The Analysis of Textile Mixtures Using Fourier Transform Infra-red Photoacoustic Spectroscopy

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Fourier transform infra-red photoacoustic spectroscopy can be used to obtain high-quality infra-red spectra of textile samples with the minimum of sample preparation. For a wool/polyester blend, the spectrum of the polyester was obtained by subtracting the spectrum of wool from that of the mixture. The resulting spectrum closely resembles that of the polyester. For a wool/nylon blend, the spectrum of the mixture obtained by adding the spectrum of wool to that of nylon was shown to be very similar to that of the actual wool/nylon blend. By quantifying the addition and subtraction processes it should be possible to analyse such binary mixtures quantitatively. A three-component blend of wool, nylon and viscose rayon was also examined and the spectral subtraction process was found to be of value in identifying the components.

INTRODUCTION

The analysis of fibre mixtures using infra-red (i.r.) spectroscopy is a well-established technique [1], which unfortunately has a number of inherent disadvantages. For the technique to be of any value, the constituents of the mixtures need to have reasonably different i.r. spectra so that analysis can be made on the basis of either (a) the presence or absence of absorbance peaks or (b) the relative heights of the various peaks compared with each other. A further difficulty lies in the mode of sampling. It is difficult to obtain reproducible data using the method of attenuated total reflectance (ATR) because of problems associated with making a good contact between the sample and the crystal in the ATR attachment [2]. If fibre mixtures have to be ground up and then incorporated into a medium (e.g. potassium bromide) then the reproducibility in preparing the samples again presents a problem [3]. Similarly, processes involving the separation of fibres before analysis by i.r. spectroscopy have inherent problems of reproducibility [4].

We wish to describe the use of Fourier transform i.r. photoacoustic spectroscopy (FTIR-PAS) for the analysis of fibre mixtures. This technique helps to alleviate some of the problems detailed, but it cannot be regarded as a panacea.

The use of FTIR-PAS is a proven technique and offers a valuable alternative to acquiring data via a dispersive

instrument [5]. Detection of the frequencies of i.r. absorption can be accomplished by means of a conventional detector or by use of a photoacoustic cell [6–8]. In the latter, i.r. radiation absorbed by the sample is detected by its re-emission with consequential heating of the gas above the sample. If the sample is contained in a hermetically sealed cell, one side of which is the diaphragm of a microphone, the increase in volume of the gas contained in the cell due to the re-emission of absorbed radiation leads to a response in the diaphragm, which then converts the pressure change into an electrical signal. An outstanding feature of this mode of detection lies in the fact that solid samples, such as powders [9–11], polymer films, solutions, fibres, pieces of cloth, etc. may often be used without any prior sample preparation. Thus we have used photoacoustic spectroscopy to study the absorption of dyes on wool in the visible region of the spectrum [12] and the absorption of fluorescent whitening agents and the photo-yellowing of wool in the near ultra-violet (u.v.) and visible regions of the spectrum [13]. The analysis of textile blends using near-i.r. photoacoustic spectroscopy has received little attention [14,15].

The use of photoacoustic detection in the mid-i.r. is a relatively recent development and utilises several important features of the interferometric technique, namely low stray light, high throughput, signal averaging and the ability to vary the modulation frequency and the scan speed to suit the experiment. Advantages such as these have brought FTIR-PAS to the stage where it is being applied for the first time in a wide variety of applications.

Recently the analysis of a textile blend using FTIR-PAS was reported [16]. In the present paper we demonstrate the power of the technique, which lies in the ease of sampling and the computational facilities associated with the spectrometer.

EXPERIMENTAL

Samples

An all-wool plain-weave challis fabric of weight 100 g/m² (Bannisters Ltd, Colne) and a plain-weave lightweight staple blend (60% wool/40% polyester) fabric (E Ward Ltd, Bradford) were chosen. Wool/nylon (80:20) and wool/nylon/viscose rayon (40:40:20) yarns were selected as being representative of typical carpet weaving yarns employed by the UK carpet industries. A 100% polyester fabric (ICI Fibres Ltd) was also tested, a medium-weight (150 g/m²) serge fabric woven from yarns spun from short-staple fibre. All the fabrics and yarns were received as-scoured and prepared for dyeing. No further preparative treatment was given.

Instrumentation

The spectra shown in Figures 1–7 were obtained using a Princeton Applied Research photoacoustic cell in the sample compartment of a Nicolet 170 SX FTIR spectrometer. The gas above the sample in the cell was air, and as a consequence all the spectra show absorptions due to carbon dioxide at ~2350 cm^{-1}. To obtain spectra, 300 interferograms were signal-averaged over an 8 min accumulation time. The computer carried out the Fourier transformation of the averaged interferogram. This gave an energy spectrum as a function of wavenumbers, which was ratioed against the energy spectrum of carbon black to remove any spectral characteristics of the interferometer and the cell. The velocity of the moving mirror for each system analysed was 0.074 cm s^{-1}.

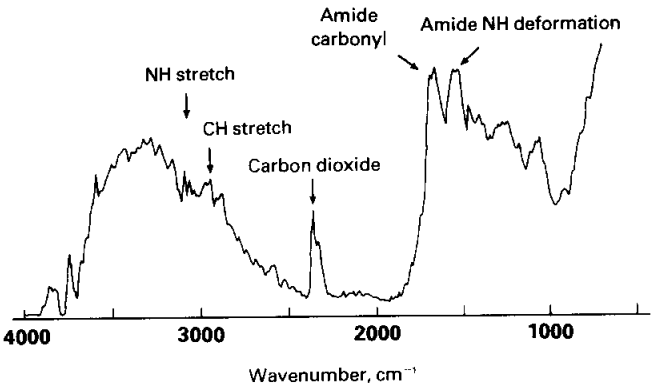

Figure 1 – FTIR-PAS spectrum of wool challis fabric

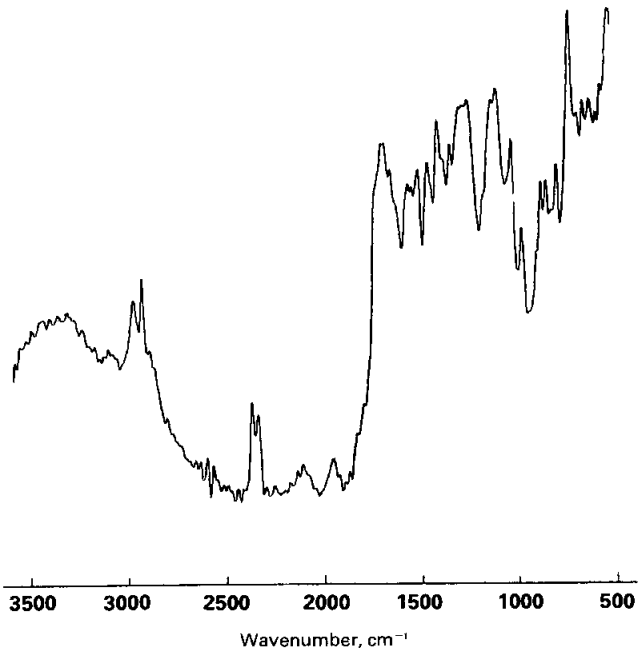

Figure 2 – FTIR-PAS spectrum of wool/polyester mixture

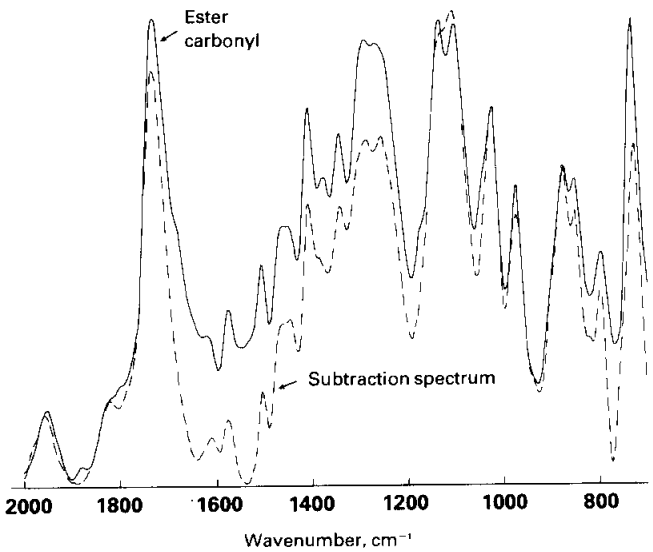

Figure 3 – Spectrum of polyester obtained by spectral subtraction, i.e. subtraction of spectrum shown in Figure 1 from that shown in Figure 2

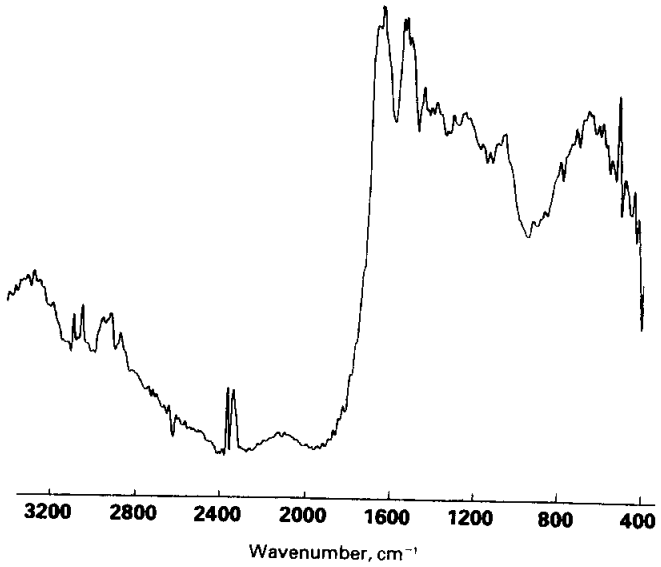

Figure 4 – FTIR-PAS spectrum of wool yarn

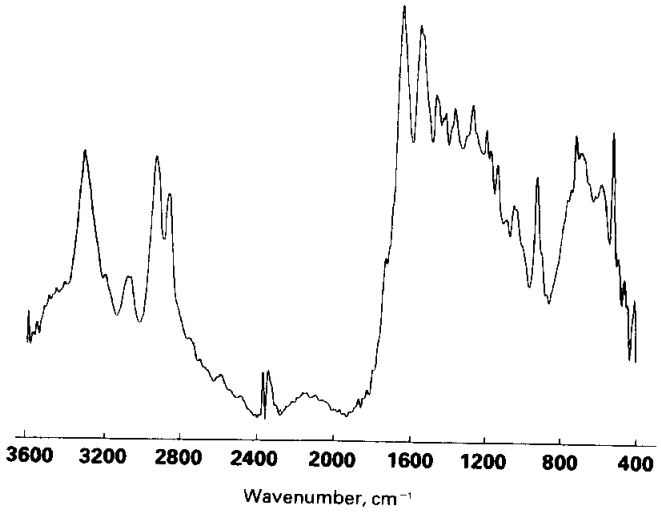

Figure 5 – FTIR-PAS spectrum of nylon yarn

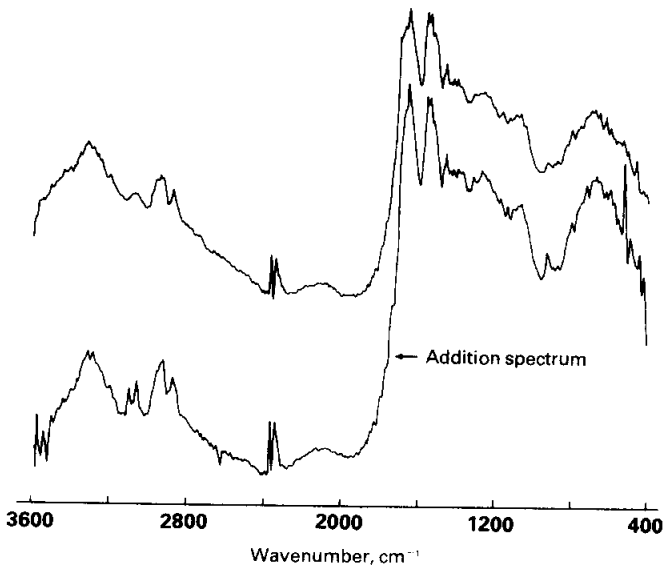

Figure 6 – Spectrum of hypothetical 80% wool/20% nylon mixture obtained by spectral addition, and FTIR-PAS spectrum of wool/nylon (80:20) yarn mixture

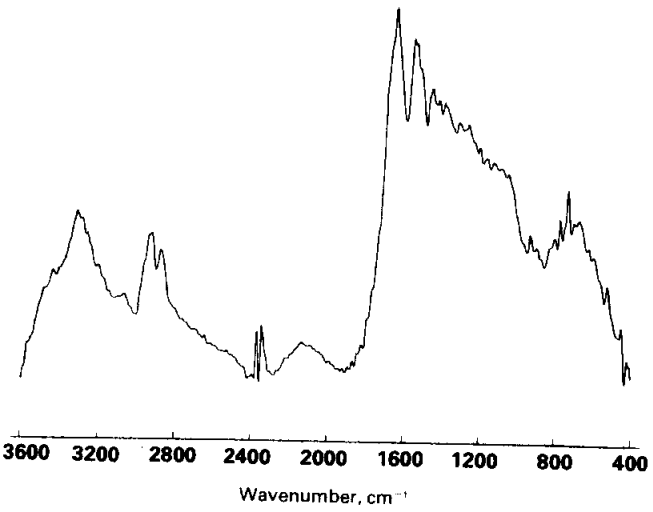

Figure 7 – FTIR-PAS spectrum of a wool/nylon/viscose rayon (40:40:20) mixture

RESULTS AND DISCUSSION

The FTIR-PAS spectrum of the wool (challis) and that of the wool/polyester blend fabric are shown in Figures 1 and 2 respectively. As can be seen from Figure 2, it is very difficult to find convenient absorption bands to enable the polyester to be identified and to determine the amount of polyester. Since the spectra are of good quality (resolution 8 cm^{-1}) the computer can be employed to subtract the spectrum of wool from that of the mixture. In principle the extent to which the subtraction is done is a measure of the wool content (in this case around 60%), although quantifying spectral subtraction is something of an art and as yet the accuracy of this process cannot be quantified. As can be seen from Figure 3, the spectrum of the polyester derived by the subtraction process is very similar to that of the authentic polyester. Thus the spectral subtraction process is particularly suitable for obtaining the spectrum of an unknown component in a two-component mixture.

Figures 4 and 5 show spectra of wool and nylon yarn respectively. In Figure 6 the spectrum of a yarn composed of 80% wool and 20% nylon is shown together with a spectrum obtained by adding (using the computer) the spectrum of wool and nylon (shown in Figures 4 and 5). To obtain the closest similarity between the synthesised and actual spectrum it is necessary to add contributions from the spectra shown in Figures 4 and 5 which correspond to the amounts of wool and nylon in the mixture. It can be seen from Figure 6 that spectral addition is another way of checking the composition of a binary mixture, if samples of the individual components are available. An interesting feature of Figure 6 is that the NH and carbonyl regions (1700 and 3000 cm^{-1}) of the synthesised spectra differ slightly from those seen in the spectrum of the mixture itself. These differences indicate that hydrogen bonding of the peptide bonds in the wool yarn may be somewhat different to that in the mixture. At this stage it is not clear whether this effect is due to molecular interaction between the peptide bonds of wool and the amide bonds of the nylon, or whether the spinning finish used with the nylon is affecting the peptide bonds of the wool. Since these spinning finishes are polyethers, it is quite possible that they interact and disrupt the hydrogen bonding exhibited by the peptide bonds of the wool.

A ternary mixture of wool and nylon and viscose rayon was also studied and its spectrum is shown in Figure 7. Subtraction of the spectrum of the wool/nylon mixture (Figure 6) from the spectrum shown in Figure 7 was

attempted in order to obtain the spectrum of viscose rayon. The result is shown in Figure 8 together with the spectrum of viscose rayon. One can clearly see that the spectrum obtained by subtraction contains a contribution from nylon (e.g. contributions at 3300, 740 cm^{-1}, etc). Difficulty in quantifying the subtraction process for this mixture was due to the fact that the viscose rayon and nylon have overlapping carbonyl bands at 1650 cm^{-1} and the nylon and the wool have overlapping bands around 1550 cm^{-1}. The use of the subtraction technique for ternary mixtures is undoubtedly more difficult to apply than for binary mixtures.

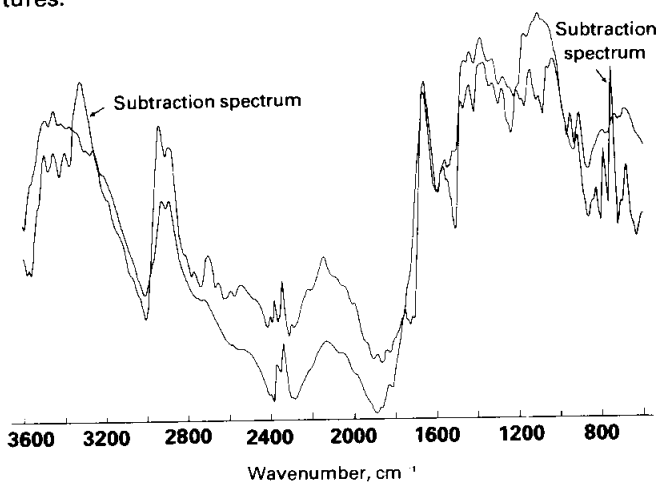

Figure 8 – FTIR-PAS spectrum of viscose rayon together with that obtained by subtracting the spectrum shown in Figure 6 from that shown in Figure 7

CONCLUSION

The results show that high-quality i.r. spectra of a variety of textile materials can be obtained using FTIR-PAS. The spectra can be used, in conjunction with the computer, to aid identification of components and to give some idea of the composition of fibre mixtures.

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