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EFFECTS OF TEMPERING TIME AND TEMPERING TEMPERATURE OF CAUSTIC MERCERIZATION ON THE SPECTRAL AND ELECTRICAL PROPERTIES OF EGYPTIAN COTTON FIBRES

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Several samples of Egyptian cotton fabric strips were subjected to various caustic mercerization conditions, varying in temperature and time. On these samples extensive measurements were undertaken, comprising X-ray diffraction, IR absorption spectra and the temperature-dependence of DC-electric conductivity.

The phase constitution, % crystallinity, IR band assignment, activation energies for conduction and energy gap were evaluated as a function of caustic mercerization. The results obtained were correlated and discussed in detail, leading to valuable information of use in the cotton fabric strip industries.

Cellulose is a polycrystalline aggregate consisting of small crystalline regions separated by amorphous regions. It is a polymorphic polymer which in its native state crystallizes in a lattice called cellulose I. The mercerizing of cotton fabrics with aqueous solutions of sodium hydroxide alters the molecular arrangement of native cellulose (cellulose I) with a decrease in the overall crystallinity. Changes in crystallinity and lattice type of cottons after both treatments have been pointed out by many workers.

A previous publication [1] described changes in the fine structure and mechanical properties of cotton fibres after mercerization in sodium hydroxide or liquid ammonia. Fibres at three levels of maturity were treated slack and under tension (small bundles). Both reagents changed the morphology and fine structure, with only slight differences between caustic and ammonia mercerizations. A comparative study of "ammonia and conventional mercerizations" as pretreatments for cotton fabric subsequently crosslinked with butadienediepoxide (BDO) was reported by Jung et al. [2]. BDO was selected as the finishing reagent because it can

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be effectively applied in basic media and the other bonds formed between it and cellulose are stable to further treatment.

Abou-Sekkina et al. [3] studied some of the physicochemical properties of caustic mercerized cotton fibres. They concluded that electric conductivity measurements could be used as a precise and rapid tool to follow small changes in crystal structure resulting from chemical treatment.

Accordingly, the major goal of the present investigation was to evaluate the effects of the soaking time, and the temperature of caustic mercerization on Egyptian cotton fabric fibres.

Experimental

Materials

Bleached Egyptian cotton cloth samples (130 gm/m^2) were utilized.

Chemical Treatment

a) Mercerization

The cloth was immersed for fixed periods at 20°, 60° and 90° in 20% (w/w) aqueous NaOH containing a few drops of Mercerol. Caustic soda was then removed by rinsing in hot water (70°), and neutralizing in very dilute acetic acid solution. The cloth samples were finally washed again with water and air-dried for 24 hr.

B) Cu–K$_\alpha$ X-ray diffraction analysis

Cu–K$_\alpha$ X-ray diffraction patterns were obtained by the method of Segal et al. [4], using a Textur–Goniometer X-ray diffractometer, Model PW 1078/25 (Philips, Eindhoven, The Netherlands).

C) IR spectra

The room-temperature infrared absorption spectra of cotton fabrics were obtained with a Perkin–Elmer double beam spectrophotometer by the KBr disk method of O'Connor et al. [5].

D) Temperature-dependence of DC-electric conductivity

This was performed using the two-probe technique under vacuum with a 616 Keithley Electrometer (U.S.A.). The samples were in the form of cotton fibre bundles. The circuit and mode of measurements used were very similar to those previously described [6]. Measurements were made at both room and elevated temperatures up to $\simeq$430 K and the reading were taken twice after 10 min for each temperature equilibration; all of the circuit components were well shielded with copper.

In all of the above measurements, the readings were taken more than once and in each case reliable data were carefully obtained.

Results and discussion

Since the commercial caustic mercerization processes are generally applied to cotton products, it is essential to know the morphology and fine structure of cotton fabric strips as a function of the temperature and the time of caustic mercerization. It is only natural that applications of the X-ray diffractometer would be found in the field of cellulose chemistry to investigate the nature of the changes brought about by caustic mercerization.

Figure 1, patterns A–E, show the Cu–K_α X-ray diffraction spectra of the untreated control (pattern A) and various caustic mercerized cotton fibres (patterns B–D).

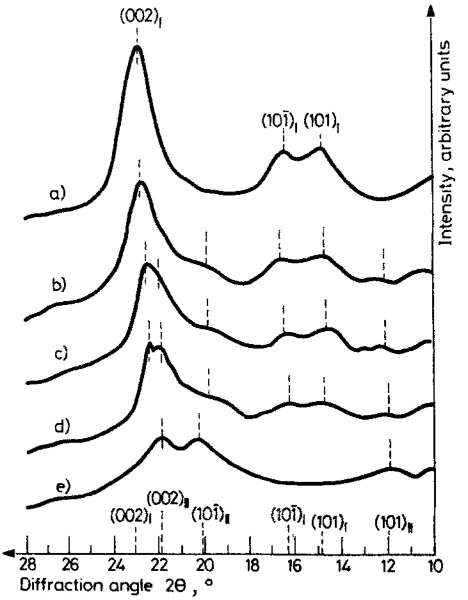

Fig. 1 X-ray patterns. A schematical representation for the room temperature X-ray diffraction patterns: A) untreated cotton fibres, B) NaOH-treated cotton (90 °C, 2 min), C) NaOH-treated cotton (60 °C, 5 min), D) NaOH-treated cotton (20 °C, 10 min), E) NaOH-treated cotton (0 °C, 60 min)

Since the coherent scattering of X-rays is a property of the crystalline portion of the cellulose, a reduction of the amount of crystalline cellulose should reasonably be expected to affect the characteristic X-ray pattern. Such an effect has been reported by Hess et al. [7, 8] and by Hermans et al. [9, 10]. Thus, the diffractometer tracings are particularly useful for several reasons.

a) Marked decreases in the intensities of the crystalline interferences which originate in the crystalline regions are readily discernible if there are reductions in the degree of crystallizing reagents; and

b) this information can be obtained in fraction of the time required to get crystallinity information by chemical or other physical means. The recorded data lend themselves to quantitative measurements; methods of handling such measurements to obtain estimates of the degree of crystallinity in cellulosic fibres are at present under study. Therefore, in a sample of cotton undergoing mercerization, the transformation of cellulose I to cellulose II (see Fig. 1) can easily be followed by means of the diffractometer. In fact, the advantages of the instrument are such as to recommend it strongly for cotton process control. Thus, the use of X-rays provides a true index of mercerization, since the positions and intensities of the interferences arise directly from the degree of change brought about in the cellulose crystal lattice by the chemical treatment. Anything that interferes with or inhibits this change is reflected in the diffractometer tracings. The diffractometer tracings of mercerized yarns in Fig. 1 illustrate how the tracings are affected by such factors. They indicate partial or complete conversion of the lattice, depending upon the conditions of treatment. A factor important in the structure or performance of textile fibres is the crystallite orientation. Thus, in the present investigation, with the rotating platform carrying the sample bundle set perpendicular to the primary X-ray beam, the diffractometer is operated in the transmission technique. A standard sized bundle of parallelized fibres is held in combs under tension and the Geiger counter of the X-ray spectrophotometer is centred on the 002 interference. As the sample rotates in the X-ray beam, the radiation diffracted by the 002 planes of the sample sweeps across the window of the Geiger counter.

As can be seen clearly from Fig. 1, the diffraction peaks at 20° values of 15.0, 16.4 and 22.8 are highly characteristics of the cellulose I lattice [11, 12].

On the other hand, the X-ray diffractograms of each sample pretreated with 20% NaOH at different dipping temperatures (20°, 60° and 90°) show peaks at $2\Theta°$ values of $\simeq 12.40$ and $\simeq 21.6$, which are characteristic of the cellulose II lattice [11, 12]. Thus, in accordance with Jung et al. [13], the conversion to cellulose II was only partial after the conventional mercerization of fabrics, whereas cellulose II exists in major amount. A complete conversion was attained on treatment with sodium hydroxide at 0° for 60 min. (Fig. 1, pattern E). Measurements of the apparent crystallite size normal to the *hkl* planed by XRD were (as usual) based upon the half-intensity breadth of the crystallographic reflection. The half-intensity breadths β were then substituted in the Scherrèr equation:

$$D_{hko} = 0.9\ \lambda/\beta \cos\theta$$

After the substraction of the amorphous background intensity from the total over the entire angular spread of the equatorial reflections [14], the average sizes of the crystallites D_{hkl} perpendicular to the reflection planes *hkl* were calculated for both these reflections. The results obtained are collected in Table 1. It was found that increase of the tempering temperature of caustic mercerization from 20° up to 90° drastically increased the size of the crystallites perpendicular to the principal equatorial reflection 002 (see Table 1). This could be explained by the fact that cellulose II has a higher degree of crystallinity than cellulose I and the high temperature of caustic mercerization favours further conversion of cellulose I to

Table 1 Characterization of representative samples of Egyptian cotton fabrik strips subjected to various treatments and their Cu–K_α–X-ray diffraction data

Sample	Phases	θ°	2θ°	I/I_0	Apparent crystalline sizes *l* to phase *hkl* *D* (Å)	Crystallite dimension *l* to planes *hkl D* (Å)	*hkl*
Untreated control	Cell. I	7.5	15.0	2.9	40	5.91	101 (I)
		8.2	16.4	2.8	42	5.40	$10\bar{1}$ (I)
		11.4	22.8	9.3	51	3.90	002 (I)
NaOH (20 °C)	Cell. I	6.0	12.0	1.3	62	7.37	101 (II)
treated cotton	+	7.55	15.1	1.92	50	5.87	101 (I)
(10 min.)	Cell. II	8.35	16.7	2.0	68	5.31	$10\bar{1}$ (I)
		10.15	20.30	2.90	51	4.37	101 (II)
		11.35	22.70	5.20	39	3.92	002 (I)
NaOH (60 °C)	Cell. I	6.0	12.0	1.2	—	7.37	101 (II)
treated cotton	+	7.6	15.2	2.0	49	5.83	101 (I)
(5 min.)	Cell. II	8.4	16.8	2.1	58	5.28	$10\bar{1}$ (I)
		11.4	22.8	5.5	45	3.9	002 (I)
NaOH (90 °C)	Cell. I	6.25	12.5	1.1	—	7.1	101 (II)
treated cotton	+	7.6	15.2	2.4	47	5.83	101 (I)
(2 min.)	Cell. II	8.45	16.9	2.36	50	5.25	$10\bar{1}$ (I)
		11.5	23.0	6.8	46	3.87	002 (I)
NaOH (0 °C)		5.95	11.9	1.38	50	7.44	101 (II)
treated cotton	Cell. II	10.05	20.1	3.44	49	4.42	$10\bar{1}$ (II)
(60 min.)		10.8	21.6	3.70	35	4.41	002 (II)

cellulose II. Another important aspect is the influence of the dipping temperature of caustic mercerization. A reduction in crystallite size perpendicular to the reflection planes 101 and 10$\bar{1}$ was noted (samples 2 and 4) when the dipping temperature was increased from 20° up to 90°. A possible reason for this behaviour is that the higher the dipping temperature, the greater the reduction of the extension of the lateral order to a few cellulosic chains.

The main value of the application of infrared spectra to cellulose is in showing spectral changes due to decrystallization and those due to changes in lattice type, resulting from treatment with 20% sodium hydroxide under various conditions, namely at fixed temperatures for fixed periods.

Spectral changes brought about by different sodium hydroxide treatments are shown in Fig. 2. Comparison of the spectra of the samples treated with sodium hydroxide at 20, 60 and 90° showed a decreased absorption band at 1430 cm^{-1} (assigned to CH_2 deformation), while the band at 893 cm^{-1} (assigned to C_1 group vibration) increased in intensity. The intensities of these bands decreased and increased, respectively, with decrease in the treatment temperature and with increase in the time of sodium hydroxide treatment. The intensity of the band at 2900 cm^{-1} (C—H stretching) appeared to be unaffected by the changes brought about in the lattice type by sodium hydroxide treatment. This band is strong and sharp in samples containing cellulose I and II and samples containing a mixed lattice. The band at 1116–1111 cm^{-1}, assigned to the C—O stretching, which is strong in the case of the untreated cotton, is reduced to a certain extent by sodium hydroxide treatment. The presence of the 1111 cm^{-1} band in mercerized cottons at 20, 60 and 90° as only a shoulder, is attributable to the development of strong,

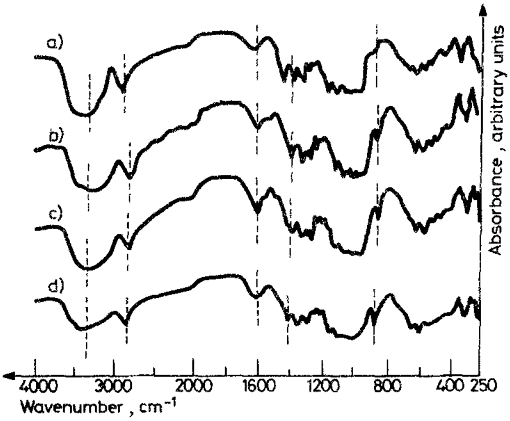

Fig. 2 The room temperature KBr infrared absorption spectra: A) untreated cotton fibres, B) NaOH-treated cotton (90 °C, 2 min), C) NaOH-treated cotton (60 °C, 5 min), D) NaOH-treated cotton (20 °C, 10 min), E) NaOH-treated cotton (0 °C, 60 min)

poorly-resolved, adjacent bands, as a result of the destruction of the cellulose I crystal lattice. The strong and sharp band at 1165 cm^{-1}, assigned to the antisymmetrical C—O—C bridge, remained constant in NaOH-treated cottons at 60 and 90°, but was reduced to a certain extent in NaOH-treated cottons at 20°. Comparison of the spectra of the samples containing the lattice types I and II in the region 1200–1400 cm^{-1} showed some strong similarities and some marked differences. The spectrum of cellulose I differs from those of cellulose II and mixed lattice I + II in the intensities of the bands at about 1370, 1325, 1280 and 1230 cm^{-1}. These observations are in agreement with those reported by Nelson and O'Connor [15], who studied the spectra of highly crystalline celluloses and amorphous celluloses.

They showed that the regions from 850–1500 cm^{-1} contained several bands which were affected essentially by the amorphous content of cellulose samples, rather than by the crystal lattice type.

In this respect, measurements of the DC-electric conductivity were carried out on bundles of cotton fabric strips by current flow through the longitudinal axis of the fibres ($\simeq 3$ cm long).

Values of DC-electric conductivity (σ, $\Omega^{-1}\,\mathrm{cm}^{-1}$) were estimated at room and elevated temperatures up to $\simeq 500$ K under vacuum (10^{-2} mm Hg). Thus, the variation of log σ of various caustic mercerized cotton fibres with the reciprocal of the absolute temperature $\left(\frac{1}{T}10^{-3}\,\mathrm{deg}^{-1}\right)$ is clearly shown in Figs 3–5. Table 3 includes obtained values of the conductivity parameters, together with the related data taken before and after mercerization of the cotton fibres. Since there is a positive temperature coefficient of electric conductivity $\left(\frac{\mathrm{d}\sigma}{\mathrm{d}t} = +Ve\right)$ for each curve (see Figs 3–5), all the investigated specimens have a semiconducting character in the investigated temperature region.

The DC-conductivity σ varies exponentially with the absolute temperature according to the well-known relation [19].

$$\sigma = \sigma_0 \bar{e}^{\Delta E}/2KT \qquad (1)$$

where σ is the electric conductivity at a given temperature T, σ_0 is the initial conductivity (or the preexponential factor), ΔE is the activation energy for conduction and K is the Boltzmann constant. The relatively slight response of σ with temperature variation involves ΔE values which can be interpreted by a simple single band model in most cases. From Figs 3–5 and Eq. (1), the ΔE values are calculated (Table 3) and assumed to correspond to the thermal activation energies for electric conduction. Figures 3–5 (all curves) comprise two activation energies (for each curve): ΔE_0 for the high-temperature region or for the intrinsic

Table 2 Characterization and band assignments of the characteristic IR absorption bands for representative samples undergoing different NaOH treatments

Sample	λ_{max}	Band assignments	Refs
Untreated control	3400s	OH stretching	
	2900m	CH stretching	
	1640m	HOH bending	
	1440m	HCH deformation	
	1380m	CH bending	
	1325m	CH rocking in ring	16, 17
	1285w		
	1250w		
	1235w		
	1205w	CO stretching	
	1165s	ring frequency	18
	1116s	COC	
	893	C_1 group vibration	
NaOH (20 °C) treated cotton (5 min.)	3420s	OH stretching	
	2900m	CH stretching	
	1640	HOH bending	16, 17
	1430	HCH deformation	
	1370	CH bending	
	1320	CH rocking in ring	
	1280		
	1260		
	1225	CO stretching	18
	1200		
	1160	ring frequency	
	1120	COC	
	893	C_7 group vibration	
NaOH (20 °C) treated cotton (10 min.)	3435	OH stretching	
	2900	CH stretching	
	1640	HOH	
	1430	HCH deformation	16, 17
	1375	CH bending	
	1320	CH rocking in ring	18
	1280		
	1260		
	1230	CO stretching	
	1200		
	1165	ring frequency	
	1120	COC	
	893	C_7 group vibration	

(**Table 2** continue)

Sample	λ_{max}	Band assignments	Refs
NaOH (60 °C)	3445	OH stretching	
(5 min.)	2895	CH stretching	16, 17
	1640	HOH bending	
	1430	HCH deformation	18
	1370	CH bending	
	1315	CH rocking in ring	
	1280		
	1265		
	1235	CO stretching	
	1200		
	1165	ring frequency	
	1116	COC	
	893	C_1 group vibration	
NaOH (60 °C)	3450	CH stretching	
(10 min.)	2900		
	1640	HOH bending	
	1430	HCH deformation	
	1370	CH bending	
	1310		
	1280	CH rocking in ring	16, 17
	1260		
	1225	CO stretching	18
	1200		
	1165	ring frequency	
	1120		
	893	C_1 group vibration	
NaOH (90 °C)	3425	OH streching	16, 17
treated cotton	2900	CH stretching	
(2 min.)	1630	HOH bending	
	1430	HCH deformation	18
	1370	CH bending	
	1315	CH rocking in ring	
	1280		
	1260		
	1230	OH stretching	
	1200		
	1160	ring frequency	
	1111	COC	
	893	C_1 group vibration	

(**Table 2** continue)

Sample	λ_{max}	Band assignments	Refs.
NaOH (90 °C) treated cotton (5 min.)	3430	Oh stretching	16, 17
	2900	CH stretching	
	1630	HOH bending	18
	1430	CH_2 deformation	
	1375	CH bending	
	1320		
	1280	CH rocking in ring	
	1265		
	1230		
	1200	CO stretching	
	1165	ring frequency	
	1111	COC	
	893	C_1 group vibration	

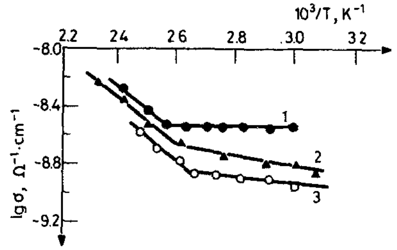

Fig. 3 A diagram showing the variation of DC-electrical conductivity Log σ °K as a function of absolute temperature ($1000/T°K^{-1}$) for samples 1, 2 and 3

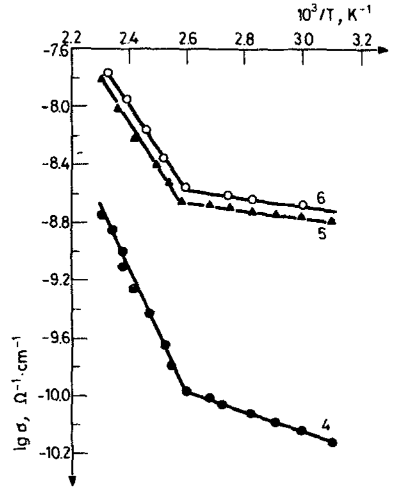

Fig. 4 The variation of log electrical conductivity as a function of temperature for samples No. 4, 5 and 6

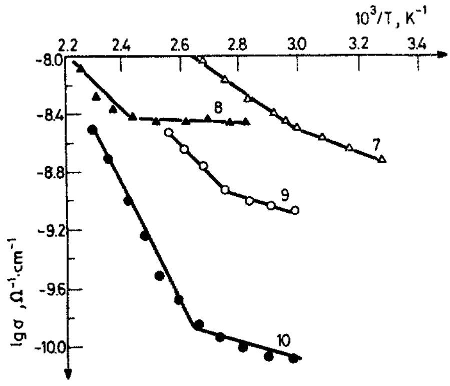

Fig. 5 The variation of log electrical conductivity as a function of temperature for samples No. 7, 8, 9 and 10

Table 3 Values of the obtained electrical conductivity (Log σ_{100} Ω^{-1} cm^{-1}, by extrapolation from the curves of Figs 3–5) and activation energy (ΔE_0) for conduction together with other data taken for cotton, fibres before and after mercerizations

Sample No.	Sample treatment	Crystalline phase	Crystallinity	Log σ_{100} (Ω^{-1}cm^{-1})	Activation energy, eV	Energy gap *Eg*, eV
1	20% NaOH, 5 min (20 °C)	Cell. I+II	67	−8.54	0.8	1.6
2	20% NaOH treated (at 0 °C)	Cell. II	60	−8.68	0.65	1.3
3	20% NaOH treated 10 min (at 20 °C)		64	−8.84	0.7	1.4
4	20% NaOH treated 5 min (60 °C)		—	−10.0	1.7	3.4
5	20% NaOH, 10 min (60 °C)	Cell. I+II	64	−8.60	1.1	2.2
7	20% NaOH treated 2 min (90°C)		—	−8.30	0.57	1.14
8	20% NaOH, 5 min (90 °C)	Cell. I+II	68	−8.44	0.66	1.32
9	20% NaOH, 15 min (20 °C)	Cell. I+II	61	−8.76	0.70	1.40
10	20% NaOH, 1 min (90 °C)		—	−9.88	1.4	2.80

conduction mechanism, and ΔE for the low-temperature region or for the extrinsic conduction mechanism for the specimens investigated. In our view, the existence of these extrinsic and intrinsic activation energies could plausibly be correlated with the presence of mixed crystalline cellulosic phases (cellulose I or cellulose II) and/or an amorphous cellulosic phase.

Consequently, the extrinsic activation energy corresponds to the minor cellulosic phase, which may act as an impurity in the major phase. The latter corresponds to the intrinsic activation energy indicating the deep impurity donor levels. In accordance with the earlier publication of Abou-Sekkina and Abou El-Enein [20], $2\Delta E$ is thus the energy gap (Eg) in eV for the investigated cotton fabric strips.

Figure 6 shows the variation of (A) electric conductivity at 100° ($\log \sigma_{100}$ °C) and (B) energy gap (Eg, eV) as a function of the tempering temperature of caustic mercerization of cotton fabric strips. From this plot (Fig. 6) it can easily be seen that the electric conductivity rises with increasing tempering temperature (curve A), reached a maximum and then becomes lower again. The behaviour of Eg with tempering temperature is the reciprocal of that of $\log \sigma_{100}$ (curve B). This behaviour is probably correlated with the change in crystalline phase and degree of crystallinity of cellulosic fibres as a function of tempering temperature.

Figure 7 presents the variation of (A) $\log \sigma_{100}$ (Ω^{-1} cm^{-1}) and (B) energy gap (Eg; eV) as a function of the tempering time of caustic mercerization of cotton fabric strips.

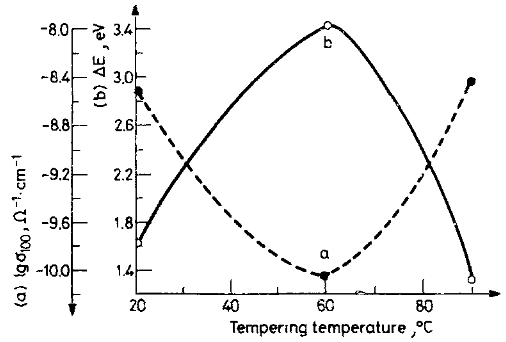

Fig. 6 An illustration showings the variation of: A) Log σ_{100} and B) Energy gap (Eg, eV) as a function of the tempering temperature of caustic mercerization of Egyptian cotton fabric strips

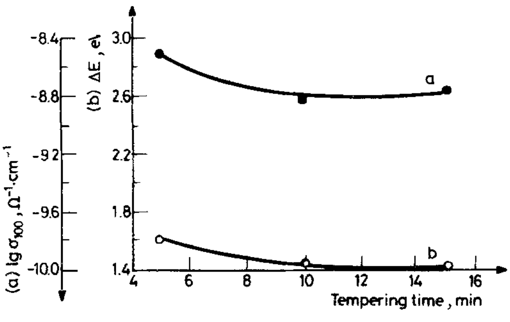

Fig. 7 A plot showings the variation of: A) Log σ_{100} and B) Energy gap (Eg, eV) as a function of tempering time of caustic mercerization of Egyptian cotton fabric strips

From Fig. 7, it is obvious that both log σ_{100} and Eg show the same trend: they decrease with increase of the tempering time till constancy or saturation is reached at about 10 minutes. Thus, 10 minutes represent the optimum tempering time for the caustic mercerization of Egyptian cotton fabric strips. Of course, this is useful information for the cotton fibre strip industries.

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Zusammenfassung — Verschiedene Proben von ägyptischen Baumwollstreifen wurden unter verschiedenen Bedingungen (Temperatur, Zeit) einer Laugung unterworfen. Die gelaugten Proben wurden eingehend durch Röntgendiffraktometrie, IR-Absorptionsspektroskopie und Messungen der Temperaturabhängigkeit der elektrischen Gleichstromleitfähigkeit untersucht. Phasenstruktur, Kristallinität, Zuordnung der Banden, Aktivierungsenergien für die Leitfähigkeit und verbotene Bänder wurden in Abhängigkeit von der Laugung ermittelt. Die erhaltenen Ergebnisse wurden miteinander verglichen und in Einzelheiten diskutiert, wobei für die Baumwollverarbeitungsindustrie wertvolle und empfehlenswerte Informationen erhalten wurden.

Резюме — Несколько образцов египетского хлопкового материала фабричного изготовления были подвергнуты мерсеризации при различных температурах и времени. Для мерсеризованных образцов хлопка проведен рентгеноструктурный анализ, измерены ИК спектры и температурная зависимость постояннотоковой составляющей электропроводности. В зависимости от условий мерсеризации проведена оценка фазового состава, процента кристалличности, отнесения ИК полос, энергии активации проводимости и энергетической щели. Приведено подробное обсуждение полученных результатов, а полученные на их основе корреляции дали обширную информацию полезную для текстильного производства.