

An Infrared Technique for the Quick Analysis of Cotton-Polyester Blends

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Synopsis

The details of an infrared technique standardized to analyze cotton-polyester blends of different compositions are reported. It is observed that particle size plays a very important role in the peak intensity of the 1725 cm^{-1} band (C=O stretching) in polyester, the absorption band recommended for analysis of cotton-polyester blends. However, the product of the band width and peak intensity is found to be dependent on particle size to a lesser extent and hence is a more reliable parameter in the measurement of percentage composition. It has further been shown that the method of sample preparation can affect the band width and hence the results. A suitable sample preparation procedure is, therefore, described whereby an accuracy of $\pm 3\%$ in the estimation of polyester content in a given blend can be achieved.

INTRODUCTION

A number of methods are available for the analysis of fiber blend composition of which the chemical method is the one usually employed when enough sample is available. Any microtechnique which requires very little sample (of the order of 10 mg or less) has the added advantage that it can be utilized to study the variation in blend composition from point to point of a blended yarn or fabric. Such variations in blend composition which are quite common in the conventional spinning process lead to streaks or variation in shade in the fabric. Cotton-polyester blends have become more popular these days, and hence it was thought worthwhile to standardize the infrared technique for determining the blend composition.

Use of infrared technique in quantitative analysis of fiber blends is well known. The method becomes very simple when each of the analytical components has a nonoverlapping absorption band. O'Connor¹ has mentioned the possibility of using the technique in the analysis of cotton-Dacron blends. However, a preliminary attempt to find out the percentage composition from peak intensity measurements (i.e., from a plot of absorbance versus percentage composition), as suggested by O'Connor, was found to lead to ambiguous results. Further, the present authors did not come across actual details of such analysis in the literature. A thorough investigation was also undertaken to identify the factors that influence such an estimation.

EXPERIMENTAL

Materials

Cotton and polyester yarns and their blends of various compositions, all spun to 50s count on conventional spinning machines in the Laboratory, were used in the study. The polyester stable fiber Futura (trade name of polyester fiber made by Indian Organic Chemicals Ltd.) used in the blends was of normal tenacity and of 1.5 denier.

The cottons used were Sudan and Hybrid 4; in each case, the blend composition was as follows: cotton (Sudan): polyester = 75:25 (SF1), 50:50 (SF2), 33:67 (SF3); and cotton (Hybrid 4): polyester = 75.25 (HF1), 50:50 (HF2), 33:67 (HF3).

Sample Preparation

Standard (Calibration) Curve. Cotton and polyester yarns were finely cut, and mechanical mixtures of different predetermined compositions were prepared by mixing required masses of cotton and polyester in a vibratory ball mill. The total mass of sample for each mechanical mixture was restricted to 15–20 mg. A ball milling time of not less than 10 min was used in each case for proper mixing and powdering of the samples. The powder in each case was pressed in a die at a pressure of 4.5 tons/cm² for 3–5 min to get a thin cake (pellet) of sample. A mass of 10 mg from this cake was mixed with 220 mg spectral-grade KBr by grinding for 10 min in an agate mortar. The sample-KBr mixture was taken in a die and pressed for 5 min under a pressure of 7.5 tons/cm². The sample was also subjected to evacuation (using an ordinary backing pump) in the die 5 min prior to and during pressing. The relative humidity of the room where the above operations (mixing with KBr and pressing) were carried out was below 40%, whereas the sample weighing and ball milling were done at 65% R.H.

The pellets prepared in the manner described above were, however, opaque and hence required further dilution. This was done by taking 10 mg mass of the above-mentioned pellet (sample + KBr) and mixing it with 90 mg KBr (weight of KBr is not very critical in this case) again by grinding in an agate mortar for 8–10 min and pressing as before. Thus, a set of pellets containing a mass of polyester less than 0.43 mg and giving transmittance in the range of 20% to 70% was finally obtained for scanning.

Analysis of Blends. The same sample preparation procedure was used as that described above. A ball milling time of 12–15 min was used for all the blends. This was, however, found to yield "broader" band with blends containing large proportions of polyester. Hence, for such samples, a longer ball milling time was used (up to 20 min), or the polyester content was brought down by adding a known weight of pure cotton to a known weight of the blend (in the proportion, say, 1:2).

In addition to the above, a set of KBr pellets containing known percentages of cotton and polyester was also prepared, using cotton and polyester samples ball milled separately.

TABLE I
Data Used in Obtaining Standard Curve Shown in Figure 1

Polyester in mechanical mixture, %	Ball milling time, min	Absorbance A at peak max.	Measured bandwidth $d\nu'$, cm^{-1}	Actual band with $d\nu = \frac{d\nu'}{2.5}$, cm^{-1}	$A \times d\nu$
20.0	10.0	0.2099	73.1	29.2	6.13
30.0	11.0	0.2792	74.0	29.6	8.26
40.0	12.0	0.3880	73.0	29.2	11.33
50.0	14.0	0.4150	81.0	32.4	13.45
60.0	16.0	0.4635	88.0	35.2	16.32
70.0	20.0	0.5211	92.5	37.0	19.28

Recording the Spectra

Spectra of the pellets were recorded using a Perkin—Elmer Model 457 spectrophotometer from 1900 cm^{-1} to 1500 cm^{-1} ; settings used were: slit, normal; scan speed, medium; and scale expansion, $\times 2.5$. For each pellet, the 1725 cm^{-1} band was scanned twice, the second scan corresponding to rotation of the pellet through 90 degrees in its own plane with reference to the first position. And if the % transmittance differed by more than 1%, it was taken to indicate nonuniformity in the mixing of samples. Such samples were rejected, and fresh pellets were prepared and their spectra recorded. The peak intensity (absorbance) in each case was measured by the baseline technique.² The band width was measured in the region of the band where the absorbance was half the maximum.

Chemical Analysis of Blends

These were done according to the method given in the *B.S. Handbook*.³ Each blended sample was analyzed twice and the average value was taken.

RESULTS AND DISCUSSION

Table I gives the data used in obtaining the standard curve. It is evident from the values that absorbance versus % composition will not yield a straight

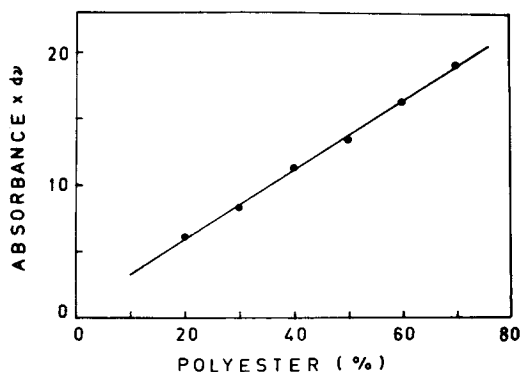


Fig. 1. Calibration curve for determination of polyester in cotton-polyester blends by measurement of product of absorbance and band width of the 1725 cm^{-1} band.

TABLE II
Blend Compositions Computed by Different Methods

Sample	Absorbance <i>A</i>	Band width <i>dν</i> , cm^{-1}	<i>A</i> × <i>dν</i>	Polyester, %			
				Nominal	Derived from		Chemical method
					Fig. 1 (method I)	Fig. 2 (method II)	
SF1	0.2320	31.2	7.24	25.0	25.0	39.0	24.8
SF2	0.4849	29.2	14.16	50.0	51.0	80.0	49.7
SF3	0.4914	36.0	17.69	67.0	66.0	81.5	66.4
HF1	0.2088	33.2	6.93	25.0	24.5	34.5	25.0
HF2	0.4125	34.4	14.19	50.0	51.0	66.5	50.8
HF3	0.4715	36.8	17.35	67.0	63.5	76.0	68.7
HF3 ^a	0.4172	30.0	12.52	44.6	45.0	—	—

^a This was obtained by mixing HF3 with pure cotton in 2:1 proportion.

line. On the other hand, the product of absorbance and band width varies linearly with the composition, as is shown in Figure 1. However, absorbance versus % composition gives a straight line (Fig. 2) when cotton and polyester are ball milled separately and then mixed at the required proportion to get mechanical blends.

The compositions of unknown blends obtained by means of Figure 1 (method I) and Figure 2 (method II) are entered in Table II along with the values determined by chemical analysis. It may be seen that the results obtained from Figure 1 give the true composition as given by the chemical method to a high degree of accuracy (within $\pm 3\%$), while those obtained from Figure 2 highly overestimate the values. This is mainly because the peak intensity is a function of the particle size. The reduction of particle size of polyester when in blend with cotton is always higher than when pure polyester is ball milled for the same or even longer intervals of time. Even though a plot of absorbance versus % composition gives a straight line when cotton and polyester are ball milled separately and mixed, the actual absorbance for the

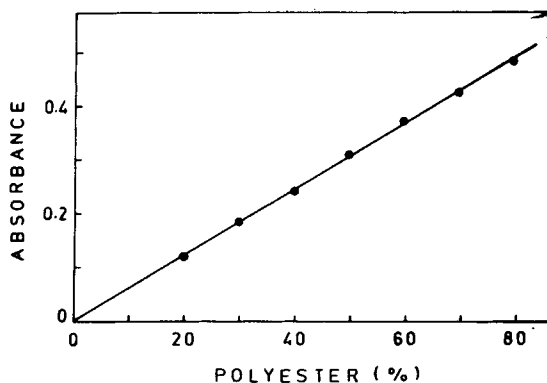


Fig. 2. Dependence of polyester percentage on the absorbance at peak max. of the 1725 cm^{-1} band (cotton and polyester ball milled separately and then blended to get the desired compositions).

same amount of polyester is low because of bigger particle size of the latter (which effectively results in a broader band).

Even for results obtained from Figure 1, the claimed accuracy is possible only when the unknown blend gives a band width ($d\nu$) which lies within the two extreme values of $d\nu$ used in getting the standard curve. This was brought out by the fact that the pure 67% nominal blend gave a higher band width (36.8 cm^{-1}), and hence a greater % error in the estimation of blend composition, when ball milled for the same extent of time as that used for blends with polyester content less than 50%. However, the accuracy is increased when the band width is reduced either by using a longer ball milling time or by adding a known weight of pure cotton (to the blend) before ball milling. Hence, for an unknown blend, if after the fixed ball milling period too broad a band is obtained, it may be that it contains more polyester than cotton, and hence one of the above procedures (preferably the latter) should be adopted.

CONCLUSIONS

The infrared method should be used to analyze cotton-polyester blends with sufficient caution. Reliable accuracy ($\pm 3\%$) can be obtained if the product of absorbance and band width is plotted against % compositions rather than absorbance against % composition. A linear plot for absorbance \times band width versus % composition is possible only when the band widths for the extreme compositions (say, 20% and 80%) do not differ by more than $7\text{--}9\text{ cm}^{-1}$. The band width obtained for any unknown blend should also lie within the extreme band widths used in getting the standard graph. The greater the deviation of the band width from the given criterion, the higher will be the % error in the estimation of blend composition. Once the standard curve is obtained for a given spectrophotometer, the analysis of an unknown blend is a matter of only a couple of hours.

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