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Optical Studies of Saudi Cotton Fibre Structure Using Scanning Electron Microscopy

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Abstract. Application of Becke-line, double-beam and multiple-beam interferometric methods were used to measure the refractive indices and birefringence of Saudi (Gizan) cotton fibres. The scanning electron microscopy was used to estimate the cross sectional parameters and to show deformations and fine fibrillar structures which cannot be resolved with optical microscopes on the fibre surface. The resulting data were utilized to calculate the polarizability per unit volume, Cauchy's dispersion constants, dispersive power, isotropic refractive index, the spiral angle, the optical orientation angle and specific volume of the fibre material. The results were found in good agreement with the previously published values of different variaties of cotton fibres. Micro-interferograms are given for illustrations.

For optically anisotropic fibres the refractive index and the double refraction are parameters characterizing the structure of the material.

Different techniques, Becke-line, double-beam and multiple-beam have been used to determine the indices of refraction and birefringence of both natural and man-made fibres (Faust 1954, Betrabet *et al.* 1963, Pluta 1972, Barakat 1971, Hindeleh 1978, Hamza 1980, Zurek & Zakrzewski 1983 and Fouda *et al.* 1986). For fibres having perfectly regular transverse cross-sections, theories have been presented [Wilkes 1985, El-Nicklawy *et al.* 1983, Fouda *et al.* 1987, El-Nicklawy & Fouda 1980 (a,b) and Fouda & El-Nicklawy 1981] for both single and multiple-index fibres, in order to obtain useful analytical formulae. For fibres having irregular cross-sections, however, these formulae cannot be directly applied.

Clearly, any method or formula derived for calculating refractive indices and other optical parameters has its own merits, and the preference for one method or formula over another is likely to be determined by the case of application.

In spite of the tremendous growth of the synthetic fibre industry in recent years, cotton still holds 71% of the world's fibre production market.

Since many of the structural features of cotton fibres are intimately related to their optical properties, the investigation was extended to the study of birefringence and fibre reversals.

Cotton fibre, with its oriented fibrillar structure, is known to be strongly birefringent. It has a higher refractive index for light vibrating parallel ($n^{\|}$) and a lower refractive index for light vibrating perpendicular ($n^{\perp}$) to the fibre axis where the difference ($n^{\|} - n^{\perp}$) gives the birefringence ($\triangle n$). In the study of the birefringent properties of cotton fibres, the most significant contribution has been made by Meredith (1946 and 1963). He has demonstrated that the specific strength and initial Young's modulus of cotton fibres are highly correlated with birefringence. Also he has observed that long, fine cottons show greater birefringence and lower spiral angle than short, coarse cottons and proposed that the original unconvoluted fibres of all varieties have the same spiral angle.

The present work provides the optical characteristics of Saudi (Gizan) cotton fibres and proves the suitability of the Becke-line method, multiple-beam and double-beam interferometry for measuring the refractive indices for light vibrating parallel and perpendicular to the fibre axis and the birefringence of fibres with irregular cross-sections, such as Saudi cotton. The resulting data of refractive indices in both parallel and perpendicular directions were utilized to calculate the other optical parameters related to the refractive indices.

Experimental

In this work, four different techniques were used to estimate the optical and geometrical parameters of Saudi (Gizan) cotton fibres.

The scanning electron microscope was used to study the longitudinal and cross-sectional shapes of the cotton samples. The fibres were attached to metal stubs with suitable adhering substance and then coated under vacuum with a conducting layer of gold about 200 Å thick.

The optical set-up for producing multiple-beam Fizeau fringes in transmission, double-beam interferometry (by Pluta microscope) and the Beck-line methods were discussed previously in detail (Faust 1954, Pluta 1972 and El-Nicklawy and Fouda 1980).

1. For multiple-beam Fizeau fringes, considering the area enclosed under the fringe shift, we used the following formula (Hamza *et al.* 1985) to overcome

any irregularity in the fibre cross-section:

$$n_a^{\parallel} = n_L + (F^{\parallel}/h)(\lambda/2A) \qquad \text{...... (1)}$$

with an analogous formula for $n_a^{\perp}$, where;

$n_a^{\parallel}$ and $n_a^{\perp}$ are the mean refractive indices for light vibrating parallel and perpendicular to the fibre axis, respectively. $F^{\parallel}$ (or $F^{\perp}$) is the total area enclosed under the fringe shift when using a monochromatic light of wavelength λ vibrating parallel (or perpendicular) to the fibre axis; h is the interfringe spacing, A is the fibre cross-section area and n_L is the refractive index of the used immersion liquid.

2. For the determination of n_a and $\Delta n_a = n_a^{\parallel} - n_a^{\perp}$, by the double beam interferometric technique, the Pluta polarizing interference microscope was used. The previous formula (1) was applied but λ/A is used instead of $\lambda/2A$ according to Hamza (1980), then

$$\Delta n_a = (F^{\parallel} - F^{\perp})\lambda/hA \qquad \text{...... (2)}$$

where $(F^{\parallel} - F^{\perp})$ is the total area enclosed under the fringe shift obtained in the non-duplicated image.

3. For the determination of the polarizabilities per unit volume, Cauchy's dispersion constants, dispersive power, isotropic refractive index, the spiral angle, the optical orientation angle and specific volume of the cotton fibre material. The resultant data of the refractive indices for light vibrating in the directions parallel and perpendicular to the fibre axis and for different wavelengths, were employed in calculating the above optical parameters according to the following relations (Samuels 1974, Ward 1975 and Ernest 1979):

$$(n^2 - 1)/(n^2 + 2) = \frac{4}{3}\pi P \qquad \text{...... (3)}$$

where P is the polarizability per unit volume,

$$n = A + B/\lambda^2 \qquad \text{...... (4)}$$

where A & B are Cauchy's dispersion constants that characterize the dispersion activity of the material, then the dispersion power is:

$$(dn/d\lambda) = -2B/\lambda^3 \qquad \text{...... (5)}$$

The isotropic refractive index n_{iso} is:

$$n_{iso} = (n_a^{\parallel} + 2n_a^{\perp})/3 \qquad \ldots\ldots (6)$$

The spiral angle θ of cotton fibres (in degrees) was calculated from the relation (Betrabet *et al.* 1963)

$$\cos^2\theta = \frac{(n_{\parallel}^{x})(n_{\parallel} - n_{\perp}^{x})(n_{\parallel} + n_{\perp}^{x})}{2_{\parallel}^{2}\ (n_{\parallel}^{x} - n_{\perp}^{x})(n_{\parallel}^{x} + n_{\perp}^{x})} \qquad \ldots\ldots (7)$$

which is deduced from the geometry of the ellipsoid for the cellulose crystallite. $n_{\parallel}^{x}$ and $n_{\perp}^{x}$ represent the values obtained for ramie (1.595 & 1.529, respectively) the most perfectly oriented natural cellulosic fibres.

The optical orientation angle γ can be found using Hermans equation:

$$\triangle n/\triangle n_0 = 1 - \frac{3}{2}\sin^2\gamma \qquad \ldots\ldots (8)$$

where $\triangle n_0$ is the birefringence of perfectly oriented cellulose and $\triangle_n$ is the birefringence of the fibre under investigation (Hermans 1946). The value of $\triangle n_0$ has been previously determined to be 0.071 (Hebert *et al.* 1970).

Hermans (Hannes 1972) has emperically established a relation between the isotropic refractive index n_{iso} of cellulosic fibres and their specific volume v, namely:

$$(n_{iso} - 1)v = \text{constant} \qquad \ldots\ldots (9)$$

Experimental verification of the above relation shows that the constant of relation (9) lies within the range 0.475 & 0.477.

Results and Discussion

Cotton cannot be considered a uniform material even though a sufficient large number of fibres may have a characterizing behaviour.

Figure 1 shows the cross-sectional shape of the Saudi cotton fibres. The most striking character of cross-sections of cotton fibres is the variability of every dimensional feature. Some authors classified cotton fibres with respect to the cross-sectional shape into three categories, circular, elliptical and linear (Mauersberger 1947). Statistical determination of the cross section area (A) for Saudi

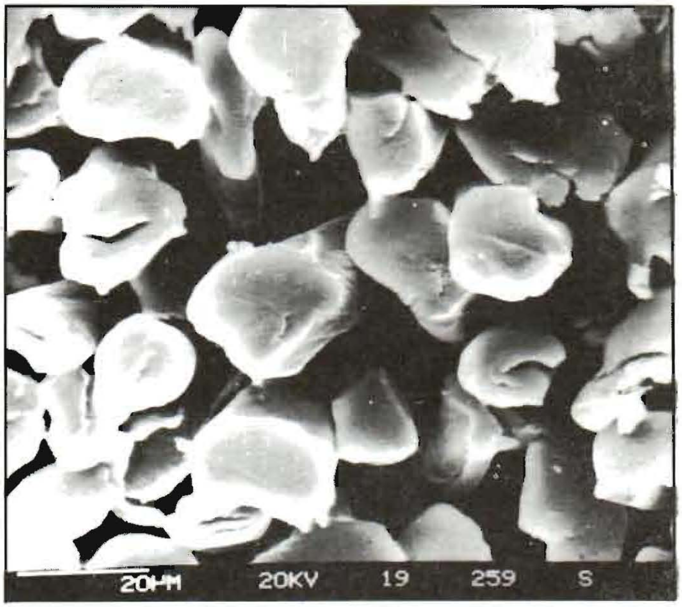

Fig. 1. Scanning electron microscopic view for cross-sections of Saudi (Gizan) cotton fibres.

(Gizan) cotton fibres shows that $A = 202.3\ \mu m^2$ as an average with tolerance of ±11.5%.

Figure 2 is a longitudinal view of Saudi cotton fibre. Except for the base and tip, the mature fibre is essentially the same throughout its length. The body of the fibre is characterized by its thickened wall and central canal. The spirals or convolutions are distinct twists about the longitudinal axis of the fibre, and the direction of the helix is frequently reversed.

Figure 3 is a view of a damaged sample of Saudi cotton fibre. Microscopical examination of fibres for effects of damage is of considerable value not only as a means of detecting the type of damage, but also as an aid in the improvement of processing to minimize damages. Practically, any sample of commercial lint contains some form of fibre damages (e.g. mechanical, chemical and biological tendering).

The mean refractive indices and birefringence:

In the present work, the mean refractive indices and birefringence of Saudi (Gizan) cotton fibres were determined by two different interferometric techniques:

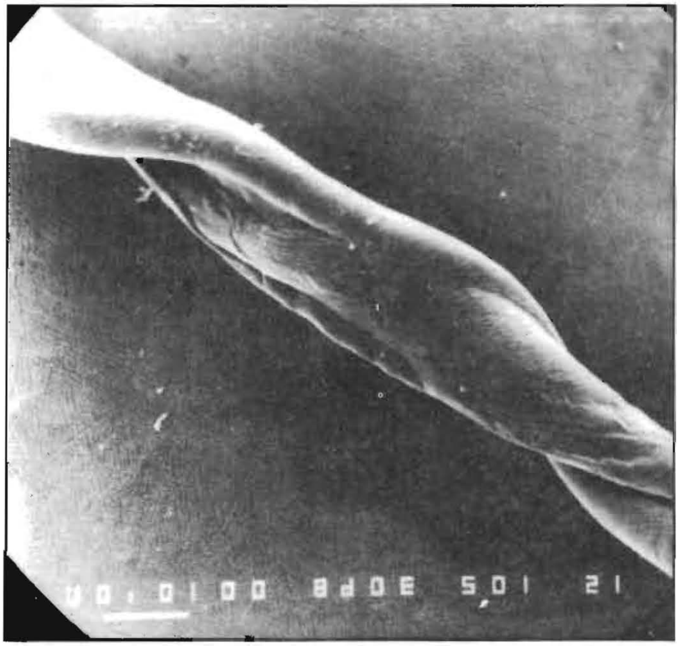

Fig. 2. Longitudinal view for Saudi cotton fibre shows convolutions along the fibre axis.

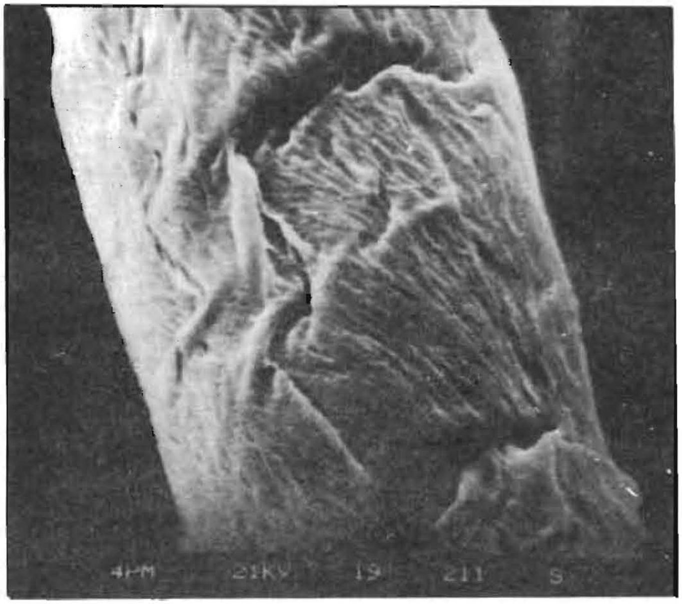

Fig. 3. Scanning electron microscopic view for surface of a Saudi cotton fibre shows damages on the fibre surface.

i) Two-beam interferometry:

Figure 4 is a duplicated image of Saudi cotton fibre produced by the Pluta (MPI-3) microscope when white light of average wavelength 550 nm was used.

The area under fringe shift was determined from a magnified print and the obtained values of $F^{\parallel}$ & $F^{\perp}$ were introduced into equation (1) (after adaptation for the two-beam interferometry), it was found that:

$$n_a^{\parallel} = 1.578 \quad \text{and} \quad n_a^{\perp} = 1.529 \quad \text{at } 21°\text{C}.$$

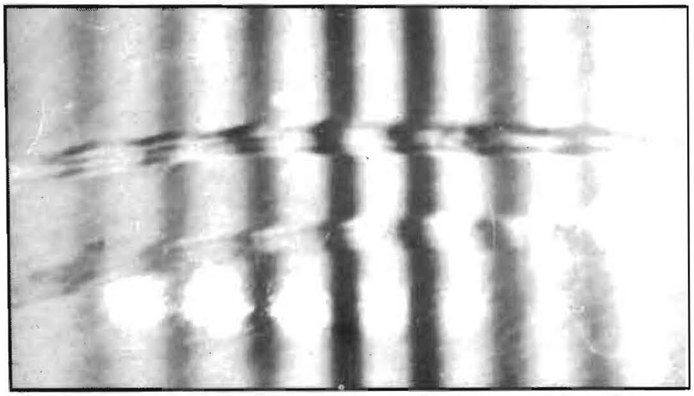

Fig. 4. Duplicated image produced by the Pluta interference microscope for Saudi cotton fibre, white light of average wavelength 550 nm is used.

Figures 5a & b are non-duplicated images of Saudi cotton fibre for white light ($\lambda = 550$ nm) and green light ($\lambda = 546$ nm), respectively. From the non-duplicated image and equation (2) it is found that the mean birefringence of Saudi cotton fibres is

$$\triangle n_a = 0.051 \text{ (for white light) and}$$

$$\triangle n_a = 0.052 \text{ (for green light)}$$

ii) Multiple-beam interferometry:

Figures 6a & b are microinterferograms for multiple-beam Fizeau fringes in transmission crossing a Saudi cotton fibre. Monochromatic plane polarized light vibrating (a) parallel and (b) perpendicular to the fibre axis was used. Introducing the area under the fringes shift shown in figures 6a & b into equation (1), one gets the mean refractive indices $n_a^{\parallel}$ and $n_a^{\perp}$. It is found that for Saudi cotton fibres:

$$n_a^{\parallel} = 1.5835 \quad \text{and} \quad n_a^{\perp} = 1.5319 \quad (\text{at } 21°\text{C and } \lambda = 546.1 \text{ nm}).$$

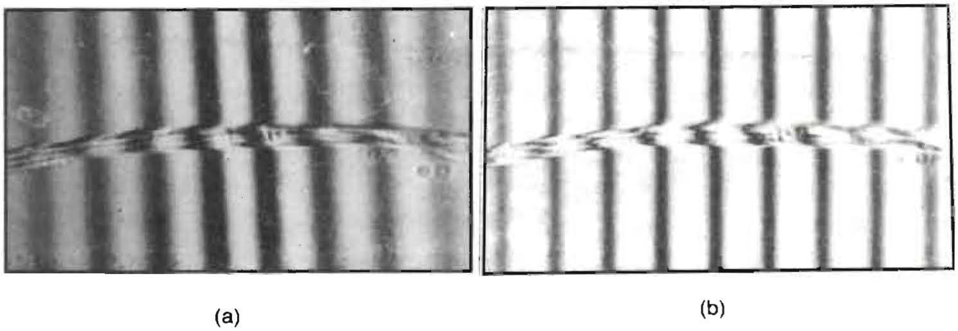

Fig. 5a&b. Non-duplicated image produced by the Pluta interference microscope for Saudi cotton fibre, (a) using white light and (b) using green light of wavelength 546 nm.

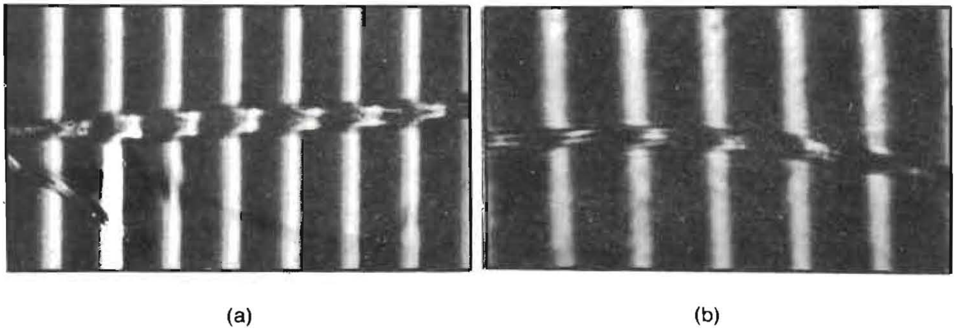

Fig. 6a&b. Microinterferograms for multiple-beam Fizeau fringes in transmission crossing Saudi cotton fibre. Monochromatic plane polarized light vibrating (a) parallel and (b) perpendicular to the fibre axis is used.

At the same conditions, the mean birefringence $\triangle n_a$ of Saudi cotton is

$$\triangle n_a = n_a^{\parallel} - n_a^{\perp} = 0.0516.$$

A comparison between values of $n_a^{\parallel}$, $n_a^{\perp}$ and ($\triangle n_a$) obtained from the two interferometric techniques show a fair agreement between them, where the small differences are within the range of accuracy differences between the two techniques used.

Refractive indices of the fibre skin layer:

By using the well known Becke-line method, it was possible to determine refractive indices $n_s^{\parallel}$ & $n_s^{\perp}$ for skin layer of Saudi cotton fibres for light vibrating parallel and perpendicular to the fibre axis, respectively. The wavelength dependence of $n_s^{\parallel}$, $n_s^{\perp}$ and $\triangle n_s$ was determined and given in table (1).

It is clear that Saudi (Gizan) cotton fibres have a remarkable dispersion.

Figures 7 and 8 show the linear behaviour of $n_s^{\parallel}$ and $n_s^{\perp}$, respectively, with $1/\lambda^2$. This linear behaviour ensures obedience of the dispersion of Saudi cotton fibres to the well known Cauchy's dispersion formula (4). From figures 7 & 8, Cauchy's constants for Saudi cotton fibres are found to be:

$$A_s^{\parallel} = 1.5604 \ , \ A_s^{\perp} = 1.5108$$

$$B_s^{\parallel} = 15 \times 10^3 \ \text{nm}^2 \ \& \ B_s^{\perp} = 14 \times 10^3 \ \text{nm}^2$$

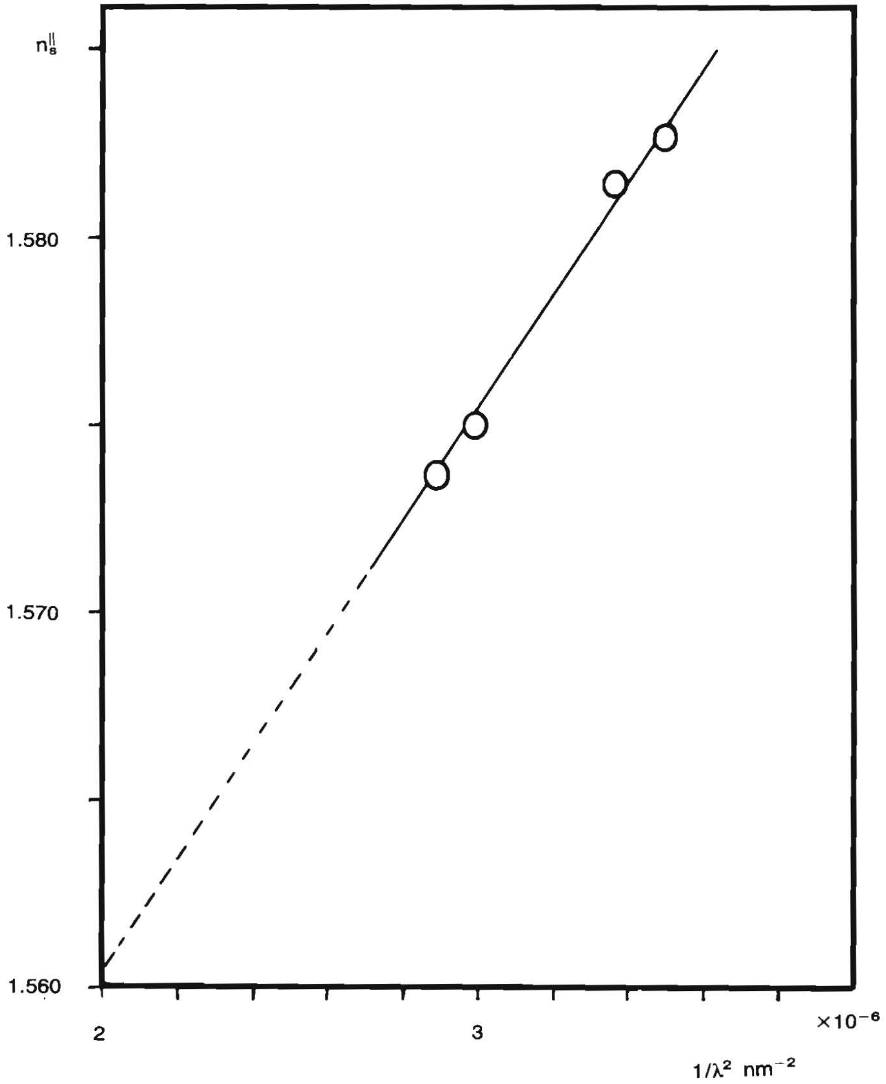

Fig. 7. The linear variation of $n^{\parallel}20_s$ with $1/\lambda^2$ for Saudi cotton fibres.

From the application of equation (5) to the results given in figures 7 & 8, the average dispersive power of skin layer for Saudi cotton fibres within the

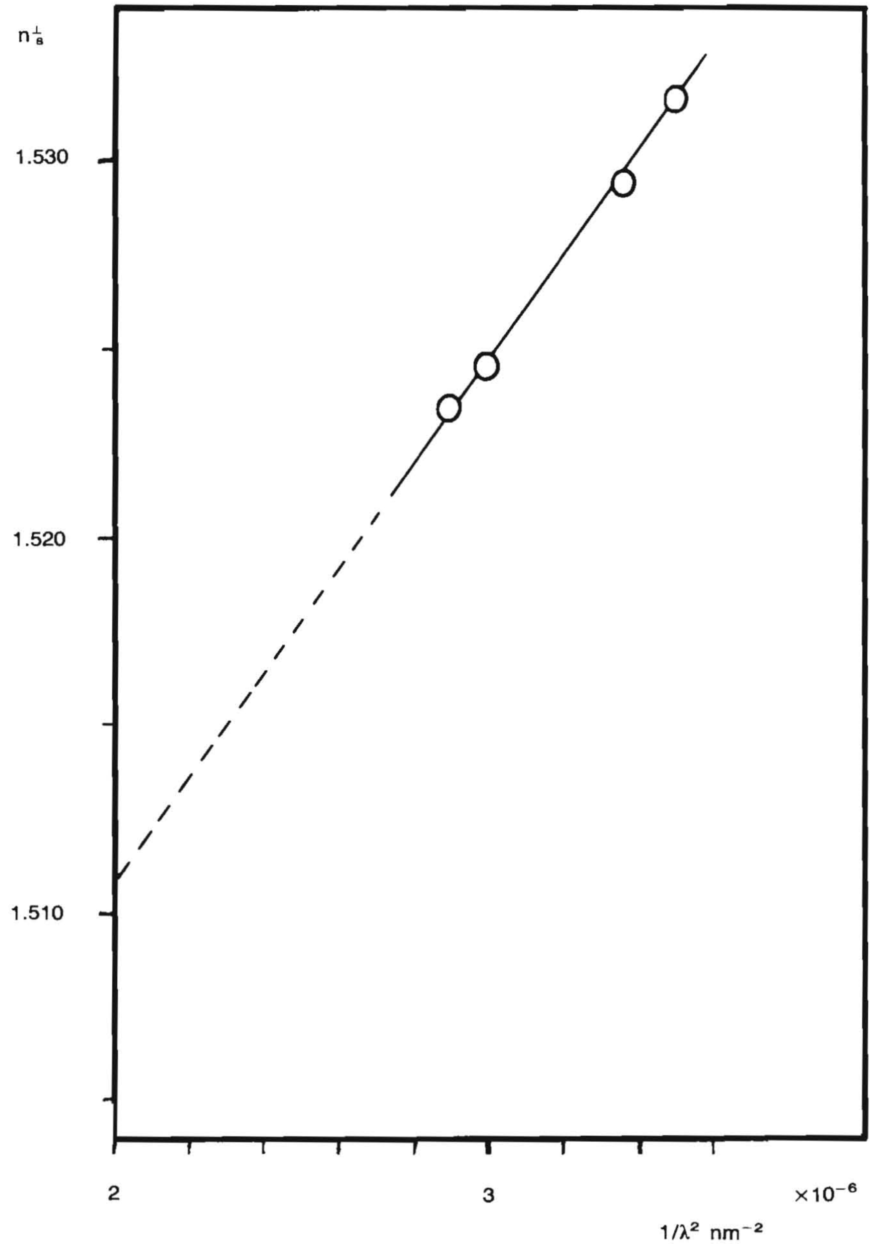

Fig. 8. The linear variation of $n_s^{\perp}$ with $1/\lambda^2$ for Saudi cotton fibres.

considered range of wavelengths was found as

$$dn_s^{\parallel}/d\lambda = -1.11 \times 10^{-4}\ \mathrm{nm}^{-1} \text{ and } dn_s^{\perp}/d\lambda = -1.04 \times 10^{-4}\ \mathrm{nm}^{-1}$$

The polarizability per unit volume:

The experimental values of the mean polarizabilities per unit volume parallel, $P_s^{\parallel}$, and perpendicular, $P_a^{\perp}$ to the fibre axis were calculated from the multiple-beam interferometric measured values of the mean refractive indices $n_a^{\parallel}$ and $n_a^{\perp}$. The calculation was done using Lorentz-Lorenz equation (3). It is found, for Saudi cotton fibres, that:

$$P_a^{\parallel} = 0.0798 \text{ and } P_a^{\perp} = 0.0740$$

Values of refractive indices for the cotton skin layer, as given in table (1), are also used to calculate the polarizabilities per unit volume of the fibre skin layer. Table (2) contains the obtained values of $P_s^{\parallel}$, $P_s^{\perp}$ and $\triangle P_s$ at different wavelengths.

The isotropic refractive index:

Table (2) shows also the isotropic refractive index of Saudi cotton fibres skin layer. Data given in table (1) was introduced into equation (6) in order to find the

Table 1. Refractive indices and birefringence of Saudi cotton fibres skin layer by the Becke-line method

λ(nm)	$n_s^{\parallel}$	$n_s^{\perp}$	$\triangle n_s$
589.3	1.5737	1.5234	0.0503
578.0	1.5749	1.5245	0.0504
546.1	1.5814	1.5294	0.0520
535.1	1.5826	1.5316	0.0510
436.0	1.5902	1.5372	0.0530

Table 2. Isotropic refractive index and polarizability per unit volume of Saudi cotton fibres skin layer

λ(nm)	n_{iso}	$P_s^{\parallel}$	$P_s^{\perp}$	$\triangle P_s$
589.3	1.5402	0.0787	0.0730	5.725×10^{-3}
578.0	1.5413	0.0789	0.0731	5.797×10^{-3}
546.1	1.5467	0.0796	0.0737	5.926×10^{-3}
535.1	1.5486	0.0797	0.0739	5.770×10^{-3}
436.0	1.5548	0.0806	0.0746	6.021×10^{-3}

values of n_{iso} for the skin layer. Figure 9 clarifies that the isotropic refractive index of the fibres under test obeys Cauchy's dispersion formula too. The Cauchy's constants of the isotropic Saudi cotton fibres were found as:

$$A_{iso} = 1.5344 \quad \text{and} \quad B_{iso} = 13 \times 10^3 \text{ nm}^2$$

Specific volume of Saudi cotton fibres:

The most accurate values of $n_a^{\parallel}$ and $n_a^{\perp}$ obtained by the multiple-beam interferometric technique, were used to calculate the mean isotropic refractive index, n_{iso} (mean), which is found as n_{iso} (mean) = 1.5491.

Introducing the value of n_{iso} (mean) into relation (9) one gets, as an average, the specific volume(v)for Saudi cotton fibres amounts to: v = 0.8669 cc/gm.

Spiral angle of the skin layer:

The obtained values of $n_s^{\parallel}$ and $n_s^{\perp}$ at different wavelengths were introduced into equation (7) in order to determine the spiral angle θ_s of skin layer for Saudi cotton fibres.

Optical orientation angle of the skin layer:

The calculated values of skin layer birefringence for Saudi cotton fibres by the Becke-line techniques were used to calculate the optical orientation angle γ_s with the help of equation (8). Figure 10 shows the behaviour of γ_s and θ_s by changing the wavelength. It is clear that the orientation angle γ_s is slightly changed within the visible range of spectrum while θ_s shows a remarkable increase by increasing the wavelength within the same range of spectrum.

Conclusion

From the measurements carried out in this work, the following conclusions may be drawn:

1. From the refractive indices measurements it was found that birefringence is positive, which indicates that the majority of the chains within the cellulose structure lie more nearly parallel than normal to the fibre axis.
2. Obtained experimental values of the skin spiral angle were found to be directly proportional to the wavelength according to a non-linear behaviour.
3. That the orientation angle was weakly changed within the visible range of spectrum.
4. Optically determined values of isotropic refractive index for Saudi (Gizan) cotton fibres showed that n_{iso} obeys Cauchy's dispersion formula.

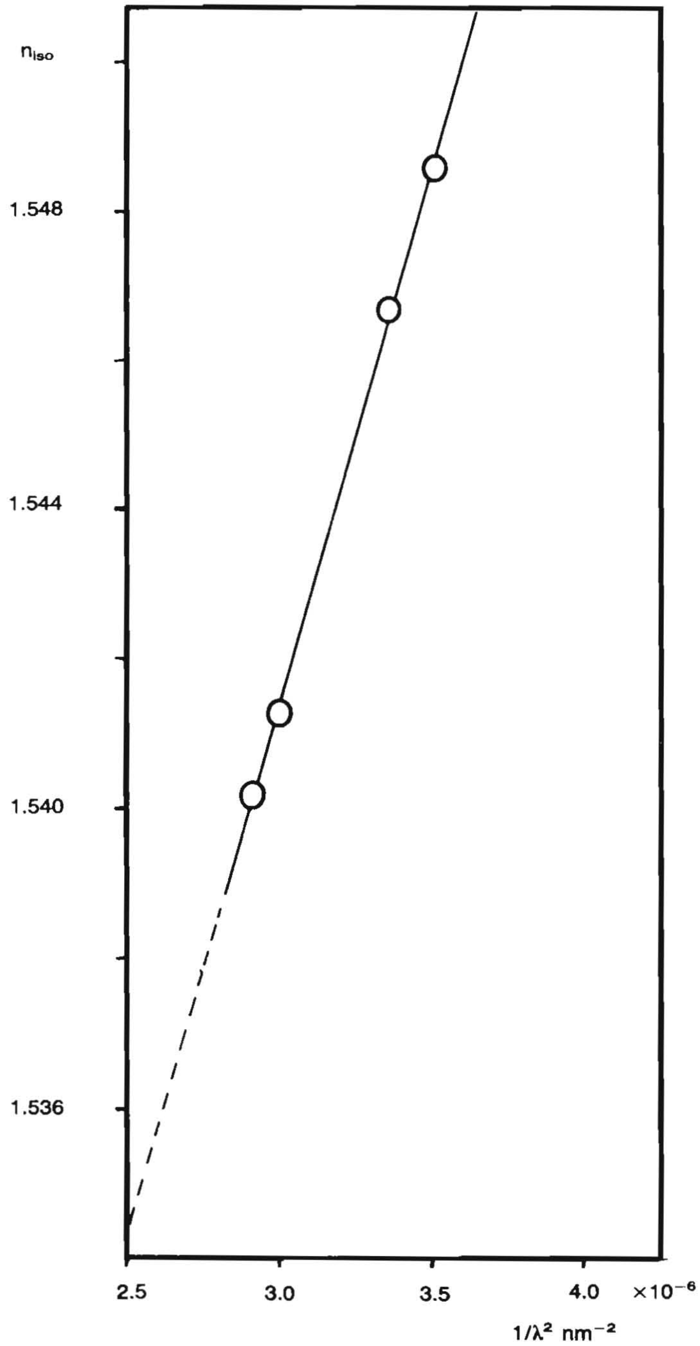

Fig. 9. The linear variation of n_{iso} with $1/\lambda^2$ for Saudi cotton fibres.

5. Variation of the fringe shift along the fibre axis could be due to the irregularities shown by the S.E.M. (Fig. 2).
6. Due to the extreme variability in the cross-section, the S.E.M. was used to estimate the mean geometrical parameters of the irregular cross-section of Saudi cotton fibres.

The presented results prove the efficiency of the Becke-line and the interferometric techniques, using recent relations ($n_a^{\parallel} = n_L + (F^{\parallel}/h)(\lambda/2A)$ and ($\triangle n_a = (F^{\parallel} - F^{\perp})\lambda/hA$), for the determination of refractive indices and

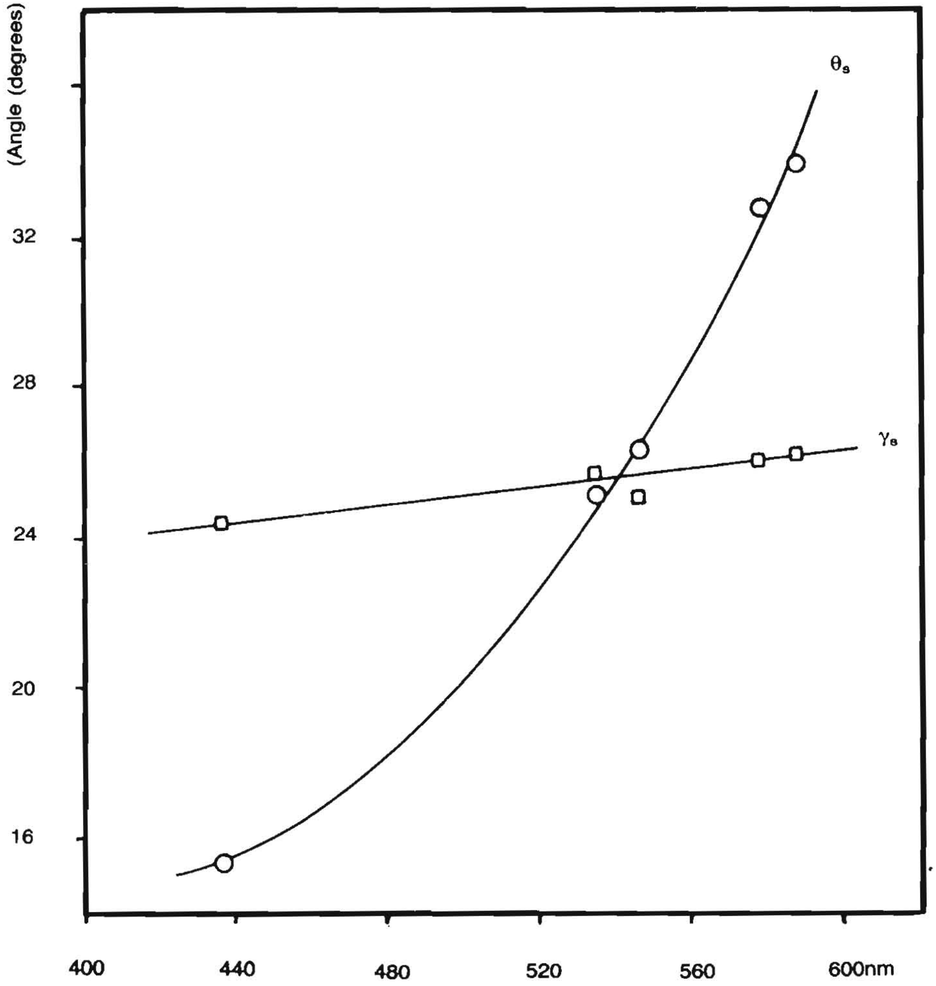

Fig. 10. The variation of spiral angle θ_s (o) and the optical orientation angle γ_s (n) of skin layer for Saudi cotton fibres by increasing the wavelength.

birefringence of fibres with irregular transverse sections, such as Saudi cotton fibres which have great variability in its cross-sectional shape

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دراسات ضوئية على التركيب الخاص للقطن السعودي باستخدام المجهر الالكتروني الماسح

ابراهيم محمد فودة و ماهر محمود التونسي و كريمال أحمد الفرحاتي

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تم في هذا البحث استخدام معادلات حديثة الإستنتاج وكذلك عدة طرق تجريبية وهي طريقة بيكا للاشارة الخطية وطريقة التداخل المتعدد لفيزو والثنائي باستخدام مجهر التداخل الضوئي لبلوتا وذلك لقياس معاملات الإنكسار والإنكسار المزدوج للقطن السعودي (من منطقة جيزان)، وقد استخدم المجهر الالكتروني الماسح لتقدير أبعاد ومقطع الشعيرات ولتوضيح الإنبعاجات والتغيرات الدقيقة على سطح الشعيرة والتي لا يمكن توضيحها باستخدام المجاهر الضوئية .

وباستخدام الاشعة المتعددة لفيزو والثنائية لبلوتا تم تعيين معاملات الإنكسارات المتوسطة في الاتجاهين الموازي والمتعامد على محور الشعيرة، وباستخدام طريقة بيكا للإشارة الخطية أمكن تعيين معاملات الإنكسار للطبقة الخارجية للشعيرة في الاتجاهين الموازي والعمودي .

وقد تم حساب درجة الإستقطابية لوحدة الحجوم وكذلك حساب ثابتي معادلة كوشي للتفرق الضوئي، قوة التفريق ومعامل الإنكسار الايزوتروبي (في حالة التماثل) وزاوية الحلزون والزاوية الضوئية للتوجيه والحجم النوعي لمادة ألياف القطن السعودي .

ولقد إتضح من النتائج مدى توافقها مع الأنواع الأخرى من القطن .

ويمكن تلخيص النتائج :

١ ـ إتضح إن معامل الإنكسار المزدوج ذو قيمة إيجابية مما يدل على أن غالبية السلسلة البنائية لتركيب السليلوز توجد موازية لاتجاه محور الشعيرة.

٢ ـ وجد أن زاوية الحلزون للطبقة الخارجية تتناسب مع الاطوال الموجية المختلفة بعلاقة غير خطية.

٣ ـ ومن النتائج وجد أن زاوية التوجيه تتغير تغيراً طفيفاً خلال مستوى الطيف المنظور.

٤ ـ كما وجد أن معامل الإنكسار المتماثل (الايزوتروبي) يتبع معادلة كوشي وتم حساب ثابتي المعادلة في حالة التماثل وذلك للطبقة الخارجية.

٥ ـ نتيجة للإنبعاجات البنائية لألياف القطن لوحظ إختلافات في شكل أهداب التداخل الضوئي على محور الشعيرة وقد تأكد ذلك من خلال الدراسات على سطح الشعيرات باستخدام المجهر الالكتروني مع الماسح.

٦ ـ نتيجة للاختلافات الحادة في أبعاد ومقطع الشعيرة استخدم المجهر الماسح لتعيين الأبعاد الهندسية المتوسطة لألياف القطن السعودي.

ويتضح مما سبق أهمية طرق التداخل المتعدد لفيزو والثنائي لبلوتا وكذلك طريقة بيكا لدراسة الترتيب الجزيئي للألياف، وأهمية استخدام المعادلات الحديثة للحصول على نتائج دقيقة وخصوصاً للألياف غير المنتظمة المقطع، كما أن استخدام طريقة التداخل الثنائي وباستعمال مجهر بلوتا تعطي نتائج سريعة دون الحاجة لتحضيرات صعبة كما في حالة استخدام طريقة التداخل للاشعة المتعددة لفيزو. وباستعمال مجهر بلوتا يمكن الحصول على معامل الإنكسار المزدوج مباشرة دون الحاجة لتعيين معاملي الإنكسار الموازي والمتعامد كل على حده.

وتم توضيح تلك الدراسة باستخدام المنحنيات والصور الشمسية.