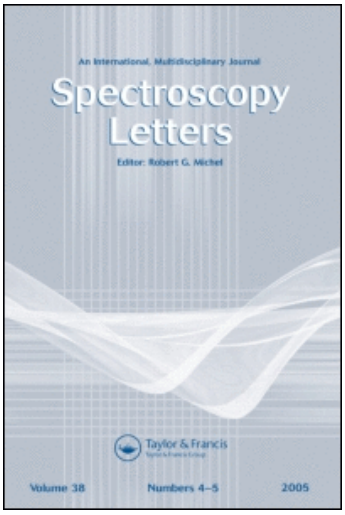

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Infrared Quantitative Study of Acetylation of Microspheres of Polystyrene

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INFRARED QUANTITATIVE STUDY OF ACETYLATION OF MICROSPHERES OF POLYSTYRENE

keywords: Infrared spectroscopy, acetylation, polystyrene, quantitative study, microsphere

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ABSTRACT

Fourier transform infrared spectroscopy has been used to study quantitatively the acetylation of monodisperse polystyrene microspheres

with diameters ranging from 7 to 9 μm. The CH_2 stretching infrared vibration mode at 2921 cm^{-1} was used as the internal intensity standard. The acetylation extent could be easily measured by comparing the relative intensities of the bands of acetyl group (1678, 1415, 1359 cm^{-1}) or the bands due to the para-substituted benzene ring to the band at 2921 cm^{-1} from the calibration curve.

Introduction

Monodispers microspheres of polystyrene has been recently found important applications in microscopical analysis, biochemical technology, and biomedicals.[1] The acetylation of the polystyrene microspheres is another important step to functionize these spheres.[2,3] However, the measurement of the acetylation extent has been difficult. In this letter we report a method of infrared quantitative measurement for the acetylation study of polystyrene. Calibration curves of the relative intensities of the acetyl bands to the 2921 cm^{-1} which was used as the internal standard band have been prepared. By measuring the relative intensities for the unknown sample, an accurate acetylating extent could be easily read from these calibration curves.

EXPERIMENTAL

Polystyrene microspheres were prepared by microemulsion polymerization. The diameters of these spheres, measured by the use of electron microscopy, were in the range of 7 to 9 μm. The polystyrene spheres were acetylated by acetyl chloride in the presence of $AlCl_3$ and Cl_2CH_2. The infrared spectra were recorded using a Perkin-Elmer Model 281B Fourier transform infrared spectrometer.

Result and Discussion

Figure 1 illustrates the infrared spectra recorded from the polystyrene (Figure 1A), the 99.2 wt% acetylated polystyrene (Figure 1C), and a polystyrene with unknown extent of acetylation.

In Figure 1C, the C=O stretching band at 1678 cm^{-1}, the asymmetric and symmetric deformations at 1415 and 1359 cm^{-1} are characteristic vibration modes for acetyl group. The band at 829 cm^{-1}, which is due to *para*-disubstituted phenyl ring, is very weak in the spectrum of Figure 1A, but is strong in Figure 1C. While the band at 760 cm^{-1}, which is due to the vibrational mode of 5 adjacent H wag for the *mono*-substituted phenyl ring, disappeared in Figure 1C. The spectral changes for Figure 1A and C indicated completely acetylation of polystyrene.

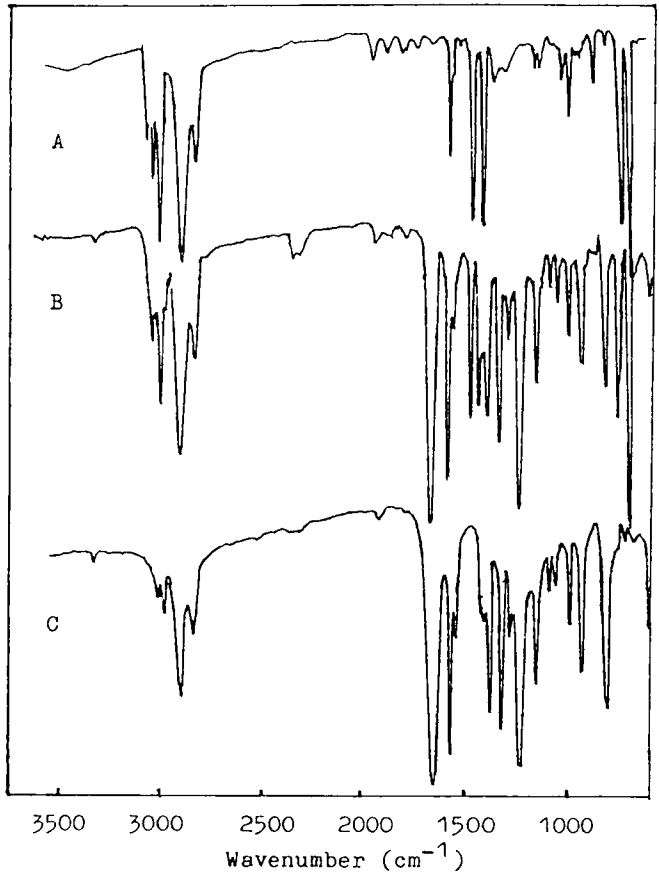

Figure 1. IR spectra of (A) polystyrene, (B) partially acetylated polystyrene, and (C) 99.2% acetylated polystyrene.

During acetylation, CH_2 group is not supposed to undergo any reaction. So the intensity of IR band for CH_2 stretching remains unchanged after acetylation. It is reasonable to use the band at 2921 cm^{-1} as the internal standard for intensity measurement. Figure 1B is a spectrum recorded from partially acetylated polystyrene microspheres. The extent for acetylation is expected to be measured by infrared quantitative study. We shall demonstrate the measurement of the unknown acetylated sample of Figure 2B by the use of calibration curves in Figure 2.

Four calibration curves are shown in Figure 2A, B, C, and D. They were prepared by plotting the relative intensities of IR bands versus the extent of acetylation by mixing the 99.2 % acetylated polystyrene and pure polystyrene in the desired ratio.

The relative intensities used for plotting the calibration curves of Figure 2A, B, C, and D are the intensity ratio of 1678, 1415, 1359, and 829 cm^{-1} bands over 2921 cm^{-1} band respectively.

Observation of the spectral intensity of corresponding bands of Figure 1B and the calibration curves shown in Figure 2A, B, C, and D, one can easily found that the extent of acetylation of the unknown

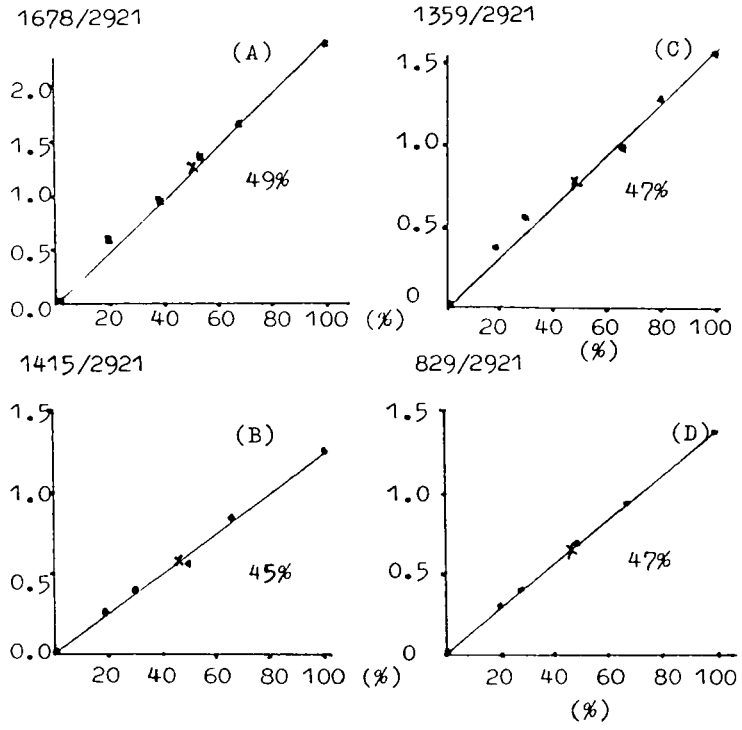

Figure 2. Calibration cureve of relative intensity versus the extent of acetylation. The intensity ratio are (A) $I_{1678\ cm\text{-}1}/I_{2921\ cm\text{-}1}$, (B) $I_{1415\ cm\text{-}1}/I_{2921\ cm\text{-}1}$, (C) $I_{1359\ cm\text{-}1}/I_{2921\ cm\text{-}1}$, and (D)$I_{829\ cm\text{-}1}/I_{2921\ cm\text{-}1}$.

sample is 49%, 45%, 47%, 47% respectively. We can see that the measured results for acetylation are quite coincide with each other, and that the calibration curve of Figure 2C or D seems more accurate for quantitative study.

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