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Characterization Techniques

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Abstract

© 2017 John Wiley & Sons, Ltd.Solid-state nuclear magnetic resonance spectroscopy (SSNMR) not only is a reliable tool to determine the structure of organic molecular compounds, but also has developed into a suitable technique to characterize graphene oxide (GO) and has the potential to make structural analysis of GO derivatives possible. Common spectroscopic techniques are introduced, and the advances of SSNMR for analyzing the structure of GO are summarized. Finally, the prospects of SSNMR are discussed. Fourier-transform infrared (FTIR) spectroscopy is a simple and robust method to analyze GO. It is not very representative, since absorption bands from the fingerprint region are almost impossible to reveal due to the overlapping of numerous signals. Also, FTIR is the most commonly misinterpreted method among all those used for GO characterization. Nevertheless, FTIR has played an important role in revealing some specific functional groups. Several real examples are discussed. X-ray photoelectron spectroscopy (XPS) is a very powerful tool in characterizing GO and its composites. The ability to detect heteroatoms makes XPS superior even when compared to SSNMR. XPS also provides the chemical or electronic state for each element. With respect to GO, XPS provides not only the elemental content, but also the nature and the relative content of the functional groups. Raman spectroscopy is a reliable tool to determine and visualize the heterogeneity of graphene-family materials, including single-layer graphene and some specific types of reduced graphene oxide (RGO). It is a highly sensitive method to determine and quantify the amount of defects in graphene. Here, the basic principles of Raman spectroscopy and the basic interpretation of spectra are given. In addition, using an example, we show how to perform statistical analysis and how to use these data as a basis for statistical Raman microscopy. Being a very powerful tool for characterizing graphene-family materials in general, Raman spectroscopy is not very informative when used on typical GO samples. The density of defects in GO is very high, and Raman spectroscopy cannot resolve different defect densities after some threshold defect density has been reached. The density of defects in typical GO samples is beyond this threshold. However, Raman spectroscopy is a very powerful tool for characterizing some specially prepared low-defect-density RGO samples. Microscopy is broadly used to characterize GO in terms of the flakes' size, thickness, number of layers, etc. High-resolution transmission electron microscopy (HRTEM) has been exploited to reveal the fine chemical structure of GO. For the first time HRTEM has confirmed the two-type-domain structure of GO. In this chapter, as well as HRTEM, the following microscopy methods are reviewed: scanning electron microscopy (SEM), atomic force microscopy (AFM) and transmission electron microscopy (TEM). Several examples of how these methods have been used to characterize GO are reviewed.

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Keywords

Graphene oxide, Infrared spectroscopy, Nuclear magnetic resonance spectroscopy, X-ray photoelectron spectroscopy