Irradiation of Solvent-Treated Cotton Fibers

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Synopsis

The infrared spectra and dielectric properties of cotton fibers pretreated for one day with toluene, methanol, and mixtures of the two have been investigated after drying the samples for 1 and 4 days. The effect of γ-radiation on the crystallinity and dielectric properties of the solvent-treated was examined. The samples were irradiated in air and also in the presence of the solvents. The data obtained indicate that the addition of toluene to methanol increases the rate of uptake of methanol by cotton. Moreover, it is found that irradiation of cotton fibers in the presence of toluene assists the oxidation of the cotton while irradiation in the presence of methanol reduces the oxidation effects of γ-radiation.

INTRODUCTION

The penetration of solvents into various textiles has been investigated by several workers.[1-3] Solvent penetration is generally an advantage for dyeing treatments, whereas for many other finishing treatments it is a disadvantage. The previous studies demonstrated that some solvents can be introduced into cellulose by solvent exchange. The treatment of textiles with solvent mixtures containing a readily sorbed and a not readily sorbed solvent results in the penetration of the fibers by both solvents. The irradiation of textile fibers in solvents indicates that the oxidation effects of γ-radiation depends on the solvent used.[4,5]

The present paper reports on a study of the infrared spectra and dielectric properties of cotton fibers treated for 1 day with solvent and γ-irradiated.

EXPERIMENTAL

The fibers were first washed and dried in an oven at 70°C for 24 h. The dried fibers were then ground in a Spex mixer mill at room temperature for short times (5 min each). The average particle size of the powder ranged from 0.076 to 0.167 mm. The solvent-treated samples were prepared by soaking the powder in analytical-grade toluene, methanol, and mixtures of the two for 1 day at room temperature (25°C). After filtration, parts of the samples were left to dry in the open air for 1 and 4 days before any measurements were made. The other part was exposed to γ-radiation at a dosage level of 15 Mrad from a ^{60}Co source, Noratom Control A-S Gamma A 3500 unit, 22.54 rad/s. A part of the untreated powder was also irradiated in the presence of the

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solvents. All treatments were carried out at a solvent/cotton ratio of 50 : 1. KBr discs were prepared from the treated samples, and the infrared spectra were recorded on Beckman IR 4220 spectrophotometer.

The samples prepared for the dielectric measurements were in the form of discs (12 mm diameter and 2 mm thick). These samples were prepared by pressing the powder under a constant pressure of 10 ton/cm^2 using a hydraulic pressing machine. An automatic precision bridge type RLCB from Rhode and Schwartz, (München, West Germany) was used for capacitance measurements from which the dielectric constant ϵ' was obtained and also for measuring the power factor tan δ at 1 kHz. The accuracy of the measurements was $\pm 0.1\%$ for capacitance and $\pm 5\%$ for tan δ. The measurements were carried out at room temperature (25°C).

RESULTS AND DISCUSSION

The infrared spectra were recorded of samples treated for 1 day with solvent and air-dried for 1 and 4 days. Examination of these spectra revealed that treatment of cotton fibers for 1 day with toluene, methanol, and mixtures of the two results in no significant changes in the spectral features of the cotton fibers. The degree of crystallinity of the treated samples was measured by using the Nelson ratio,[6] A_{1370}/A_{2900} cm^{-1}. The values obtained are given in Table I and indicate that all the treated samples had similar values, treatment with an 80/20 toluene/methanol mixture being marginally lower. Table I also indicates that solvent treatment causes no significant changes in the crystallinity of the samples left to dry for 4 days.

Table I also shows that the sample treated with 80/20 toluene/methanol mixture and left to dry for 1 day is characterized by the highest value of the dielectric constant (ϵ') and dielectric loss (ϵ''), where $\epsilon'' = \epsilon' \tan \delta$. Although the degrees of crystallinity of the samples left in air for 4 days are similar, values of ϵ' and ϵ'' decrease with increasing methanol concentration. This is

TABLE I
Effect of Absorbed Solvents on the Crystallinity Index, Dielectric Constant, and Dielectric Loss of Cotton Fibers

	After 1 day			After 4 days		
Solvents	Crystallinity index	ϵ'	ϵ''	Crystallinity index	ϵ'	ϵ''
Dry	0.537	3.35	0.084	0.532	—	—
Toluene	0.544	5.66	0.147	0.530	3.99	0.101
Toluene/ methanol, 4/1 v/v ratio	0.484	5.84	0.151	0.540	3.85	0.098
Toluene/ methanol, 1/1 v/v ratio	0.513	4.44	0.116	0.525	3.38	0.085
Toluene/ methanol, 1/4 v/v ratio	0.554	5.56	0.146	0.550	3.23	0.081
Methanol	0.500	4.52	0.117	0.500	3.14	0.079

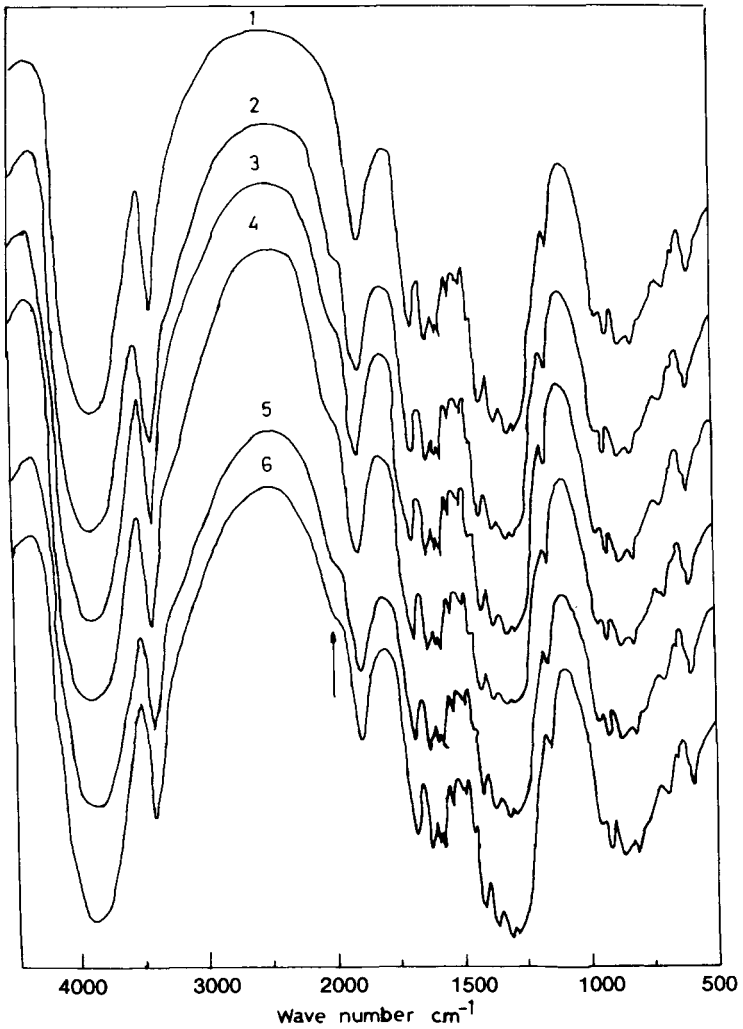

Fig. 1. IR spectra of solvent-treated cotton fibers, left to dry for 1 day and subsequently γ-irradiated: (1) dry cotton; (2) toluene; (3) toluene/methanol, 4/1 v/v ratio; (4) toluene/methanol, 1/1 v/v ratio; (5) toluene/methanol, 1/4 v/v ratio; (6) methanol.

contrary to expectation since ϵ' for toluene and methanol are 2.38 and 33.62, respectively. This may be attributed to the greater evaporation rate of methanol. It is also noted that ϵ' and ϵ'' are less after 4 days, probably due to further evaporation. (After 4 days, ϵ' and ϵ'' start to increase, which may be due to the absorption of water vapor from the atmosphere).

The solvent-treated samples, which has been left to dry for 1 day, were then exposed to γ-radiation at a dosage of 15 Mrad. Figure 1 shows that irradiation of cotton samples pretreated with solvents causes no changes in their spectral features apart from the appearance of a weak shoulder at about 1720 cm^{-1} due to the absorption of carbonyl and carboxylic groups. The IR spectra of the irradiated samples were measured 1 and 4 days after irradiation. The degree of crystallinity of these samples are shown in Table II. It is clear from

TABLE II
Effect of Absorbed Solvents and γ-Irradiation on Crystallinity Index, Dielectric Constant, and Dielectric Loss of Cotton Fibers

	After 1 day of irradiation			After 4 days of irradiation		
Solvents	Crystallinity index	ϵ'	ϵ''	Crystallinity index	ϵ'	ϵ''
Dry	0.530	4.28	0.101	0.53	—	—
Toluene	0.484	5.79	0.184	0.502	4.77	0.126
Toluene/ methanol, 4/1 v/v ratio	0.491	6.76	0.183	0.516	4.33	0.114
Toluene/ methanol, 1/1 v/v ratio	0.542	5.71	0.152	0.535	3.98	0.105
voluene/ methanol, 1/4 v/v ratio	0.527	5.78	0.154	0.515	3.93	0.103
Methanol	0.520	5.49	0.146	0.550	3.68	0.097

Table II that irradiation of solvent-treated cotton fibers results in slight decreases in the crystallinity of the samples solvent-treated with toluene and with the 80/20 toluene/methanol, although the degree of crystallinity of these two samples increases slightly when they are left in air for 4 days after irradiation.

Table II also shows that for any given solvent or mixture of solvents, the ϵ' and ϵ'' values for γ-radiation solvent-treated samples are always higher than those of the unirradiated samples. Also, both ϵ' and ϵ'' for the samples measured after 1 day from irradiation are higher than those for the samples measured after 4 days from irradiation. Furthermore, the sample treated with the 80/20 toluene/methanol mixture and measured after 1 day from irradiation is characterized by the highest values of ϵ' and ϵ''.

Samples of cotton fibers were also irradiated in the presence of toluene, methanol, and mixtures of the two. The IR spectra of these samples are illustrated in Figure 2. This figure shows that exposure of cotton fibers to 15 Mrad γ-doses in the presence of toluene results in the formation of a well-defined peak at 1720 cm^{-1} associated with the absorption of C=O groups. The intensity of this C=O band decreases as the percentage of methanol in the mixture increases and finally entirely disappears from the spectrum of the sample irradiated in methanol. This means that while exposure of cotton fibers in the presence of toluene helps the oxidation of cotton, exposure in the presence of methanol reduces the oxidation effect of γ-radiation. Table III indicates also that irradiation of cotton fibers in presence of toluene produces a slight decrease in its crystallinity.

Comparison of ϵ' and ϵ'' in Tables II and III reveals that the samples irradiated in presence of solvents assume higher values of ϵ' and ϵ'' than the solvent-treated samples irradiated in air. The sample irradiated in the presence, of the 80/20 toluene/methanol mixture and measured after 1 day from irradiation is characterized by the highest values of ϵ' and ϵ''. Tables I and II

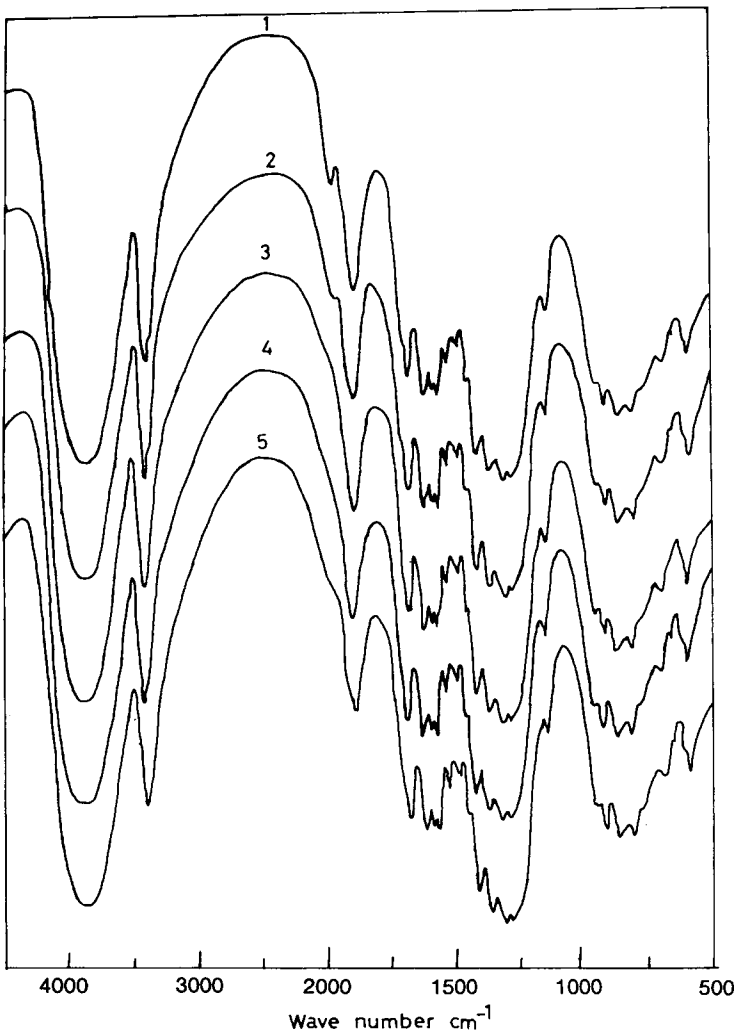

Fig. 2. IR spectra of cotton fibers γ-irradiated in the presence of solvents: (1) toluene; (2) toluene/methanol, 4/1 v/v ratio; (3) toluene/methanol, 1/1 v/v ratio; (4) toluene/methanol, 1/4 v/v ratio; (5) methanol.

also show that ϵ' and ϵ'' for the samples measured after 4 days of drying decrease as the percentage of toluene in the mixture decreases.

The penetration of solvents into various textiles has been discussed by several research workers,[1-3] who have stated that some solvents can be introduced into cellulose by the solvent exchange method. They found that treatment of textiles with solvent mixtures containing both a readily sorbed and a not readily sorbed solvent leads to penetration of the fibers by both solvents. Holt[1] found that a 4-h treatment of dry cotton fabric with anhydrous perchloroethylene (PER) at 20°C gives less than 0.05% uptake of PER, but when 10% methanol is added, the PER uptake increases to 6%.

Since ϵ' of toluene is smaller than that of cotton, its contribution to the values measured in this study is not clear. Addition of toluene to methanol

TABLE III
Crystallinity Index, Dielectric Constant, and Dielectric Loss of Cotton Fibers γ-Irradiated in the Presence of Solvents

Solvents	After 1 day of irradiation			After 4 days of irradiation		
	Crystallinity index	ϵ'	ϵ''	Crystallinity index	ϵ'	ϵ''
Toluene	0.480	9.15	0.268	0.480	5.57	0.743
Toluene/ methanol, 4/1 v/v ratio	0.569	11.42	0.348	0.50	5.18	0.137
Toluene/ methanol, 1/1 v/v ratio	0.50	7.56	0.218	0.544	4.85	0.123
Toluene/ methanol, 1/4 v/v ratio	0.536	5.73	0.162	0.52	4.75	0.119
Methanol	0.538	7.53	0.216	0.50	4.68	0.117

increases ϵ' and ϵ'' of 1-day solvent-treated cotton. This result leads to the conclusion that the addition of toluene to methanol increases the rate of uptake of methanol by cotton. This result correlates well with the results reported in the literature. Moreover, irradiation of cotton fibers in the presence of toluene assists the oxidation of cotton fibers while irradiation in the presence of methanol reduces the oxidation effects of γ-radiation.

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