# 食品接触材料纸和纸板中非故意添加物质分析方法的研究进展

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**摘 要:** 食品接触材料中的非故意添加物质是指包装材料与食品之间化学反应的副产物或新形成的化合物、低聚物、降解产物、原料中的杂质。在食品接触材料中, 纸和纸板是除塑料之外最常用的材料, 而纸和纸板中的非故意添加物质主要来自印刷油墨、粘合剂、上浆剂和表面涂料。本文主要对食品接触材料纸和纸板中非故意添加物质分析方法的研究进展进行了概述, 旨在给国内相关研究提供参考, 并有助于后续食品接触材料纸和纸板中非故意添加物质分析工作的开展。

**关键词:** 食品接触材料纸和纸板; 非故意添加物质; 分析方法

## Research progress on analytical approaches of non-intentionally added substances in food contact materials paper and board

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**ABSTRACT:** Non-intentionally added substances in food contact materials refer to by-products of chemical reaction between packaging materials and food, or newly formed compounds, oligomers, degradation products and impurities in raw materials. Among the food contact materials, paper and cardboard are the most commonly used materials besides plastics, while the non-intentionally added substances in paper and cardboard mainly come from printing inks, adhesives, sizing agents and surface coatings. This article mainly summarized the research progress of the analysis methods of non-intentionally added substances in food contact material paper and paperboard, in order to provide reference for domestic related research, and help to carry out the subsequent analysis of non-intentionally added substances in food contact material paper and cardboard.

**KEY WORDS:** food contact materials paper and board; non-intentionally added substances; analytical method

## 1 引 言

食品接触材料（food contact materials, FCM）在运输和存储过程中对食品进行包装和保护, 以延长食品的保质期。食品接触材料包括与食品接触的所有材料, 是由原材料和有意添加物质(intentionally added substances, IAS)共同生产加工而成, 添加有意添加物质是为了提高食品接触材料本身的稳定性和机械性能。有意添加物质包括单体, 预聚物, 抗氧化剂, 润滑剂, 表面活性剂和紫外稳定剂。除有意添加物质外, 食品接触材料还可能含有非故意添加物质

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(non-intentionally added substances, NIAS), 这些物质可能来源于包装材料与食品之间的化学反应的副产物或新形成的化合物、低聚物、降解产物、原料中的杂质[1]。在食品包装材料中, 纸和纸板是除塑料之外最常用的材料, 因此消费者很可能会吃到用纸和纸板包装的食物。食品包装毋庸置疑应该是安全, 但是纸和纸板的多孔特性使其无法阻碍化学物质迁移到食品中[2,3]。

由于欧盟法规 2011/10 第 19 条中提到非故意添加物质, 因此公众加强了对非故意添加物质的关注[4], 欧盟法规 2011/10 规定: 非故意添加物质允许存在于最终的制品中, 但应由制造商根据国际公认的科学原则对其进行风险评估。由于目前没有参考标准, 通常无法对非故意添加物质进行定量分析。在风险评估方面, 仅考虑分子量达到 1000 Da 的非故意添加物质[4]。本文主要对食品接触材料纸和纸板中非故意添加物质分析方法研究方面的文献报道进行了梳理与概述, 以期在为过国内相关学者提供一定的研究参考。

## 2　非故意添加物质分类

非故意添加物质的来源各不相同, 可以分为反应副产物或新形成的化合物、低聚物、分解或降解产物, 原料中的杂质以及在生产或回收过程中产生的污染物。降解过程是非故意添加物质形成的最常见途径。不仅食品接触材料的基材本身可能会发生降解, 而且用于改善食品接触材料物理化学性能的添加剂也会发生降解。降解产物可进一步分为聚合物降解和添加剂降解。微波炉加热、其他加热方式、出于灭菌目的的紫外线照射、消费者的误使用、自然老化过程都会使食品接触材料产生较低分子量的化学物质[5]。与较高分子量的化学物质相比, 较低分子量的化学物质具有更高的扩散系数, 因此迁移到食物中风险更高[1]。

食品接触材料中添加添加剂以增强其性能, 比如抗氧化剂和光稳定剂。Alin 等[6]和 Burman 等[7]分别对抗氧化剂降解产物进行研究。亚磷酸三(壬基酚)酯用于增强某些聚合物树脂的性能, 氧化后会产生烷基酚、辛基酚和壬基酚。烷基酚也可能来自聚乙氧基壬基酚的降解, 而聚乙氧基壬基酚是生产塑料瓶常用清洁剂中的表面活性剂[8,9]。

偶氮颜料本身是用于配制油墨的故意添加物质, 用于印刷油墨的偶氮颜料中存在伯芳族胺(primary aromatic amines)和 *β*-萘酚, 这 2 种化学物质作为杂质存在于偶氮颜料和最终油墨配方中, 因此伯芳族胺、*β*-萘酚是非故意添加物质[10,11]。

副产品或新形成的非故意添加物质可能在食品接触材料制造过程中或是在消费者使用食品接触材料过程中产生。这些化合物也可能是食品接触材料中化合物与食品之间相互作用的产物。例如使用频率较高的聚氨酯粘合剂一般是通过多元醇和二异氰酸酯单体的聚合反应制成, 若粘合剂未正确固化或组分未正确混合, 会造成聚合反应的不完全, 并且剩余的未聚合芳族异氰酸酯会与水反应形成伯芳族胺[12], 形成新的非故意添加物质就是伯芳族胺[13]。环氧漆可能含有双酚 A 和双酚 A 二缩水甘油醚, 双酚 A 二缩水甘油醚可以与食物蛋白反应形成副产物也是非故意添加物质[14]。

污染物也属于非故意添加物质, 污染物有可能迁移到食品中, 因此需要将污染物包括在风险评估中[15]。污染物与杂质不同, 从某种意义上说, 在食品接触材料的生产过程中或是使用过程中会产生污染物, 例如来自于印刷油墨中的矿物油饱和烃和矿物油芳烃、全氟化合物(全氟酸和全氟酸磺酸盐)是其他类型的非故意添加物质。

## 3　非故意添加物质分析方法

非故意添加物质分析过程的第一步是收集食品接触材料纸和纸板中可能存在的非故意添加物质和有意添加物质的化合物的信息。Van-Bossuyt 等[16]公布了食品接触材料纸和纸板中已知和被使用的物质清单, 根据安全性和理化数据评估了 6073 种化合物, 并将这些化合物与其他官方清单进行比较, 在所有已标识和分类的化合物中, 42%被归类为单一物质, 20%归为聚合物, 18%归为混合物, 20%归入其他物质(金属络合物和无机物质)。纸和纸板中发现的非故意添加物质主要来源是印刷油墨, 粘合剂, 上浆剂, 表面涂料, 生产过程原料中的杂质[5,17]。在纸和纸板中经常检测到的非故意添加物质是芳香胺、双酚 A、双酚 A 二缩水甘油醚、全氟化合物、邻苯二甲酸盐、印刷油墨和矿物油。采用适当的分析技术对非故意添加物质进行化学分析, 收集非故意添加物质的信息。

### 3.1　靶标分析法

经过前处理的已知存在的非故意添加物质可以使用靶标分析法进行分析。尽管在大多数情况下使用质谱, 但是分析方法和检测器的选择还是以分析的非故意添加物质类别为基准。表 1 概述了食品接触材料纸张和纸板中用靶标分析法检测到的不同类别的非故意添加物质。

挥发性的非故意添加物质一般采用气相色谱-质谱(gas chromatography-mass spectrometry, GC-MS)的方法进行分析[20,26,30,38,41], 半挥发性和不挥发性的非故意添加物质合物则采用气相色谱和液相色谱-质谱(liquid chromatography-mass spectrometry, LC-MS)进行分析[12,31]。

Fierens 等[38]研究了比利时市场上销售的 400 种食品和食品包装中是否存在邻苯二甲酸酯化合物, 将样品分为高脂食品、低脂食品、饮料和包装材料, 使用了 4 种不同的提取方法, 采用 GC-MS 进行分析。Parigoridi 等[20]通过 GC-MS 分析了 3 种再生纸板中 5 种有机污染物的存在情况, 还应用了超声波辅助溶剂萃取的提取方法。Bradley 等[41]采用气相

**表 1 靶标分析法测定食品接触材料纸和纸板中不同类别的非故意添加物质**

**Table 1 Determination of different types of non-intentionally added substances in food contact material paper and paperboard by target analysis method**

| 化合物 | 样品 | 分析技术 | 参考文献 |
|---|---|---|---|
| 胶粘剂 | 层压纸制基材 | 大气压气相色谱串联四极杆飞行时间质谱(Atmospheric pressure gas chromatography tandem quadrupole time-of-flight mass spectrometry, APGC-QTOF-MS) | [18] |
| 蒽、二苯甲酮、邻苯二甲酸二甲酯、硬脂酸甲酯和五氯苯酚 | 纸和纸板 | 气相色谱火焰离子化检测器(gas chromatography flame ionization detector, GC-FID) | [19] |
| 二苯甲酮、2,6-二异丙基萘和 2,7-二异丙基萘、间三联苯和邻三联苯 | 再生纸板 | 气相色谱-质谱(gas chromatography-mass spectrometry, GC-MS) | [20] |
| 双酚 A | 纸和纸板 | 高效液相色谱-质谱(high performance liquid chromatography-mass spectrometry, HPLC-MS) | [21] |
| 双酚 A、双酚 A 二缩水甘油醚、双酚 F、双酚 F 二缩水甘油醚 | 再生纸 | 超高效液相色谱-串联四极杆飞行时间质谱(ultra Performance liquid chromatography-tandem quadrupole time-of-flight mass spectrometry, UPLC-QTOF-MS) | [22] |
| 双酚 A、邻苯二甲酸二辛酯 | 再生纸和纸板 | GC-MS | [23] |
| 双酚 A、双酚 A 类似物 | 纸 | GC-MS-MS | [24] |
| 化学污染物 | 纸板 | GC-MS | [25] |
| 矿物油 | 纸和纸板 | HPLC-GC-FID | [26] |
| 矿物油 | 纸板 | GC-FID | [27] |
| 矿物油 | 再生纸 | GC-FID | [28] |
| 矿物油 | 纸和纸板 | HPLC-GC-FID | [29] |
| 多环芳烃和正烷烃 | 纸张回收过程中产生的灰尘 | GC-MS | [30] |
| 全氟烷基磺酸盐 | 可微波爆米花袋 | UPLC-QTOF-MS(负模式) | [31] |
| 全氟烷基磺酸盐 | 爆米花袋 | UPLC-QTOF-MS | [32] |
| 全氟烷基磺酸盐 | 纸 | 液相色谱串联质谱(liquid chromatography tandem mass spectrometry, LC-MS-MS) | [33] |
| 全氟烷基磺酸盐 | 纸板 | PIGE 光谱法 | [34] |
| 全氟烷基磺酸盐 | 纸 | UPLC-MS-MS | [35] |
| 光引发剂 | 纸板 | UPLC-MS-MS | [36] |
| 光引发剂 | 纸 | LC-MS-MS | [37] |
| 邻苯二甲酸盐 | 纸和纸板 | GC-MS | [21] |
| 邻苯二甲酸盐 | 食品和纸板 | GC-MS | [38] |
| 邻苯二甲酸盐 | 纸板 | GC-MS | [39] |
| 邻苯二甲酸盐 | 纸 | GC-MS-MS | [33] |
| 芳香胺 | 聚氨酯纸粘合剂 | GC-MS-MS | [12] |
| 芳香胺 | 纸/塑料层压板 | 超高效液相色谱-高分辨质谱(ultra performance liquid chromatography - high resolution mass spectrometry, UPLC-HRMS) | [40] |
| 印刷油墨 | 纸和纸板 | GC-MS | [19] |
| 印刷油墨化合物: 二苯甲酮、4-甲基二苯甲酮、2-甲基二苯甲酮、3-甲基二苯甲酮、4-羟基二苯甲酮、2-羟基二苯甲酮、4-苯基二苯甲酮、2-苯甲酰基苯甲酸甲酯、1-羟基环己基苯基酮、2-异丙基噻吨酮、4-异丙基噻吨酮、 2,4-二乙基-9H-噻吨-9-酮、2,2-二甲氧基-2-苯基苯乙酮、2-甲基-40-(甲硫基)-2-吗啉代苯丙酮、4-(4-甲基苯硫基)二苯甲酮、乙基-4 - 二甲基氨基苯甲酸酯、2-乙基己基-4-(二甲基氨基)苯甲酸酯、N-乙基 - 对甲苯磺酰胺、磷酸三苯酯和富马酸二(2-乙基己基)酯 | 食品包装印刷纸/纸板食品包装盒和里面容纳的食品 | GC-MS | [41] |

色谱-质谱法分析了 350 种用印刷纸或纸板包装的不同食品中的油墨化合物, 测定了所有食品溶剂提取物中 20 种特定的印刷油墨化合物。Nguyen 等[42]研究了印刷油墨化合物从纸和纸板间接迁移到食品的过程, 提出了食品与纸板之间被塑料层隔开时的迁移机制。

### 3.2　非靶标分析法

对于未知的非故意添加物质的标识采用非靶标分析法。在完成已知非故意添加物质的筛选分析后, 通常会面对未知的非故意添加物质的“峰林”, 需要有信息量很大、及时跟新的化合物数据库和软件工具进行标识[43]。Biedermann 等[44]甚至表示, 不可能通过非靶标分析法标识食品接触材料纸和纸板中的所有非故意添加物质, 只是标识了用于包装食品的再生纸板中与健康有关的潜在组分, 采用乙醇和正己烷 1:1(*V*:*V*)的混合液中浸泡 3 d 后提取, 将提取液浓缩在乙醇中, 并通过高效液相色谱分馏成 7 个部分, 采用全二维气相色谱结合飞行时间质谱, 检测出超出 10 μg/kg 检测限的非故意添加物质超过 250 种。Canellas 等[45]采用 GC-MS 和超高效液相色谱结合飞行时间质谱标识多层纸中水基生物降解胶粘剂的非故意添加物质, 用 GC-MS 分析挥发性的非故意添加物质, 而不挥发性的非故意添加物质则用 UPLC-QTOF-MS 分析。表 2 概述了食品接触材料纸张和纸板中用非靶标分析法检测到的不同类别的非故意添加物质。

## 4　结　论

对非故意添加物质的分析具有较大难度, 因为它们的存在和化学结构通常是未知的。采用靶标分析法用于分析已知的非故意添加物质; 非靶标分析法用于分析未知的非故意添加物质。所有分析方法均应定性定量分析食品接触材料中存在的非故意添加物质。对于分析已知的非故意添加物质较为容易, 但由于缺乏参考标准, 对于未知的非故意添加物质存在较多困难。尽管非故意添加物质的化学结构通常很难确定, 但是使用高灵敏度的先进分析技术可以检测大多数非故意添加物质。对食品接触材料纸和纸板中非故意添加物质分析研究仍需进一步深入和拓展, 以便为后续制定污染限值以及科学评估对人体健康的影响提供依据。

**表 2　非靶标分析法检测食品接触材料纸和纸板中的不同类别的非故意添加物质**

**Table 2　Determination of different types of non-intentionally added substances in food contact material paper and paperboard by nontarget analysis methods**

| 化合物 | 样品 | 分析技术 | 参考文献 |
|---|---|---|---|
| 2,6-二叔丁基-4-羟基甲苯、二叔丁基苯酚、二苯甲酮、4,4'-双(二甲基氨基)二苯甲酮、三苯甲烷 | 再生纸和纸板 | 顶空 GC-MS<br>GC-MS<br>高效液相色谱-二极管阵列检测器(high-performance liquid chromatography-diode array detection, HPLC-DAD)<br>电感耦合等离子体质谱(inductively coupled plasma mass spectrometry, ICP-MS) | [46,47] |
| 双酚 A、甲基苯甲酸、松香酸、双酚 A 二缩水甘油醚 | 非再生纸和再生纤维 | HPLC(用于分馏)<br>UPLC-MS/MS(标识) | [5] |
| 脱氢松香酸和松香酸 | 再生纸板 | GC-MS<br>LC-MS | [48] |
| 矿物油: 矿物油芳烃、矿物油饱和烃 | 再生纸板 | 在线 HPLC-GC-FID | [49] |
| 矿物油: 矿物油芳烃、矿物油饱和烃 | 纸板 | 在线 HPLC-GC-FID | [50] |
| 矿物油 | 纸和纸板 | 在线 HPLC-GC-FID<br>GC×GC-MS | [26] |
| 污染物 | 再生披萨盒 | UPLC-QTOF-MS<br>GC-QTOF-MS | [51] |

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