

Wheat Straw as a Source of Cellulose Pulps

M. K. Trivedi,

Introduction

The shortage of cellulose pulp for various end-uses has been so alarming in the recent past that we must continually search and investigate all possible sources of fibrous raw materials capable of providing pulp. The agricultural residues are one of the oldest sources exploited for paper manufacture but the advancement made in pulping of woods and attendant chemical recovery reduced their importance. It is well-known that the non-cellulosic content of these materials is so high that the traditional methods of chemical recovery are unpracticable. New techniques of pulping need to be examined with an aim of recovery of useful by-products to make the processing of these materials economically attractive. In recent years bagasse and rice straw have received more consideration for use in pulp and paper than any other agricultural residue^{1,2}. Wheat straw ranks only second to rice in availability in our country and if the seasonal availability of these materials is considered, the wheat straw appears to be an equally important fibrous source

Dr. M. K. Trivedi,
Asst. Professor,
Dept. of Chemical Engineering,
Indian Institute of Technology,
Bombay-400076.

Wheat straw being richer in non-cellulosic constituents as compared to other materials, can serve as a useful source of cellulose pulps only if processes aiming at by-product recovery are visualised. An attempt has been made to process wheat straw with simple equipment and common chemicals whereby pulps ranging from paper pulps to dissolving grade pulps could be obtained with possibilities of lignin and furfural recovery from spent liquors.

for pulps. Even with conservative estimates about 40 million tonnes of this material can be obtained. Assuming that 50 percent of it is used as cattle fodder, a sizable quantity, which is normally used as fuel can be made available for pulping⁴. The work presented here attempts to show how various non-cellulosic components of wheat straw can be gainfully utilised while obtaining a cellulose pulp of good quality with simple equipment and common chemicals.

Experimental

The wheat straw obtained from Malwa region of Rajasthan was cut to pieces 3 to 4 cm in length and was stored over saturated sodium chloride solution to maintain a uniform moisture content. All pulp samples were similarly stored to have uniformity. Except commercial Hydrochloric acid other chemicals used were of reagent grade. Analysis of all samples was done according to Tappi standards. All recorded results are average of six consistent

observations. The analysis of raw materials is given in Table 1.

Table 1
Analysis of wheat straw
(% based on O. D. material)

1. Hot water solubles	9.5
2. 1% Alkali solubles	40.2
3. Alkali benzene solubles	2.9
4. Ash	6.0
5. Lignin	21.0
6. Pentosans	23.0
7. Cross and Bevan cellulose	44.2
8. 'Alpha' cellulose	32.0
9. (Beta+Gamma) cellulose	5.0

Treatment of straw with Alkali

About 25g of wheat straw exactly weighed was presoaked in water overnight and boiled with aqueous alkali solution using 10% alkali on straw and solid to liquor ratio of 1:20 for one hour. The pulp obtained was washed free of alkali and weighed. The amount of alkali, solid to liquor ratio and time of treatment were varied and results are recorded in Table 2. The condition giving maximum loss of material were repeatedly confirmed to obtain the pulp which was analysed (column-1

Table-6) and used for further delignification.

Treatment of straw with Hydrochloric acid and alkali

About 10 g of straw was treated with commercial Hydrochloric acid (strength 10.2 N, Sp. Gravity 1.15) at ambient temperature for 1 hour with a solid to liquor ratio of 1:10. After the treatment the residue was filtered out, washed and weighed. Loss in weight for varied time, concentration and solid to liquor ratio was noted. The results are recorded in Table 3.

The residue so obtained was boiled with aqueous alkali solution under different conditions of time, concentration and amount of alkali. The pulp obtained was thoroughly washed and weighed. Results are recorded in Table 4. The analysis of pulp obtained under optimum conditions is given in column No. 3 of Table 6.

Delignification and bleaching of pulps

The pulps obtained in above treatments were further delignified under conditions given in Table 5. Data in column 1 is for pulp after presaking and alkali boil and in No.2 is for pulp after acid-alkali treatment. Conditions were so chosen that maximum delignification may be obtained. The final pulps were treated with millinormal Hydrochloric acid overnight, washed and analysed. The results are tabulated in Table 6. Column 2 and 4 denote analysis of alkali boiled and acid-alkali treated pulp respectively. The

Table 2
Pre-soaking and Alkali boil of straw

S. No.	Percent alkali used	Time of treatment hours	S.L. ratio	Percent yield
1	10	1	20	70.3
2	15	1	20	68.4
3	20	1	20	62.9
	25	1	20	61.8
5	30	1	20	60.0
6	20	0.5	20	66.0
7	20	1	20	63.0
8	20	1.5	20	60.2
9	20	2.0	20	60.2
10	20	2.5	10	59.5
11	20	1.5	10	64.5
12	20	1.5	15	62.3
13	20	1.5	20	60.3
14	20	1.5	25	61.5
15	20	1.5	30	62.0

Table 3
Treatment of Straw with Acid

Sl. No.	Strength of acid N	Time hours	S.L. ratio	percent yield
1	10.2	1	20	68.5
2	10.2	2	20	65.0
3	10.2	3	20	65.1
4	10.2	4	20	63.7
5	10.2	5	20	63.5
6	10.2	6	20	62.9
7	15.0	4	20	83.3
8	6.0	4	20	76.2
9	7.0	4	20	71.1
10	8.0	4	20	66.6
11	9.0	4	20	62.8
12	10.2	4	6	68.2
13	10.2	4	10	65.4
14	10.2	4	12	62.3
15	10.2	4	15	62.5
16	10.2	4	20	62.7

bleached pulp of No.4 was further treated with 17.5% sodium hydroxide for 1 hour at 20°C, washed, treated with dilute hydrochloric acid, washed acid free and analysed. The results are given in last column of Table 6.

Discussions

A study of the analysis indicates that the cellulose content of wheat straw is barely 32 percent, and the components like lignin pentosans are quite high thereby causing an imbalance of heat and chemicals if conventional processes of pulping and recovery are adopted. A simple method has been suggested by Kamath and Sidhanty⁵ to process bagasse and rice straw to obtain a variety of pulp grades. When this method is investigated for wheat straw (Table 2), it is observed that if water pre-soaked material is boiled with 20% alkali for 1.5 hours and a solid to liquor ratio of 1:20, a good pulp in about 60% yield is obtained. As the analysis of this pulp indicates (Table 6, column 1), the lignin removal under these conditions is quite high and is about 80% of that present in straw. The pulps with higher lignin contents can be obtained by much lesser amount of alkali used. This pulp can be further delignified to 1.2% lignin content by a sequence of treatments outlined in Table 5 column. 1. A glance at the analysis of this pulp (Table 6-columns-2) shows that the pentosans content of this pulp is very high though the lignin and ash are considerably reduced. An acid prehydrolysis seems

Table 4
Alkali boil of residue after acid treatment

S. No.	Percent alkali	Time hours	S.L. ratio	Percent yield on straw
1	10	1.0	20	37.1
2	20	1.0	20	36.0
3	30	1.0	20	35.8
4	40	1.0	20	35.4
5	20	0.5	20	39.5
6	20	1.5	20	36.4
7	20	2.0	20	37.0
8	20	1.0	10	37.2
9	20	1.0	15	37.0
10	22	1.0	18	36.1
11	20	1.0	20	36.0
12	20	1.0	18	36.1

Table 5
Treatments for delignification and bleaching

Treatments		1 Alkali Pre-soaking boil	2 acid-alkali boil
1	Pulp g	50	50
2	Chlorine demand%	8.5	4.5
3	Chlorination		
a	chlorine added%	6.5	3.6
b	Time min.	60	3.0
c	Temperature °C	27	27
d	PH	2.4	2.5
4	Alkali Extraction		
a	Alkali%	1.5	1.0
b	Time min.	60	60
c	Temperature °C	75	70
d	PH	11.0	11.0
5	Hypochlorite		
a	Chlorine%	2.0	0.9
b	Time min.	90	90
c	Temperature	40	40
d	PH	9.5	9.5

Table 6
Analysis of pulps

Sl. No.	Percent on pulps	1	2	3	4	5
1	yield% (on straw)	60.3	40.9	36.0	33.1	32.4
2	Pentosans	24.2	23.0	2.9	2.2	1.2
3	Lignin	5.8	1.2	0.8	0.6	0.1
4	Ash	1.3	0.5	0.6	0.5	0.3
5	'Alpha' cellulose	53.0	78.2	93.0	92.3	98.4
6	(Beta+Gamma) cellulose	—	—	3.2	4.8	—

essential if a pulp denuded of pentosans is required.

A simple method of acid treatment at ambient temperatures and pressure has been evolved through data obtained in Table 3. Hydrochloric acid (sp. gravity 1.15) used 12 times the weight of straw for 4 hours results in maximum loss of constituents from the straw. A time of 4 hours seems necessary for removal of pentosans though the greater part of solubilisation occurs within first hour of treatment. The material is partially opened up and a semi pulp is obtained at this stage. The results are fairly reproducible and on average a loss of 37% is indicated at this stage. The extract which is obtained at this stage can be worked up for recovery of acid and furfural which is a valuable by-product⁶. It seems that concentration of acid as well as liquor ratio can be considerably reduced if a little shear is given to the material at this stage. The time may be reduced to an hour and lesser quantity of acid may be required. This however, could not be studied due to lack of proper equipment.

The alkali treatment of this residue was also carried out at ambient pressures. It was observed that boiling with alkali solution for 1 hour using 20% alkali and a solid to liquor ratio of 1:18 leads to maximum loss of constituents. Delignification and Deashification are the main reactions at this stage. The small

amount of fats and oils present is also saponified at this stage. The unbleached pulp obtained by the treatments was in about 36% yield with about 93% alpha cellulose, 0.6% ash and 2.9% pentosans. (Table 6 column-3). The bleaching of this pulp (Table 5 column-2) resulted in further removal of ash, lignin and pentosans (Table 6-column-4). The purity of alpha-cellulose of this pulp was confirmed by treating it with 17.5% sodium hydroxide and analysing the resultant pulp (Table 6-column-5). A pulp very rich in 'alpha' cellulose associated with 0.3% ash, 0.1% lignin and 1.2% pentosans is obtained. This can be further evaluated as dissolving grade pulp. The alkaline liquors can be treated for recovery of lignins and promising results have been obtained in this direction⁷.

The work has indicated that wheat straw can be pulped with simple method and equipment to obtain pulps of different types. Extent of delignification can be controlled by treatment with alkali and chlorine based chemicals, whereas any step for maximum removal of pentosans needs acid treatment which can be conveniently carried out. The possibility of the by-product recovery makes the processes attractive for further trials and scaling up experiments.

Conclusions

1. A pre-soaking with water followed by alkali boil gives good pulp from wheat straw at ambient pressure.

2. Pulps with very low lignin content can be obtained with proper treatments.
3. An acid pre-treatment stage with commercial hydrochloric acid results in maximum removal of pentosans.
4. A pulp with very high alpha cellulose content, and low ash, pentosans and lignin can be obtained by combined acid-alkali treatments.

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References

1. Atchison, J.E. Ind. Pulp, Paper 78, 10 (1974)
2. Aggrawala, J. C. *et al*, Ippta, 8, 3 (1971)
3. Clark, T.F. and Bagby, M.O. Ippta 7, 16 (1970)
4. Trivedi, M.K., M. Tech Dissertation, I.I.T. Bombay, 2 (1962)
5. Kamath, N.R. and Sidhanty, A.R., Ind. Pulp. Paper, 23, 265 (1968).
6. Vishnoi, R., Ph. D. Thesis, I.I.T., Bombay, 37 (1965).
7. Trivedi, M.K., Ph. D. Thesis, I.I.T. Bombay 214 (1973)