

Quantitative Analysis of Wool/Cotton Blends: An Infrared Method

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Synopsis

An infrared method is described for the analysis of wool/cotton blends. The method employs the absorption peak of wool at 1520 cm^{-1} and that of cotton at 1160 cm^{-1} simultaneously to get a reliable accuracy greater than $\pm 3\%$ in the determination of blend composition in the analysis of actual blends.

INTRODUCTION

The usefulness of semimicro methods, such as those based on infrared absorption, for quantitative estimation of blend composition cannot be overemphasized. In a previous communication,¹ the authors reported details regarding standardization of an infrared method for the analysis of polyester/cotton blends. In the course of an investigation in this laboratory to assess the suitability of cotton processing systems for spinning wool/cotton blends and to arrive at optimum blend compositions for desired end uses, it became necessary to determine the proportion of components at various stages of spinning. The studies on the analysis of blend composition were therefore extended to cover wool/cotton blends.

In the light of observations made in connection with the analysis of polyester/cotton blends, the analysis of wool/cotton blends seemed to be quite straightforward, as wool has a strong absorption peak at 1520 cm^{-1} ,* while cotton does not absorb in this region. However, on trying out the method suggested for polyester/cotton blends,¹ the plot of $A \times d\nu$ (absorbance \times band width) versus % wool for a series of cotton/wool mixtures failed to yield a straight line even when $d\nu$ varied only over a very small range. The reason for this behavior appears to be the very large band width ($\approx 75\text{ cm}^{-1}$) on account of which the peak intensity varied much faster than the band width with change of particle size. Hence, it became necessary to have strict control over the particle size utilized in getting the calibration graph for the instrument. We have been able to judge this by using the cotton peak at 1160 cm^{-1} along with the wool peak at 1520 cm^{-1} .

* The 1650 cm^{-1} band of wool was not used, as this band appears to be influenced by the adjacent band at 1630 cm^{-1} due to sorbed water.

EXPERIMENTAL

Sample Preparation

For Standard (Calibration) Graph. Cotton and wool (total weight 20 mg) of known proportions were mixed thoroughly and powdered by grinding in a vibratory ball mill for about 5 min. As the percentage of the wool in the mechanical blend increased, a slightly higher ball milling time was given. This was necessary to keep the scatter of points about the regression line to a minimum, especially for the wool peak. A known amount of the mechanically blended sample was mixed thoroughly with a known weight of KBr; and by a process of successive dilution the amount of blend, of various known concentrations, in the final pellets was adjusted to 1.25 mg.

Analysis of Unknown Blends. Unknown blends comprised yarns processed in this laboratory and fabrics obtained commercially. The yarn samples, which were products of the investigation on wool/cotton blends referred to in the introduction, had been spun from Hybrid-4 cotton and scoured, carbonized wool blended together at the drawing stage. In both cases (yarns or fabrics), the samples were finely cut, ground in the ball mill (20 mg for 5 min), and then subjected to progressive dilution in exactly the same manner as the mechanical blends used for getting the calibration curve so that the final pellet in this case, too, contained 1.25 mg of the blend.

Recording of Spectra

Spectra were recorded by using the Perkin-Elmer Model 457 grating spectrophotometer. In all cases the region from 1800 to 800 cm^{-1} was scanned with normal slit program and at medium scan speed. The absorbances for the two peaks of interest were determined by employing the usual baseline method.

Chemical Analysis

This was carried out according to a standard procedure², in which the wool part of the blend is dissolved away completely by boiling in 5% (w/w) sodium hydroxide for $\frac{1}{2}$ hr. The percentage loss of weight of cotton due to alkali boiling was also determined independently, and correction to this effect was applied.

RESULTS AND DISCUSSION

Figure 1 shows the absorption spectra of wool/cotton blends for two different blend compositions in the 1800–800 cm^{-1} range. The absorption peaks used in the blend analysis are indicated in the figure along with the baselines. The standard calibration graphs obtained on the basis of wool peak (1520 cm^{-1}) and cotton peak (1160 cm^{-1}) for the wool.cotton blends are given in Figures 2 and 3.

Table I shows data on variation in intensity of the bands at 1520 and 1160 cm^{-1} in relation to ball milling time for pure wool, cotton, and a blend of cotton and wool. It is evident from the table that as a result of vibratory ball milling, which results in progressive reduction of particle size, the absorbance A of the wool peak (1520 cm^{-1}) increases sharply without significant change in band width, such

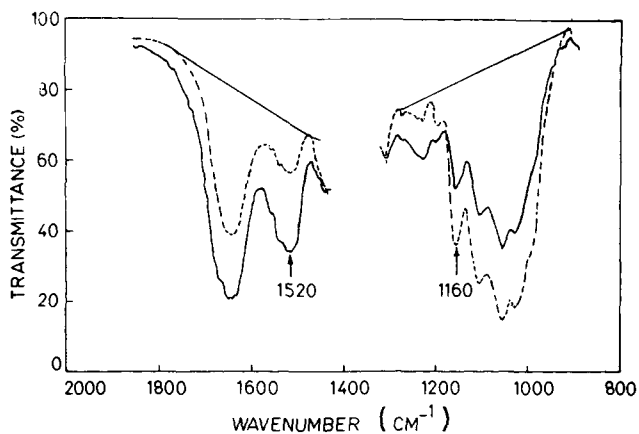


Fig. 1. Infrared spectra of wool/cotton blends in the range of 1800–800 cm^{-1} for two different blend compositions. Baselines for the 1520 and 1160 cm^{-1} bands used in the present investigation are also shown.

that $A \times d\nu$ shows a net rise. It is necessary, therefore, to ensure equal particle size for wool in all the mechanical blends so as to obtain a straight calibration curve of absorbance versus concentration. It is also necessary to maintain the same particle size in the unknown blend in order to arrive at the correct composition. However, consistency in particle size cannot be easily achieved because of uncontrolled variations in the vibrational amplitudes even though the ball milling time, quantity of sample, and number of balls in the vial, etc., are kept constant. Moreover, there is no direct way of ascertaining whether the ball milling carried out has resulted in the optimum size of particles required for correct blend estimation.

Exclusive use of the cotton peak at 1160 cm^{-1} is equally unsatisfactory because the change in intensity of this band with increase in concentration is not large enough to permit accurate quantitative analysis. Further, this peak also shows

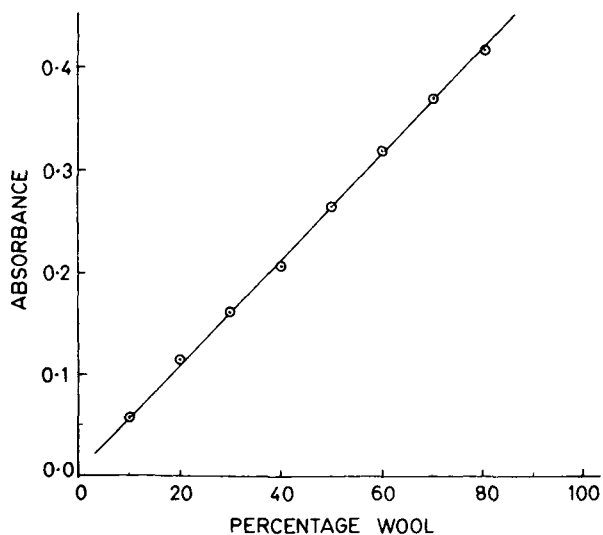


Fig. 2. Standard calibration graph for the estimation of wool percentage in wool/cotton blends (based on 1520 cm^{-1} band of wool).

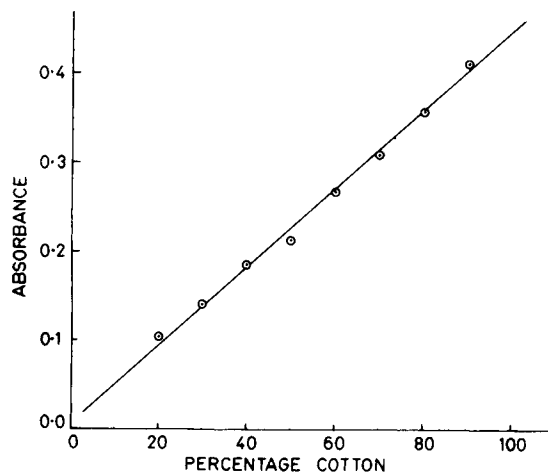


Fig. 3. Standard calibration graph for the estimation of cotton percentage in wool/cotton blends (based on 1160 cm^{-1} band of cotton).

a small but definite rise in intensity with ball milling, though the effect tends to level off beyond a certain ball milling time.

It is also evident from the data in Table I that wool fibers, however finely cut by means of scissors, give rise to a shallow peak, unsuitable for the analysis. Although the 1160 cm^{-1} cotton peak is relatively intense even before grinding, it is still necessary to subject the sample to ball milling for achieving uniform dispersion in the KBr matrix. Moreover, grinding also helps to increase the

TABLE I
Variation of Peak Intensity with Ball Milling Time for the Peaks at 1160 and 1520 cm^{-1} in Pure Cotton, Pure Wool, and in a Wool/Cotton Blend

Sample	Weight of sample in the pellet mg.	Ball milling time, min.	Absorbance A due to band at		$d\nu$ of wool peak, cm^{-1}	$A \times d\nu$ (wool peak)
			1160 cm^{-1} (cotton)	1520 cm^{-1} (wool)		
Wool	0.25	0.0	—	0.0370	—	—
	0.25	10.0	—	0.1083	74.0	8.0142
	0.25	20.0	—	0.1933	70.0	13.5310
Cotton	0.50	0.0	0.1130	—	—	—
	1.25	0.0	0.2522	—	—	—
	1.25	5.0	0.4427	—	—	—
	1.25	10.0	0.4493	—	—	—
	1.25	20.0	0.4658	—	—	—
Mechanical blend (60% wool + 40% cotton)	1.25	0.0	0.1133	0.0378	—	—
	1.25	10.0	0.2116 (47) ^a	0.3774 (72) ^a	73.0	27.550
	1.25	15.0	0.2201 (49) ^a	0.4260 (81.5) ^a	72.0	30.672

^a The cotton/wool percentages obtained from the respective absorbances are given in parenthesis.

TABLE II
Comparison of Blend Composition of Blended Yarns Obtained by Infrared Analysis and Chemical Analysis

Sample	Serial no. for analysis	% Wool (by chemical analysis)	Absorbance at		% Cotton from cotton graph (c)	% Wool by infrared method		Mean = $(w + w')/2$
			1520 cm^{-1} (wool peak)	1160 cm^{-1} (cotton peak)		From wool graph (w)	From cotton graph (w')	
Yarn I	1	59.5	0.3038	0.1859	40.8	57.8	59.2	58.5
	2	59.2	0.2944	0.1743	38.5	56.0	61.5	58.8
	3	60.2 (59.6) ^a	0.3257	0.1880	41.5	62.0	58.5	60.2 (59.2) ^a
Yarn II	1	40.1	0.2405	0.2795	62.5	45.5	37.5	41.5
	2	39.3	0.2361	0.2784	62.0	44.5	38.0	41.2
	3	39.0 (39.5) ^a	0.2196	0.2773	62.0	41.5	38.0	39.8 (40.8) ^a
Yarn III	1	25.2	0.1549	0.3632	79.0	29.0	21.0	25.0
	2	25.2	0.1404	0.3374	74.0	26.0	26.0	26.0
	3	25.7 (25.3) ^a	0.1417	0.3242	72.5	26.2	27.5	26.9 (26.0) ^a
Fabric (wool + cotton)	1	39.9	0.2130	0.2896	64.5	40.0	35.5	37.8
	2	38.6	0.2034	0.2855	63.5	38.2	36.5	37.4
	3	39.5 (39.3) ^a	0.2121	0.2961	66.0	40.0	34.0	37.0 (37.4) ^a

^a Average of each of the two different methods is given in parenthesis at the bottom in respective columns.

change in peak intensity with concentration, thereby leading to an increase in the accuracy of estimation of cotton component in the blend. Thus, in both cases ball milling becomes indispensable.

From the above discussion it follows that if the wool component in an unknown blend happens to be overground whereby the particle size becomes smaller than that in the mechanical blend used for obtaining the calibration graph, then the composition derived from the wool peak will evidently be an overestimate. Similarly, an overground cotton component would give rise to a peak from which the estimate of cotton component would also be high. The net effect of the above particle size differences will be that the estimate of blend composition derived independently from the two peaks will generally differ. It is also evident from Table I that with increase in ball milling time, the overestimate of wool percentage from the wool peak increases faster than that of cotton from the cotton peak. Thus, with the results obtained from the two absorption bands, it is possible to say whether adequate degree of grinding has been effected during ball milling. If not, a different ball milling time, higher or lower, depending on the requirement, may be chosen so that the disagreement between the two independent estimates of composition is reduced.

The results of independent estimation of composition from the wool and cotton peaks are gathered in Table II for three differently blended yarns and for a commercial fabric, along with the estimates of wool percentage by chemical analysis. Infrared data were obtained with the help of the calibration curves shown in Figures 2 and 3. It is evident from the data in Table II that if the two estimates for the percentage of wool obtained from the wool and cotton peaks do not differ by more than 10 units, then the average of the two estimates would be within ± 3 of the actual value obtained by chemical analysis. Thus, a simultaneous determination of the blend composition from the wool and cotton peaks helps to reduce errors that may arise from estimates with a single peak which, as stated earlier, is sensitive to particle size. If, however, sufficient sample is available, the determinations may be repeated with various other ball milling times so that the estimates from the wool and cotton peaks eventually coincide, thus giving a greater accuracy of estimation of the components.

CONCLUSIONS

A simultaneous use of the wool peak at 1520 cm^{-1} and the cotton peak at 1160 cm^{-1} renders the analysis of wool/cotton blends easy and also reduces the uncertainty in the determination of blend composition based on a single peak. The average of the two independent estimates of percentage composition from the above peaks gives the actual value to an accuracy of better than ± 3 units, even when the values of independent estimates differ by about 10. This accuracy can be further improved by repeating the analysis with suitable ball milling time (when sufficient sample is available) so that the values of blend composition determined from the two different peaks nearly coincide.

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References

1. P. Bhama Iyer, K. R. Krishna Iyer, and N. B. Patil, *J. Appl. Polym. Sci.*, **20**, 591 (1976).
2. E. Horowitz and J. Mandel, *Am. Dyest. Rep.*, **43**, 613 (1954).

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