ON THE STRUCTURE OF HYDRATED CELLULOSE OBTAINED FROM RAW JUTE FIBRE.

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ABSTRACT.

X-ray investigation of the structure of raw jute fibre first treated with NaOH solutions of different concentrations, both under tension and without tension, washed in water and then dried in free air for several days reveals that when tension is applied to the fibre during the treatment even a 30% NaOH solution converts only a part of the native cellulose into hydrated cellulose, but if no tension is applied the cellulose is wholly converted into hydrated cellulose. The structure of this hydrated cellulose is slightly different from that obtained by previous workers from raw cotton or ramie fibre. The dimensions of the unit cell of this hydrated cellulose are: $a = 8{\cdot}8$ A.U., $b = 10{\cdot}3$ A.U., $c = 9{\cdot}5$ A.U., and $\beta = 57° 54'$. This treatment is found to make the fibre softer and to diminish its thermal conductivity, so that some of its physical properties are almost the same as those of coarse wool.

INTRODUCTION.

It is well known that when cotton or ramie fibre is treated with NaOH solution, washed in water and dried in free air the native cellulose in the fibre is either partially or wholly converted into hydrated cellulose, the percentage of native cellulose present after the treatment depending upon the strength of the solution used and its temperature. The question has been investigated thoroughly by Sisson and Saner (1941). They have shown that raw cotton fibre is completely converted into hydrated cellulose by the action of 18% NaOH solution at the room temperature, no tension being applied to the fibre during the treatment, but above 65°C. the native cellulose in the fibre is only partially converted into hydrated cellulose even by 50% NaOH solution. Bleached cotton was found to give the same results as raw cotton fibre. The analytical composition of the hydrated cellulose has been observed to be the same as that of native cellulose, but the crystal structure of the former is different from that of the latter. The dimensions of the unit cell of dried hydrated cellulose as determined by Andress (1929) are: $a = 8{\cdot}1$ A.U., $b = 10{\cdot}3$ A.U., $c = 9{\cdot}1$ A.U. and $\beta = 62°$. It is, however, not known whether raw jute fibre, which contains about 10% to 13% of lignin besides native cellulose, behaves in the same way as cotton fibre when treated with NaOH solution. The present investigation was therefore undertaken to study the crystal structure of the product obtained by treating raw jute fibre with NaOH solutions of different concentrations both under tension and without applying any tension, washing it with water and drying. The values of thermal conductivity of the hydrated cellulose obtained by treatment with 30% NaOH solution and of the raw jute fibre have also been determined.

EXPERIMENTAL.

The treatments which small bundles of 'white top' raw jute fibre had undergone before their structures were analysed with the help of X-rays are enumerated below:—

(*a*) The small bundle was kept immersed in 18% NaOH solution for half an hour without applying any tension and then washed in tap-water and dried in free air for more than a week.

(*b*) A second group of fibres was slightly stretched with the help of weights and the fibres were treated as in (*a*).

(*c*) The process (*a*) was repeated in the case of a third bundle, using 30% NaOH solution.

(*d*) The process (*b*) was repeated, using a fourth bundle and 30% NaOH solution.

(*e*) The product obtained by treatment (*c*) was again kept immersed in 1% NaOH solution for a few hours, washed in water and dried in free air for a few days.

(*f*) A second sample of the product mentioned above was kept immersed in water at about 65°C. for a few hours and dried in free air for a few days.

(*g*) A third sample of the same product was dried at about 106°C. for three hours in an electrically heated chamber.

X-ray pattern of the product after each of the treatments mentioned above was photographed by exposing a group of about a dozen strands selected from the sample to unfiltered Cu-radiation from a Hadding tube. The strands were held parallel and close to each other with their lengths vertical in a special holder. The width of the bundle was wholly covered by the cross-section of the incident X-ray beam. A slit system consisting of a cylindrical bore about 0·5 mm. in diameter and 4 cm. in length along the axis of a lead rod was used. An exposure of about 8 to 10 hours was necessary for obtaining a good photograph.

As the fibre subjected to treatment (*c*) mentioned above resembled coarse wool the thermal conductivities of this treated fibre and of the original raw jute fibre were also measured[1] using an apparatus used previously by Niyogi and Basu Mallik (1942) and modified recently by Bhattacharyya, P. K., of this laboratory. The results obtained in all these investigations are discussed in the following section.

Results and Discussion.

The X-ray diffraction pattern of the fibre obtained after treatment (*c*) is reproduced in Fig. 2, Plate I, while that for the original raw jute fibre is shown in Fig. 1. The spacings of the planes giving reflections in the equatorial layer line are given in column 4, Table I, these planes being marked A_1, A_2 and A_3 respectively starting from the innermost one. If these are identified with (101), (10$\bar{1}$) and (002) planes respectively, as has been done by previous workers in the case of hydrated cellulose obtained from cotton or ramie, the dimensions of the unit cell given in the same column are arrived at. For the reflections in the other layer lines the relation

$$\frac{4\sin^2\theta}{\lambda^2} = 0{\cdot}018h^2+0{\cdot}01544l^2-0{\cdot}0177hl+0{\cdot}00943k^2$$

is found to be satisfied. The spacings of A_1, A_2 and A_3 and the dimensions of the unit cell observed in the case of dry hydrated cellulose by Andress (1929) are given in column 3, Table I. It can be seen from Fig. 2 that practically the

Table I.

	Water cellulose Sakurada and Hutino.	Hydrated cellulose dried (Andress).	Hydrated cellulose from raw jute (present authors).
A_1	8·98 A.U.	7·32 A.U.	7·96 A.U.
A_2	4·41 ,,	4·45 ,,	4·42 ,,
A_3	3·95 ,,	4·03 ,,	4·03 ,,
a	10·03 ,,	8·14 ,,	8·8 ,,
b	10·3 ,,	10·3 ,,	10·3 ,,
c	9·98 ,,	9·14 ,,	9·5 ,,
β	52°	62°	57° 54′

[1] The authors' thanks are due to Mr. S. K. Mukherjee for carrying out these measurements.

whole of native cellulose in jute fibre is converted into hydrated cellulose by treatment (*c*) in which 30% NaOH solution is used and no tension is applied to the fibre. The results given in Table I further show that this hydrated cellulose when dried in free air has a structure different from that found by Andress (1929) in the case of dry hydrated cellulose obtained from other sources. The structure is also different from that of water cellulose obtained by Sakurada and Hutino (1936) by treating ramie with 18·5% NaOH solution, washing it in water and without allowing the product to dry, as can be seen from column 2, Table I. They pointed out that in the case of the hydrated cellulose obtained by them some water molecules penetrated inside the lattice while the sodium atoms were removed by washing the treated fibre in water, and consequently, the unit cell was larger in the moist state than in the dry state. The moist hydrated cellulose which was called by them 'water cellulose' showed a (101) spacing of 8·98 A.U., but when it was allowed to dry in free air for three days this spacing was reduced to 7·66 A.U. and when dried at 105°C. for about three hours the same spacing was further reduced to 7·32 A.U. It is, however, observed in the present investigation that the (101) spacing in the hydrated cellulose obtained from jute fibre by treatment (*c*) and dried in free air for more than a week is 7·96 A.U. which is greater than 7·66 A.U. observed by Sakurada and Hutino in the case of hydrated cellulose obtained from ramie and dried in free air. When the hydrated cellulose obtained in the present investigation is dried at about 106°C. in an electrically heated chamber, it is found to be partly converted into native cellulose and the spacing of the (101) plane of the remaining hydrated cellulose changes to 7·42 A.U. The pattern obtained after this treatment is shown in Fig. 8, Plate I. The presence of (101) and ($10\bar{1}$) reflections due to native cellulose is clearly seen between the (101) and ($10\bar{1}$) reflections of hydrated cellulose and the widening of the (002) reflection indicates the presence of (002) reflection from native cellulose corresponding to a spacing of 3·92 A.U. superposed on that due to hydrated cellulose. Further treatments (*e*) and (*f*) do not alter the structure of the hydrated cellulose obtained by treatment (*c*) as can be seen from the corresponding patterns shown in Figs. 6 and 7. It has also been found that ageing for three months does not alter the structure (Fig. 9).

When tension is applied to the raw jute fibre during treatment with 30% NaOH solution washed in water and dried in free air, the major portion of native cellulose is converted into hydrated cellulose having the structure given in column 4, Table I, but part of the native cellulose remains unchanged as can be seen from the pattern reproduced in Fig. 3, Plate I. The proportion of such unchanged cellulose observed after treatments (*a*) and (*b*) (with 18·5% NaOH solution) is still larger as can be seen from patterns shown in Figs. 4 and 5. Hence the behaviour of raw jute fibre is different from that observed by Sisson and Saner (1941) in the case of cotton fibre.

The thermal conductivity K of the hydrated cellulose obtained from jute fibre in the present investigation is given in Table II.

TABLE II.

Substance.	K in $\frac{\text{B.T.U. in}}{\text{ft.}^2\,\text{hour.}\,^\circ\text{F.}}$
Hydrated cellulose from jute	0·24
Raw jute	0·28
Pure wool	0·24

It can be seen that the thermal conductivity of hydrated cellulose is smaller than that of raw jute fibre and is the same as that of pure wool. This hydrated cellulose is much softer than raw jute fibre. Hence it is quite suitable for being used as a cheap substitute for coarse wool in making warm fabrics.

Acknowledgments.

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Explanation of Plate I.

Fig. 1. Raw High Top jute fibre.
Fig. 2. Fibre subjected to treatment (*c*), (30% NaOH without tension).
Fig. 3. Fibre subjected to treatment (*d*), (30% NaOH with tension).
Fig. 4. Fibre subjected to treatment (*a*), (18% NaOH without tension).
Fig. 5. Fibre subjected to treatment (*b*), (18% NaOH with tension).
Fig. 6. Product of treatment (*c*) subjected to treatment (*e*), (washed in 1% NaOH solution).
Fig. 7. Product of treatment (*c*) subjected to treatment (*f*), (steeped in water at 65°C.).
Fig. 8. Product of treatment (*c*) dried at 106°C. for three hours.
Fig. 9. Product of treatment (*c*) dried in free air for three months.

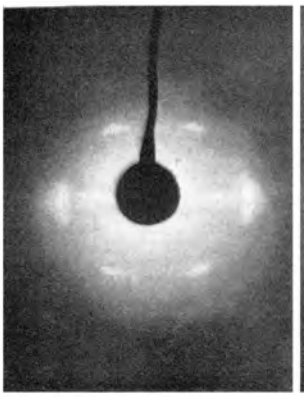


FIG. 1.

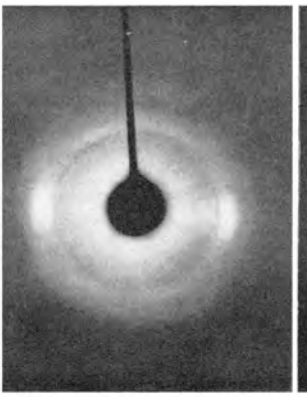


FIG. 2.

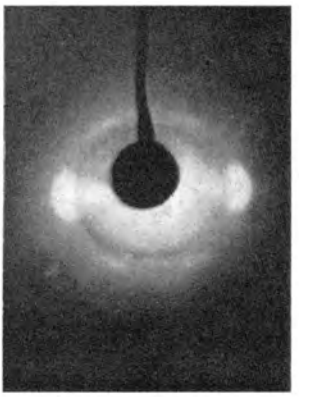


FIG. 3.

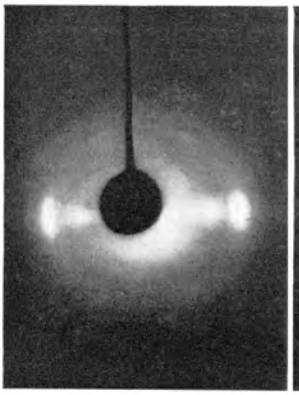
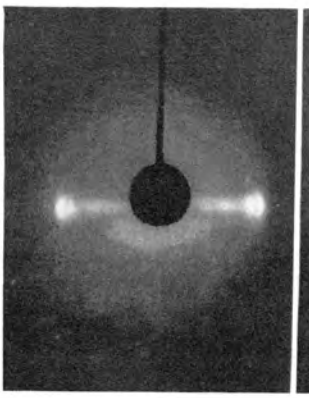


FIG. 4.



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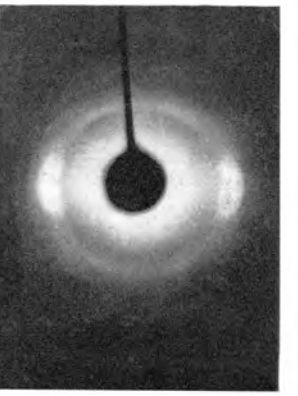
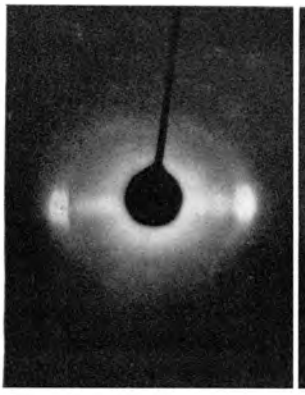


FIG. 6.



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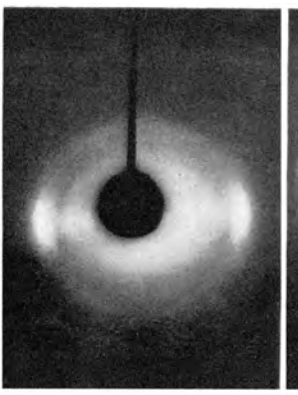

FIG. 8.

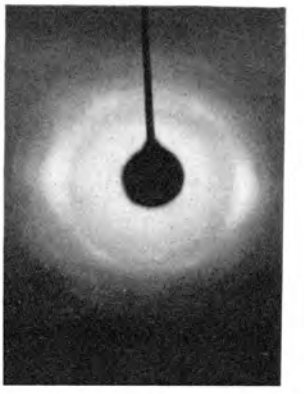

FIG. 9.