

<http://www.pjbs.org>

**PJBS**

ISSN 1028-8880

**Pakistan  
Journal of Biological Sciences**

**ANSI***net*

Asian Network for Scientific Information  
308 Lasani Town, Sargodha Road, Faisalabad - Pakistan

## Estimation of Lignin in Jute by Titration Method

<sup>1</sup>M. Ashraf Hussain, <sup>1</sup>M. Emdadul Huq, <sup>1</sup>Sarder Matiur Rahman and <sup>2</sup>Zakaria Ahmed

<sup>1</sup>Chemistry Division, <sup>2</sup>Microbiology and Biochemistry Division,

Bangladesh Jute Research Institute, Manik Mia Avenue, Dhaka-1207, Bangladesh

**Abstract:** The standard method for determining the Kappa Numbers of paper pulps has been evaluated, modified and applied for estimation of Lignin in different samples of jute. The values of the lignin content as determined from the Kappa Numbers of different samples of jute showed conformity with the values of Klason lignin estimated by sulphuric acid method. The method consisted in determining by titration the amount of potassium permanganate reduced by the lignin content of jute fibre. The developed method is easier to follow in comparison with the tedious gravimetric analysis of lignin using corrosive inorganic acid.

**Key words:** Estimation, pulp, Klason lignin, kappa number, kappa lignin, titration, hydrolysis, slope

### Introduction

Jute is a lignocellulosic bast fibre which contains about 12-14% lignin, a natural resinous material which acts as a binder for the cellulose and other materials involved in the structural skeleton of jute and other land plants. Jute fibre is now a days being chemically modified for newer and diversified textile uses. For the purpose, lignin content of jute fibre is either modified or degraded partially which becomes more sulphonated and goes into solution during hydrolysis with sulphuric acid.

Of the various methods for quantitative determination of lignin in land plants, the use of 72% (w/w) sulphuric acid to hydrolyze all carbohydrates excepting phenyl propane residues popularly known as Klason lignin has been widely accepted (Klason, 1908; 1910). The method often gives erroneous results due to incomplete hydrolysis of cellulosic contents. It is reported that this unhydrolyzed cellulosic materials may form furfural compounds which condense with the lignin under acidic conditions and thus falsify the results (Brauns, 1952). Moreover, some amount of the lignin content dissolves in the sulphuric acid liquor forming soluble lignin sulphonic acid (Brauns and Brauns, 1960). Considering this fact, the method has been modified in order to determine lignin precisely.

The uncertainty in the determination of lignin in raw jute and chemically modified jute is more pronounced when they contain low percentage of lignin. Above all, the sulphuric acid method of lignin estimation is tedious, time consuming and may be hazardous if the acid liquor splashes during work.

It is known that grading of wood pulps is assessed from the degree of delignification estimated by determining their Kappa Number which is a measure in number the milliliter of decinormal solution of potassium permanganate consumed by 1 gram of pulp under the specified standard conditions (Tasman and Berzins 1957). The present study was undertaken to develop an easier modified Kappa Number method for the estimation of lignin in raw jute and modified jute fibre.

### Materials and Methods

The experiment was carried out in the chemistry laboratory of Bangladesh Jute Research Institute, Maniak Mia Avenue, Dhaka-1207, Bangladesh during the year 1999-2001. Hard jute cuttings, normal retted raw jute fibre, over retted jute fibre, mercerized jute fibre, sulphonated jute fibre and bleached jute fibre were used as experimental samples. The chemical reagents used were potassium permanganate solution (0.1N and 0.05N), sulphuric acid solution (4N), potassium iodide solution (1N), sodium thiosulphate solution (0.1N) and starch indicator solution (0.2%). The apparatus included a mortar and a pestle made of porcelain or glass, a temperature bath for maintaining constant temperature of 25° C in the reaction vessel, 1000 ml beaker, 500 ml graduated cylinder, 25 ml and 5 ml pipettes, 50 ml burette, some conical flasks and a stop watch. The results of Kappa Number determination by standard method

are corrected to 50% consumption of the permanganate added. This standard method was modified to make it applicable to jute fibre and suitable for small sample with low permanganate consumption. The following modified recipe was used:

Maximum weight of jute sample	0.5 gm
Volume of water	600 ml
Volume of 0.1N potassium permanganate solution	75 ml
Volume of 4N sulphuric acid solution	75 ml
Total volume	750 ml
Temperature of the mixture	25° C
Time of reaction	10 minutes
Volume of 1N potassium iodide solution	15 ml
0.1N sodium thiosulphate solution	as required

Since 0.5 gm weight of all fibre samples did not consume the same amount of permanganate solution, it was by trial that an amount of the fibre to the nearest of 0.1 mg was taken for consumption not less than 10%, but preferably not less than 30% and not more than 50% of the permanganate used in the experiment. In this case, the results were corrected to 50% consumption of the permanganate solution added using the values of the correction factors from Table 1.

Using Table 1 as a guide, 0.5 gm of the fibre sample were dispersed in 30 ml distilled water and ground to fine paste by using mortar and pestle. The disintegrated sample was transferred to 1000 ml conical flask and distilled water was added to make the total volume to 600 ml. Seventy five ml of potassium permanganate solution and 75 ml of sulphuric acid solution were mixed together and added immediately to the disintegrated fibre sample. Thus, the total volume was made to 750 ml. The reaction was allowed to proceed at 25° C temperature for exactly 10 minutes. Then 15 ml of potassium iodide solution was added and the free iodine was titrated with standard sodium thiosulphate solution using starch as an indicator. A blank titration was made using the same volume of water and reagents. The Kappa Number was then calculated from the following equation:

$$\text{Kappa Number (K)} = \frac{p \times f}{W}$$

Where

- p = Milliliter of 0.1N potassium permanganate consumed by the experimental jute sample.  
 w = Weight in gram of moisture free jute sample.  
 f = Factor for correction to 50% permanganate consumption, which has been shown in the Table 1.

Klason lignin contents of different jute samples were determined by sulphuric acid method described under the references (Norman and Jenkins, 1934; Bhuiyan *et al.*, 1979).

Table 1: Factors 'f' in the equation below for correction of different percentages of permanganate consumed

% consumed	0	1	2	3	4	5	6	7	8	9
10	0.911	0.913	0.915	0.918	0.920	0.923	0.925	0.927	0.929	0.931
20	0.934	0.936	0.938	0.941	0.943	0.945	0.947	0.949	0.952	0.954
30	0.958	0.960	0.962	0.964	0.966	0.968	0.970	0.973	0.975	0.977
40	0.979	0.981	0.983	0.985	0.987	0.989	0.992	0.994	0.996	0.998
50	1.000	1.002	1.004	1.006	1.009	1.011	1.013	1.015	1.017	1.019
60	1.022	1.024	1.026	1.028	1.030	1.033	1.035	1.037	1.039	1.042
70	1.044									

**Results and Discussion**

The Klason lignin, Kappa Numbers and Kappa lignin was determined for eight different jute samples as shown in the Table 2. The Klason lignin was found to vary from 14.60% to 3.50%, the Kappa Numbers from 91 to 30 and Kappa lignin from 14.10% to 4.65%. The highest results were obtained with raw jute sample like hard jute cutting and the lowest values with chemically modified fibers like double bleached jute.

Table 2: Klason lignin, Kappa Number and Kappa lignin of different jute samples

The samples	Klason lignin (%)	Kappa Number	Kappa lignin (%)
Hard jute cutting	14.60	91	14.10
Raw jute fibre	12.50	78	12.09
Over retted jute	11.60	74	11.47
Mercerized jute	12.00	79	12.24
Sulphonated jute	8.50	67	10.38
Half bleached jute	7.20	60	9.30
Bleached jute	6.70	52	8.06
Double bleached jute	3.50	30	4.65

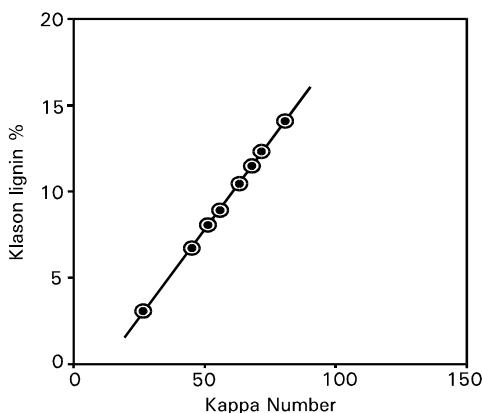


Fig. 1: Relation between Kappa Number and Klason lignin

Klason lignin contents and Kappa Numbers of fibre samples taken from raw jute and chemically treated jute were determined which have been shown in Table 2. The results have been extrapolated in Fig. 1 from which it is observed that the Kappa Number of jute fibre has essentially a straight line relationship with its Klason lignin and that the slope of the line is 0.155, which conforms approximately to the standard relationship (Tasman and Berzins 1957).

In compliance with the Kappa Number-lignin relationship, the Kappa Numbers of different samples multiplied by the slope, 0.155 of the straight line in the Fig. 1 gave indirectly the lignin contents of the samples, which have been shown in Table 2, naming them as Kappa lignin to distinguish from Klason lignin. It is observed from Table 2 that the Klason lignin contents of the natural jute fibers are slightly higher and those of the chemically treated ones are slightly lower than the Kappa lignin contents. This explains the fact that some unhydrolyzed solid cellulosic materials were added to the actual weight of the lignin contents specially when the fibers were raw and matured during gravimetric analysis of lignin by sulphuric acid method and that some amount of the lignin contents of the fibers were lost as soluble lignin sulphionate specially when the fibers were pretreated chemically. So, it may be

suggested that lignin contents of all kinds of jute and jute products may be determined precisely from the modified relationship,

$$\text{Lignin content} = \text{Kappa Number} \times 0.155$$

provided that the conditions for Kappa Number determination are properly maintained.

Brauns and Brauns (1960) suggested that in the studies of the lignin determination, concentration of sulphuric acid was the best which give the lowest yield of lignin with the highest methoxyl content. It is true that a concentration that is too low does not completely hydrolyze the carbohydrates and thus gives a higher yield of apparent lignin, with a lower methoxyl content; on the other hand, when the acid is too concentrated, dehydration may occur and give residues with a higher carbon content, a lower hydrogen content, and a higher methoxyl content; in this case the lignin yield will be lower and methoxyl content higher- but a drastically changed and decomposed lignin will result. For the determination of lignin in annual plants and in forage by sulfuric acid method, it is necessary to remove proteins and some carbohydrates before treatment with strong mineral acids because it is known that proteins interfere in the lignin determination and that some carbohydrates may form furfural compounds which condense with the lignin under acid conditions and thus falsify the results. Since alcohol extracts some lignin from wood, an alcohol extraction prior to the lignin determination should be avoided. In some cases, some lignin may be extracted even by an alcohol-benzene mixture, with the amount varying with the ratio of ethanol to benzene- the higher the ratio of benzene, the less lignin will be dissolved (Brauns and Brauns, 1960). In the study of lignin determination of cereal straws (Brauns and Brauns, 1960), it was found that 72% sulfuric acid gave the minimum yield with the maximum methoxyl content. A comparison of the 72% sulfuric acid and 42% hydrochloric acid methods showed that the former gave slightly higher lignin yields with straws pretreatment with 1% hydrochloric acid; when the pretreatment was omitted, the difference in yields varied by 2-3%. The determination of lignin in young pine shoots was also studied and it was found that the residues obtained on hydrolysis with 72% sulfuric acid and 42% hydrochloric acid contained large amounts of condensation products (from polyuranides and other carbohydrates), having relatively high oxygen contents but no aromatic groups.

**References**

Bhuiyan, A.M., M. Ali and M. Rahman, 1979. Studies on Changes in Chemical Composition of Jute Fibre During Retting. *Bangla. J. Jute Fib. Res.*, 4: 33-39

Brauns, F.E., 1952. The chemistry of lignin. New York, Academic press, pp: 137

Brauns, F.E. and D.A. Brauns, 1960. The chemistry of lignin: VI. The determination of lignin. Supplement Volume, Academic press, New York, pp: 128-129

Klason, P., 1908. Chemical Composition of Deal (Fir wood). *Ark. Kemi. Mineral. Geol.*, 3: 1-10

Klason, P., 1910. Determination of lignin in sulphite wood pulp. *Papierfabr.*, 8: 1285-6

Norman, A.G. and S.H. Jenkins, 1934. The determination of lignin. *Biochem. J.*, 28: 2147-2160

Tasman, J.E. and V. Berzins, 1957. The Permanganate Consumption of Pulp Materials, *Tappi*, 40: 691-704.