INFRARED AND RAMAN SPECTOSCOPIC STUDY OF ACETYLATION OF RAW COTTON FOR APPLICATION IN SORPTION OF OIL SPILLS

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Abstract: Raman and Fourier transform infrared spectroscopy (FTIR) have been used to investigate the acetylation of raw cotton in an effort to develop hydrophobic, biodegradable, sorbent materials for cleaning up oil spills. Both Raman and FTIR spectroscopy indicate successful acetylation though the Raman bands are much weaker. A linear correlation was observed between the degree of acetylation measured by Raman spectroscopy and that obtained from FTIR data.

Sorbent materials for cleaning up oil spills are presently being developed in our laboratory. A review of various porous materials that have been or are presently being developed for oil spill cleanup operations was published recently [1]. Natural sorbents such as rice straw, cotton, wood, wool fibres etc. have relatively high sorption capacity and biodegradability and are more economical to produce than the synthetic polymeric materials that are commonly used. Like the acetylation of rice straw that was reported previously [2], the acetylation of cotton is expected to yield a product that has a relatively high hydrophobicity and oil sorption capacity. Fourier transform infrared spectroscopy was previously reported [3] to be highly sensitive and reliable for the determination of the level of acetylation. The level of acetylation was determined as the ratio R between the intensity of the acetyl C=O stretching band at 1740-1745 cm^{-1} and the intensity of C-O stretching vibration of the cellulose backbone at about 1020-1040 cm^{-1}. Raman spectroscopy has also been used to determine the structure of cellulose in cotton [4,5] and the level of acetylation in modified starches [6,7]. In particular, Raman spectroscopic technique has been found to allow faster determination of the degree of acetylation and is non-destructive and less susceptible to interference from residual impurities than the currently used traditional wet chemistry methods [6,7]. In this paper, we report the use of infrared and Raman spectroscopic techniques for the investigation of the acetylation of raw cotton samples.

The FTIR spectra of the raw cotton samples show evidence of acetylation with three ester bands appearing and/or enhanced at 1740-1745 (carbonyl C=O stretching of ester), 1368 [C-H in -O(C=O)-CH_3] and 1234 cm^{-1} (C-O stretching of acetyl group). The lowering of intensities of OH stretching band at 3337 cm^{-1} and OH in-plane bands at 1337, 1310 and 1200 cm^{-1} also provide further evidence of successful acetylation. The degree of acetylation was estimated from the FTIR data by calculating the ratio R_x between the intensity of the acetyl C=O stretching of ester at 1740-1745 cm^{-1} and the intensity of C-O stretching vibration of the cellulose backbone at about 1020-1040 cm^{-1}. Further evidence of successful acetylation was also provided by Raman data with peaks observed at about 1735 (C=O stretching), 1460 (CH_3 symmetric deformation), 835 (H_3C-C stretching) and 655 cm^{-1} (O-C=O in-plane deformation). However, these Raman peaks indicating acetylation are much weaker than the IR acetylation bands. The CH_2 symmetric and asymmetric stretching band at about 2900 cm^{-1} was also observed to increase in intensity due to acetylation (Fig. 1). Thus, the degree of acetylation from the Raman spectra was estimated by calculation the ratio R_y between the intensity of the 2900 cm^{-1} peak and the intensity of COC β-glycosidic asymmetric stretching link band observed at about 1093 cm^{-1}. R_y was observed to follow almost a linear relationship with R_x (Fig. 2) indicating the correlation of the Raman data with FTIR data.

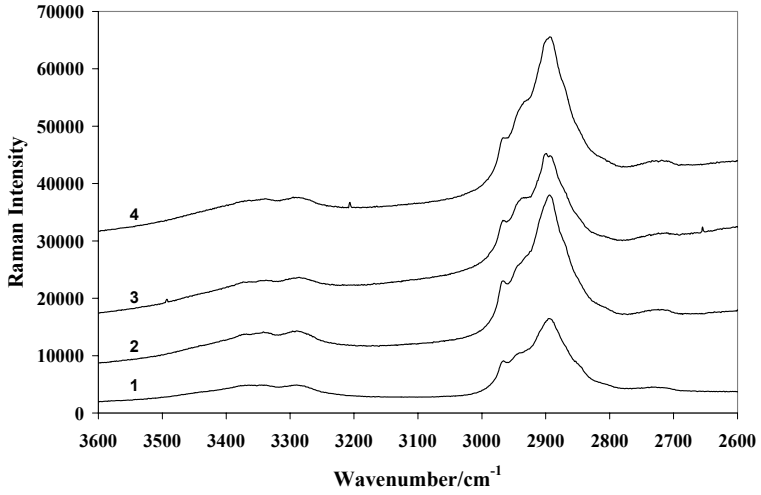

Fig. 1. Raman spectra of untreated raw cotton (spectrum 1) and acetylated raw cotton (spectra 2,3,4) with the level of acetylation decreasing in the order 4>3>2

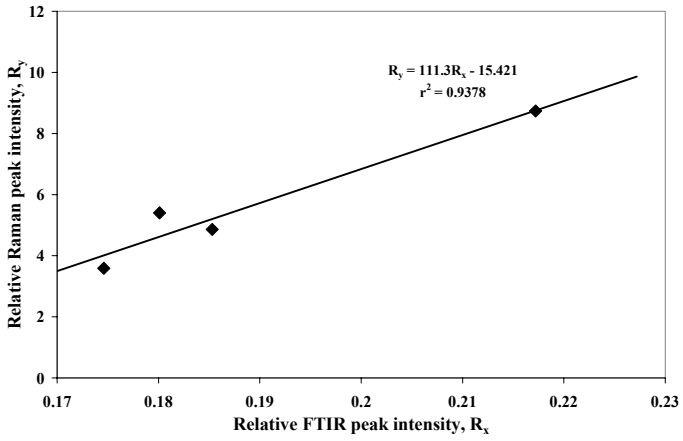

Fig. 2. The plot of degree of acetylation, R_y obtained from Raman data against the extent of acetylation R_x calculated from FTIR data

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