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磁性分子印迹聚合物在食品安全检测中的应用

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摘 要： 磁性分子印迹聚合物是将分子印迹技术与磁性材料相结合制备的物质。分子印迹技术是一种新型高效分离及分子识别技术，具有特异的识别性和选择性；而磁性材料又具有超顺磁性，能在外加磁场的作用下将其从溶液中快速分离，还可以通过共聚或表面修饰等途径使其表面有多种反应官能团，以吸附或共价键合的方式与目标分子相结合。两者结合后制备的磁性分子印迹聚合物，兼备了磁性材料和分子印迹聚合物的共同优点，具有特异的识别性和选择性，同时也避免了分子印迹聚合物需要离心或抽滤才能从溶液中分离出来的缺点，具有快速分离的特点。本文重点综述了磁性分子印迹聚合物的制备方法及其优缺点，以及磁性分子印迹聚合物在食品中的农药残留、生物医药残留和兽药残留等方面的检测应用及其研究进展。

关键词： 分子印迹；磁性分子印迹聚合物；食品安全

Application of magnetic molecularly imprinted polymers in the analysis of food safety

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ABSTRACT: Magnetic molecularly imprinted polymers (MMIPs) were prepared by the molecular imprinting technology (MIT) combined with a magnetic material. MIT was a new type of high efficient separation and molecular recognition technology, and had the specific recognition and selectivity. Magnetic materials had superparamagnetism, which could be separated from the solution under additional magnetic field fast, with their surface getting a variety of reaction functional group through the way such as copolymerization or surface modification, which could be combined with target molecules by adsorption or covalent bonds. After the combination, MMIPs had the common advantages of magnetic materials and molecularly imprinted polymer, such as specific recognition and selectivity, and could be separated fast from the solution. This paper summarized the preparation methods of MMIPs and their advantages and disadvantages, and the application of MMIPs in the analysis of pesticide residues, biological medicine residues and veterinary drug residues in foods.

KEY WORDS: molecular imprinting; magnetic molecularly imprinted polymer; food safety

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1　引　言

分子印迹技术 (molecularly imprinted technology, MIT) 是指制备对某一目标分子具有特异选择性的聚合物, 分子印迹聚合物具有特异的识别性和选择性, 能从复杂样品基质中选择性分离富集目标分子及其结构类似物[1-4]。MIT 的缺点是在聚合物与溶液分离的过程中需要经过抽滤或离心等步骤, 比较复杂。当 MIT 与磁性材料相结合则可以避免这些不足。磁性材料比较常用的是 Fe_3O_4, 一方面, 可通过共聚、表面改性等途径, 赋予其表面多种反应官能团, 通过吸附或共价键合的方式与目标物结合; 另一方面, 由于其具有超顺磁性可以很方便地在外加磁场作用下从介质中分离。将磁性材料与 MIT 相结合, 利用分子印迹聚合物包覆无机磁性粒子制备的磁性分子印迹聚合物 (magnetic molecularly imprinted polymers, MMIPs)[5-7] 即具有磁性材料的超顺磁性, 也显示了印迹聚合物较高的特异选择性。MMIPs 提取出目标分析物后, 可以在外部磁场的作用下更容易和快速地从复杂的体系中将聚合物分离出来, 不用通过离心或抽滤等繁琐的步骤, 其操作简单且易于分离。由于其表面积大, 具有独特的物理和化学性质, 磁性印迹聚合物已广泛应用于许多领域, 如食品安全、环境检测、生物分离和药物传递等[8-17]。

本文重点综述了磁性印迹聚合物的制备方法及其优缺点, 以及磁性分子印迹聚合物对食品中的农药残留、生物医药残留和兽药残留等方面的检测应用, 并对目前所存在的问题和未来的发展进行了展望。

2　磁性分子印迹聚合物的制备

磁性分子印迹聚合物主要分为悬浮聚合法、乳液聚合法, 分散聚合法。

2.1　悬浮聚合法

磁性分子印迹聚合物的合成方法比较常用的是悬浮聚合法[18], 在含有磁性粒子的反应体系中, 加入单体、引发剂、稳定剂, 通过高温热引发或低温光引发的加热方式[19,20]进行制备。悬浮聚合的优点是可以制备大粒径的微球, 能根据自己的需要对为求得粒径进行调整, 缺点是悬浮聚合的微球粒径分布比较宽[21]。

2.2　乳液聚合法

乳液聚合法[22-25]也是制备分子印迹聚合物的重要方法, 主要分为种子乳液聚合法、无皂乳液聚合法、微乳液聚合法、反相微乳液聚合法。反应体系的主要组分为单体、引发剂、乳化剂和分散介质, 制备出的聚合物粒径分布较悬浮聚合法分布窄。粒径较小, 但产物中含有乳化剂不易洗净。

2.3　分散聚合法

分散聚合法[26]是通过在反应体系中加入单体、引发剂、稳定剂和分散介质进行合成, 此方法制备出的印迹聚合物分散性比较好, 但影响合成的因素较多, 且粒径的均匀性不易控制。

3　磁性印迹技术在食品安全检测中的应用

磁性印迹技术在食品安全检测中的应用大致可以分为农药残留、兽药残留、生物医药等几方面, 具体介绍如下:

3.1　食品中农药残留的检测

为了提高农作物产量, 人们一般都会使用杀虫剂, 而杀虫剂在除掉害虫的同时也会残留部分在农作物中, 对人体造成危害。毒死蜱和二嗪农都属于有机磷杀虫剂, 能较好地防治多种作物的害虫。Ma 等[27]以毒死蜱为模板, 甲基丙烯酸为功能单体, 三羟甲基丙烷为交联剂制得磁性印迹聚合物, 用于从大米中提取毒死蜱, 并用高效液相色谱法对提取的毒死蜱进行分析测试, 经分析得到: 检出限为 0.0072 μg/g, 回收率为 81.2%~92.1 %。该方法不仅易于分离, 还克服了选择性低的问题。Davoodi 等[28]以二嗪农为模板制得的磁性印迹聚合物, 用液相色谱法分析从黄瓜组织中提取出的二嗪农, 得到的回收率为 82%~110%。农药灭草隆主要用于土壤处理, 防除葡萄、甘蔗、棉花和大田作物中的单子叶和双子叶杂草。韩爽等[29]以灭草隆为模板, 在包覆 SiO_2 的 Fe_3O_4 微球颗粒表面上进行分子印迹, 制备了分散均匀的磁性核壳分子印迹聚合物微球, 并用双光束紫外可见分光光度计进行分析, 结果表明磁性印迹聚合物对灭草隆具有良好的选择性和特异识别功能, 最大吸附容量达到 80 mol/g, 使样品中的灭草隆很好的富集, 且制备过程简单, 能够快速分离。

3.2　食品中兽药残留的检测

二甲硝咪唑是畜禽的促生长剂和高效广谱抗原虫药。因为二甲硝咪唑现在被认为具有遗传毒性、致突变和致癌的副作用, 所以许多国家已经禁止在动物的养殖中使用。Hu 等[30]利用溶胶凝胶法结合表面印迹技术合成了核-壳型磁性印迹聚合物。以二甲硝咪唑为模板, 甲基三甲氧基硅烷和 3-氨丙基三乙氧基硅烷为功能单体, 正硅酸乙酯为交联剂合成磁性印迹聚合物。分析结果表明: 对猪饲料的加标回收率为 90.3%~106.2%, 相对标准偏差小于 4.54%。

磺胺由于其抗菌谱广、疗效好等优点被广泛用于兽药临床。黄镭等[31]以磺胺为模板分子, 甲基丙烯酸为功能单体利用悬浮聚合法制备出磺胺磁性印迹聚合物复合微球, 最大表观结合量为 280.99 μmol/g, 平衡离解常数为 1.58×10^{-3} mol/L。Chen 等[32]制备的磁性印迹聚合物可以从水环境中提取四种磺胺类抗生素, 经测定聚合物的饱和磁化强度值为 16.7 emu/g, 用外加磁场可以轻易地从环境水样中脱离出来, 大大降低了预处理时间。用液相色谱-串联质谱法测定目标分析物, 得到的检出限为 0.38~1.32 ng/L, 日间和日内的相对标准偏差分别为 1.3%~6.8% 和 1.7%~9.1%, 回收率为 31.4%~82.0%。Karimi 等[33]用磺胺甲嘧啶为模板合成的磁性印迹聚合物从鸡肉中提取出磺胺类药物, 用高效液相-紫外检测的方法进行测定, 检测限为 0.1~0.5 μg/L, 回收率为 95.5%~103.0%, 显示出了良好的选择性。An 等[34]制备出对恩氟沙星具有识别作用的磁性分子印迹硅胶, 研究表明, 磁性分子印迹硅胶与非印迹硅胶对恩氟沙星的吸附量分别为 45.74 mg/g 和 35.55 mg/g, 可见磁性分子印迹硅胶能更好地结合恩氟沙星。

3.3 食品中生物医药残留的检测

药物进入环境已成为在过去的十年全球关注的主要环境问题。由于其高持久性、潜在的毒性和生物降解性低, 越来越多的药物及其衍生物在各种环境介质中被发现。抗癫痫药卡马西平作为一种研究最多的药物, 已经在饮用水中被发现[35,36]。Zhang 等[37]为了开发出可以快速地从水样中有选择性地分离出卡马西平的前处理方法, 合成了核-壳型的卡马西平磁性印迹聚合物。通过高效液相法进行分析, 结果表明: 核-壳型磁性印迹聚合物对卡马西平具有较高的萃取能力。磁性聚合物微球具有磁响应性和特异的表面功能团, 能与其他小分子、大分子或细胞等结合[38], 在磁场的作用下可定向运动到指定部位, 或从周围介质分离出来。苏立强等[39]以壳聚糖修饰的四氧化三铁为载体, 制备了磁性胰蛋白酶分子印迹聚合物, 最大吸附量为 162 mg/g。You 等[40]合成了能够提取姜黄素的热响应性磁性印迹聚合物, 经高效液相色谱法分析后, 结果显示相对标准偏差小于 4%, 回收率为 94%~98%, 检出限为 0.02 μg/mL。氯霉素是一种广谱抗生素, 对革兰氏阳性菌和革兰氏阴性菌均有抑制作用。然而, 高剂量则会造成细胞缺乏症、再生障碍性贫血等[41,42]。Chen 等[43]以氯霉素为模板合成磁性印迹聚合物, 用于萃取蜂蜜中的氯霉素, 在液相色谱-串联质谱的测试条件下, 得到检测限为 0.047 ng/g, 回收率为 84.3%~90.9%。He 等[44]利用磁性分子印迹聚合物提取人体尿液中的氟喹诺酮类药物, 回收率为 83.1%~103.1%, 相对标准偏差为 0.8%~8.2%。

3.4 食品中其他方面的检测

3.4.1 塑化剂

在食品安全检测方面, 一个有效的样品前处理有助于提高分离和检测。磁性分子印迹方法既可以快速分离又具有特异选择性。邻苯二甲酸酯类(PAEs)增塑剂是使用最广泛的增塑剂[45-47], 如今大多数食品包装都是塑料制品, 所以塑料包装在为消费者提供方便的同时也带来了食品安全隐患。Qiao 等[48]利用磁性印迹固相萃取测定塑料瓶装饮料中的 PAEs, 用气相色谱法测定从维他命饮料中提取的塑化剂, 分析结果表明: 样品的回收率为 89.5%~101.3%, 检测限为 0.53~1.2 μg/L, 分离效果好, 回收率高。

3.4.2 三聚氰胺

He 等[49]以三聚氰胺为模板制备的磁性分子印迹聚合物提取牛奶中的三聚氰胺, 用液相色谱-串联质谱法进行分析, 检测限为 2.6 ng/mL, 回收率为 88.0%~95.8%。

3.4.3 双酚 A

双酚 A 是世界上使用最广泛的工业化合物之一, 广泛应用于罐头食品和饮料的包装、奶瓶、水瓶、牙齿填充物所用的密封胶、眼镜片以及其他数百种日用品的制造过程中。Ji 等[50]利用细乳液聚合法合成磁性印迹聚合物, 提取环境水样和牛奶中的双酚 A, 并用高效液相色谱法进行测定。结果显示: 磁性印迹聚合物对环境水样的检测限为 14 ng/L, 相对标准偏差为 2.6%~4.7%, 回收率为 89%~106%; 对于牛奶样品的检测限为 0.16 μg/L, 相对标准偏差为 6.5%~7.5%, 回收率为 95%~101%。

4 展 望

磁性分子印迹技术不仅能选择性地分离富集目标分析物, 有效降低杂质的干扰, 而且能够运用磁性在外加磁场的作用下达到快速的分离。因此它是一种快速、经济、高选择性的样品前处理方法。但是在磁性分子印迹技术中, 由于磁性纳米粒子需要被包覆在印迹聚合物里面, 存在一个磁泄露问题, 即部分磁性粒子不能被包覆; 同时磁性印迹聚合物的粒径分布比较宽, 这些问题都有待解决。相信随着合成技术的不断改进, 磁性分子印迹技术在化学、模拟酶催化、药物控制释放、生物传感等众多领域都会有较好的应用前景。

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