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### **Structure Elucidation of Textile Fabrics by Fourier Transform Infrared Photoacoustic Spectroscopy**

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STRUCTURE ELUCIDATION OF TEXTILE FABRICS BY  
FOURIER TRANSFORM INFRARED PHOTOACOUSTIC SPECTROSCOPY

Key Words: Photoacoustic Spectroscopy, Infrared,  
Fourier transform, Textile Fabrics

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ABSTRACT

A photoacoustic detection system mainly based on a Fourier transform infrared spectrometer has been constructed and some tentative evaluations of its performances have been made. Infrared photoacoustic spectra of cotton and nylon cloths have been obtained without troublesome sample preparations which are necessary in conventional infrared spectrometry. It can be said that the Fourier transform infrared photoacoustic spectroscopy is useful for the structure elucidation of organic polymers.

## INTRODUCTION

It is well known that the infrared spectroscopy is a powerful analytical method for the characterization of various kinds of substances. There are, however, several kinds of substances which are difficult to examine by conventional infrared measurement techniques. Although photoacoustic spectroscopy(PAS) is proved to be a useful method for the analysis of bulk materials,<sup>1)</sup> most of the studies on PAS are limited to the uv-visible spectral range for which powerful excitation sources are readily available. On the other hand, high power continuum sources do not exist at present in the infrared region. In order to overcome the above disadvantage for infrared PAS, photoacoustic detections using a Fourier transform infrared spectroscopy(FTIR) have been demonstrated recently,<sup>2-4)</sup> since FTIR spectrometer has a greater throughput of radiation than the conventional dispersive infrared spectrometer.<sup>5)</sup> In the present investigation, some discussions on the characteristics of the FTIR-PAS and preparative results of the FTIR-PAS for the structure elucidation of organic polymers are presented.

## EXPERIMENTAL

The PAS system used in this study was mainly based on a Digilab FTS-15 Fourier transform infrared spectrometer. The light source was a nichrome wire and the

infrared radiation was modulated with a Michelson interferometer in which a movable mirror was scanned at a constant velocity. The modulated beam was focussed on to the sample in the PAS cell which was placed in the sample compartment of the FTS-15 spectrometer. The PAS cell was machined from brass and its internal volume was approximately  $0.95 \text{ cm}^3$ . A KBr plate (22mm  $\phi$ , 3mm thick) was used as a cell window and a 1/2 inch B&K model 4165 condenser microphone was used as a pressure transducer. The photoacoustic signal from the microphone was amplified with a Brookdeal model 9454 ac amplifier and was fed to the MCT (HgCdTe) detector amplifier section of the FTS-15 spectrometer. All of the spectra were measured at  $4 \text{ cm}^{-1}$  resolution using 400 scans which took about ten minutes. A schematic drawing of the PAS cell is shown in FIG. 1. As shown in FIG. 1, the microphone is connected to the cell

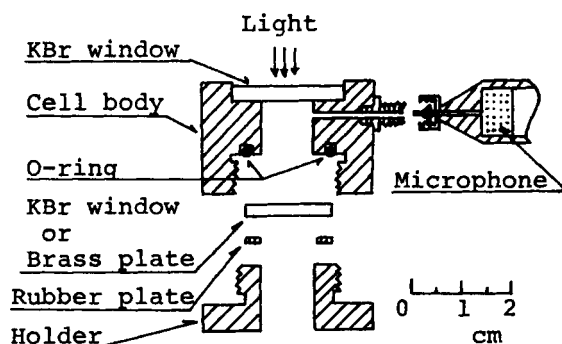


FIG. 1

Schematic drawing of the photoacoustic cell.

body simply with a plug and a nut to examine easily the effect of the length of the acoustic guide in photoacoustic signals.

### RESULTS AND DISCUSSION

The photoacoustic spectrum of carbon black(benzene soot) and the source spectrum obtained by a pyroelectric detector(TGS: triglycine sulfate) are shown in FIG. 2. The carbon black photoacoustic spectrum is known to be

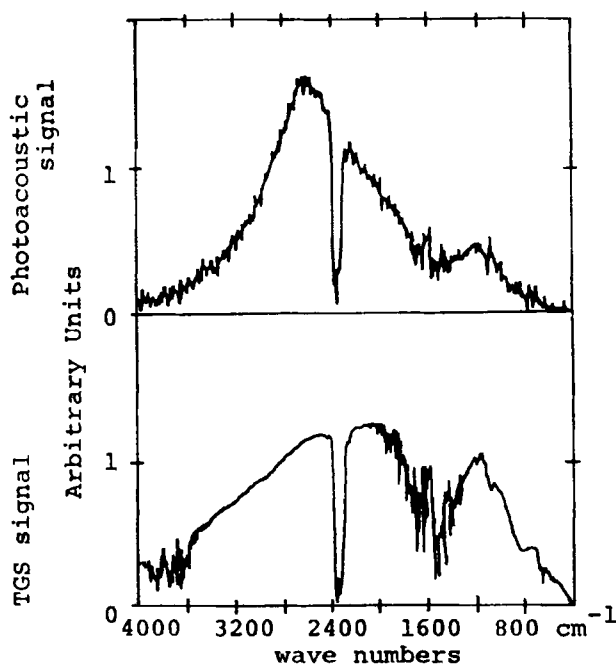


FIG. 2

Comparison of the carbon black photoacoustic spectrum (upper) with the source spectrum measured by a pyroelectric(TGS) detector(lower).

virtually identical with the source spectrum measured by a pyroelectric detector such as TGS in the dispersive PAS system with fixed frequency light modulation.<sup>6)</sup> In the case of FTIR-PAS, the above-mentioned identity is not observed as shown in FIG. 2. This result can be attributed to the difference of the light modulation methods between the dispersive PAS and the FTIR-PAS. The modulation frequencies in the FTIR-PAS vary with the spectral frequencies (wave numbers). In other words, the frequency of the radiation being emitted from the source,  $\nu[\text{cm}^{-1}]$ , corresponds to the modulation frequency,  $\omega[\text{sec}^{-1}]$ , through the relationship,  $\omega=2V\nu$ , where  $V[\text{cm}\cdot\text{sec}^{-1}]$  represents the velocity of the moving mirror in a Michelson interferometer.<sup>5)</sup> The mirror velocity used for the measurement of spectra is 0.3 cm/sec in our spectrometer. Therefore, the spectral range between 400 and 4000  $\text{cm}^{-1}$  corresponds to the modulation frequency range between 240 and 2400 Hz. Taking the  $\omega^{-1}$  dependence of the carbon black photoacoustic signals<sup>7)</sup> into consideration, carbon black photoacoustic signals for FTIR-PAS must be larger in the lower wave number region. However, this tendency is not observed as shown in FIG. 2. The photoacoustic spectra in FTIR-PAS are considered to be affected greatly by Helmholtz resonance effect,<sup>8)</sup> since the carbon black photoacoustic spectral features are found to vary depending on the length of the acoustic guide in the PAS cell.

These effects, however, can be compensated when the normalization for the photoacoustic spectra of substances is carried out using a carbon black photoacoustic spectrum measured by a same PAS cell as a reference. In FIG. 2, negative going features at ca. 2350 and 1600  $\text{cm}^{-1}$  are due to the atmospheric  $\text{CO}_2$  and  $\text{H}_2\text{O}$  absorptions in the optical path, respectively.

Infrared photoacoustic spectra of several kinds of textile fabrics have been measured and as an example of FTIR-PAS, photoacoustic spectra of cotton and nylon cloths are shown in FIG. 3 (A) and (B), respectively.

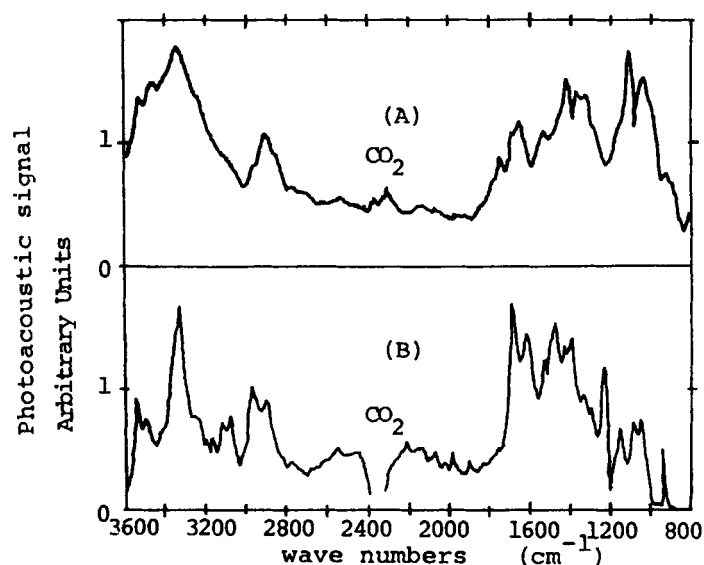


FIG. 3

Infrared photoacoustic spectra of cotton(upper;A) and nylon(lower;B) cloths.

In FIG. 3 (A), the bands at ca. 3300, 2900 and 1100  $\text{cm}^{-1}$  are assigned to OH stretching, CH stretching and CO stretching modes, respectively. These bands are characteristic of celluloses. In FIG. 3 (B), the bands at ca. 3350, 2900, 1650 and 1550  $\text{cm}^{-1}$  are assigned to NH stretching, CH stretching, C=O stretching and NH bending modes, respectively. These bands are characteristic of polyamide structure. The features of these spectra are fairly similar to those of the published spectra measured by a KBr method or a thermally pressed film method.<sup>9)</sup> In this study, infrared spectra can be obtained by putting a small piece of sample in the cavity of the PAS cell. FTIR-PAS is therefore attractive for the structure elucidation of organic polymers without destructive procedures and troublesome sample preparations which are necessary in conventional measurement techniques.

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