Application of photoacoustic FT-IR spectroscopy in textiles—studies on synthetic fabrics

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A simple, direct and non-destructive method for recording infrared spectrum of synthetic fabrics is described. The method is based on the photoacoustic fourier transform infrared (PA-FT-IR) spectroscopy and has great potential as a quality control/assurance tool for online monitoring of the quality of finished textile products. The method is suitable for comparison purposes as spectral characteristics change due to factors like shade variation, dyeing and composition of individual fibre components which can be used as quality indicators. A method for determining the acrylic fibre component in a nylon-acrylic fabric (dyed or undyed) has also been reported. The method does not require any reference standards (fibres or fabric) and is applicable for the composition ranges reported. The sample-cum-detector system can be custom built even for online monitoring.

Keywords: Acrylic fibre, Nylon-acrylic fabric, Photoacoustic FT-IR spectroscopy

1 Introduction
Modern optical spectroscopy has a great potential as a routine and automated tool, specially in quality control. Most of the currently practiced quality control measures are manual and those used for characterization are time consuming and destructive in nature. Because of the nature of the textile products (e.g. scattering in nature, or opaqueness in the case of dyed cloth), the infrared spectroscopy could not find a place in this well established industry specially as a non-destructive characterization tool. However, with the development in instrumentation (e.g. FT-IR, PAS, etc) and ability to combine two independent techniques (like GC, HPLC, PAS) the scope and extent of application has increased considerably. The present paper describes a simple, direct and non-destructive method for characterization of finished textile products that can be used as a quality control tool using photoacoustic FT-IR spectroscopy. The additional advantage of this technique is that it is possible to characterize the structural/chemical quality of the component fibres (or its parent copolymer in case of synthetic cloth) of the finished product. The work reported here is based on the studies carried out on the synthetic cloth made from nylon and acrylic yarns.

2 Materials and Methods
Transmission (TR), diffuse reflectance (DR) and photoacoustic (PA) spectra of the cloth sample woven from nylon and acrylic yarns were recorded on Bruker’s FT-IR spectrometer (Measurement parameters used were: No. of scans, 64; apodization, 4P; and velocity, 7).

DR spectrum was recorded under N₂ on Bruker’s IFS-88 model with MCT detector using Praying Mantis model (Harrick Corpn, USA) DR accessory. A mirror was used as a reference. The sample spectrum was also taken by keeping the sample on the mirror.

Transmission spectrum was recorded under vacuum on Bruker’s FT-IR 113V model.

PA spectrum was recorded using MTEC-PA cell on Bruker’s FT–IR 113V model. Carbon black was used as a reference. The absorbance spectrum of the sample was obtained after subtraction of the PA spectrum without any sample (i.e. the atmosphere used as a conducting medium in the PA cell) from the absorbance spectrum.

3 Results and Discussion
The IR spectra of cloth obtained using different accessories are shown in Figs 1-3. The major peaks, no. of peaks and the range of signal amplitude observed in each spectrum are given in Table 1.
As the sample consisted of nylon and acrylic fibres, the chemical constituents expected to contribute to the IR spectrum are nylon-6, polyacrylonitrile, methyl acrylate and methallyl sulphonic acid. The important band assignment to different functional groups are \( \nu_{NH} (3300 \text{ cm}^{-1}) \), \( \nu_{CH} \) methylene (\( \approx 2900 \text{ cm}^{-1} \) doublet), \( \nu_{C-N} (\approx 2240 \text{ cm}^{-1}) \), combination of \( \nu_{CO} \) and \( \nu_{CO} \) (amide I) (\( \approx 1700 \text{ cm}^{-1} \)), ester group of MA (between \( 1200-1100 \text{ cm}^{-1} \)), and \( \text{SO}_3 \) (\( \approx 540 \text{ cm}^{-1} \)).

A comparative study of these spectra indicates that (i) the bands mentioned above are present in all the spectra but with varying degrees of resolution, (ii) the signal amplitude (y-axis) range in the case of DRS in nearly 4 times than those of transmission and PA spectra, showing better sensitivity of the latter (Table 1, Figs 1-3), (iii) the resolution of PA spectrum appears to be superior and follows the sequence: PAS > DRS > Transmission (Figs 1-3), and (iv) the number of peaks obtained by peak picking for each spectrum indicates the following order: PAS > DRS > Transmission.

The FT-IR spectrum of the cloth represents a combined spectrum of its components, viz. nylon fibre, acrylic fibre and the dye (in case of dyed samples). The facility to carry out mathematical manipulations (like subtraction of standard component spectra from that of the sample) may further help in the identification of particular component responsible for the poor quality of the product (cloth). For instance, the IR spectrum of each component can be obtained by quantitative subtraction of IR spectrum of individual components from that of blended fabric and then matching the resultant spectrum with that of the corresponding individual component.

The photoacoustic FT-IR spectra of two undyed samples having different shades but from the same fabrics are shown in Figs 4 and 5. A visual comparison shows a marked difference between the two, specially in the region 500-1500 cm\(^{-1}\). This difference can be used as an indicator of difference in quality of the fabric in these areas. Similarly, Figs 3 and 4 show the spectra of the same
portion of the fabric with and without dye (the dye used was methylene blue). The difference between the two is also clearly observed. These examples show the potential of this new technique as a routine quality control tool in textiles.

A semi-quantitative method for determining the composition of acrylic fibre component from the IR spectrum of the blended fabric (Fig. 3) has also been developed. The method is essentially the same as that reported earlier for single fibres except that the technique used here is PA-FT-IR instead of microscopic-FT-IR and the infrared spectrum used for the purpose is that of blended fabric rather than single fibre. Though the details of the method have been reported earlier, a brief account of the procedure is given below.

IR spectrum of the fabric contains well resolved bands at 2240 cm\(^{-1}\) and 1720 cm\(^{-1}\) corresponding to \(\nu C=O\) and \(\nu C=\equiv N\) which are also representative of the PAN and MA components of acrylic fibre present in the fabric. To reduce the error due to physical variations in the sample, optical density ratio (ODR) of the two bands was used in the final calculations. ODR is given by

\[
ODR = \frac{\text{Absorbance } \nu C=O}{\text{Absorbance } \nu C=\equiv N}
\]

The absorbance of each of the above bands was determined from the spectrum as described below:

Tangents were drawn from the bases of the two bands (Fig. 3) at 2310 cm\(^{-1}\) and 2200 cm\(^{-1}\) for 2240 cm\(^{-1}\) band and 1780 cm\(^{-1}\) and 1680 cm\(^{-1}\) for 1720 cm\(^{-1}\) band and perpendiculars were drawn from the corresponding apex of the two bands. The distance from the base (zero line) to the point of intersection on the base line (i.e. tangent) \((I)\) and that to the apex of the peak from zero line \((I_0)\) were measured. The absorbance ratios and the corresponding ODR were calculated using the above data (these measurements can also be carried out through interactive display) and used to determine % MA content \((x)\) in the acrylic yarn of the blend using the following relationship

\[
x = 16.3y - 8.5y^2 + 2.24y^3 - 1.78
\]

where \(y = ODR\)

The MA content determined by this method has been reported in Table 2. It is evident from the data that the method is valid as the MA content in the dyed and undyed samples from the same batch is same as expected. However, the variation from sample to sample can be taken as an indication of the fact that the samples are from different production batches.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>MA content, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dyed fabric</td>
<td>Undyed fabric</td>
</tr>
<tr>
<td>227</td>
<td>9.60</td>
</tr>
<tr>
<td>228</td>
<td>9.99</td>
</tr>
<tr>
<td>229</td>
<td>9.30</td>
</tr>
<tr>
<td>230</td>
<td>9.12</td>
</tr>
</tbody>
</table>

4 Conclusion

Photoacoustic FT-IR spectroscopy is one of the most suitable technique for analyzing cloth samples. It provides a simple and non-destructive method that can be used as a routine method for quality control/assurance in finished products. If needed, a custom-built online monitoring instrument can also be developed which can record/monitor the quality of the finished product at fixed distances/intervals automatically using computer-
ized data analysis which is in-built in the instrument. The only requirement is a PA-FT-IR system.

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