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Some Observations on the Shrinkage of Jute

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INTRODUCTION

The swelling in water and the swelling and mercerizing behavior in alkali show certain characteristics of jute which are different from those of a pure cellulose fiber such as ramie or cotton.[1,2] In view of the strong correlation between swelling and shrinkage, as apparent from the evidence of other cellulose fibers,[3-5] an investigation into the shrinkage behavior of jute seems worthwhile, as it is likely to clarify certain points arising from the results on swelling. For example, for a similar degree of swelling, the shrinkage of jute should show a higher value in dilute alkali, and the corresponding portion of the shrinkage curve be similar in shape to the swelling curve; also a lower shrinkage in strong alkali and attainment of maximum at a lower concentration are to be expected. On the other hand, looking upon shrinkage as a longitudinal phenomenon similar to axial swelling and as distinct from transverse swelling, it should be lower in dilute alkali. The study of shrinkage as a function of alkali concentration would thus indicate which is the more reasonable view, and how far the speculation about the presence of an extra structural material is justified.[1] Secondly, as part of the soda-cellulose I of jute is reconverted to cellulose I on removal of alkali,[6-8] measurements of permanent contraction would show whether a corresponding part of the alkali shrinkage is also restored on washing out of the alkali. If so, assuming that a relation between orientation and shrinkage exists such that, other things remaining equal, a more oriented fiber would shrink more, the mercerized or optimum alkali-treated jute should exhibit a residual positive shrinkage when subsequently put into alkali, and the dilute alkali-treated fiber should show a reduced value of shrinkage on account of its reduced orientation, as found in connection with the study of anisotropy.[9]

EXPERIMENTAL

The materials used in this investigation were dewaxed white jute; purified ramie; viscose rayon; jute, ramie, and viscose pretreated in bulk and in singles with various concentrations of NaOH solution for 1/2 hr. at room temperature (78 ± 2°F.); jute delignified to different extents; and jute and ramie freely swollen in 17.5% NaOH solution for 1/2 hr. at room temperature, stretched to different degrees in a stretching frame, washed, and air-dried in the stretched state.

The following measurements were made: (*1*) shrinkage of single jute and ramie fibers in NaOH solutions of concentrations in the 0–28% range; (*2*) shrinkage of treated jute and ramie fibers in a limited range of NaOH solutions so as to determine the maximum shrinkage; permanent contraction resulting from treatment of jute, ramie, and rayon fibers in dilute and strong NaOH solutions, viz., 5–16.5%; diameter and axial swelling for determining the swelling orientation factor according to the formula

$$f_s = (Q - 1)/(Q + 1/2)$$

where Q is the ratio of specific diameter swelling/specific longitudinal swelling.[9-11] In addition, some x-ray photographs of treated jute, ramie, and rayon were taken.

For measuring the shrinkage in alkali the specimens were prepared as follows. A small bundle of parallelized fiber was cut into 12-cm. and 7-cm. lengths, the former for water and dilute alkali and the latter for strong alkali solutions. Individual fibers free from adhering branches were picked out, and paper clips were gummed at the two ends, the grip length being kept at 10 cm. in one instance and 5 cm. in another. In order to prevent any action of alkali beyond the specified length, a paraffin wax coating was given to the clips. The specimen was then mounted in an extensometer, the construction and performance of which have been previously described in connection with the measurements of longitudinal swelling in water.[1] As the delignified fiber was highly tendered when steeped in alkali, it was not possible to make a systematic study of the maximum shrinkage for

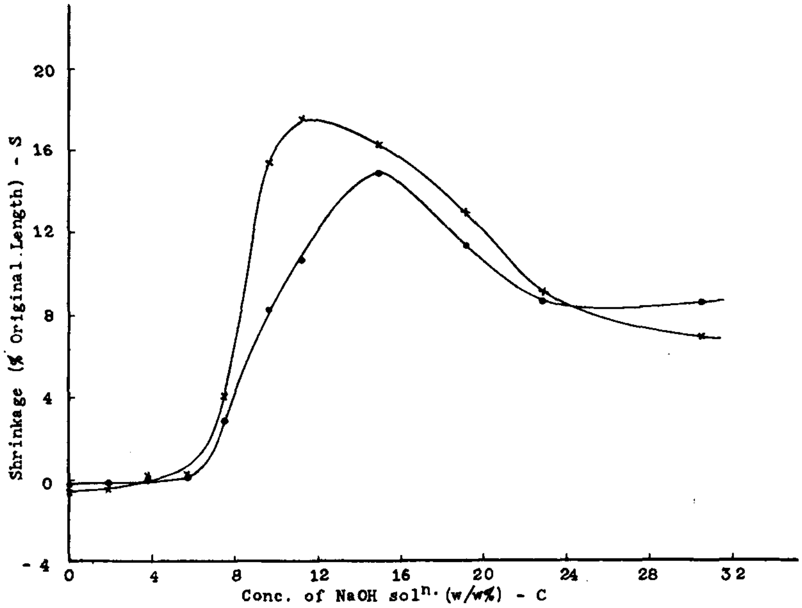

Fig. 1. Variation of shrinkage with alkali concentration: (●) jute; (×) ramie.

this sample except taking a few indicative measurements.

For measurement of permanent contraction on removal of alkali (and drying), the length of the specimens was 10 cm. In all cases, 20 specimens were taken to represent a sample corresponding to a particular concentration of alkali.

The measurements of diameter and longitudinal swelling were the same as have been reported earlier.[1]

RESULTS AND DISCUSSION

A. Shrinkage of Dewaxed Jute and Purified Ramie in Alkali

The shrinkage behavior of dewaxed jute and purified ramie in 0–28% NaOH solutions is represented in Figure 1. The shrinkage starts in jute and ramie at 5–6% alkali, while the corresponding concentration reported for cotton is about 8%.[12] Jute shrinks less than ramie through most of the alkali concentrations, except beyond 23–24%, when it shrinks more, and the maximum for jute is reached at a slightly higher concentration. It is interesting to note that the difference between jute and ramie in this regard is similar to that between a yarn and its constituent fibers.[5] The general shape of the curve for jute is, however, similar to that for ramie, particularly below 23–24% alkali, and is conspicuous by the absence of a higher value, and a concavity towards the concentration axis in the dilute range; this has been found to be a special feature of the swelling curve of jute vis-a-vis ramie and cotton.[1,2] For comparison with ramie, the reported results[2] for the cross-sectional swelling of jute and ramie should, of course, be represented graphically.

The starting of measurable shrinkage in cotton at about 8% alkali has been explained as due to the removal of convolutions of the fiber when in alkali, the observed effect being the resultant of an elongation due to untwisting and of a contraction due to coiling of the chain elements.[12] Although a certain degree of untwisting is observable in both jute and ramie, it may not be of any major consequence for these fibers, and the critical alkali concentration is presumably determined by the balance between the normal longitudinal swelling and contraction of the chain elements which increases with increasing concentration.

The lower shrinkage of jute as compared to ramie up to 23–24% is in line with the expectation that the shrinkage is also subject to a similar restraint as swelling. But the similarity of the shrinkage characteristics of jute as compared to ramie with those of a yarn as compared to its constituent fibers strongly indicates that the aforesaid restraint is comparable to a twisting strain which holds the fibers together in a yarn.[5] It is true that the comparison does not get one nearer to the origin of the restraint but, nevertheless, gives an idea regarding its operational behavior.

The lower shrinkage and the absence of concavity in the shrinkage curve, as contrasted with swelling characteristics of jute in dilute alkali, clearly indicate that the fiber contains some material which contributes to swelling but not to shrinkage. This is in agreement with the observation made in connection with the study of water swelling of jute.[1] It would likewise be reasonable to conclude that the material concerned is extrastructural in nature, apparently constituting a separate phase independent of the structural continuum. This result would further indicate that for jute, at least, axial swelling and shrinkage are to be regarded as belonging to the same category of phenomena concerning longitudinal changes, and they are more closely associated with the structural framework of the fiber than the transverse swelling.

B. Permanent Contraction of Jute, Ramie, and Rayon Treated in Alkali

1. Effect of Single Alkali Treatment on Jute, Ramie, and Rayon. The permanent changes in length resulting from treatment of jute with 7.5 and 16.5% NaOH, ramie with 9 and 16.5% NaOH, and viscose rayon with 5, 10, and 16.5% NaOH solutions are shown in Table I. It will be seen that the permanent contraction due to premercerizing alkali treatment is negligibly small in jute and ramie; individual measurements show that the original length of 10 cm. hardly decreased to less than 9.9 cm. It is, however, quite appreciable in rayon treated with 5% alkali, and the sample of rayon also shows a noticeable decrease in crystalline orientation. Since both jute and ramie show appreciable shrinkage when steeped in alkali solutions, these results would indicate that

TABLE I
Permanent Contraction of Jute, Ramie, and Rayon on Single Alkali Treatment

Material	Treatment	Permanent contraction, %
Jute	7.5% NaOH	1.0
Jute	16.5% NaOH	15.3
Ramie	9% NaOH	1.0
Ramie	16.5% NaOH	21.0
Rayon	5% NaOH	7.9
Rayon	10% NaOH	23.9
Rayon	16.5% NaOH	24.6

the shrinkage in premercerizing alkali is practically completely reversible in these fibers. On the other hand, comparison of the value of permanent contraction in ramie resulting from strong (i.e. mercerizing) alkali treatment with the shrinkage measured in similar concentration indicates that the latter would appear to be completely irreversible; in fact, the permanent contraction is a little higher than the alkali shrinkage. This difference is, however, probably due to the fact that the fibers were shrunk under different conditions in the two instances; they were fixed at either end in one instance and completely free in the other. As these considerations should also apply to jute, one would reasonably expect a similar difference between the permanent contraction and the alkali shrinkage of the fiber. However, in view of the fact that a part of the alkali shrinkage of jute measured in optimum concentration is reversible, as we shall see below, it is not possible to detect the effect in this fiber.

Aside from the question of any effect arising from morphological differences between ramie and rayon, the above results would indicate that permanent contraction occurs in a fiber characterized by large and numerous crystallites, e.g., ramie, only when the structure is disrupted by an optimum degree of swelling. On the other hand, permanent contraction may occur in a poorly crystalline fiber like rayon even with less disturbance. The case of jute in this context, however, appears to be particularly complicated. While in view of comparable crystallinity[2] with rayon one would expect a permanent contraction in jute on dilute alkali treatment, the effect is found to be limited to a slight disorientation only, without any permanent contraction. This can be accounted for by assuming a rigidity in the structure of jute. But the said rigidity is not completely eliminated, even on strong alkali swelling, as indicated by partial reversal of shrinkage in mercerized jute; the resistance offered by the crystalline domains in ramie, however, is possibly completely withdrawn under similar circumstances.

2. Effect of Repeated Alkali Treatments of Jute and Ramie. The permanent contractions in jute and ramie repeatedly treated in 16.5% NaOH solution are shown in Table II. Figure 2 represents the x-ray photographs of jute and ramie corresponding to first and fifth treatments. It will be seen (Table II) that the permanent contraction continually increases in either fiber on repeated alkali treatments, but the increase is

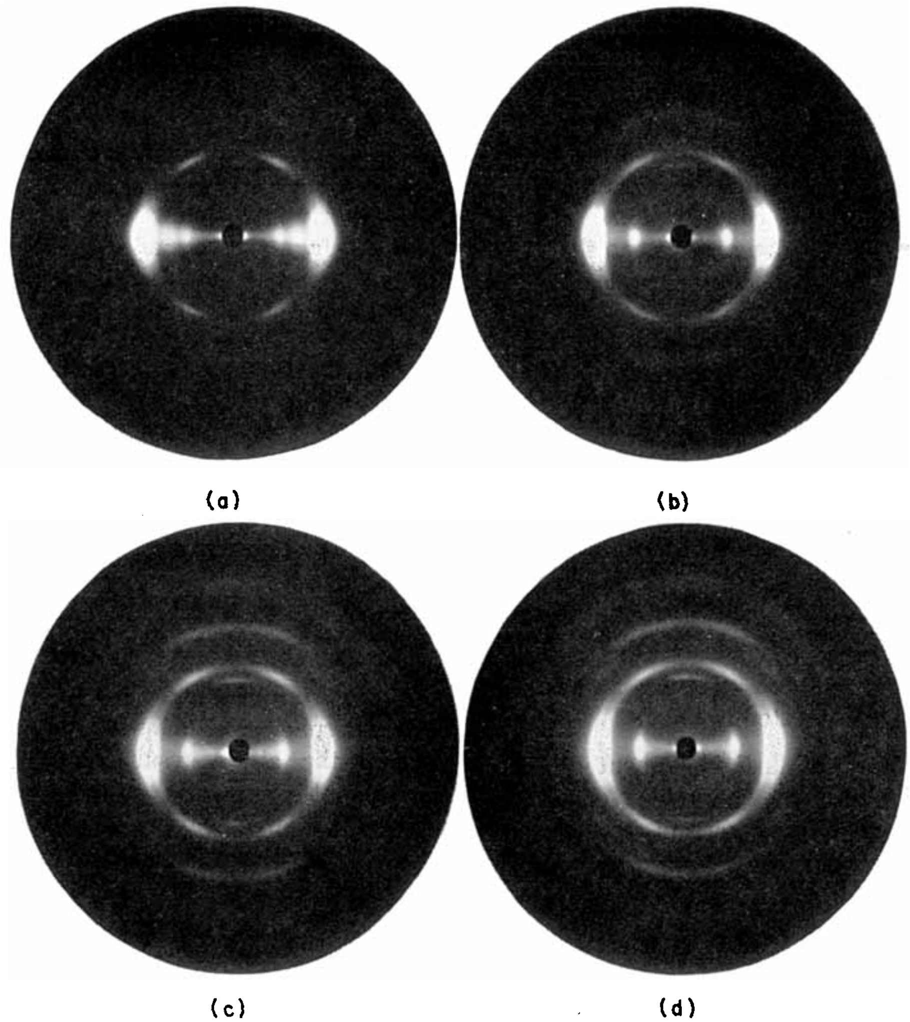

Fig. 2. X-ray photographs of jute and ramie treated with 16.5% NaOH: (*a*) jute, treated once; (*b*) jute, treated five times; (*c*) ramie, treated once; (*d*) ramie, treated five times.

definitely slowed down after the second treatment. The crystalline orientation has markedly decreased in both jute and ramie, and an additional change

TABLE II
Permanent Contraction of Jute and Ramie on Repeated Alkali Treatments

Material	No. of treatments with 16.5% NaOH	Permanent contraction, %
Jute	1	15.3
"	2	17.5
"	3	18.0
"	4	18.6
Ramie	1	21.0
"	2	23.2
"	3	24.1
"	4	25.0

in jute is that it is almost completely mercerized after the fifth treatment.

The progressive increase in permanent contraction on repeated alkali treatment is an interesting result which demonstrates a progressive weakening of the cohesiveness arising from the interchain frictional resistance. As one would normally expect, this results in increased disorientation of the structural elements and indicates that disorientation and permanent contraction go hand in hand. Accordingly, the disorientation effected in dilute alkali-treated jute without being attended with any permanent contraction, as previously noted, appears to be a singular feature of the structure of jute. On the other hand, the evidence that complete mercerization of jute follows repeated alkali treatments suggests yet another method of successfully overcoming the antimercerizing influence of its structure, the other procedures being delignification[6–8] hydrolytic pretreatment with boiling sulfuric acid,[7] and mercerization with perchloric acid.[8,13] It is also noteworthy that jute mercerized by this process is the least tendered and retains the fiber form.

C. Maximum Shrinkage of Pretreated Jute and Ramie in Alkali

1. Freely Alkali-Treated Jute and Ramie and Delignified Jute. The maximum shrinkage (i.e., in optimum NaOH solution) of alkali-pretreated jute and ramie is shown in Figure 3. It will be seen (Fig. 3) that the maximum shrinkage of jute is appreciably reduced on very dilute alkali pretreatment, and, starting from 1% NaOH, the reduction is more or less constant throughout the premercerizing range of alkali. On the other hand, the maximum shrinkage of similarly pretreated ramie is not affected until the strength of alkali is 10%, i.e., until the start of the attack on the crystallites. It is interesting to note that the two fibers behave in this regard in the same way as towards orientation, as noticed elsewhere.[9] The parallel between shrinkage and orientation is further indicated by the results on delignified jute. It has been found that delignification up to a certain stage does not involve any significant change in orientation,[9] and the present work shows that the maximum shrinkage of delignified jute is the same as, if not a little more than, that of the untreated one. The results of strong alkali pretreatment show that, whereas the maximum shrinkage of mercerized ramie is negative, that of mercerized jute is positive, having a value of approximately 3.1%, indicating that a part of the total shrinkage undergone in the first immersion in alkali has been restored. It may be pointed out that this partial reversion of the maximum shrinkage of jute is an exact replica of the phenomenon of partial reversion of the soda-cellulose I of jute to cellulose I when the alkali is washed out; the difference between the values of the maximum shrinkage of native and mercerized jute bears to the former approximately the same ratio as the proportion of hydrate formed in jute under ordinary conditions of mercerization in alkali,[6] i.e.:

$$\frac{\text{(Maximum shrinkage of native jute} - \text{Maximum shrinkage of mercerized jute)}}{\text{Maximum shrinkage of native jute}} = \frac{15.6 - 3.1}{15.6} = \sim 80\%$$

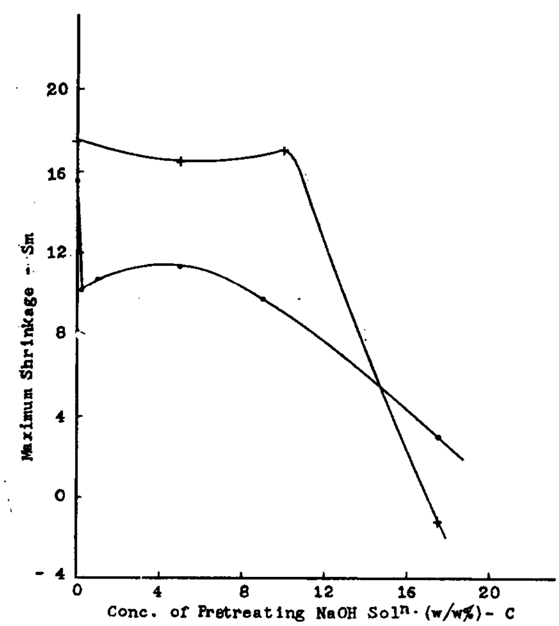

Fig. 3. Dependence of shrinkage on pretreating alkali concentration: (●) jute; (×) ramie.

The lower value and partial reversal of optimum shrinkage of jute are also suggested by the less disoriented state of its crystallites when the diffraction arcs in the corresponding x-ray patterns of jute and ramie are compared (Figs. 2*a*, 2*c*).

An obvious conclusion from the above results is that shrinkage is strongly correlated with orientation, and, in a particular fiber representing a definite morphology, a reduction in orientation is followed by a reduction in shrinkage. Accordingly, the reversal of a part of the optimum shrinkage of untreated jute can be associated with a corresponding partial reversal of the optimum disorientation. It should be pointed out, however, that although the correspondence between the proportions of reversible shrinkage and reversible soda-celullose I of jute suggests that the restraint might be completely held in check so long as the fiber was in alkali (the inference has been drawn from a study of the soda-cellulose of jute in the x-ray diagram of which no native cellulose could be detected[7]), the lower values of both shrinkage and swelling in alkali indicate that it will not be reasonable to regard the movements of the chain elements of jute as unfettered as in ramie. We have already pointed out the subdued nature of the swelling of jute of alkali.[1]

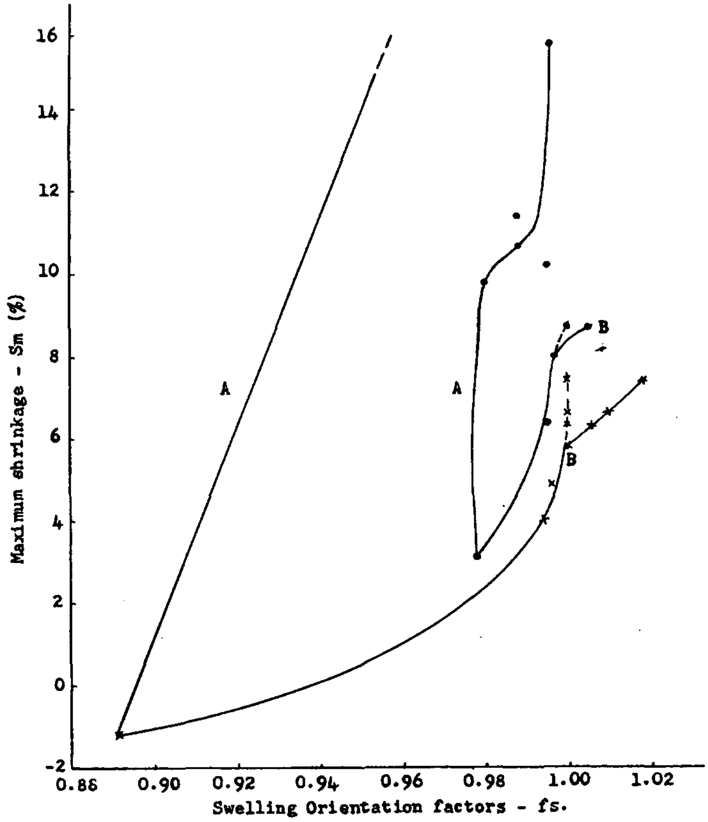

Fig. 4. Dependence of shrinkage on orientation: (●) jute; (×) ramie.

2. Reoriented Jute and Ramie. The maximum shrinkages of native, disoriented (i.e., freely mercerized) and reoriented (i.e., mercerized and stretched) jute and ramie fibers are plotted against the respective swelling orientation factors in Figure 4. It will be seen that the curve for each fiber has two sections, one (A) denoting the change from the native to the disoriented state and another (B) denoting the change from the disoriented to the maximum reoriented state. In this regard, the use of f_s as an expression for the oriented state of these fibers has been discussed in another report, and it has been pointed out that the value of $f_s \geqq 1$ should be more reasonably ascribed to some metastable state of the chain molecules by virtue of which the fiber shows positive shrinkage even in pure water.[9] Because of its contribution to shrinkage, however, it may not be reasonable to limit the orientation factor to unity and modify the curves as indicated by the dotted portions.

It will be seen (Fig. 4) that the nature of the curve as represented by section (B) is roughly similar for both jute and ramie, but that represented by section A is different for the two fibers; the shrinkage of jute decreases with disorientation produced on dilute alkali treatment and the corresponding portion of the curve shows inflection. This feature has been noted in the previous section describing the results for freely treated fibers. The interesting aspect of these results is that the maximum shrinkage of the reoriented samples is considerably less than that of the native fiber, whereas the degree of orientation of the reoriented specimen, especially of the maximum stretched one, is much higher. This difference is more prominent in ramie than in jute. It has been found by comparing the x-ray photographs that when the stretched fiber is subsequently treated in optimum alkali solution used for producing the maximum shrinkage, it is much less disoriented than the freely mercerized sample. The corresponding permanent contractions are also considerably less, e.g., 7.1% against the original value of 21% for ramie. However, the transformation of the small cellulose I residue (in reoriented ramie) to cellulose II is not prevented.

The increase in the value of maximum shrinkage with the increase in orientation clearly demonstrates the dependence of one upon another, as previously indicated for the freely treated fibers; the non-rectilinear nature of the curves suggests that the progressive mechanical strain is not confined merely to inducing alignment of the chain elements but also modifies the network structure considerably. The nature of the modification induced is, however, difficult to envisage. The swelling behavior of the reoriented samples, studied elsewhere,[9] suggests that the mechanical strain might have led to the formation of certain junction points in the intercrystalline areas. It is remarkable that, if hydrogen bonds are involved in the process, the resistance to shrinkage and disorientation observed here would require that they are stable against the action of strong alkali. On the other hand, the successful conversion of the cellulose I residue in ramie would indicate that these bonds must differ from those of which we are aware in the structure of jute and which hold their own against strong alkali swelling and resist complete mercerization. If the resistance to mercerization is regarded primarily as a resistance to rotational movement of the chains and is intrafibrillar in origin, acting as a longitudinal strain as postulated for jute,[1,8,14] one would wonder if the bond developed on mechanical stretching may be interfibrillar in character.

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Synopsis

Measurements have been made of the shrinkage in alkali and of the permanent contraction resulting from alkali treatment. The results supported by x-ray evidence following the changes in orientation and degree of mercerization show that the shrinkage of jute is subject to a similar restraint as swelling, and the shrinkage behavior of jute compared to ramie is as that of a yarn compared to that of its constituent fibers. Unlike swelling, the shrinkage of jute at dilute alkali is less than that of ramie and the corresponding region of the shrinkage curve is conspicuous by the smoothness of swelling curve, indicating that the shrinkage refers primarily to the structural framework of the fiber, whereas an extrastructural material may contribute to swelling. Although the crystallinity of jute is comparable to that of rayon, no appreciable permanent contraction occurs in the fiber treated in dilute alkali, and this points to a restraint in the structure of jute similar to that exercised by the crystallites in ramie. Whereas the resistance in ramie is completely neutralized in mercerizing alkali and the whole of the alkali shrinkage remains as permanent contraction, the restraint still operates in jute, and the permanent contraction is in deficit of the alkali shrinkage. The respective disorientations are also of the same order. Both permanent contraction and crystalline disorientation continually increase on repeated alkali treatments of jute and ramie; the additional effect found in jute is its complete mercerization after a number of treatments. The shrinkage as indexed by its maximum value is strongly correlated with the oriented state of the fiber, and the disorientation produced in jute on dilute alkali treatment is followed by a lower maximum shrinkage. Incomplete disorientation of mercerized jute shows up in positive shrinkage and its proportion is the same as that of unconverted cellulose I residue. In spite of a very high degree of orientation, the reoriented fiber shows a considerably low maximum shrinkage compared to the native. This is suggested to have been due to the formation of some junction points when the freely swollen plasticized fiber is mechanically strained.

Résumé

On a mesuré le retrécissement et la contraction permanente résultant du traitement aux alcalis. Les résultats confirmés par rayons-X, suivant les changements d'orientation et le degré de mercérisation, montrent que le rétrécissement du jute est sujet à un empêchement semblable au gonflement et que le comportement de ce rétrécissement du jute vis-à-vis de celui de la ramie, est comparable à celui d'un fil vis-à-vis des fibres qu'il constitute. Mais lors de goflement différent, le rétrécissement du jute dans les alcalis dilués est plus faible, comparé à celui de la ramie et la région correspondante de la courbe de rétrécissement se caractérise par l'absence de bosse observée dans la courbe de gonflement, montrant que le rétrécissement se rapporta d'abord à la charpente structurale de la fibre; toutefois un facteur extra-structural peut contribuer au gonflement. Bien que la cristallinité du jute soit comparable à celle de la soie, il n'y a aucune contraction permanente appréciable dans le cas des fibres traitées par les alcalis dilués et ceci indique un empêchement de structure de la jute semblable à celui exercé par les cristallites dans la ramie. Mais, bien que la résistance dans la ramie soit complètement neutralisée dans un alcali

mercérisant et que la totalité du rétrécissement d à l'alcalie demeure sous forme de contraction permamente, ln̂'empèchment agit encore dans le jute et la contraction permanente est plus faible que le rétrécissement dǔ à l'alcali. Les désorientations respectives sont également de même ordre. La contraction permanente ainsi que la désorientation cristalline augmentent continuellement lors des traitements répètés par les alcalis dans le cas du jute et de la ramie; l'effet additionnel trouvé dans le cas du jute est dǔ à sa complète mercérisation après un certain nombre de traitements. Le rétrécissement, ainsi que le montre sa valeur maximum, est en relation étroite avec l'état orienté de la fibre et la désorientation produite dans le jute par le traitement aux alcalis dilués est suivie apr un rétrécissement moins prononcée. La désorientation incomplète du jute mercérisé révèle un rétrécissement certain et sa proportion est la même que dans le cas d'un résidu cellulosique non transformé. Malgré un degré fort élevé d'orientation, la fibre réorientée indique un rétrécissement maximum beaucoup plus faible comparé à la fibre originale. Ceci est probablement dû à la formation de quelques points de jonction lorsque la fibre plastique librement gonflée est tendue mécaniquement.

Zusammenfassung

Es wurden Messungen der Schrumpfung in Alkali und der durch Alkalibehandlung hervorgerufenen permanenten Kontraktion durchgeführt. Die Resultate werden durch Röntgenuntersuchungen der Änderung der Orientierung und des Mercerisierungsgrades gestützt und zeigen, dass die Schrumpfung von Jute einer ähnlichen strukturellen Beschränkung unterworfen ist wie die Quellung und dass das Schrumpfungsverhalten von Jute im Vergleich zu Ramie dem eines Garnes im Vergleich zu seinen Fasern entspricht. Die Schrumpfung von Jute in verdünntem Alkali ist jedoch, im Gegensatz zur Quellung, in Vergleich zu Ramie kleiner und der entsprechende Bereich der Schrumpfungskurve fällt durch das Fehlen der bei der Quellungskurve beobachteten Ausbuchtung auf; dies weist darauf hin, dass die Schrumpfung in erster Linie mit dem strukturellen Skelett der Faser in Beziehung steht, während zur Quellung ein ausserstrukturelles Material beitragen kann. Obwohl die Kristallinität von Jute mit der von Rayon vergleichbar ist, tritt keine wahrnehmbare permanente Kontraktion in der Faser bei Behandlung mit verdünntem Alkali auf; dies weist auf einen strukturellen Zwang bei Jute hin, ähnlich wie ihn die Kristallite bei Ramie ausüben. Aber während die Strukturbeständigkeit bei Ramie durch die alkalische Mercerisierungslauge vollständig aufgehoben wird und die gesamte alkalische Schrumpfung als permanente Kontraktion bestehen bleibt, ist bei Jute der Zwang noch immer vorhanden und die permanente Kontraktion ist geringer als die alkalische Schrumpfung. Die entsprechende Desorientierung liegt in der gleichen Reihenfolge. Sowohl die permanente Kontraktion als auch die kristalline Desorientierung nimmt bei wiederholter alkalischer Behandlung von Jute und Ramie zu; als zusätzlicher Effekt wird bei Jute eine vollständige Mercerisierung nach einer Anzahl von Behandlungen gefunden. Die durch ihren Maximalwert charakterisierte Schrumpfung zeigt einen ausgeprägten Zusammenhang mit dem Orientierungszustand der Faser; die durch Behandlung mit verdünntem Alkali erzeugte Desorientierung in Jute verursacht ein niedrigeres Schrumpfungsmaximum. Unvollständige Desorientierung von mercerisierter Jute zeigt sich in einer positiven Schrumpfung und ihr Verhältnis ist das gleiche wie bei unkonvertiertem Cellulose-I-Rückstand. Trotz des hohen Orientierungsgrades zeigt die reorientierte Faser ein wesentlich niedrigeres Schrumpfungsmaximum als die ursprüngliche. Dies wird auf die Bildung von Vernetzungsstellen bei der mechanischen Dehnung der frei gequollenen, weichgemachten Faser zurückgeführt.

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