

THE ORIENTATION AND SIZE OF MICELLES IN JUTE FIBRE.

By S. C. SIRKAR, *D.Sc., F.N.I.*, and N. N. SAHA, *M.Sc.*, Department of Physics,
University College of Science, Calcutta.

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ABSTRACT.

The percentages of micelles oriented in directions making different angles with the axis of the fibre have been determined in the case of high and low quality Tossa and White jute of known strength. It is found that although the height of mode of the curve obtained by plotting percentage of oriented micelles against angle between direction of orientation and axis of the fibre is larger in stronger fibre, the heights are not actually proportional to the strengths, because the height observed in the case of the bleached fibre is not so very small as to signify the negligible strength of the fibre which is actually observed. The size of the micelles has also been determined using Laue's equation for orthorhombic lattice in the case of high and low quality Tossa and high quality Bogi jute before and after the removal of lignin. It is observed that the length of the micelle is larger in Tossa jute than in Bogi jute which is weaker than Tossa jute, but the length is about one-tenth that found in ramie. It is concluded that the length of micelles determines partly the strength of these fibres.

INTRODUCTION.

It was pointed out in a previous paper (Sirkar, Saha and Rudra, 1944) that the X-ray diffraction patterns yielded by different qualities of jute fibre are not exactly identical in all respects although the spacings of planes are found to be the same in the different qualities studied. It was observed that the width and length of the spots were not the same in patterns due to different qualities of the fibre and also the background was slightly different in some cases, e.g., a faint ring passed through the 002 reflections in some low quality fibres, but it was much weaker in the case of other qualities. It was also pointed out that since the width and length of the spots are connected respectively with the size and orientation of the micelles along the fibre axis, it might be possible to correlate some of the physical properties of the fibre with the size and orientation of the micelles if these were determined quantitatively. The object of the present work was to determine these properties of the micelles quantitatively in a few qualities of the fibre and to find out whether these could be correlated with the strength of the fibre.

EXPERIMENTAL.

In order to determine the orientation of the micelles in different qualities of the fibre, the intensity of the (002) reflection along the arc of the circle passing through this reflection and with its centre at that of the central spot of the diffraction pattern was measured at different points at intervals of five degrees from the midpoint of the arc. The usual method of photographic photometry was used for this purpose. In order to obtain the microphotometric records of the blackening of different points at known angular distances, the film was mounted on a stage of special type in the self-recording (Kipp & Zonen type) microphotometer in such a way that the centre of the central spot in the film coincided with the centre of a vertical graduated circle. In order to convert the blackenings into intensities, intensity marks were obtained on a film with different known times of exposure and keeping the current in the X-ray tube absolutely steady. The intensities were assumed to be proportional to the times of exposure and with the help of microphotometric records of these blackenings a blackening-log intensity curve was drawn. It was found later that a second such curve for the low intensity region was also required for measuring the size of the micelles and accordingly a second curve was drawn with lower intensities.

The intensity of the background on the film was determined at each point of the (002) reflection studied and it was subtracted from the observed total intensity of the (002) reflection at the particular point. In this way the intensities at the midpoint of the arc and at angular distances of 5° , 10° , 15° , 20° from the midpoint were determined and these were converted into the corresponding intensities taking that at the midpoint as 100. A curve was then drawn with angular distances from the midpoint as abscissa and corresponding intensities as ordinates in the case of each quality studied. The curves obtained for the different qualities studied are shown in Figs. 1-5.

In order to compare with each other the results obtained with different qualities from different photographs of the patterns due to these qualities of the fibre the percentages of orientation of the micelles along different directions were calculated in each case and these were plotted against the angles made by the micelles with the fibre-axis. For this purpose the whole area enclosed between the 'angle of inclination' axis and the curve was taken as 100 in each case. The midpoint of the (002) reflection was found to correspond to the axis of the fibre and therefore the angular distance of any point in the (002) reflection from its midpoint along the (002) ring gave the angle of inclination of the axis of micelles responsible for the intensity of scattering at that point. In this way the curves shown in the figures 6-10 were obtained. Some of the photographs from which these data were obtained are reproduced in Plate I. The photographs were taken with a very fine slit of 0.5 mm. bore and 5 cm. in length. In order to find out whether there is any correlation between either the orientation of the micelles or their size and the strength of the fibre, the strength of samples cut from the same strands from which the fibres irradiated were taken was measured * in the case of a few samples.

In order to determine the size of the micelles Laue's method (Laue, 1926) was employed. The angular distance B between the position of maximum intensity and that of half the maximum intensity was determined for reflections from the three planes (002), (120) and (031); and the values of m_1 , m_2 and m_3 were determined from the relation,

$$B \cos \frac{\chi_m}{2} = 0.9 \lambda \left(\frac{\frac{h^2}{m_1^2 a^4} + \frac{k^2}{m_2^2 b^4} + \frac{l^2}{m_3^2 c^4}}{\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}} \right)^{\frac{1}{2}} \dots \dots \dots (1)$$

where χ_m is the angle between the incident beam and the direction of maximum intensity in the beam reflected from the plane h, k, l and m_1, m_2, m_3 are pure numbers indicating how many times the edges a, b, c of the unit cell are repeated along the respective axes in the micelle. The value of the angle β of the lattice has thus been assumed to be 90° although it is actually 84° . The term containing higher power of $\cos (\chi_m/2)$ has been neglected, in equation (1). In calculating the half-widths of the reflections from the planes mentioned above care was taken to take into account the intensity of the background. As it is intended to carry on this work by the new method developed by Hosemann (1939a), the sizes of the micelles were determined only in a few qualities of the fibre. Unfiltered Cu radiation and the narrow slit system mentioned earlier were used. No correction was, however, made for the absorption in the sample. The results obtained are discussed in the following section.

RESULTS AND DISCUSSION.

PART I.—Orientation of micelles in jute fibre.

The percentages of micelles oriented in directions making angles 0° , 5° , 10° , 15° and 20° were calculated for untreated high and low quality Tossa, high and low quality White and high and low quality Bogi jute. The intensity distribution along

* The authors are indebted to the Director, T.R.L., Indian Central Jute Committee, for kindly getting these measurements made in his laboratory and supplying the data.

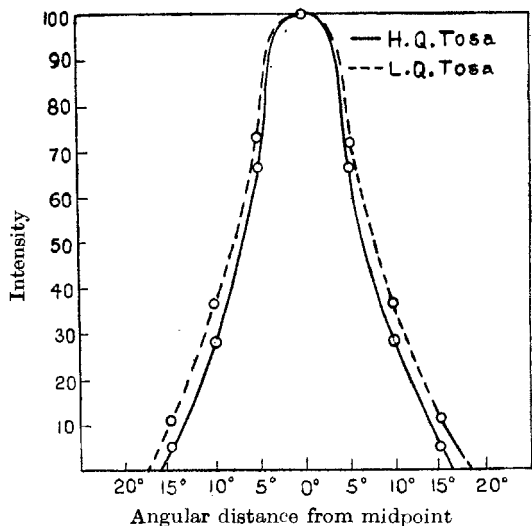


FIG. 1.

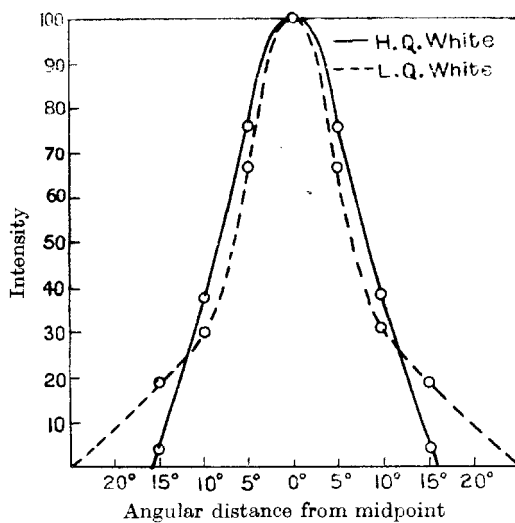


FIG. 2.

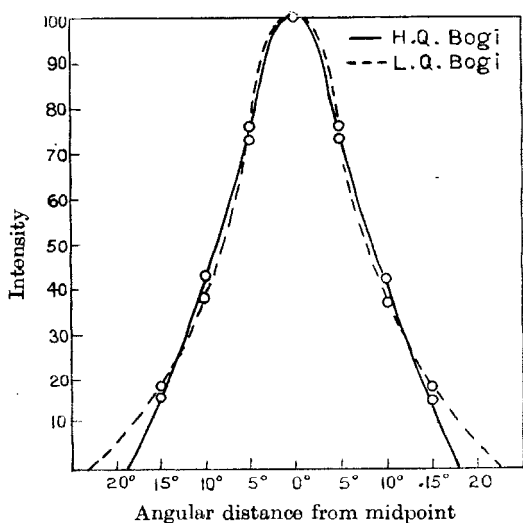


FIG. 3.

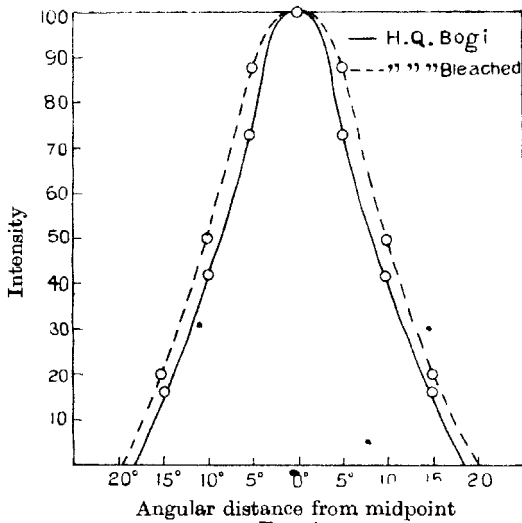


FIG. 4.

the length of (002) reflections is shown in figures 1, 2 and 3 respectively and the percentages of orientation of the micelles are plotted in figures 6, 7 and 8 respectively. It will be seen from these latter figures that in the case of each class the height of mode is less in the case of low quality than that for the high quality. This does not signify, however, that the heights of the mode obtained for different classes are strictly proportional to the strength of the fibre. In Table I the height of the mode and the strength of the fibre in terms of (breaking load)/(mass per unit length) are given for high and low qualities of two well-known classes of jute fibre, viz., Tossa (olitorious) and White (capsularies). Curve III in fig. 5 represents the intensity distribution observed in the case of high quality white jute treated with 10% phenol and formalin solution as mentioned in the previous paper (Sirkar, Saha and Rudra, 1944). Since it was observed that the cellulose was converted into cellulose II, the height of mode was not determined in this case.

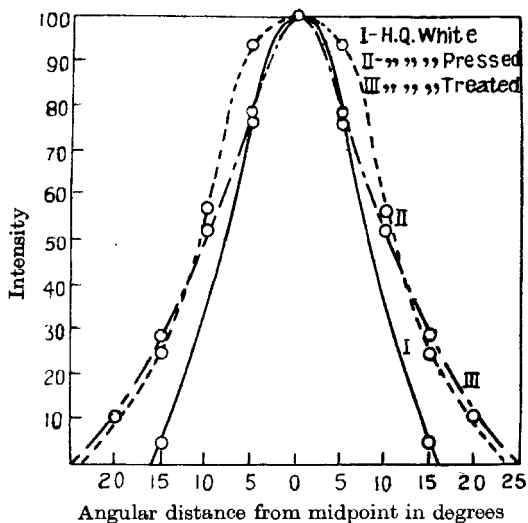


FIG. 5.

TABLE I.

Type of jute fibre.	Height of mode.	(Breaking load)/(mass per unit length).
High quality Tossa	33	4.43 ± 0.009
Low quality Tossa	28.3	2.90 ± 0.146
High quality White	28.6	2.79 ± 0.115
Low quality White	27.2	2.05 ± 0.098

From Table I it can be seen that the height of mode in the case of high quality Tossa is the greatest of all these heights observed in the four qualities. The strength observed for high quality Tossa is also larger than that of any of the other three qualities. There seems to be some relation between the strength and the orientation of micelles. It should be mentioned here, however, that the results given in

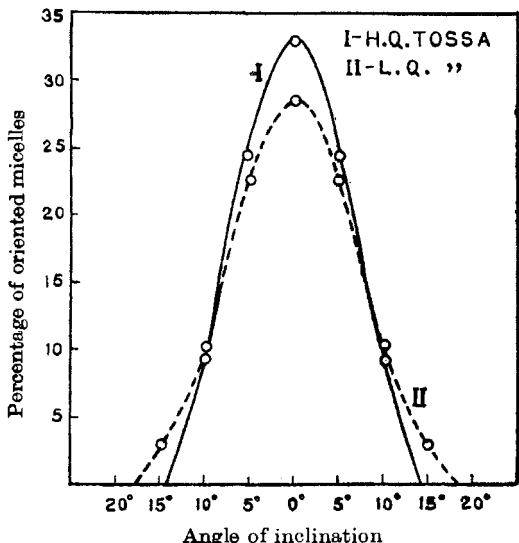


FIG. 6.

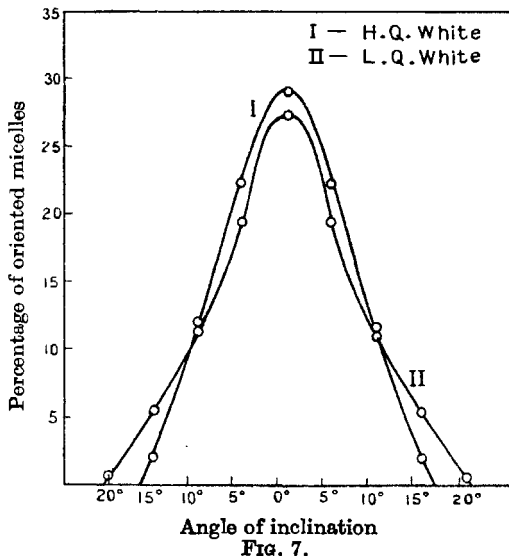


FIG. 7.

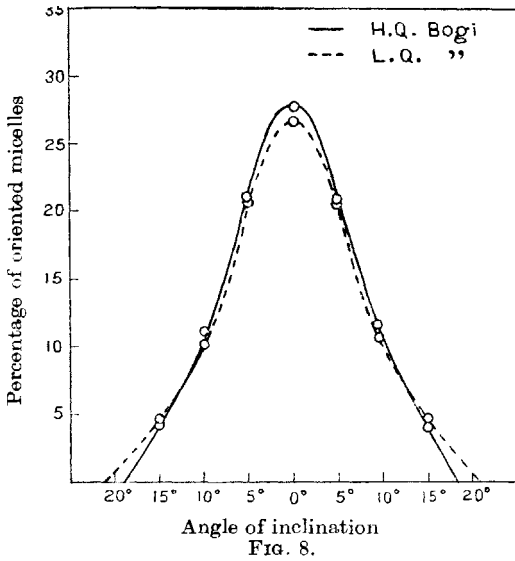


FIG. 8.

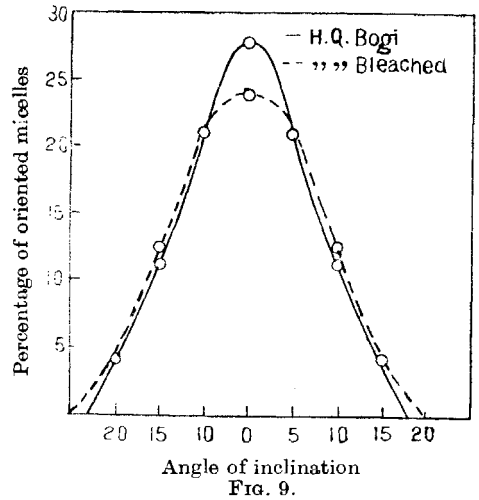


FIG. 9.

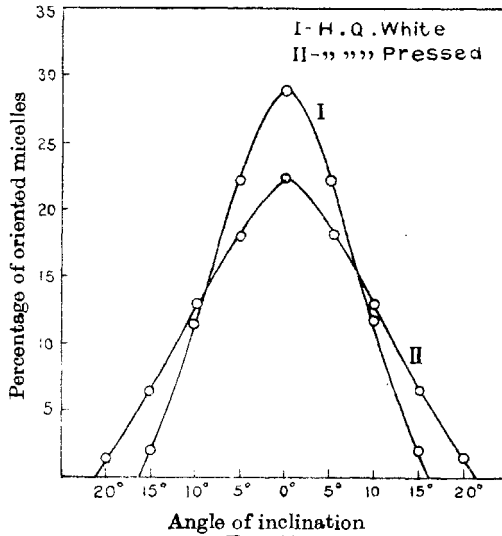


FIG. 10.

Table I are not of statistical nature, because they were not obtained for a very large number of samples of same quality. These statistical results will be reported in a future communication.

The results given in figures 5, 6, 9 and 10 relate to some qualities of fibre subjected to some particular treatment. Although elaborate investigations of this type on treated fibres are being carried out, the results of which will be reported in a future communication, these results obtained in the case of two types of treatment are included here in order to understand the significance of the results obtained for the untreated fibre. It can be seen from Fig. 9 that the height of mode for bleached fibre is less than that for unbleached one. But since the tensile strength for the bleached fibre in the wet condition is almost negligible the difference in heights of mode cannot be solely responsible for the difference in the strengths of the fibre in the two states. The fact that the removal of lignin which acts as a cementing material by which the micelles are attached to each other diminishes the strength enormously indicates apparently that the presence of lignin is responsible for the

strength of the fibre. On the other hand, it is well known that the percentage of lignin in ramie fibre is very small (Norman, 1936) while its strength in the wet condition is larger than that of bleached jute fibre in the same condition. This indicates that besides the presence of lignin as a cementing material there must be some other factor which increases the strength of such fibres. This point has been discussed in the next section.

Fig. 10 shows the orientation of micelles in some quality of white jute and that of the same fibre subjected to high mechanical pressure under a screw press. It can be seen that the orientation of the micelles is disturbed by the mechanical pressure and the height of mode is diminished considerably. The strength of the pressed fibre has been actually measured and found to be much smaller than that of the original fibre.

PART II.—Size of the micelles.

Owing probably to small experimental errors which could not be avoided in the measurement of intensities, the values m_1, m_2, m_3 , the number of times the unit cell is repeated along a, b and c -axis respectively as calculated from Laue's equation, were sometimes found to be fractional and the nearest integral numbers were taken in those cases. These values were calculated only in the case of high and low quality Bogi, low quality Tossa and high quality Bogi jute bleached by ClO_2 solution in water. The results are given in Table II.

TABLE II.

Type of jute.	Half-width in radian.			m_1	m_2	m_3
	(002)	(031)	(120)			
High quality Bogi	·0197	·016	·019	6	9	9
Low quality Bogi	·023	·019	·021	6	8	8
Low quality Tossa	·025	·020	·023	6	7	7
High quality Bogi (bleached)	·0257	·026	·027	5	6	7

It can be seen from Table II that the length of micelle is only at most nine times the primitive translation along the b -axis. The values of m_1, m_2, m_3 were found previously by Banerjee and Ray (1941) in the case of a particular variety of jute (X-early) in the delignified condition to be 3, 6 and 5 respectively. The value of m_2 found by them agrees with the results obtained in the present investigation in the case of high quality Bogi jute in the bleached condition. The value of m_2 , however, is not constant in different qualities and it is large in fibres having greater strength.

The size of the micelles in cellulose in other native fibres found by Mark and Meyer (1929) and also by Hess *et al.* (1931) in the case of ramie is much larger than that found in the case of jute fibre in the present investigation. According to those authors the micelles in ramie are 60–70Å wide and at least 1000Å long. In the case of jute fibre the micelles are at most about 100Å long, but their width is almost the same as that of micelles in other fibres. Probably this explains the difference in the strength of the bleached jute fibre and ramie; as the micelles are about ten times longer in ramie than in jute fibre even the absence of cementing material does not make the strength of ramie as small as that of bleached jute fibre in wet condition. Although this method of finding the size of the micelles is not very accurate as pointed by Hosemann (1939) the results obtained by him in the case of other fibres by the new method developed by him are not in disagreement with those obtained by Laue's method (Hosemann, 1939b). Hence from the results obtained in the present investigation it can safely be concluded that the length of the micelles in jute fibre is much smaller than that in ramie and *it is the length of the micelle which determines partly the strength of these fibres.* The length of the micelle,

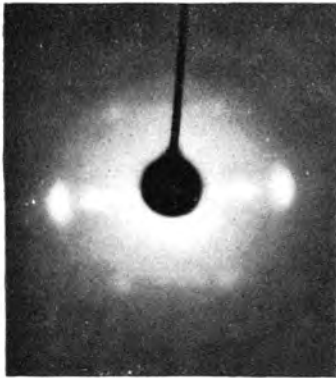


FIG. 11. High quality Tossa.

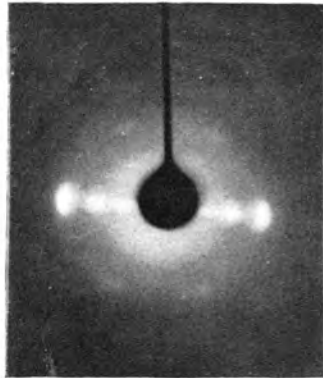


FIG. 12. Low quality Tossa.

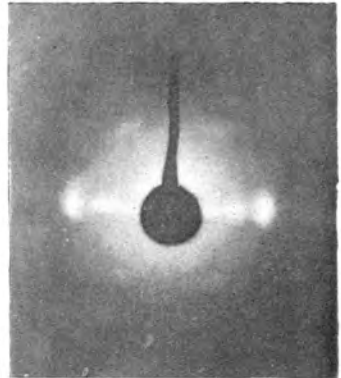


FIG. 13. High quality White.

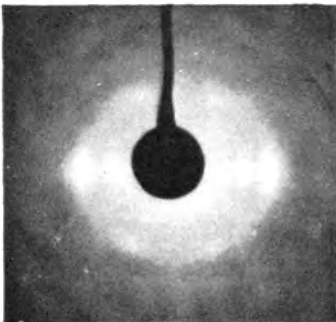


FIG. 14. Low quality White.

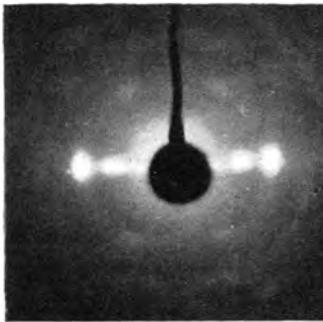


FIG. 15. High quality Bogi.

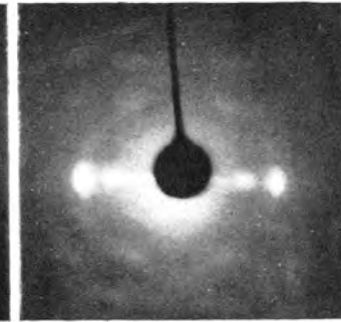


FIG. 16. High quality Bogi (bleached).

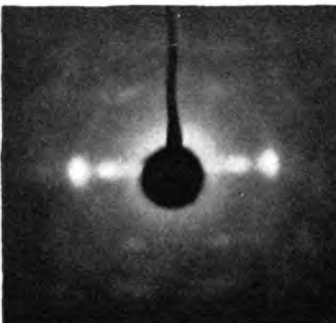


FIG. 17. Low quality Bogi.

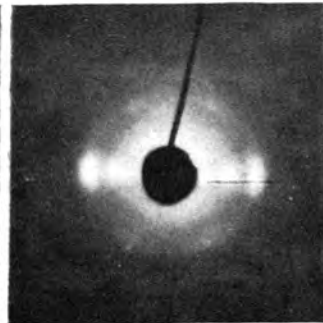


FIG. 18. High quality White (pressed).

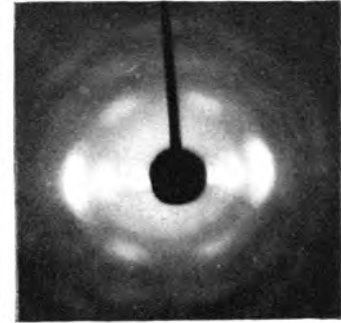


FIG. 19. High quality White treated in phenol-formalin.

X-ray patterns due to jute fibre.

however, seems to be diminished when the fibre is bleached. This question is being investigated more thoroughly and the results will be reported in a later communication. These results are significant for this reason that in future any attempt at improving the strength of the fibre will probably reduce to the biological problem of increasing the length of the micelles in jute fibres during the growth of the plant. If this problem can be solved bleached jute in wet condition will not be as weak as it is now.

ACKNOWLEDGMENT.

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REFERENCES.

- Banerjee, K. and Ray, A. K. (1941). The structure of jute fibre by X-ray diffraction method. *Proc. Nat. Inst. Sc. India*, **7**, 377.
- Hosemann, R. (1939a). Theory of X-ray Scattering by groups of particles. *Z. Physik*, **113**, 751.
- Hosemann, R. (1939b). New X-ray method of determining submicroscopic fine-structure of substances. *Z. Physik*, **114**, 133.
- Hess, K., Trogus, C., Akim, L. and Sekurade, I. (1931). Morphology and chemistry of cellulose fibres. *Ber.*, **64**, 408.
- Laue, M. Von (1926). Lorentz-factor and Intensity distribution in Debye-Scherrer ring. *Z. Krist.*, **64**, 115.
- Mark, H. and Meyer, K. H. (1929). Over the structure of crystallised portions of cellulose. *Z. Physik Chem. (B)*, **2**, 115.
- Norman, A. G. (1936). Composition of vegetable fibres with particular reference to jute. *Biochem. J.*, **30**, 831.
- Sirkar, S. C., Saha, N. N. and Rudra, R. M. (1944). X-ray Analysis of jute fibre of different qualities under various conditions. *Proc. Nat. Inst. Sc. India*, **10**, 325.