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A COMPARATIVE STUDY ON SOME CHEMICAL CHARACTERISTICS OF JUTE AND *HIBISCUS* FIBRES

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A comparative study has been made on the chemical composition as well as on the characteristic functional groups of jute and fibres of the *Hibiscus* group, namely Mesta, Bimli and Kenaf, with a view to differentiating these fibres on the basis of chemical properties. From analyses of a number of different samples it is confirmed that the yield of lignin or holocellulose, obtained from jute, is significantly different from those of other allied fibres. Although a wide variation is observed in the carboxyl, existing in different states, *i.e.* free and combined, in jute and *Hibiscus* fibres, no definite conclusion can be drawn from the results obtained. The acetyl content, total acidity and the methoxyl associated with the carbohydrate fraction of jute are significantly lower than those of fibres of the *Hibiscus* group, and these values may be taken as additional criteria for differentiating jute and other allied fibres. Further, since the acetyl content and the total acidity of *Corchorus capsularis* fibre (White jute) are considerably high compared with those obtained from *Corchorus olitorius* fibre (Tossa or Daisee jute), it seems possible that from the variations in these values the two botanical species of jute fibre can be differentiated from each other.

A number of bast fibres, closely allied to jute which belongs to *Corchorus* species, can be successfully processed on jute mill machinery. The most common of these substitute fibres are derived from the *Hibiscus* plant, usually from the species known as *H. cannabinus*, and are described by a variety of different names but, in India, are usually known as either Mesta or Bimli and in other countries as Kenaf. The quality of *Hibiscus* fibres, so far as the spinning property is concerned, varies similar to that of jute, but generally speaking, these fibres are coarser and more brittle than jute and, as a result, are less suitable for the spinning of fine yarns.

The principal characteristic feature of jute and the *Hibiscus* fibres is, that although cellulose forms the main structural component, it does not occur in the pure state but is associated predominantly with lignin and hemicelluloses. The chemical compositions as well as the physical and chemical properties of these fibres are so akin to each other that it is extremely difficult to differentiate them. Since the *Hibiscus* fibres are coarser and stiffer than jute, differentiation of these fibres is usually made by visual examination and by feel. The present work was initiated with the object of differentiating these fibres not only on the basis of chemical composition but also on the characteristic functional groups which until recently have been considered of little importance.

It has been reported by Powrie and Speakman (*J. Text. Inst.*, 1943, **34**, T77) and more recently by Das, Mitra and Wareham (*ibid.*, 1951, **42**, T181) that jute contains a higher percentage of lignin in comparison with Mesta and Bimli while the variation in the other main constituents, *i.e.*, α-cellulose, pentosan calculated as xylan, polyuronide and hexosan, of jute from those of other allied bast fibres is of such a small order that it can be regarded as insignificant. The procedure employed by Das *et al.* (*loc.cit.*) for the determination of lignin is not very accurate, since the lower yield obtained by this method has been found to be due to a partial solubility of lignin in the reagent used

for its isolation (Sen Gupta and Callow, *ibid.*, 1949, **40**, T650; Macmillan, Sen Gupta and Roy, *ibid.*, 1952, **43**, T103). A more correct value, however, is expected to be obtained by the method suggested by Sen Gupta and Callow (*loc.cit.*). It was considered of importance therefore to re-examine the lignin content of these fibres with a more accurate procedure in order to ascertain whether the difference between the values of lignin in jute and other allied fibres was really significant.

In addition to the major constituents, jute fibre contains a number of characteristic functional groups, namely carboxyl, acetyl and methoxyl, which are generally regarded as of minor importance, and until recently, have received no serious attention. More recent investigations (Bhattacharjee and Callow, *ibid.*, 1952, **43**, T53; Macmillan and Sen Gupta, this *Journal*, 1952, **29**, 737) have shown that the acidity of jute is due to both uronic carboxyl and acetyl groups. Uronic carboxyl groups have been found to exist in three different states, a small portion (3 m.e./100 g., approx.) being in the free state and the major portion in the combined form, of which a part is occupied with cations (9 m.e./100 g.) and the rest (12 m.e./100 g.) in the ester combination with the hydroxyl group of lignin (Sarkar, Chatterjee and Mazumdar, *J. Text. Inst.*, 1947, **38**, T318). The acetyl, which amounts to approximately 3.5% on jute, occurs in combination with the hydroxyl groups, mainly, if not entirely, associated with the carbohydrate fraction (Macmillan and Sen Gupta, *loc.cit.*) and is easily hydrolysed by the action of dilute caustic soda solution at room temperature. It has been demonstrated that of the methoxyl groups in jute, the major portion is associated with the lignin residue, the remainder being attached to the hemicellulosic fraction (Sen Gupta and Callow, *J. Text. Inst.*, 1951, **42**, T382), possibly the uronic acid residue (Sarkar, Mazumdar and Pal, *Textile Res. J.* 1952, **22**, 529). Although a considerable amount of work has been carried out on the functional groups in jute, no data appear to be available on the occurrence of these groups in other allied fibres, and there is a possibility that such groups will vary not only in amounts but also in proportions, existing in combination with different fibre components. In the present investigation therefore, attention was particularly directed towards the relative proportions as well as the mode of occurrence of these groups with a view to obtaining some additional evidence for differentiating jute more positively from the fibres of the *Hibiscus* group.

EXPERIMENTAL

Three different samples of jute, three of Mesta, two of Bimli and one of Kenaf were employed in the present investigation. The White and Tossa varieties of fibre, belonging to *C. capsularis* and *C. olitorius* species respectively, were of good quality jute obtained from Pakistan, while the samples of Daisee jute (also *C. olitorius*) and those of Mesta were procured from different parts of West Bengal; Bimli fibres were obtained from Madras and Kenaf, which was the only sample available, was imported from Australia. Representative samples, obtained from the middle portions of the strands of different fibres, were de-waxed by extracting the materials with a mixture of alcohol-benzene (1 : 2), washed successively with alcohol and water, and air-dried. Experiments were conducted with the air-dried samples and the analytical results expressed on the oven-dry (at 105°) weight of the fibres.

Lignin was determined by the 72% sulphuric acid procedure, as modified by Sen Gupta and Callow (*J. Text. Inst.*, 1949, **40**, T650).

For isolation of the holocellulose fraction, the fibre sample was subjected to two successive treatments with hot (98°) sodium chlorite solution (0.7%, using a liquor ratio 50:1) maintained at p_H 4, the duration of each treatment being 2 hours, followed by treatment with 2% sodium bisulphite.

The free carboxyl and that occupied with cations were determined by the procedure suggested by Sarkar, Chatterjee and Mazumdar (*loc. cit.*); the uronic carboxyl occurring as ester was obtained from the measure of the increased acidity of the fibre after a hydrolytic treatment with caustic soda (Macmillan and Sen Gupta, *loc.cit.*).

The acetyl content was ascertained by hydrolysing the material with caustic soda and then measuring the steam-volatile acetic acid by the procedure followed by Macmillan and Sen Gupta (*loc.cit.*).

The total acidity (*i.e.* both due to uronic and acetic acid residues) was ascertained directly by allowing the fibre to react with an excess of caustic soda and estimating the amount of alkali required for neutralisation (Macmillan and Sen Gupta, *loc.cit.*).

The methoxyl content was determined by a modified Zeisel method, as previously used by Sen Gupta and Callow (*loc. cit.*).

The values of lignin, uronic carboxyl existing in different states, acetyl and the total acidity of the various samples analysed are recorded in Table I.

TABLE I*

Lignin, acetyl and uronic acid contents, and total acidity of jute and Hibiscus *fibres.*

Material.	% Lignin.	Uronic carboxyl (m.e./100 g.). Free.	Occupied with cation.	Occurred as ester.	%Acetyl.	Total acidity (m.e./100 g.).
Jute (White)	14.29	2.03	8.12	10.64	3.80	113.1
,, (Tossa)	13.90	...	...	...	3.17	100.0
,, (Daisee)	12.80	...	...	...	3.25	101.7
Mesta (Sample 1)	9.49	1.63	5.52	12.09	5.21	143.2
,, (Sample 2)	10.47	...	...	...	5.16	139.2
,, (Sample 3)	9.70	...	...	...	5.09	138.8
Bimli (Sample 1)	9.88	1.69	6.91	11.86	5.09	138.7
,, (Sample 2)	10.87	...	...	...	5.26	146.3
Kenaf	10.80	2.11	15.14	9.25	4.46	127.8

* Results are expressed on oven-dry de-waxed fibre.

DISCUSSION

In addition to the results obtained in the present investigation, analyses of various samples of jute carried out in the Ijmari laboratories over a period show that the lignin content of jute generally varies from 12 to 14.5%, as determined by the modified 72% sulphuric acid procedure, some difference in the values being noted on changing the method of estimation. It is seen from the results in Table I that the lignin content of the other allied bast fibres studied lies within the range of 9.5 to 10.8%. Jute therefore

appears to contain a higher percentage of lignin, compared with fibres of the *Hibiscus* group, and the difference in the values for jute and those of the other bast fibres examined can be considered as significant.

The uronic acid content of jute, Mesta and Bimli is approximately the same, while that of Kenaf is appreciabely higher. A wide variation has been observed in the values of carboxyl occurring in different states and no definite conclusion can, however, be drawn from the results obtained.

Besides the results recorded in Table I, various samples of White, Tossa and Daisee jute, analysed in these laboratories, demonstrate that the acetyl content and the total acidity of jute are always within the limit of 3.1 to 3.8% and 100 to 114 m.e./100 g. respectively, the corresponding values for *Hibiscus* fibres being 4.5 to 5.2% and 128 to 146 m.e./100 g. Both the acetyl content as well as the total acidity of the other allied bast fibres are significantly higher than those obtained from jute, and these values may therefore be taken as additional criteria for differentiating jute from fibres of the *Hibiscus* group. The difference in the total acidity of these fibres, however, appears to be mainly due to variation in the acetyl content of the individual fibre samples. Further, the values of acetyl content and total acidity of White jute (*C. capsularis*) have been found to vary from 3.6 to 3 8% and 110 to 114 m.e./100 g. respectively, while those obtained from other variety of jute (*C. olitorius*) always lie within the range of 3.1 to 3.25% and 100 to 102 m.e./100 g. It seems possible therefore that from the variations of these values jute fibre belonging to two botanical species can be differentiated from each other.

Holocellulose, isolated from any of the fibres by one treatment with sodium chlorite, as recommended by Chattopadhyay and Sarkar (*Proc. Nat. Inst. Sci., India*, 1946, **12**; 23), was found to be contaminated with 1.8 to 2.0% lignin (on the weight of the holocellulose). Two such treatments, however, effect a considerable reduction in the lignin content, as shown in Table II, without any undue loss in the hemicellulose content, as is evident from the summation of lignin and holocellulose of the fibre approaching very near to 100 (Tables I and II). Any attempt to reduce the lignin content still further, by increasing the number of treatments, resulted in a marked elimination of hemicellulosic constituents. In the present work therefore the holocellulose content was determined by two treatments of acidic sodium chlorite.

The methoxyl content was estimated on individual fibres as well as on lignin and holocellulose, isolated from the respective samples. From the value of methoxyl of the isolated lignin and the lignin content of the corresponding fibre, the amount of methoxyl associated with the lignin *in situ* (expressed as a per cent on fibre) was obtained. A theoretical value of the methoxyl associated with the carbohydrate fraction was then calculated from the difference of the methoxyl content of the fibre and that of the lignin *in situ*. This value, however, will tend to be higher if lignin loses some of its methoxyl groups during the process of isolation (Dorée, "The Methods of Cellulose Chemistry", 1947, p. 366). In order to obtain the methoxyl attached to carbohydrate from a direct estimation of holocellulose, a correction was applied for the presence of residual lignin, an

assumption being made that the methoxyl content of the residual lignin was identical with that of the lignin isolated from the untreated fibre. This value is also expected to be deviated from the correct methoxyl content of carbohydrate by a number of factors. For example, a positive error is likely to be involved owing to the possibility of holocellulose being contaminated with the lignin degradation products containing methoxyl (Sen Gupta and Callow, *J. Text. Inst.*, 1951, **42**, T382) and a negative error if a partial demethylation of either the residual lignin or the carbohydrate fraction or both, occurs during delignification. Since it is not known which one of the values, *i.e.* the calculated and that obtained by the direct estimation of holocellulose, will afford a more correct indication of the actual methoxyl content of the carbohydrate fraction, both values of the *Hibiscus* fibres have been compared with those obtained from jute. A summary of the results is shown in Table II.

TABLE II*

Holocellulose content, and associated methoxyl in lignin and carbohydrate fractions of jute and Hibiscus *fibres.*

Material.	Holocellulose.	Lignin in holocellulose.	%OMe associated with lignin on		%OMe associated with carbohydrate.	
			** lignin.	fibre.	Calc.	Found
Jute (White)	85.25%	0.54%	19.25	2.75	0.89	0.95
Mesta (Sample 1)	91.07	0.54	21.19	2.01	1.66	1.27
Bimli (Sample 1)	90.67	0.50	19.03	1.88	1.48	1.34
Kenaf	89.75	0.55	18.83	2.03	1.36	1.26

* Results are expressed on oven-dry de-waxed fibre.

** Results are expressed on oven-dry lignin.

As expected from variations in the lignin contents of jute and other allied fibres, the results in Table II show that the difference in the yield of holocellulose of these fibres is also significant. The methoxyl content of lignin (as a per cent on lignin), isolated from different fibres, is more or less the same. The observed variations in the values of methoxyl in lignin (per cent on fibre) are obviously due to difference in the lignin content of the individual fibres. Analyses of various samples of jute reveal that methoxyl in carbohydrate varies within the limit of 0.85 to 0.95% while in the case of *Hibiscus* fibres the values have always been found above 1.25%. The methoxyl associated with the carbohydrate fraction of jute therefore seems to be appreciably lower than that of allied fibres, the difference being more marked in calculated values.

It appears possible from the results † described that jute can be differentiated from fibres of the *Hibiscus* group by the variation in the values of lignin or holocellulose, acetyl, total acidity and the methoxyl associated with the carbohydrate fraction.

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