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傅里叶变换红外光谱在微塑料检测中的应用

丁钰祥[1] 张家铭[1] 张洪伟[2] 吴申申[1] 陈汉清[1] 陈 瑞[1*]

(1. 首都医科大学 公共卫生学院，北京 100069；
2. 首都医科大学 燕京医学院，北京 101300)

摘 要 塑料制品的生产在为日常生活提供便利的同时也产生了大量塑料废物，其降解产生的微塑料(Microplastics，MPs)的健康效应和生态系统效应已成为全球关注的环境问题。伴随 MPs 污染问题的严重性和日益突出的环境影响，开发 MPs 的有效检测技术或方法尤为重要。傅里叶变换红外光谱(Fourier Transform Infrared Spectroscopy，FTIR)技术开发已较为成熟，因其高灵敏度、快速分析、非破坏性等优点，在 MPs 检测中得到广泛的应用。焦平面阵列-傅里叶变换红外光谱(Focal Plane Array Fourier Transform Infrared Spectroscopy，FPA-FTIR)技术作为 FTIR 的一项新技术，具有短时间内对大量样品进行高通量光谱扫描的优势，目前用于 MPs 的鉴定、分析与表面特性研究，为研究 MPs 与环境间的相互作用机制提供了新的研究方法。对 FPA-PFTIR 技术在 MPs 检测中的应用进行着重介绍，同时对其在未来的发展前景进行展望，以实现 FTIR 技术在识别 MPs 污染问题上的新突破。

关键词 微塑料；检测技术；傅里叶变换红外光谱技术；焦平面阵列-傅里叶变换红外光谱技术

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Application of Fourier Transform Infrared Spectroscopy in Micro-Plastics Detection

DING Yuxiang[1], ZHANG Jiaming[1], ZHANG Hongwei[2],
WU Shenshen[1], CHEN Hanqing[1], CHEN Rui[1*]

(*1. School of Public Health, Capital Medical University, Beijing 100069, China;*
2. Yanjing Medical College, Capital Medical University, Beijing 101300, China)

Abstract The production of plastic products provides convenience for daily life but also generates a large amount of plastic wastes, and the health and ecosystem effects of microplastics (MPs) generated by their degradation have become a global environmental concern. Given the seriousness of the MPs pollution problem and its increasingly prominent environmental impact, it is particularly important to develop effective detection techniques for MPs. Fourier transform infrared spectroscopy (FTIR) technology has

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作者简介：丁钰祥，女，在读学生，主要从事预防医学研究。E-mail：dingyuxiang_ccmu@outlook.com

* **通信作者**：陈瑞，男，教授，主要从事环境化合物健康效应的生物学机制和评价体系研究。E-mail：ruichen@ccmu.edu.cn

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been widely used in MPs detection due to its high sensitivity, rapid analysis, and non-destructiveness. Focal plane array fourier transform infrared spectroscopy (FPA-FTIR), a new technological advancement of FTIR, offers high-throughput spectral scanning of many samples in a short period. It is currently used for the identification, analysis, and surface characterization of MPs, providing a new method to explore the interaction mechanism between MPs and environment. This paper highlights the application of FPA-FTIR technology in MPs detection and looks forward to its future development prospects to achieve new breakthroughs in identifying MPs contamination by FTIR technology.

Keywords microplastics; detection technology; Fourier transform infrared spectroscopy; focal plane array-Fourier transform infrared spectroscopy

大量塑料垃圾通过各种途径进入环境后，在生物、物理和化学作用下缓慢分解，产生许多较小的塑料碎片和颗粒[1]。这些塑料碎片和颗粒被称为“微塑料(Microplastics, MPs)”，MPs 粒子直径为 1 μm～5 mm[2]。目前已使用多种方法对 MPs 的物理表征和化学性质进行研究，包括视觉和光学显微镜方法(目视分析)、尼罗河红染色-荧光显微镜、扫描电子显微镜、能量色散 X 射线光谱仪、原子力显微镜、光谱分析法、气相色谱-质谱法、标记法、热成像、热分析法等[3-5]。上述方法应用于不同的检测场景，也存在各自的局限性。

伴随 MPs 污染问题的严重性和日益突出的环境影响，为了保护人类健康与生态系统，开发 MPs 颗粒物的有效检测技术尤为重要。MPs 体积小、分布广，因此对其检测和表征具有挑战性。傅里叶变换红外光谱(Fourier Transform Infrared Spectroscopy, FTIR)技术对于 MPs 检测有重要的实用价值，可用于识别和量化各种环境样本中的 MPs，目前已得到广泛应用[6]。本文将对 FTIR 在微塑料检测中的应用进行综述。

1 傅里叶变换红外光谱技术的概况

FTIR 技术是一种利用红外辐射与物质的相互作用，对干涉后的红外光进行傅里叶变换的原理而开发的分析化学技术。目前被广泛地应用于纳米材料[7]、高分子[8]、医药[9]、生物学[10]、化学和环境科学[11]等多个领域。FTIR 技术发展经历三次更新迭代(图 1)。

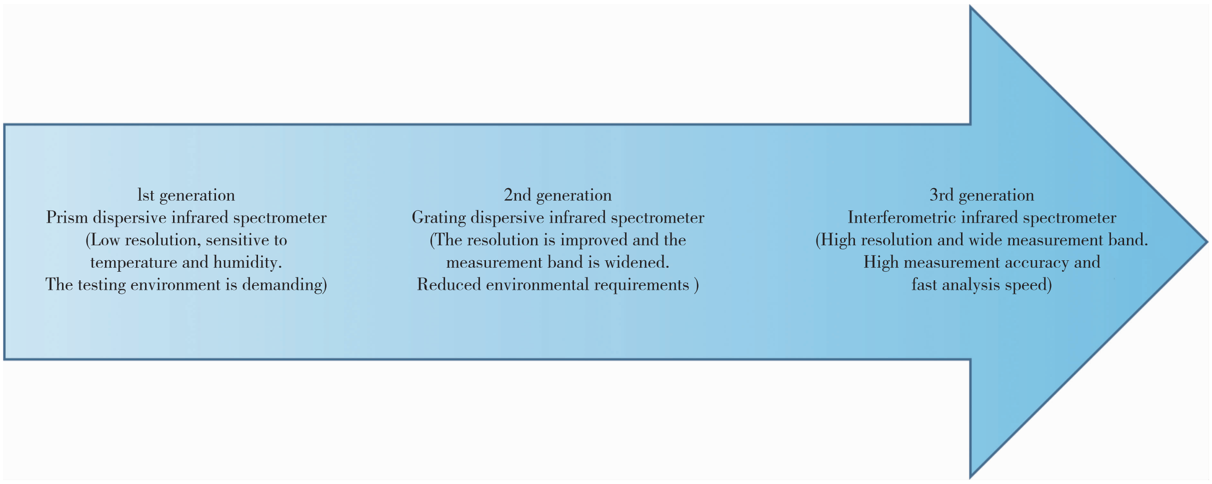

图 1 FTIR 技术的发展历程

Figure 1 The history of FTIR technology.

1.1 FTIR 的原理

FTIR 主要由红外光源、迈克尔逊干涉仪、样品室、探测器、激光器、电子计算机和记录仪等部件组成。FTIR 区分于其他光谱仪的重要组成部分是迈克尔逊(Michelson)干涉仪，它由两个反射镜(固定反射镜和移动反射镜)和分束器组成[12]。FTIR 由光源向外发出红外辐射光，通过干涉仪产生干涉光。待测样品在受到规定范围的红外辐射后，可根据样品不同物质成分和分子结构激发振动，检测器得到带有样品信息的干涉图，包含光源的全部频率和与

该频率相对应的强度信息。随后,收集到的信号被引导至探测器,用计算机处理和转换系统使用傅里叶变换进行数据处理,可将干涉图转换为红外吸收图谱[13],进而可分析样品的化学成分和结构信息(图 2)。

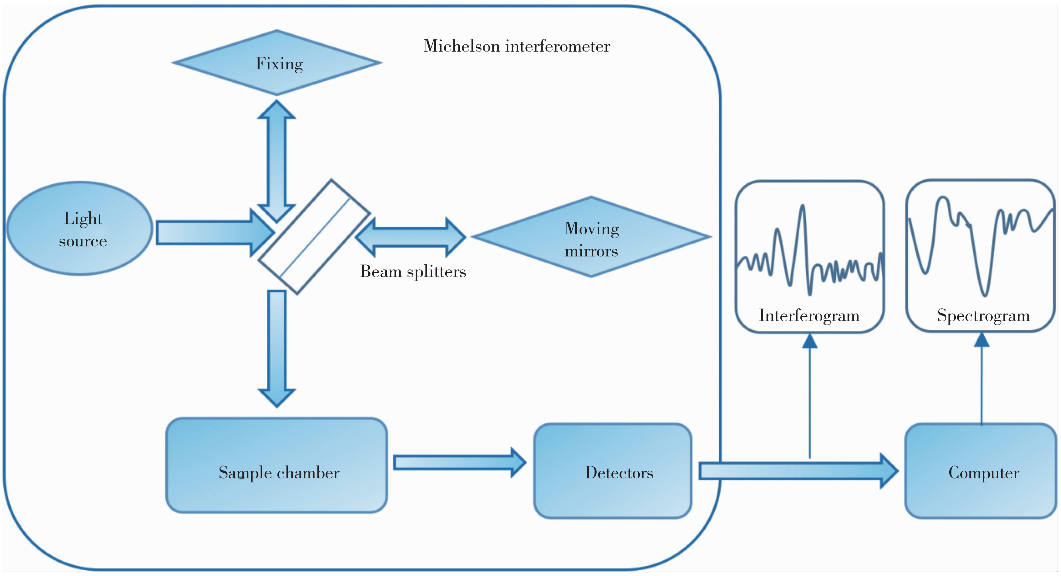

图 2　FTIR 的原理

Figure 2　Principles of FTIR.

传统红外光源在亮度、偏振性方面存在一定不足,同步辐射红外光源的亮度比传统光源高 2~3 个数量级。基于同步辐射的傅里叶变换红外(SR-FTIR)显微光谱具有高亮度、良好的偏振性、高聚焦性能等特点,使其在最高空间分辨率下可提供更好的信噪比[14]。用于检测样品时,可以获得空间分辨率低至衍射极限的分子图[15],同时可以提高分子信号的灵敏度。

1.2　FTIR 的技术优势

首先,FTIR 技术扫描速度快,在短时间内能获取大量化学信息并进行高通量分析,可用于追踪快速反应过程,或测定不稳定物质的红外光谱[16]。其次,FTIR 技术因其灵敏度高[17],可测试弱信号光谱样品,并可有效检测样品成分中微量 MPs,加之杂散辐射低,在成像过程中受到的干扰小,测量结果精确度高,因此,可有效、准确地分析样品并区分各种聚合物类型[18]。FTIR 技术在检测过程中对样品无需进行特殊处理,对待测样品无损,可用于不同环境介质(包括水源、沉积物、生物群、土壤和空气)样品中 MPs 的检测[13]。因其能与多种仪器联用,可更方便、快捷、准确地测试样品,如热重红外联用技术、气相色谱-FTIR 联用技术、差示扫描量热-FTIR 联用技术等。

2　傅里叶变换红外光谱技术的实验方法

FTIR 技术可用于气态、液态、固态三种状态的物质定性、定量、形貌特征表征分析。FTIR 根据探测技术可分为点探测与面探测。点探测技术包括透射 FTIR(Transmission Fourier Transform Infrared Spectroscopy,T-FTIR)、衰减全反射 FTIR(Attenuated total reflection Fourier Transform Infrared Spectroscopy, ATR-FTIR)和漫反射 FTIR(Diffuse reflectance Fourier Transform Infrared Spectroscopy, DRIFTS)三种模式[19],面探测的代表技术为 FPA-FTIR(Focal Plane Array Fourier Transform Infrared Spectroscopy, FPA-FTIR)(表 1)。

表1　FTIR技术的实验方法对比

Table 1　Comparison of experimental methods for FTIR techniques (Including detection technology, Mode, Principle, Apply, Advantages and Disadvantages)

Detection technology	Modes	Principle	Applies	Advantages	Disadvantages	References
Point detection	T-FTIR	Transmission spectroscopy is used to analyze the composition, structure and properties of substances	Suitable for the analysis and identification of solid sample components	Low spectral interference, wide analysis range, high analysis precision, good imaging quality	High requirements for sample pretreatment, high technical requirements for instrument use, requires the sample to have a certain degree of transmissibility	[20]
	ATR-FTIR	After multiple total reflections of the incident light, a spectrum is plotted based on the absorption of the spectrum by the sample	Analytical identification of liquids, pastes, powders, films, and solids	No sample pretreatment is required, in-situ analysis of samples, no requirement for sample thickness	The cost and operation are highly professional, the detection speed is slow	[21]
	DRIFTS	Spectroscopy of the surface reflection of the sample is used to analyze the properties of the substance	Inspect samples with rough surfaces (e.g. powders, fibers, metal coatings, etc.)	No sample pretreatment is required, no requirements for sample surface smoothness and transparency	The accuracy and reliability of the analysis are slightly lower, the sample uniformity and instrument use technology are required	[22]
Area detection	FPA-FTIR	A new technology derived from FT-IR, the FPA array detector simultaneously collects spectral data from multiple points on a single plane	Scan large areas of samples in a short period of time to obtain information on particle count, size, and polymer type	Fast detection speed, collect spectral information from thousands of sample pixels simultaneously, increase the detection area and improve the detection efficiency	Detection of small samples is not possible due to the problem of infrared diffraction limit	[23]

3 FTIR 技术在微塑料样品检测中的应用

目前,在 MPs 检测中应用较多的是 ATR-FTIR 技术和 FPA-FTIR 技术,因此本文对这两项技术的应用进行综述。

3.1 ATR-FTIR 技术

ATR-FTIR 技术可高精度分析大粒径 MPs 样品的化学组成,如其中的添加剂、填充剂、染料等;每种塑料都具有特征性红外吸收峰,通过与数据库比对或标准品对比,可以确定 MPs 样品的塑料组成类型及其含量,如聚乙烯(PE)、聚丙烯(PP)、聚氯乙烯(PVC)、聚苯乙烯(PS)等[24]。ATR-FTIR 技术仅可单独处理相对较大粒径的颗粒(约＞100 μm)。为了扩展 ATR-FTIR 技术检测 MPs 的范围,在原有 FTIR 光谱仪外增加显微镜来检测小粒径 MPs,显微镜和 ATR-FTIR 的组合通常被称为微型傅里叶变换红外光谱仪(ATR-μFTIR)[23]。ATR-μFTIR 可用于识别盐、尘埃和生物群中小粒径 MPs(10～500 μm)的聚合物类型[25-27]。ATR-μFTIR 能原位观察和表征单个 MPs 颗粒,更加直观地了解它们在环境基质中的空间分布和相互作用,也可对大于 10 μm 的 MPs 颗粒进行量化。然而,该技术仅配备单晶(IR)检测器,一次检测只能在样品上获得单点 FTIR 光谱,因此成像速度慢,检测耗时较长。

3.2 FPA-FTIR 技术

3.2.1 FPA-FTIR 技术的优点

FPA-FTIR 技术是目前 FTIR 光谱学的前沿技术[23],其有望成为一项非常有前途的 FTIR 技术。首先,FPA-FTIR 技术能在短时间内对大面积样品进行高通量的光谱扫描,对于富含颗粒的样品而言,FPA 检测器相较传统的逐粒扫描方法更加快速、准确[28-29],可在几分钟内获得数千个样品的红外光谱,并根据收集样品的自动映射获得颗粒的化学(光谱)和空间信息,快速成像并分析数据,显著提高分析效率[30]。FPA-FTIR 技术以高分辨率进行多区域快速测量并自动分析计数,提高数据质量的同时实现对样品的全面分析[31],高效获取 MPs 丰度[32-34]。FPA 探测器可以收集非均质 MPs 粒子的空间和光谱信息,克服了 FTIR 无法对较厚 MPs 检测的弊端[35]。此外,FPA-FTIR 有详细、无偏、高通量分析优势,检测前无需人工对 MPs 颗粒进行预分拣。因此,FPA-FTIR 技术在 MPs 的自动分析中具有巨大的潜力。

3.2.2 FPA-FTIR 技术在 MPs 检测中的应用

FPA-FTIR 技术在 MPs 检测中的应用分为定性检测和定量检测,表 2 列出了近年来的一些具体应用案例。

表 2　FPA-FTIR 技术在 MPs 检测中的应用

Table 2　Application of FPA-FTIR technology in MPs detection(Including Analytical Methods, Analyze the data, Chemical/physical properties of MPs, Application value)

Technology	Analytical methods	Analyze the data	Chemical/physical properties of MPs	Application value	References
FPA-FTIR	Qualitative testing	The characteristic absorption frequency of the sample group	The shape, size, and shape of MPs particles	Detecting the surface characteristics of MPs, including chemical weathering, degradation, surface chemical composition, presence of functional groups, surface function, pollutant adsorption, etc. It is helpful to understand other toxic pollutants, toxic organic compounds and microorganisms adsorbed and bound by MPs, and to provide a basis for the study of the interaction mechanism between MPs and the environment	[34]
	Quantitative testing	The strength of a specific absorption peak corresponding to a polymer functional group	The content, distribution, and source of MPs in environmental samples	The amounts of MPs in environmental samples was quantified against a known concentration calibration curve to detect the type and content of plastics. The primary and secondary sources of MPs can be analyzed to determine the proportion of pollutants in different plastic types. Several studies have been used to detect MPs levels in different ecosystems and waste management systems	[36]

在定性检测的应用中，SIMON-SÁNCHEZ 等[37]在 2022 年开展基于 FPA-FTIR 技术检测沿海 MPs 的研究，使用软件对生成的高光谱图像进行分析，得到颗粒丰度以及每个颗粒的详细物理化学信息（聚合物组成、二维尺寸、估计体积和质量）。ZHOU 等[38]于 2022 年采用基于 FPA-FTIR 和扫描电子显微镜相结合的方法，使用数据库匹配方法定量评估外卖食品容器中的 MPs 含量，发现外卖食品容器中最常见的塑料类型为纤维素、聚酰胺（PA）、聚氨酯（PU）和聚苯乙烯（PS），并对 MPs 的大小、形状分布和特性进行进一步鉴定。

在定量检测的应用中，LORENZ 等[39]于 2019 年通过将有效的样品制备与最先进的 FTIR 和自动分析技术相结合，检测并分析北海南部含有的 MPs 沉积物和地表水样品，测量出 MPs 浓度并得到所涉及聚合物组成的可靠数据。通过 FPA-FTIR 检测技术得到的 MPs 化学成分及其含量，可用于评估环境质量和污染程度，为环境保护和污染治理提供数据支撑，同时为塑料制造与应用阶段选择污染程度更小的塑料提供参考依据。

3.2.3 FPA-FTIR 技术与智能图像处理算法联合在 MPs 检测中的应用

近年来，研究人员开始开发基于 FTIR 光谱图像的智能图像处理算法，用于快速、自动地检测和分类 MPs 颗粒。这些算法可以识别出 MPs 颗粒的光谱特征，并将其与大量样品数据库进行比对，以实现高效的检测和分类[40]。HUFNAGL 等[41]于 2021 年报道了一种基于随机决策森林模型的机器学习方法[42]，可以用于分析环境样本中大量 FPA-μFTIR 数据集。该模型可应用于区分 20 多种不同的聚合物类型，适用于复杂基质检测，如空气、水体、土壤等环境中的 MPs 污染情况。相关研究发现，已经开发的 MPs 识别（μIDENT）算法与 FPA-FTIR 技术组合在识别 MPs 方面效果显著，基于 FTIR 的自动 MPs 鉴定精度可提高到 96%[43]。此类算法为高通量检测 MPs 提供了更多的选择和可能性，有望在环境保护和 MPs 污染研究中发挥重要作用。

4 FTIR 技术的前景与挑战

FTIR 技术在 MPs 检测中存在一定局限性，如检测前样品制备复杂[44]；检测过程中受到技术本身分辨率的限制，需要与其他技术联合使用分析复杂样品；检测的数据结果较为复杂，需要经验丰富的分析人员进行数据解释和化学信息的提取分析。

FTIR 技术在检测微米级的微塑料样品时，由于空间分辨率的衍射极限无法探测。红外散射扫描近场光学显微镜（Infrared Scattering-type Scanning Near-field Optical Microscopy IR s-SNOM）提供了更精细的空间分辨率，成为纳米尺度检测的有力工具。此外，原子力显微镜-红外光谱（Atomic Force Microscopy-infrared，AFM-IR）系统以及纳米-FTIR（Fourier Transform Infrared Nanospectroscopy，nano-FTIR），均可提供纳米级空间分辨率的光谱成像，突破红外光的衍射极限，进一步提高 FTIR 技术空间分辨率。纳米技术的进一步发展有望提高空间分辨率和信噪比（消除伪影）[45]。使得纳米微区（$<100\ \mu m$）化学成像和红外光谱采集成为可能，实现快速、无损和高空间分辨率的纳米物质化学成分检测。

5 结语

随着各项技术的发展，FTIR 仪器不断更新迭代，仪器的分辨率、灵敏度和数据处理能力得到了显著提高。在未来大数据时代，FTIR 技术在检测 MPs 领域仍有广泛应用前景，可以通过开发自动检测方法，将 FTIR 与自动化样品处理系统和机器学习算法相结合，简化 MPs 的检测和分析，提高效率和准确性。通过设立标准化和方法验证确保使用该技术生成的 MPs 数据可靠性强，提高实验可重复性。可见，FTIR 技术将会在 MPs 的检测中迎来新的机遇。

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