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PHOTOACOUSTIC SPECTROSCOPY MEASUREMENTS OF FLUORESCENTLY WHITENED WOOL FABRIC

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Abstract - Detection of fluorescent whitening agents on wool and analysis of a wool/nylon mixture was carried out using UV/visible and FTIR PAS respectively.

We have previously shown that photoacoustic spectroscopy (PAS) can be used to obtain the absorption spectra of dyes on wool fabrics. The absorption spectra of various dyes in two-dye and three-dye combinations can easily be distinguished using this technique. Current research in this department indicates that the technique is sensitive to glossy surfaces. However, at the level of instrument sensitivity used it was found that small differences in fabric weave did not significantly affect the signal amplitude, i.e. plane weave flannel and worsted fabrics, treated in the same manner, gave similar photoacoustic and reflectance spectra. Because of the difficulties in obtaining absorption spectra of fabrics using conventional techniques, reflectance spectroscopy has generally been used for the study of dyes on fabric.

Although reflectance spectroscopy is of great value for visual colour evaluation, it cannot act as a substitute for absorption spectroscopy. The advantages of obtaining absorption spectra of fabrics is most clearly demonstrated by fabrics treated with fluorescent dyes. Such treatments are becoming more commercially important. As this laboratory has been investigating the effects of fluorescent whitening agents (FWAs) on the photoyellowing of wool fabrics it was felt that a commercial FWA could be used to exemplify the potentials of PAS for the measurement of fluorescent compounds on fabrics.

Whilst UV/visible spectroscopy is ideal for studying surface treatments containing appropriate chromophores, FTIR-PAS is well suited for obtaining IR spectra of fabric samples due to ease of sampling. As a consequence, an exploration of the applicability of this technique to the analysis of textile mixtures was undertaken.

Experimental

The wool fabric chosen for this study was scoured serge (200g/m²). Uvitex CF200 (Ciba-Geigy Ltd.) was applied to the fabric using a John Jeffreys' Dyemaster with a liquor to wool ratio of 30:1. Sodium hydrosulphite (5% on weight of fabric), formic acid (to pH 3.5) and the FWA (1% on wool) were added. The temperature was raised to 95°C at 1°C/minute, and held for 30 minutes at 95°C. The wool was then rinsed with cold water. The exhaustion, for the FWA used, was found to be above 95%.

To obtain bleached wool a pad/batch method was used. The bleach bath contained Detergyl EDC (10g/l, Alliance-Francolor), urea (300g/l), Solvitose OFA (8g/l, Avebe) dispersed in propan-2-ol, and sodium bisulphite (20g/l). The wool was padded and expressed to 100% liquor retention and then the wool was batched overnight at room temperature.
The wool fabric was irradiated for 16 hours at room temperature by exposure to long wave UV fluorescent tubes, with a $\lambda_{\text{max}}$ at approximately 350nm. PA spectra were obtained from an EDT Model OAS 400 spectrometer. FTIR-PA spectra were run on a Nicolet 7000 series instrument fitted with a Princeton Applied Research photoacoustic cell.

Results and Discussion

Fig. 1 shows the PA spectrum of a silica standard. This exhibits some absorption between 300-500nm. Serge wool fabric absorbs light below 550nm. When this fabric is bleached with sodium hydrosulphite the amount of light absorbed between 350-550nm is reduced (Fig. 1). When the FWA is applied to the fabric the whiteness of the fabric is visibly improved. The presence of the FWA on the fabric can be readily detected by PAS as shown by Fig. 2. Unlike reflectance spectroscopy, the measurement of the absorption spectrum of the FWA is unaffected by the fluorescence of the FWA ($\lambda_{\text{max}}$ 437nm).

Natural wool yellows on exposure to sunlight, and this process is accelerated by bleaching the wool, by water, and by the application of FWAs\textsuperscript{3}. This yellowing can be assessed by PAS (Fig. 3). When the wool is irradiated, the surface of the wool facing the light yellows to a greater extent than the surface facing away from the light.

The PA spectrum of the irradiated wool with an applied FWA (Fig. 4), shows that photoproducts have been produced which absorb light below 550nm, and consequently give the material a yellow cast. The spectrum also shows that the absorption band, which is characteristic of the FWA, has disappeared, suggesting that much of the absorption below 420nm is due to photoproducts. It appears that, on irradiation of the whitened wool, the FWA is being destroyed, causing a decrease in absorption below 420nm, but this is offset by the formation of photoproducts (from either the wool or the FWA) in this region.

The above results, together with those obtained from fabrics dyed with compounds which absorb in the UV-visible region\textsuperscript{1} demonstrate the usefulness of UV-visible PAS in textile chemistry.

IR-PAS has also been shown to be of value in the analysis of textile mixtures\textsuperscript{6}. For IR spectroscopy to be of any use, the constituents of the mixture must have reasonably different IR spectra so that analysis can be made on the basis of the presence or absence of absorption peaks, or the relative heights of one peak, when compared with another. Sampling presents a further problem for conventional IR spectroscopy. It is particularly difficult to obtain reproducible results using attenuated total reflectance (ATR) methods due to the problems associated with making a good contact between the sample and the crystal with ATR attachment. In an alternative approach, the fibres are ground up and then incorporated into a potassium bromide disc. Once again the reproducibility in preparing samples presents a problem. By way of contrast, the preparation of samples for FTIR-PAS is extremely simple. Small samples are cut from the cloth or yarn and inserted into the sample holder. To obtain good results it is essential not to push the sample hard down into the cell. There is a limited range of FTIR spectrometers which can be used with PAS since it is necessary to keep the velocity of the mirror in the interferometer down to quite low values. The Nicolet instrument is well-suited for this purpose. Furthermore, its powerful computer allows one to both add and subtract spectra.

Fig. 5 shows a spectrum of wool-polyester mixture. The spectrum was easily obtained with a very short accumulation time. The spectral resolution is good, but, even so, it is difficult to assign peaks unequivocally to either the wool or the ester and to quantify the data. By subtracting the spectrum of pure wool from the spectrum shown in Fig. 5 the spectrum of the polyester was obtained. The result is shown in Fig. 6 together with the spectrum of an authentic sample of the polyester. The two spectra are very similar and this result shows that spectral subtraction can be used to good effect. Obtaining spectra in this way aids identification of the
Fig. 1 PA spectra of wool
Fig. 2 PA spectra of fluorescently whitened wool

Fig. 3 The surface effect of irradiating bleached wool
Fig. 4 PA spectra of irradiated fluorescently whitened wool
Fig. 5 FT-IR of challis + polyester

Fig. 6 FT-IR of polyester by subtraction
unknown ester and since the amount of subtraction required to eliminate absorbances from the wool can be quantified, the composition of the mixture can be obtained. This example shows that FTIR-PAS can provide high quality IR spectra of textile mixtures and aid in their analysis.

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References

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