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Review

A review of recent trends in polymer characterization using non-destructive vibrational spectroscopic modalities and chemical imaging

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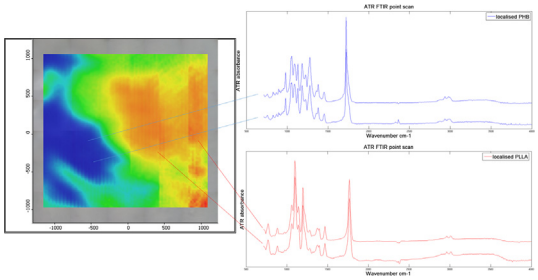
HIGHLIGHTS

- Traditional methods of polymer characterization with emphasis on advantages of vibrational spectroscopic methods highlighted.
- Chemical Imaging & data processing for spatial imaging introduced briefly.
- Applications of chemical imaging for polymer coatings, compositional studies, crystallinity, phase homogeneity/heterogeneity.
- The future of chemical imaging with regards to polymer characterization is discussed.

GRAPHICAL ABSTRACT



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ABSTRACT

This review focuses on the recent developments in vibrational spectroscopy and chemical imaging (i.e. Raman, Near Infrared, Mid Infrared) to characterize polymers in diverse forms, their behaviour and transient phenomenon. First, important polymeric properties and traditional methods of their characterization are outlined. Then relative advantages & disadvantages have been presented of different characterization methods are presented. This is followed by a detailed review of applications of chemical imaging and spectroscopic techniques in polymer characterization, including the limitations encountered. The article ends with a discussion on the future of chemical imaging with regards to polymer characterization.

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1. Introduction

In order to tailor polymers to different applications e.g. thin films for packaging, coatings for drugs, scaffolds for biomedical engineering, it is necessary to conduct a thorough study of their physico–chemical properties such as molecular mass, structure, morphology, thermal characteristics, and mechanical characteristics. Synthetic polymers are commonly created in different forms such as pellets, composites, blends, films, colloids, coatings. Therefore, instrumental techniques capable of accurate, reliable and rapid evaluation of polymers are required. Traditional methods for polymer characterization (summarized in Table 1) typically involve laborious sample preparation, are time consuming, destructive in nature and/or invasive, all of which may lead to loss of crucial information of polymer in its *natural state*. Therefore, there is a need for technologies which bypass above mentioned shortcomings and allow for a prediction based approach for characterization with minimum loss of information.

1.1. Vibrational spectroscopy

Vibrational spectroscopy is a well-established area of applied spectroscopy that enables molecular characterization of materials based on the vibrations of atoms and molecules [1–6]. Infrared & Raman spectroscopy are the most widespread vibrational spectroscopic modalities in common use [7–9]. When infrared radiation passes through a sample, bonds between atoms vibrate due to absorption of energy Different molecules and functional groups exhibit characteristic absorption peaks corresponding to the frequency of a vibration in a given molecule [10,11]. Based on the range of frequencies studied, infrared spectroscopy is subdivided into three categories: the near infrared (NIR) region within the 400 nm–2500 nm or 25,000–4000 cm^{-1} range, which is sensitive to overtone and combinations of vibrational vibrations; the mid infrared (MIR) region within the 2500 nm–25000 nm or 4000–400 cm^{-1} range to study fundamental vibrations and the far infrared (FIR) region spanning 25000–1000,000 nm or 400–10 cm^{-1} range to study rotations.

MIR spectra are typically obtained using interferometer based Fourier Transfrom Infrared (FTIR) systems. For samples thicker than a few 100 um, a large amount of IR energy absorbed by the sample results in a low signal to noise spectrum. This can be overcome by the use of Attenuated Total Reflection (ATR), where a polished crystal surface (e.g., diamond, germanium) contacts the sample surface, IR light passes through the crystal, generates an evanescent wave through the sample [12].

Table 1
Traditional methods of polymer characterization.

Characterization method	Property investigated	Nature of analysis
Gel Permeation Chromatography(GPC) [45]	Mass analysis: Number average molecular mass, molecular mass distribution	Destructive for sample
Mass Spectroscopy(MS) [45]	Structural and functional group analysis: identification of chemical moieties and conformational state	Destructive for sample
X-Ray Diffraction(XRD) [30]	Structural and functional group analysis, degree of crystallinity.	Destructivity depends on sample preparation method used
Nuclear Magnetic Resonance(NMR) [29]	Structural and functional group analysis, identification of exact structure, chemical moieties, and conformational state	Laborious sample preparation, non-destructive
Small Angle X-ray scattering(SAXS) [45]	Microscopic domain analysis	Minimum sample preparation, non-destructive
Electron Microscopy (Scanning, Transmission): SEM, TEM [3]	Microscopic domain analysis	Laborious sample preparation, destructive for sample
Atomic Force Microscopy(AFM) [21]	Measures physical surface characteristics such as roughness	Laborious sample preparation, destructive for sample
Transmission Optical Microscopy [28]	Structural analysis	Requires transparent samples
Differential Scanning Calorimetry(DSC) [40]	Determines phase transition, melting point, reaction kinetics of polymers	Destructive for sample
Thermogravimetric Analysis (TGA) [40]	Determines thermal degradation pathway of polymers	Destructive for sample
Dynamic Mechanical Analysis (DMA) [45]	Mechanical analysis(tensile strength, Young's Modulus)	Destructive for sample
Viscometry [45]	Mechanical analysis(tensile strength, Young's Modulus)	Destructive for sample
Thermomechanical Analysis (TMA) [31]	Thermo-mechanical behaviour	Destructive for sample
X-Ray Photoelectron Spectroscopy(XPS) [46]	Structural and functional group analysis	Requires Vacuum, destructive for sample

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