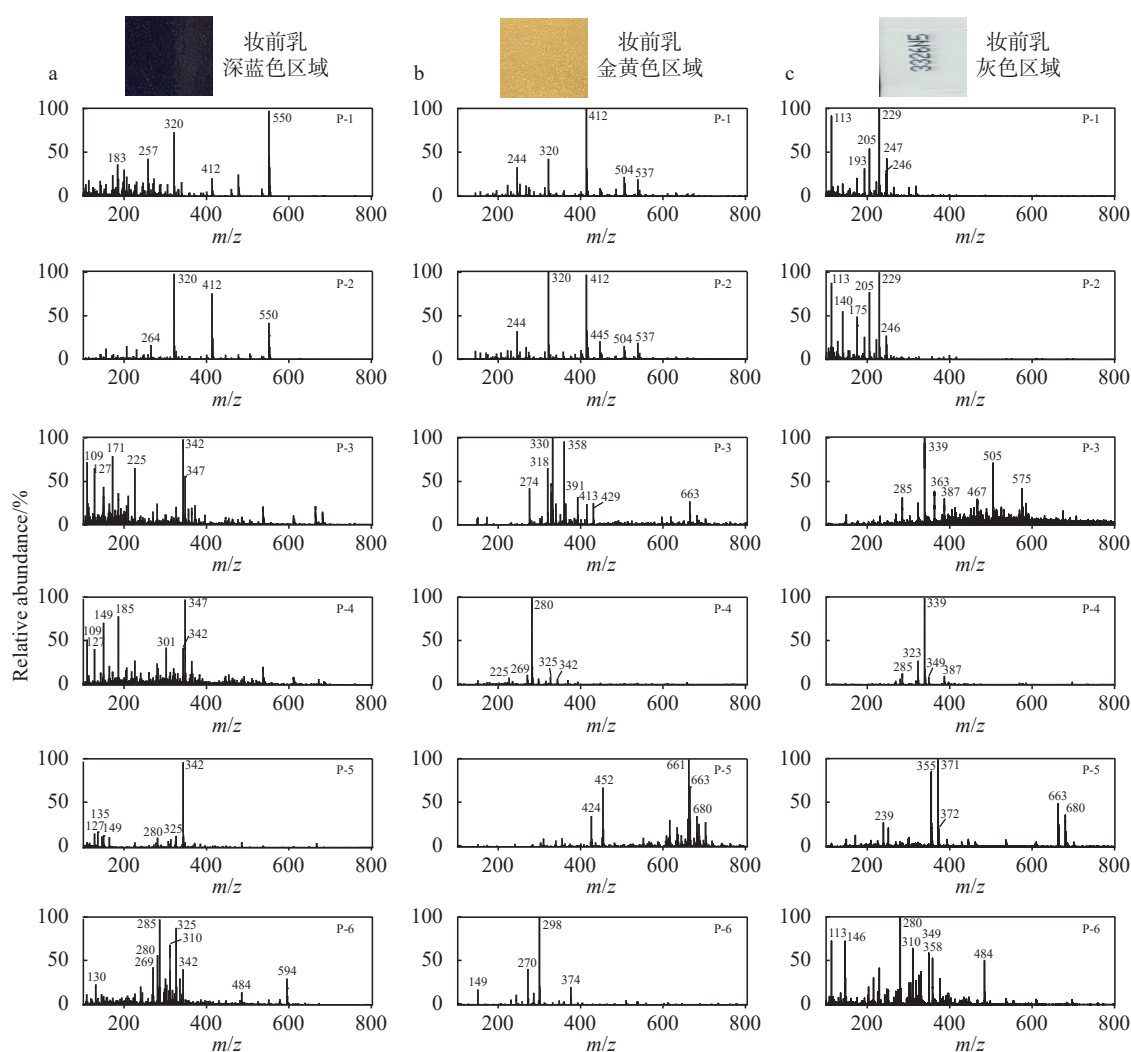
Fig. 3 Sampling area location schematic

2 结果与讨论

2.1 妆前乳正品和赝品的 LACFI-MS 质谱图比较

选择妆前乳包装 3 个不同颜色区域进行分析, 其 LACFI-MS 质谱图示于图 4。可见, 在不同批次间, 正品虽存在特征峰强度的细微差异, 但特征峰信号一致, 表明其包装材料组成稳定。其中, 深蓝色与金黄色区域均检出 m/z 320、412, 表明这 2 个区域的基底材料可能相同; 而深蓝色区域的特有峰 m/z 550, 以及金黄色区域的特有峰 m/z 537、504, 则可能源于不同的着色或涂层成分。值得注意的是, 正品灰色区域未检测到上述共有特征峰, 推测其涂层较厚或基底材质不同, 导致表面化学组成存在差异。

相比之下, 赝品包装的质谱图信号杂乱, 不同样本间的特征峰差异显著, 缺乏一致性。值得注意的是, 部分赝品在相同颜色区域显示出共有特征峰: 如 P-3、P-4、P-5 与 P-6 在深蓝色区域均出现 m/z 342 特征峰; 其中 P-3 与 P-4 不仅在深蓝色区域均出现 m/z 347 特征峰, 而且在灰色区域均出现 m/z 339 特征峰。这些共有特征峰表明相应赝品可能来源于同一造假作坊, 生产工艺与原料具有一定的相似性。



注: a. 深蓝色区域; b. 金黄色区域; c. 灰色区域; P-1、P-2 为正品; P-3、P-4、P-5、P-6 为赝品

图 4 妆前乳正品和赝品的 LACFI-MS 质谱图

Fig. 4 LACFI-MS mass spectra of genuine and counterfeit primer

2.2 洁面泡沫正品和赝品的 LACFI-MS 质谱图比较

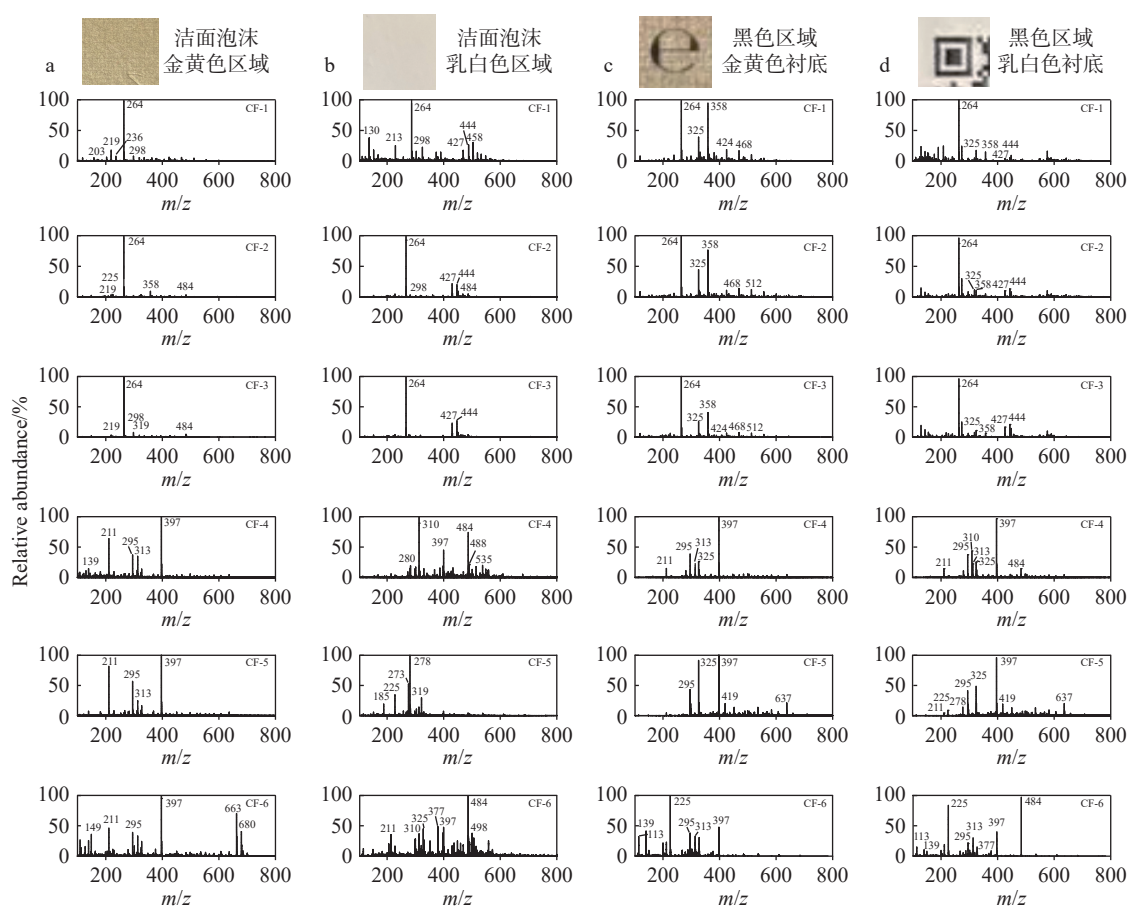
对洁面泡沫外包装 3 个不同颜色区域进行分析,其质谱图示于图 5。结果表明,在不同批次间,正品表现出高度一致的特征峰信号,证实其包装材料组成稳定、工艺可控。其中, m/z 264 在 3 个颜色区域均有检出,表明这 3 个区域可能采用相同的基底材料。颜色区分可通过各区域特征峰实现:如,乳白色区域的特征峰为 m/z 444、427,黑色区域的特征峰为 m/z 358、325,进一步表明了着色层或表面处理工艺的差异。

相比之下,赝品包装的质谱信号杂乱,且样本间差异显著,反映了其原料与生产工艺的不稳定。值得注意的是,在送检样本中,编号为 CF-4

与 CF-6 的 2 批赝品,其质谱图呈现高度相似的特征峰分布与相对信号强度,提示二者可能源于同一造假源头或生产工艺,为追踪假冒商品流通过程提供了重要线索。

综上所述,在不同批次间,虽然正品的质谱峰信号强度有细微差异,但具有相同的关键特征峰,表明正品生产工艺和材质的高度一致性和可重复性。而赝品的质谱图杂乱,且不同赝品之间的特征峰差异显著,缺乏一致性。同时,在赝品中也发现一些潜在的关联:如 P-3、P-4 和 P-5 显示出相似的特征峰;CF-4 和 CF-5 金黄色区域和黑色区域的质谱图高度相似。

值得关注的是,通过跨品类比较发现,妆前乳 P-5 与洁面泡沫 CF-6 在谱图中均出现 m/z 663、



注: a. 金黄色区域; b. 乳白色区域; c. 黑色区域金黄色衬底; d. 黑色区域乳白色衬底; CF-1、CF-2、CF-3 为正品; CF-4、CF-5、CF-6 为赝品

图 5 洁面泡沫正品和赝品的 LACFI-MS 图

Fig. 5 LACFI-MS mass spectra of genuine and counterfeit facial cleansing foam

680 等共有特征峰,提示二者可能源自同一造假生产源头,这不仅为化妆品真伪鉴别提供了依据,更凸显了 LACFI-MS 技术在造假溯源方面的应用潜力。

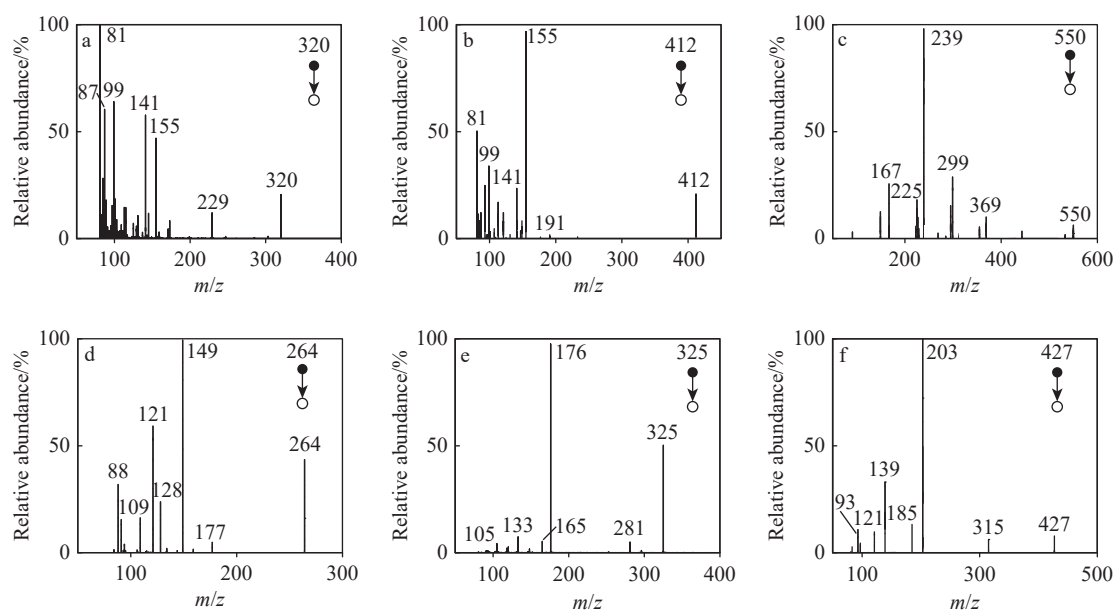
另外,本研究对洁面泡沫包装表面的黑色区域进行分析。该区域不仅出现在金黄色区域,亦分布于乳白色区域。为验证不同颜色区域的差异,分别对上述 2 个区域的黑色区域进行检测。结果表明,对于正品洁面泡沫包装,位于乳白色区域上的黑色区域不仅能检出乳白色衬底的特征峰信号(如 m/z 427、444),也能检出黑色区域的特征峰信号(如 m/z 325、358),但其信号强度显著低于金黄色衬底上的黑色区域。这一现象可能与乳白色涂层的物理或化学抑制效应有关。该结果进一步验证了前述推测,即金黄色、乳白色和黑色区域均采用相同的基底材料,并通过涂覆相应颜色涂层或印刷层来实现视觉区

分。而赝品虽大致符合上述假设,但不同样本之间缺乏一致性,表明其基底与涂层材料或工艺不稳定。

2.3 对特征峰的初步鉴定

为进一步证明 LACFI-MS 方法的可行性,对初步筛选出的特征峰进行原位串联质谱(MS/MS)分析,分别在碰撞诱导解离(collision-induced dissociation, CID)和电子活化解离(electron activation dissociation, EAD)模式下获得特征峰的碎片离子信息,示于图 6、7。结果表明,这些特征峰并非偶然噪声,而是由特定化合物产生的信号,增强了鉴定结果的科学性和可信度。

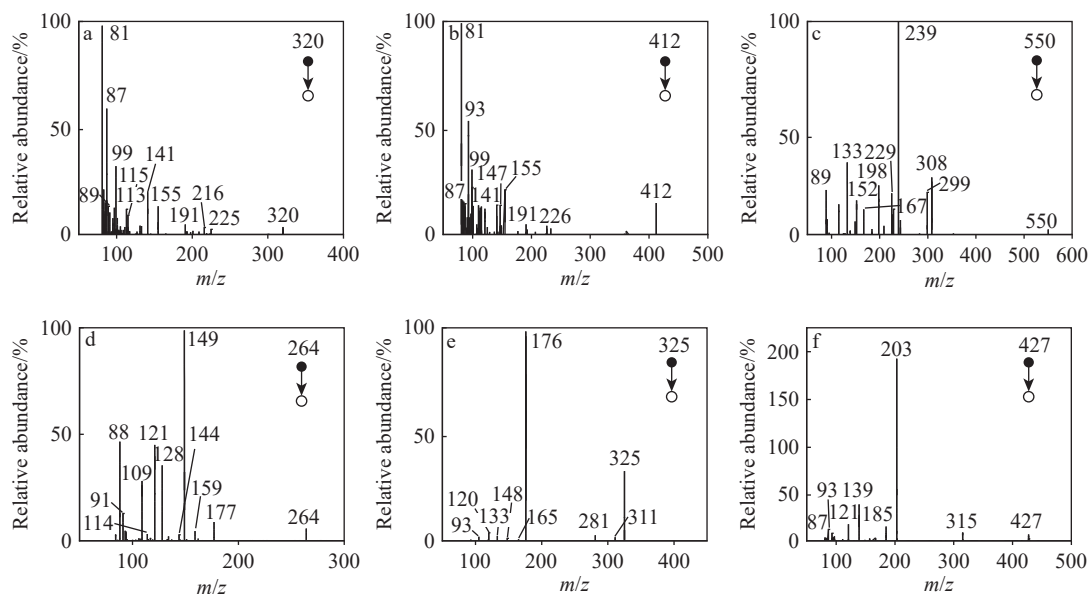
赝品的不同颜色区域均呈现出相较正品更复杂的特征峰分布,提示其包装材料或印刷体系中存在额外或非同质的化学组分。结合相关报道^[31]可推断,这些差异峰主要来源于印刷油墨及涂层体系中使用的特定添加剂,包括光引发剂、



注: a. m/z 320; b. m/z 412; c. m/z 550; d. m/z 264; e. m/z 325; f. m/z 427

图 6 CID 模式下各特征峰的串联质谱图

Fig. 6 Tandem mass spectra of each characteristic peak under CID mode



注: a. m/z 320; b. m/z 412; c. m/z 550; d. m/z 264; e. m/z 325; f. m/z 427

图 7 EAD 模式下各特征峰的串联质谱图

Fig. 7 Tandem mass spectra of each characteristic peak under EAD mode

塑化剂、润滑与脱模剂, 以及可能的染料或颜料助剂等。值得注意的是, 部分赝品在相同颜色区域出现高度一致的峰群, 表明其可能采用了相同来源或相似配方的油墨、涂层或基材, 反映出潜在的共同供应链或造假工艺路线。这些“共有化学标签”不仅有助于真伪区分, 也为追溯假冒包装的来源提供了线索。

3 结论

本研究采用 LACFI-MS 法快速鉴别化妆品包装的真伪, 无需样品预处理即可实现对化妆品包装的原位、快速检测, 能够有效区分正品与赝品包装。妆前乳和洁面泡沫的正品包装均显示出稳定、重复性强的特征峰; 而赝品信号杂乱且存在显著差异, 部分共同特征峰提示可能相同的

造假来源。该方法操作简便、灵敏度高,具有良好的应用前景,可为化妆品市场监管与真伪鉴别提供有力的技术支持。需要指出的是,本研究仅通过串联质谱对部分特征峰进行初步结构解析,仍需进一步结合标准品比对等分析手段,以实现更精确的结构确证与来源归属;此外,本研究的样本量相对有限,结论的普适性需进一步验证。

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