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SCIENTIFIC GRADING OF JUTE

Part IV.—Determination of Copper Number and Nitrogen Content of Different Grades of Jute

M. MANZOOR-I-KHUDA, A.S.M. SERAJUDDIN, M.M. ANWARUL ISLAM, MD. NURUL AMIN, M. Bose, Abdul Aziz Khan and Md. Shahjahan

Department of Chemistry, Technological Research Board, Jute Research Institute, Dacca 15

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The copper number of different grades of white (Corchorus capsularis) and tossa (Corchorous olitorus) jute increases from higher to the lower grades of jute in each variety. Tossa has comparatively higher copper number value than white jute. Results show better grades have lower copper number values thus indicating that the reducing end groups are present in comparatively larger number in the inferior grades of jute. Nitrogen content of all varieties of jute appears to vary between 0.12 to 0.26% and do not show any clear distinction as to the grade or variety of jute.

Our previous communications1-3 on the scientific grading of jute indicated that the lustre, cellulose content and lignin content of different grades of white and tossa variety of Pakistani jute differ significantly with the difference in grades. Some of the physical characteristics of jute e.g. tensile strength of spun yarn, fibre bundle strength, tenacity of single fibre, tenacity of single fibre at a fixed percentage extension, fineness and bursting energy are now being investigated to establish if some of these can serve as a scientific basis for distinguishing between different grades of jute. Present investigation deals with chemical analyses of some of the measurable factors applicable to jute namely, copper number4,5 and nitrogen5,7 content.

Copper number represents the reducing power of a cellulosic material due to the aldehydic end groups of the molecular chain. The values are of a low order with normal cotton cellulose, and any change in the cellulose, resulting from heat, action of acids, action of alkalis and action of oxidative or hydrolytic agents, is normally well marked by an increase in copper number. One cellulose may differ substantially in its analytical characteristics from another kind. This deviation of cellulosic fibre may also result from its growing conditions, method of its isolation, and the action of physical and chemical agencies upon it. Such changes are due to alterations in the structural balance of cellulose chain along with shortening the average chain length. Oxidative or of hydrolytic agent causes the fibre to develop reducing properties within it. The determination of this reducing power by estimation of copper number is, therefore, of considerable significance for determining specification of cellulose and also of the fibre. The present investigation was carried out to determine the copper number of different grades of Pakistani white and tossa jute.

The copper number of the different grades of Pakistani jute-white (Corchorus capsularis) and tossa (Corchorus olitorius)-was determined by conventional chemical methods. The tossa variety has been found to have higher copper number than the white variety. The top grades of both the varieties have lower copper number than the respective lower grades up to grade D. The E grades of both the varieties, show a lower copper number value compared to the higher grade D. This is possibly due to the fact that the bottom grades contain coarser and more matured fibres with higher percentage of bonded end groups, additionally, grade E appears to be a mixture of rejects from all grades, thus reducing the analytical significance of its copper number value. The bottom portion of jute in each grades of the both varieties of jute has higher copper number than of its top portion.

Besides cellulose, lignin, hemicellulose and pectin, jute fibres also contain small amount of nitrogenous substances. Present analysis indicates that the very small amount of nitrogenous substances present does not play any significant role in determining the quality or grades of jute fibres.

Experimental

The pucca graded bales were purchased through the Pakistan Jute Association.

Each jute sample was cut into bottom and top portions and each portion cut to fine smaller pieces. These fragmented jute samples were

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then dried in an oven at 110°C to a constant weight and stored for the following treatment.

Determination of Copper Number.—The following solutions were prepared: (A) Copper sulphate (CuSO₄,5H₂O₆) 100 g; distilled water to 1000 ml. (B) Sodium bicarbonate 50 g; sodium carbonate (anhydrous) 130 g; distilled water to 1000 ml. (C) Iron alum 100 g; concentrated sulphuric acid 140 ml; distilled water to 1000 ml. (D) Potassium permanganate solution of approximately N/25strength (1.008), 1 ml being equivalent to 2.57 mg of reduced copper. (E) 2N Sulphuric acid.

Jute sample (0.5 g) was accurately weighed and taken in a 250-ml conical flask. Solution B (95 ml) was taken in another conical flask and to it added 5 ml of solution A. The resulting solution was boiled and then poured over the cellulosic material, air bubbles being expelled carefully with the help of a glass rod. A pear-shaped glass bulb was then fitted to the conical flask and the

conical flask was immersed up to the neck in a boiling water bath for 3 hr. The flask was then removed from the water bath, cooled in tap water and finally filtered through a suction funnel. The cellulose impregnated with red cuprous oxide was washed thoroughly with hot distilled water and 1% sodium carbonate solution and again with distilled water, till it became free from soluble copper salts (ferrocyanide test). The cuprous oxide was then dissolved by treating the cellulose on the funnel with solution C (25 ml) and filtered. The material was then well washed with 2N sulphuric acid (75 ml) and the combined filtrates and washings were titrated with solution D. The results are shown Tables 1 and 2.

Determination of Nitrogen.—The dry sample (1.5 g) was taken in a Kjeldahl flask, and potassium sulphate (10 g), copper sulphate (0.5 g) and concentrated sulphuric acid (25 ml) were added to the sample. The flask was shaken until the contents were well mixed. The flask was fitted with a

TABLE	IDETERMINATION	OF	COPPER	NUMBER	PAK	WHITE	VARIETY.
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Variety of jute	Sample (bottom B or top T)	Wt of dry jute (g)	1.0080 N/25 KMnO ₄ reqd (ml)	Cu No. (g/100 g)	Mean	Mean of bottom and top
Pak White 'Special'	$\begin{array}{c} B_{I} \\ B_{2} \\ T_{I} \end{array}$	0.5000 0.5000 0.5113	1.30 1.30 1.06	0.6682 0.6682 0.5328	0.6682) } 0.5981
	T ₂ B ₁	0.5106	1.04 1.46	0.5235	0.5201	J
Pak White 'A'	B_2 T_1 T_2	0.5085 0.5121 0.5071	I.33 I.42	0.6721 0.7126 0.7044	0.7025 0.7085	> 0.7055
D L WILL	B ₁ B ₂	0.5104	1.83 1.85	0.9214	0.9265]
Pak White B.	$\begin{array}{c} T_{1} \\ T_{2} \end{array}$	0.5126 0.5121	1.10 1.17	0.5710	0.5790	} 0.7528
Pak White 'C'	B_1 B_2 T	0.5000	2.58 2.46	1.2644 1.2612	1.2628	} 0.9025
	T_2	0.4708	0.98	0.5390 0.5455	0.5422	j
Pak White 'D'	$ \begin{array}{c} B_{I} \\ B_{2} \\ T_{I} \end{array} $	0.5108 0.5115 0.5020	2.46 2.50 2.15	1.2377 1.2560 1.0990	1.2469	1.1680
	T ₂ Br	0.5125	2.20	1.0800	1.0890	j
Pak White 'E'	$\begin{array}{c} B_2 \\ T_1 \\ T_2 \end{array}$	0.5175 0.5121 0.5124	1.83 2.42 2.47	0.9088 1.2144 1.2193	0.8911	} 1.0539

Variety of jute	Sample (bottom B or top T)	Wt of dry jute (g)	1.0080 N/25 KMnO4 reqd (ml)	Cu No. (g/100g)	Mean	Mean of bottom and top
Pak Tossa 'Special'	B	0.5121	2.10	1.0538		- <u>-</u>
	B ₂	0.5109	2.03	1.0211	1.0375	0000
	T ₁	0.5122	1.45	0.7275		> 0.0000
	T_2	0.5122	1.50	0.7525	0.7400	J
Pak Tossa 'A'	Br	0.5104	2.31	1.1631	0)
	B ₂	0.5127	2.40	1.2030	1.1831	
	T	0.5125	I.40	0.7020		> 0.9488
	T_2	0.5126	1.45	0.7269	0.7145	j
Pak Tossa 'B'	Br	0.5115	2.73	1.3535	C)
	B ₂	0.5116	2.73	I.3337	1.3536	610 M 10 10 10 1
	T.	0.5115	1.82	0.9144	60	> 1.1502
	T_2	0.5117	1.95	0.9743	0.9468	J
	Br	0.5115	4.20	2.1102		7
Pak Tossa 'C'	\mathbf{B}_{2}	0.5113	4.17	2.0954		
	B ₂	0.5119	4.00	2.0533	2.0770	
	B	0.5117	4.08	2.0491		F 1.8925
	T_{T}^{*}	0.5118	3.40	1.7073	-	
	T_2	0.5117	3.40	1.7073	1.7073	
Pak Tossa 'D'	B,	0.5113	5.20	2.6137	C-0-	7
	B ₂	0.5113	5.18	2.6037	2.6087	
	Tĩ	0.5119	3.04	1.5262	all the second	2.0099
	T_2	0.5120	3.06	1.5359	1.5310	J
Pak Tossa 'E'	B,	0.5117	4.69	2.3555		r
	B ₂	0.5121	4.70	2.3587	2.3571	
	T	0.5122	3.00	1.5052		► 1.9324
	T_2	0.5122	3.01	1.5102	1.5077	J

TABLE 2.—DETERMINATION OF COPPER NUMBER OF PAK TOSSA VARIETY.

pear-shaped glass valve and heated in a fuming cupboard with a small flame until the foaming ceased. Then the flame was increased till the contents started to boil. The heating was continued for 3-4 hr till the mixture was completely colourless. The mixture was cooled, diluted carefully with distilled water (200 ml) and transferred to an ammonia distillation apparatus. A few fragments of granulated zinc were added to avoid bumping during the distillation. The flask was fitted with an efficient splash-head connected to a Liebig's condenser delivering to a 400 ml conical flask containing 10 ml of N/10 sulphuric acid and 50 ml distilled water to immerse the nozzle of the condenser, which prevented any loss of ammonia. Now 50% sodium hydroxide solution (100 ml) was added very carefully along the side of the Kjeldahl flask. The flask was then heated on a Bunsen burner, and the distillation was stopped when the volume of the distillate was

about 150 ml. The excess acid in the receiver was then titrated with N/10 sodium hydroxide solution using methyl red indicator. A blank determination was carried out as above, but without using jute sample, and was substracted from experimental value for necessary correction.

The percentage of nitrogen was calculated from the formula:

% Nitrogen =
$$\frac{(V_2 - V_1) \times \mathcal{N}_a \times 0.01401 \times 100}{W}$$

where V_2 =volume (ml) of standard alkali used in the titration; V_1 =Volume (ml) of standard alkali used in the blank titration; N_a =normality of the acid: and W=weight of the dry sample.

The results are shown in Tables 3 and 4.

Scientific Grading of Jute. Part IV

Grade	Nos.	Sample taken (g)	1.1054 א/10 Sulphuric acid for absorption (ml)	0.9612 N/10 NaOH required for neutra- lisation (ml)	NaOH (ml)	Corres- ponding equivalent acid (ml)	Nitrogen (%)	Average (%)
Pak White 'Special'	1 2 3 Blank	1.1188 1.4160 1.4412	$ \begin{array}{r} 10.0 \\ 10.0 \\ 10.0 \\ 10.0 \end{array} $	9.80 9.35 9.70 11.25	1.45 1.90 1.55 0.25	1.26 1.66 1.35	$\left. \begin{array}{c} 0.17 \\ 0.18 \\ 0.15 \end{array} \right\}$	0.17
Pak White 'A'	1 2 3 Blank	1.2180 1.0356 1.0148	10.0 10.0 10.0 10.0	9.95 9.90 10.05 11.25	1.30 1.35 1.20 0.25	1.13 1.17 1.04	$\left. \begin{array}{c} 0.12 \\ 0.12 \\ 0.11 \end{array} \right\}$	0.12
Pak White 'B'	1 2 3 Blank	1.4168 1.5592 1.5406	10.0 10.0 10.0 10.0	7.95 8.56 7.95 10.60	2.65 2.04 2.65 0.90	2.30 1.77 2.30	$\left.\begin{array}{c} 0.25 \\ 0.17 \\ 0.23 \end{array}\right\}$	0.22
Pak White 'C'	1 2 3 Blank	1.0394 1.1538 1.2252	10.0 10.0 10.0 10.0	8.10 8.00 8.00 10.00	1.90 2.00 2.00 1.50	1.65 1.74 1.74	$\left. \begin{array}{c} 0.24 \\ 0.23 \\ 0.22 \end{array} \right\}$	0.23
Pak White 'D'	1 2 3 Blank	1.2076 1.2296 1.4948	10.0 10.0 10.0 10.0	9.30 9.00 8.00 11.25	1.95 2.25 3.05 0.25	1.70 2.00 2.07	$\left. \begin{array}{c} 0.22 \\ 0.25 \\ 0.22 \end{array} \right\}$	0.23
Pak White 'E'	1 2 3 Blank	$1.3850 \\ 1.7680 \\ 1.1354$	10.0 10.0 10.0 10.0	8.50 7.50 8.70 11.00	2.50 3.50 2.30 0.50	2.18 3.00 2.00 0.00	$\left. \begin{array}{c} 0.25 \\ 0.26 \\ 0.27 \end{array} \right\}$	0.26

Table 3.—Nitrogen Content in White Variety of Jute.

TABLE 4.—NITROGEN CONTENT IN TOSSA VARIETY OF JUTE.

Grade	Nos.	Sample taken (g)	1.1054 N/10 Sulphuric acid for absorption (ml)	0.9612 N/10 NaOH required for neutra- lisation (ml)	NaOH (ml)	Corres- ponding equivalent acid (ml)	Nitrogen (%)	1	Average (%)
Pak Tossa 'Special'	1 2 3 Blank	1.7170 1.5792 1.9350	10 10 10 10	9.35 9.25 9.05 11.50	2.15 2.25 2.45 0.00	1.87 1.95 2.10	0.17 0.19 0.17	}	0.18
Pak Tossa 'A'	1 2 3 Blank	$1.6340 \\ 1.5240 \\ 1.3384$	10 10 10 10	7.55 8.45 9.10 11.25	3.95 3.05 2.40 0.25	3.22 2.43 1.87	0.30 0.25 0.22	}	0.26
Pak Tossa 'B'	1 2 3 Blank	0.9510 1.0640 1.0202	10 10 10 10	10.20 10.10 10.25 11.25	1.05 1.15 1.00 0.25	0.91 1.00 0.90	0.15 0.15 0.14	}	0.15
Pak Tossa 'C'	1 2 3 Blank	0.9536 1.1638 1.3172	10 10 10 10	10.30 9.92 9.78 11.25	$ \begin{array}{r} 1.20 \\ 1.58 \\ 1.72 \\ 0.25 \end{array} $	0.87 1.15 1.28	0.14 0.15 0.15	}	0.15
Pak Tossa 'D'	1 2 3 Blank	1.0852 1.2132 1.2512	10 10 10 10	9.95 9.70 10.30 11.20	1.55 1.80 1.20 0.30	1.35 1.57 1.04	0.19 0.20 0.13	}	0.17
Pak Tossa 'E'	1 2 3 Blank	1.5860 1.9560 1.7950	10 10 10	8.00 8.20 8.40 11.25	3.50 3.30 3.10 0.25	2.82 2.65 2.48	0.27 0.21 0.21	}	0.23

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References

- M. Manzoor-i-Khuda, M. A. Karim, Manzurul Huq and Shahidullah, Pakistan J. Sci.Ind. Res., 12, 414 (1969).
- M. Manzoor-i-Khuda, A.S.M. Serajuddin, M. Nurul Amin and A. Aziz Khan, *Ibid.*, 12, 418 (1970).
- M. Manzoor-i-Khuda A.S.M. Serajuddin, M. Bose and Md. Shahjehan *Ibid.*, **13**, 153 (1970).
- C. Doree, Methods of Cellulose Chemistry, 2nd edition, (Chapman and Hall, London, 1947), pp. 10-39.
 A.I. Vogel, A Text Book of Quantitative In-
- 5. A.I. Vogel, A Text Book of Quantitative Inorganic Analysis, 3rd edition (Longmans, London, 1961). pp, 256-57.
- London, 1961). pp, 256-57.
 6. A.L. Steyermark, *Quantitative Organic Microanalysis* (Academic Press, New York and London, 1961), p. 188.
- London, 1961), p. 188.
 7. E.P. Clark, Semi-micro Quantitative Organic Analysis (Academic Press, New York, 1943), pp. 40-42.

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