

# An approach to minimize pollution problems through vapour phase pulping process

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## SUMMARY

Today a 30 tpd mill based on agricultural residues consumes about 300 m<sup>3</sup> of water per tonne of paper produced. Practically all this water goes as effluent. About 25-30% of the total volume of the effluent is generated through digester and brown stock washers. In conventional liquid phase pulping in spherical or tumbling digesters the cooking liquor to material ratio maintained is very high due to bulky nature of straws. This leads to generation of large quantities of spent liquor with low solids concentration. Immediate efforts till suitable chemical recovery system emerges, would be to minimize the quantity of effluents discharged. In the present study with an advantage of the open structure of straw and bagasse, an attempt to apply vapour phase pulping process for these raw materials is made. The vapour phase pulping involves presoaking of the material in 25-35g/l NaOH solution with 1 : 3.5 material to liquor ratio, followed by cooking at 160° C. In vapour phase pulping no black liquor was generated. It was observed that during washing of the vapour phase pulp less quantity of water was required. The quantities of effluents and organic load in the vapour phase pulping effluents were reduced significantly. The properties of vapour phase pulps were comparable to conventional liquid phase pulp. In the case of bagasse, vapour phase pulps had better strength properties compared to liquid phase pulps. Further pilot plant trials have indicated that by adopting vapour phase process there is a wider scope to reduce the pollution load and steam consumption.

Energy and environment have profound influence on the performance of both small and integrated paper units. Large number of small units based on agricultural residues are operating today. Due to lack of economically viable chemical recovery system severe problems of pollution arises. Average pollution load generated from small mills producing bleached grade pulp, is nearly two to three times the pollution load generated by 100 tpd kraft mill with recovery system<sup>1</sup>. Average waste water discharged is about 300 m<sup>3</sup>/t of the product<sup>2,3</sup>. The effluent quantity of about 80 m<sup>3</sup>/t paper is discharged through digester and brown stock washings<sup>3</sup>.

E. L. Keller<sup>4</sup> in his studies on liquor to wood ratio has indicated that the liquor to wood ratio is a significant variable, if the waste liquors are to be treated to recover chemicals or to reduce stream pollution where concentrated liquor is advantageous. He has concluded that pulps made at 5 : 1 and 2 : 1 liquor ratio appear to be interchangeable.

Some of the mills in India are using lower material to liquor ratio during pulping either in continuous digesters or in tumbling digester in conjunction with presteaming tube. This process is close to

vapour phase cooking. The effect of liquor to wood ratio on heat consumption during cooking has also been discussed in one of the articles<sup>5</sup>. The heat consumption of 60,000 k. cal/adt at 1 : 4 ratio was drastically reduced to 10,000 k. cal/adt when liquor to wood ratio was brought down to 1 : 3.1. It was observed that normally with higher cooking liquor ratios the average steam consumption is about 5t/t pulp and 15 m<sup>3</sup> of black liquor is generated per tonne of the pulp<sup>6</sup>.

In the present study vapour phase cooking process was applied to straw and bagasse pulping. Various advantages of this process over conventional liquid phase pulping were investigated both on laboratory and pilot plant scale. It is a known fact that the vapour phase process has advantages, but in the present study a systematic approach to analyse the merits and demerits of this process, has been made.

## Results and Discussion

(a) Pulp quality : Table-1 shows the cooking conditions and pulp quality in liquid and vapour phase pulping of straws.

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For vapour phase pulping the straw was soaked in varying concentration of sodium hydroxide solution. It was observed that the material to cooking liquor ratio of 1 : 3.5 was desired for uniform absorption of chemical. After soaking there was no cooking liquor left over. For liquid phase pulping material to liquor ratio required was 1 : 7 so as to achieve complete submergence of straw in cooking liquor. Results in Table-1 shows that

vapour phase pulps had yields in the range of 62.7% to 49% by varying the chemical dosage from 8 to 14.9%. With a chemical dose of 12.5% the vapour phase pulping produced about 5% more yield than liquid phase pulping with same amount of chemical charge. E.L. Keller<sup>4</sup> has also observed that the pulp yield increased with reduced material to liquor ratios. The kappa number of vapour phase pulps was slightly on higher side. The reject content of vapour phase pulp was on lower side compared to the corresponding liquid phase pulp, indicating the uniform cooking in vapour phase process also. The chemical consumption was slightly on higher side in the case of vapour

phase process. The fibre classification shows that the vapour phase pulps had relatively small proportion of +28 fraction indicating better fibre separation. Table-2 shows the pulping conditions and pulp quality of bagasse. The bagasse used in this study had about 18% pith content.

Results show that in case of bagasse pulping also the material to liquor ratio of 1:3.5 and 1 : 7 was required in vapour and liquid phase respectively. The increased yields were obtained in vapour phase pulping. In this case also the vapour phase pulping process consumed slightly higher amounts of chemical. Fibre classification shows that vapour phase pulps had small proportion +28 fraction, indicating better fibre separation. In liquid phase pulping more amounts of fines were generated as indicated by-200 fraction.

(b) Physical properties of the pulps : Table-3 shows the strength properties of vapour and liquid phase straw pulps.

The strength properties of vapour phase pulps were superior with a soaking liquor concentration

TABLE—1 PULPING OF RICE STRAW

Particulars	Cooking Process			
	Vapour phase			Conventional liquid phase
Cook No.	1	2	3	4
<b>1. Cooking :</b>				
Chemical charge, % as NaOH	8.0	12.5	14.9	12.5
Straw to cooking liquor ratio	1 : 3.5	1 : 3.5	1 : 3.5	1.7
Spent liquor solids g/l	—	—	—	73
Chemical consumed, %	8.8	11.8	13.3	10.8
<b>2. Pulp qualities :</b>				
Total pulp yield, %	62.7	59.9	49.0	55.1
Kappa number	26.0	26.2	16.8	22.9
Reject, %	5.5	2.9	Nil	3.5
Fibre classification (Bauer McK. Nett)				
+28 fraction %		8.89		25.07
—28/48 "		22.68		20.51
—48/+150 "	—	14.23	—	9.44
—150/+200,,		9.67		4.52
—200 "		44.83		40.46

Cooking temperature — 160° C, 60 minutes at 160° C.

TABLE—2 PULPING OF BAGASSE

Particulars	Cooking Process			
	Vapour phase			Conventional liquid phase
Cook No.	5	6	7	8
<b>1. Cooking</b>				
Chemical charge % as NaOH	15.5	16.5	15.5	16.5
Time at top temp. min.	90	90	90	90
Straw to liquor ratio	1 : 3.5	1 : 3.5	1 : 7	1 : 7
Spent liquor solids, g/l	—	—	74	79
Chemical consumed, %	14.1	15.2	12.6	13.1
<b>2. Pulp qualities</b>				
Total pulp yield, %	58.2	55.9	55.6	55.2
Kappa number	26.5	22.4	23.5	18.5
Reject, %	Nil	Nil	0.9	1.0
Fibre classification (Bauer McK Nett)				
+28 fraction, %	3.06		14.40	
—28/+48	45.0		21.54	
—48/+150	16.38		23.25	
—150/+200	8.75		10.76	
—200	26.81		30.05	

Cooking temperature—160°C, 90 minutes at 160°C.

TABLE—3 STRENGTH PROPERTIES OF RICE STRAW PULPS

Strength properties	Vapour phase				Liquid phase	
	1	2	3	4		
Cook No.	1	2	3	4		
Freeness CSF, ml	155*	120*	225*	120	175*	135
Drainage time, secs	28.70	28.20	9.20	23.60	10.80	24.20
Apparent density, g/cm <sup>3</sup>	0.68	0.69	0.68	0.72	0.69	0.71
Burst Index, kPam <sup>2</sup> /g	2.05	2.45	2.45	3.50	2.10	2.95
Tensile Index, N. m/g	36.5	41.0	42.0	60.0	38.5	50.5
Stretch, %	4.1	4.3	3.8	4.6	3.2	4.0
Fold, (Kohler-Molin) log.	1.86	2.06	1.70	2.44	1.52	2.08
Tear Index, mN. m <sup>2</sup> /g	5.00	4.70	4.00	4.35	4.05	4.25
Air resistance, Gurley s/100 ml	192	180	45.5	336.0	66.0	159.2

\*Initial freeness of the pulps.

of 35 g/l (14.9%). The vapour phase pulps produced with 12.6% chemical charge had slightly lower strength properties compared to the corresponding liquid phase pulps. Decrease in strength properties might be due to dissolution of more hemicellulose with lower liquor ratio during vapour phase pulping as indicated by Keller<sup>4</sup>. Results also indicate that with a slight increased chemical dosage it is possible to produce pulps having strengths comparable to liquid phase pulps. Table-4 shows strength properties of bagasse pulps.

In the case of bagasse the vapour phase pulps had better strength properties compared to corresponding liquid phase pulps. The initial freeness values of bagasse vapour phase pulps were on

higher side.

From the quality of pulps produced it is evident that by adopting the vapour phase pulping processes the strength properties are not affected significantly. Strength wise it appears that pulps made from 1 : 3.5 and 1:7 liquor ratios are interchangeable. In case of straw, by slight adjustment of chemical dosage, it is possible to produce satisfactory pulps.

(c) Characteristics of effluents : Table-5 shows the quantity and quality of effluents generated in vapour phase and liquid phase pulping of straw. The volume of effluent generated in liquid phase pulping is about two times the volume in vapour phase process.

TABLE—4 STRENGTH PROPERTIES OF BAGASSE PULPS

Strength properties	Vapour phase						Liquid phase	
	1		2		3		4	
Cook No.	430*	160	395*	185	360*	195	380*	150
Freeness CSF, ml	590	21.50	6.40	18.40	6.90	19.40	6.30	25.50
Drainage time, secs	0.68	0.81	0.70	0.77	0.77	0.83	0.78	0.85
Apparent density, g/cm <sup>3</sup>	2.95	4.70	3.10	4.20	2.70	3.50	2.05	3.50
Burst Index kPa. m <sup>2</sup> /g	45.5	66.0	50.5	66.0	46.5	54.5	42.0	52.0
Tensile Index N.m/g	4.1	4.5	3.4	4.8	4.3	5.0	3.8	4.3
Stretch, %	2.17	2.62	2.35	2.57	2.0	2.23	1.85	2.20
Fold Kohler, M. Log.	5.55	5.00	5.55	5.90	5.20	5.10	4.95	4.85
Tear Index mN. m <sup>2</sup> /g	126	>1800	194	>1800	334	>1800	128	>18000
Air resistance, Gurley, s/100 ml.								

\*Initial freeness of the pulp.

TABLE—5 CHARACTERISTICS OF EFFLUENTS FROM RICE STRAW PULPING

Particulars	Pulping process		
	Vapour phase		Liquid phase
Cook No.	1	2	4
Pulp yield, %	62.7	59.9	53.2
Black liquor generated. m <sup>3</sup> (a)	Nil	Nil	8.9
Total volume of effluents generated, m <sup>3</sup> *	32	31	57
Total dissolved solids, kg	672	806	1026
COD, kg/m <sup>3</sup> of effluent	21	47	30.5
COD, kg/t	670	1473	1796

All results expressed on per tonne of B. D. pulp

(a) Black liquor obtained after squeezing

\* Spent liquor and washing

Increased volume of effluents is due to increased demand of water in washing stage. It was observed that the vapour phase pulps required about 30 m<sup>3</sup> of water during washing stage while the liquid phase pulps required about 47 m<sup>3</sup> of water. Reduced quantity of water during washing stage is presumably due to easy removal of water soluble adsorbed organics on the fibre surface. The total solids and COD load is much more on lower side in vapour phase effluents. The solids load is attributed to total yield of the pulp. The increased COD load, in liquid phase pulping effluents might be due to presence of more oxygen consuming organic compounds formed by degradation of lignin and carbohydrates.

Results in Table-6 shows that in the case of

bagasse also the quantity of effluents generated is on lower side.

However there was not much change in the quantity of water required during washing of vapour phase & liquid phase pulps. In spite of comparable pulp yield the bagasse liquid phase pulping effluents had COD load increased by above 40% compared to vapour phase effluents. The laboratory results confirm that by adopting the vapour phase pulping process there is a wider scope to reduce the organic load and quantity of effluents. In straw about 46% and in bagasse about 20% of the effluent quantity was reduced.

(c) Results of pilot plant trials: The results, of pilot plant trials of vapour phase and liquid phase pulping of rice straw are given in Table-7.

TABLE-6 CHARACTERISTICS OF EFFLUENTS FROM BAGASSE PULPING

Particulars	Pulping process		
	Vapour phase		Liquid phase
Cook No.	5	6	8
Pulp yield %	58.2	55.9	55.2
Black liquor generated, m <sup>3</sup> (a)	Nil	Nil	7.8
Total volume of effluents generated, m <sup>3</sup> *	45	47	55
Total dissolved solids, kg	675	620	770
COD, kg/m <sup>3</sup> of effluent	21	21	31
COD, kg/t	931	992	1694

All results expressed on per tonnes of B. D. pulp

(a) Black liquor obtained after squeezing

\* Spent liquor and washing

TABLE-7 RESULTS OF PILOT PLANT TRIALS OF STRAW PULPING

Particulars	Liquid phase	Vapour phase
Raw material A. D. kg	945	250
Moisture, %	9.5	24
Raw material O. D. kg	855	190
NaOH applied, %	13.7	13.6
NaOH, conc., kg/m <sup>3</sup>	28	51.6
Cooking liquor, m <sup>3</sup>	4.18	0.5
Straw to cooking liquor ratio	1:5	1:3:1
Cooking temp. °C	160 ± 2	160 ± 3
Steam pressure, kg/cm <sup>2</sup>	8-9	6-8
Cooking time at top temp. hr.	1	1
Steam tonnes/t O.D. pulp	4.8	1.7
Black liquor, * m <sup>3</sup> /t pulp	15.5*	7.3*
Black liquor solids, kg/t	1492	1350
Effluent, m <sup>3</sup> /t pulp	61	38
COD, kg/t pulp	1300	857
<b>Pulp Properties:</b>		
Pulp yield %	46.7	52.4
Kappa number	14.6	13.8
Freeness, CSF, ml	200	120
Tensile Index, N, m/g	55.5	46.5
Burst Index kP a. m <sup>2</sup> /g	—	3.0
Tear Index, mN. m <sup>2</sup> /g	4.15	3.65

\* Includes both free and black liquor occluded in pulp.

The objective of the pilot trials was to determine the quantity of steam consumption, amount of yield increase and the quantity of the effluents generated. During the trials moisture content of straw in vapour phase pulping was kept slightly on higher side to achieve uniform penetration of cooking liquor. It was observed that in vapour phase process, steam pressure of about 6-8 kg/cm<sup>2</sup> was sufficient as against 8-9 kg/cm<sup>2</sup> in liquid phase, to maintain the constant temperature. From the mass balance it was found that the steam consumption figures were 4.8 and 1.7 tonnes per tonnes of pulp, in liquid phase and vapour phase pulping respectively. The quantity of black liquor generated in liquid phase was almost two times the quantity generated in vapour phase. With a continuous digestion unit, it might be possible to reduce further this generation of black liquor in vapour phase process by avoiding excess steam condensation.

On pilot plant scale also it was confirmed that the quantity of effluents generated in liquid phase process is much more compared to vapour process. The COD load in liquid phase pulping effluents was higher by about 34%. About 6% more pulp yield was obtained in vapour phase process. On pilot plant scale also, it was observed that there was decrease in strength properties of vapour phase pulps. The pilot plant trials have indicated that by adopting vapour phase process there is a greater scope to minimize the pollution problems. Once the quantity of effluents is reduced then the capital inputs for treatment facilities will also be reduced. The vapour phase pulp properties could be maintained by adjusting the chemical dosage.

## CONCLUSIONS

- 1 In vapour phase pulping process higher pulp yields were obtained with a substantial reduction in quantity of effluents and pollution loads.
- 2 Vapour phase bagasse pulps had superior strength properties compared to liquid phase pulps, while in the case of straw there was a slight decrease in strength properties of vapour phase pulps. It can be concluded that strength wise vapour phase and liquid phase pulps are interchangeable.
- 3 Pilot plant trials indicate that there is a substantial reduction in steam consumption in vapour phase process.
- 4 From laboratory and pilot plant scale experiments it is evident that by adopting vapour phase process there is a wider scope to improve

steam economy and pollution problems.

- 5 Existing mills should try to adopt vapour phase process. Even if suitable recovery system emerge it is expected that the vapour phase process can give concentrated black liquors.

## EXPERIMENTAL :

**Raw material :** Rice straw was collected from local paddy growers. The paddy stalk free from husk was manually chopped to 2"-3" size and dried. The bagasse was collected from M/s. Mandya National Paper Mills Ltd., Belgula (Karnataka). The bagasse bales were broke manually and stored. The pith content of bagasse was estimated according the method described (7).

**Conventional pulping :** The conventional liquid phase pulping was carried out in series digester consisting of six bombs each of 2.5 litre capacity. The bombs were heated in polyethylene glycol bath. The material to cooking liquor ratio of 1 : 7 was selected in liquid phase process so as to achieve complete submergence of the straw. After cooking the liquor was separated by squeezing the pulp on nylon cloth. The pulp was washed with predetermined quantity of fresh water after disintegrating for 5 minutes. The washings were determined to obtain constant kappa number.

**Vapour phase pulping :** In vapour phase pulping the straw was soaked in 25-35 g/l NaOH solution at 30°C, with soaking liquor to straw ratio of 3.5 : 1. After soaking the material was cooked at 160°C in series digester. During cooking stage no liquor or water was added. The pulp was disintegrated and washed with predetermined quantity of fresh water. The quantity of fresh water for washing was fixed at the stage where constant kappa number was obtained.

**Spent liquor analysis :** The analysis was carried out as according to procedure described in Tappi Standard Methods T-625-ts-64. The chemical oxygen demand was determined as per the method described in 'Waste Water Analysis' a course manual of National Environment Engineering Research Institute, Nagpur.

**Pulp analysis :** The kappa number of the pulp was determined according to Tappi method. T-236-Os-76.

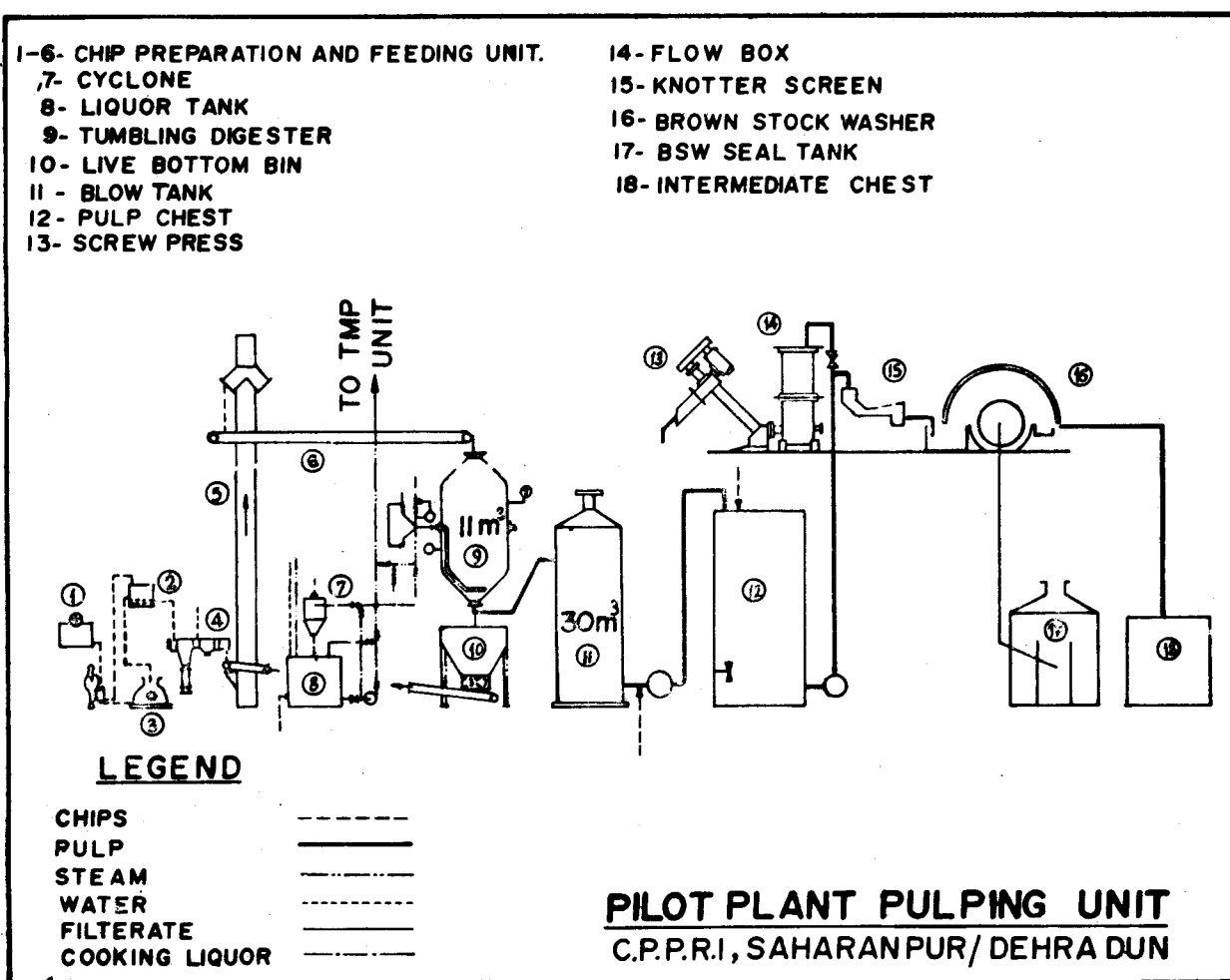
**Pulp evaluation :** Pulp evaluation and paper testing were carried out according to standard methods given in laboratory manual<sup>8</sup>.

**Pilot plant trials :** The pilot plant trials of vapour phase and liquid phase were conducted at Saharanpur pilot plant unit of CPPRI. The flow sheet of pilot plant pulping unit is shown in the

Fig. 1. The raw material was charged to a stainless steel tumbling digester of 11m<sup>3</sup> capacity. Cooking liquor from liquor preparation tank was sprayed from the top of digester. After cooking the pulp along with liquor was blown into 30 m<sup>3</sup> blow tank.

Then the pulp was diluted to about 6% consistency and taken to brown stock washer. For actual yield calculations about 125 g of straw was placed in a wire mesh basket which was placed in the strainer fixed at the top of digester. In the

vapour phase pulping the straw and chemical charges were done simultaneously to achieve uniform chemical distribution. The objective of the pilot plant trials was to assess the amount of steam condensation during vapour and liquid phase pulping process. The total quantity of washings collected at brown stock washer seal tank was noted. For calculating the liquid to material ratio after cooking the sample from blow tank after dilution with known quantity of fresh water, was taken. From blow tank consistency the quantity of black liquor generated was calculated.



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