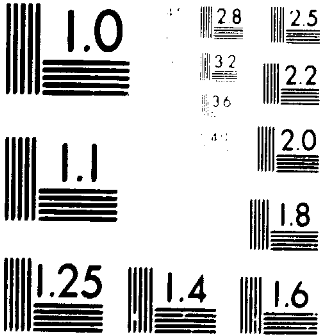


MICROCOPY RESOLUTION TEST CHART

11157 R. Joedodibroto 1981

TESTING OF A NEW PULPING PROCESS (AMMONIA PROCESS) FOR AGRICULTURAL RESIDUES

INTRODUCTION

The testing of a new pulping process for agricultural residues, project US/RAS/79/158, was carried out at the Cellulose Research Institute, Bandung, Indonesia at the request of UNIDO, to study the various possibilities of the process. Work was started on 17th June 1981 and concluded on 27th August 1981. References used in carrying out the experiments were :

1. A letter, dated October 1979, from the UNIDO to the Secretary of the Coordinating Committee for International Technical Cooperation.
2. The contract signed by the Cellulose Research Institute.
3. The note for the file from Dr. M. Judt.
4. Claims made by the inventor in his publications and patents.
5. Our own experiences.
6. Reports of other researchers.

The claims made by the inventor were not supported by experimental data sothat this was not available for reference up to the end of the experimental work. Most of the experiments were performed on rice straw, since this is considered the most potential agricultural residue in developing countries, especially in Asia, as well as the easiest raw material to pulp. Tests were also carried out on bagasse, but not as extensive as on rice straw. The pulping on bamboo was made just to demonstrate the different responses of this raw material on ammonia and soda pulping.

Special attention was paid on the recoverability of ammonia from the black liquor.

The work performed for evaluation of this process were more extensive than planned during early negotiations in 1979 as well as according to the contract signed. It is regretted that the inventor did not come with any data from his own experiments and was lacking in using a scientific approach in his methods.

It is hoped that this work could in some way be useful in showing the advantages as well as the disadvantages of the ammonia process.

MATERIAL AND METHODS

Rice straw and bagasse were mainly used for the experiments. The rice straw came from rice fields near Bandung which were harvested early May. The bagasse came from a sugar mill in West-Java which arrived in mid-June. Part of the rice straw was depithed to a yield of 84.4 %. The bagasse was also depithed and the yield obtained was 66.4 %. Bamboo chips made by hand was also made available for the pulping test. Technical grade ammonia (25% NH_3 content)and caustic soda (96 - 98 %) were used as cooking chemicals.

The pulping experiments were performed in three different kinds of stainless steel digesters : (a) digesters of three liters capacity rotating in hot air, (b) electrically heated digesters of four liters and (c) one gas-heated 15 liters tumbling digester. The digesters of four liters and 15 liters were equipped with valves for gas release. The ammonia process and the soda process were applied at various cooking conditions. After the cooks, the pulps were run through a refiner at 70 mils clearance to help defiberization, then washed and screened on a Sprout Waldron screen. This is the standard procedure applied at the Cellulose Research Institute for chemical pulp.

The pulp evaluation was carried out by beating in a Niagara beater of 10 liters capacity. For small samples the PFI mill was used for evaluation.Sheets were made in a British Standard sheet-former and the sheets tested for its physical properties according to TAPPI Standard Methods.

Some of the pulps as well as the raw material were analyzed for its chemical composition. Microscopical studies were also made on some of the pulps and the fiber dimensions determined on the raw material.

The black liquor was examined for its ammonia recoverability and solid content. Some of the pulps were bleached with hypochlorite in one stage or with CEH in three stages.

RESULTS AND DISCUSSION

1. Fiber Dimensions

The fiber dimensions were determined on the various raw materials used for pulping.

	straw	bagasse	bamboo
Average fiber length, mm	0.88	1.12	1.97
Average width of fibers, microns	10.1	14.0	19.9
Average fiber wall: thickness, microns	3.6	4.4	4.7
lumen width, microns	3.9	5.1	5.4
Fiber length, mm			
minimum	0.37	0.62	0.92
maximum	1.75	2.50	3.70
average	0.88	1.12	1.97
Fiber diameter D, micron	10.1	14.0	19.9
Lumen diameter, micron	3.9	5.1	5.4
Fiber-wall thickness, micron	3.1	4.4	4.8
$\frac{L}{D}$	87	80	99

The straw has very short fibers. The bagasse fibers were short also, while the bamboo fibers were of medium length. The bamboo fibers have the highest length to diameter ratio giving it a better apportunity for felting.

2. Digestion

Straw

The ammonia process was applied on straw at a rather wide range of digestion conditions. Ammonia solutions of 1 - 4 % concentrations at raw material to liquor ratios of 1 : 5.7 to 1 : 11, so that 7% to 45% ammonia based on raw material were used for the digestion experiments. The temperature for digestion was varied from 120° - 145° C at 30 - 120 minutes. During cooking in the electrically heated digesters (cook no. 1 - no. 12) the time to reach maximum temperatures varied from 95 - 220 minutes due to some defects and difficulties to control the temperature. These digesters, however, were equipped with valves for gas release, making them appropriate to study the ammonia recovery by gas release. The temperature when cooking in the rotating digesters heated by hot air were easier to control. The cook numbers in these digesters were written in roman numbers (I - XXXVII etc.).

Cook no. 1 was a preliminary cook using 17.5 % NH_3 based on raw material at 135° digestion temperature for 45 minutes (Table 1). A total yield of 53% gave a very low screened yield (13%) at a kappa number of 42. A higher liquor ratio (11 : 1) for cook no. 2 was then used sothat 44% ammonia based on straw was applied. A lower total yield was obtained (48%), the screened yield highly improved (32%), but the kappa number fell in the same range (39). By increasing the digestion temperature and prolonging the digestion time (no. 3, 4), no improvement was made. Longer digestion time lowers the yield and a slight increase in the kappa number is observed. When lower amounts of chemicals was used during digestion (no. 5, 6, 7) the adverse effect of high temperatures (145 - 150) on the kappa number is more pronounced, giving a screened pulp with kappa numbers as high as 80. Cook no. 1 to no. 7 were made on undepithed straw.

For comparison a soda cook was made (no. 8) using 10% caustic soda based on straw, at 135°C digestion temperature for 120 minutes.The liquor ratio used was 5.7 : 1. Total yield obtained was 46%, and the screened yield 45% at a kappa number of 11.9. This shows the large difference in cooking behavior between the ammonia and the soda process. In the ammonia cooks even large amounts of chemicals used were not able to give an acceptable bleachable pulp.

Cooks no. 9 - 12 were made at low chemical concentrations, 7 - 14 % NH_3 based on straw, aiming at a semi chemical pulp. The kappa numbers fell in the same range even if the digestion temperature was lowered from 135°C to 125°C.

Cook no. 13 was made in the 15 liters gas heated digester,in an effort to scale up the experiments. The screened accepts of 40.25 % were obatined by running through a refiner with 70 mils clearance. The second accepts after refining at 20 mils clearance twice was 18.39 %. During the cook the pressure rose to more than 10 kg/cm^2 sothat the gas had to be released a few times to maintain 10 kg/cm^2 working pressure. Cook no. 17 was aiming at 125°C digestion temperature. This temperature was however hard to maintain and rose to 143°C at the end of the cook . This was also observed on cook no. 5, which reached 150°C.

Table 1.

Digestion conditions on rice straw*

Cook no.	Raw material	Process	Liquor ratio	Liquor conc. %	Chemicals on bd raw material %	Max. Temp °C	Max. Press. kg/cm^2	Time to max. temp. minute	Time at max. temp. minute	Pulp Yield: Total yield %	Pulp Yield: Screened yield %	Kappa number on Screened pulp
1.	Straw (undepithed)	NH_3	5.5 : 1	3.17	17.55	135	5	95	45	52.79	13.14	41.66
2.	Straw (undepithed)	NH_3	11 : 1	4.06	44	135	7.4	139	45	48.23	32.17	39.79
3.	Straw (undepithed)	NH_3	10.6 : 1	3.93	41.5	140	8	159	90	48.52	31.96	43.32
4.	Straw (undepithed)	NH_3	8.6 : 1	4.81	42	140	8	155	120	45.75	30.49	46.42
5.	Straw (undepithed)	NH_3	5.7 : 1	1.65	9.77	125	-	170	90	51.60	34.43	79.64
6.	Straw (undepithed)	NH_3	5.9 : 1	2.46	14.5	145	9.1	220	120	51.16	27.71	79.85
7.	Straw (undepithed)	NH_3	5.9 : 1	3.48	20.5	145	9.7	160	130	56.94	27.45	62.32
8.	Straw (depithed)	NaOH	5.7 : 1	-	10	135	6.7	155	120	45.79	44.75	11.90
9.	Straw (depithed)	NH_3	5.7 : 1	2.40	13.7	135	6.8	105	120	56.28	12.2	75.35
10.	Straw (depithed)	NH_3	5.7 : 1	1.29	7.34	135	7.6	170	90	53.80	49.99	67.15
11.	Straw (depithed)	NH_3	5.7 : 1	1.26	7.2	135	9.1	170	90	59.33	58.03	64.37
12.	Straw (depithed)	NH_3	5.7 : 1	1.24	7	125	5.8	150	90	55.07	53.46	65.76
13.	Straw (depithed)	NH_3	5.9 : 1	3.06	17.44	130	10	130	120	59.06	40.25 16.30	66.19
17.	Straw (depithed)	NH_3	8.1 : 1	3.38	26.9	125	8.9	120	90	55.1	53.12	56.2

* Digestion in electrically heated shaking digesters of 4 liters capacity.

Cooks no. 8 - 17 were carried out on depithed straw. The ammonia cooks gave pulps of 55 - 59 % yield and 65 - 75 kappa numbers. These pulps were treated as a semichemical pulp by running through a laboratory Sprout-Waldron refiner at 70 mils and twice at 20 mils clearance.

Results of cooks made in the rotary digesters are shown on table 2. The digestion conditions here were easier to control and maximum temperatures could be maintained at the desired time. Cooks no. I - XII were treated like chemical pulps. the screened yields of the ammonia pulps were low since they should actually be considered as semichemical pulps. The kappa numbers were between 40 - 50 when 6 - 17.5 % NH_3 based on straw were used in the digestions, at 135°C and 125°C digestion temperatures. At these temperatures soda cooks using 10% NaOH based on straw could give 45.4 % and 25.5 % screened yields at 14 and 22 kappa numbers respectively. They could be considered as chemical pulps. Digestions with cook numbers XIII and higher were treated as semichemical pulps by additional refining at smaller clearance (20 mils), sothat high screened yields were obtained. The total yields of cooks with depithed straw were higher than when undepithed straw was used.

The soda process applied for comparison, showed that it could give a better delignification than the ammonia process, giving bleachable pulps when 10% NaOH based on straw was used. To make a better comparison a semichemical soda pulp should be made which could give similar properties as the ammonia pulp. This was obtained with cook no. XV (5% NaOH, 120°C) which was similar to cook no. XVI (24.4 % NH_3. 125°C).

Since high temperatures affect ammonia pulping adversely as shown in table 1, special attention was paid on digestions at low temperatures. Results showed that no improvement could be made when low amounts of chemicals were used in the digestions. At high chemical concentrations (cook no.XXVII),35.4 % NH_3 on straw and 125°C digestion temperature a relatively low kappa number was obtained. Cook no. XXVIII was a duplicate of no. XXVII to test the reproducibility (not shown on table 2). Results were very similar.

The lowest kappa numbers (26.3) were obtained on cook no. XXXIII , XXXIV and XXXV which were made at similar cooking conditions with good reproducibility also. The average values are shown on table 2 on cook no. XXXIII, 43.7 %

Table 2.

Digestion conditions on rice straw*

Cook no.	Raw material	Process	Liquor ratio	Liquor conc. %	Chemicals on bd raw material %	Max. Temp °C	Max. Press. kg/cm^2	Time to max. Temp. minute	Time at max. Temp. minute	Pulp Yield: Total yield %	Pulp Yield: Screened yield %	Kappa number on screened pulp
I	Straw (undepithed)	NaOH	5.7 : 1	-	10	135		90	120	49.0	45.5	14
III	Straw (undepithed)	NH_3	5.8 : 1	1.36	7.9	135		90	120	56.4	14.03	47.6
II	Straw (undepithed)	NH_3	5.7 : 1	3.17	17.55	135		90	45	62.8	12.7	39.9
IV	Straw (undepithed)	NaOH	5.7 : 1	-	10	125		90	120	44.3	25.5	22.5
V	Straw (undepithed)	NH_3	5.7 : 1	2.42	13.79	125		90	120	56.02	6.98	42.1
VI	Straw (undepithed)	NH_3	8 : 1	1.78	13.92	125		90	120	80.6	5.3	40.6
VII	Straw (undepithed)	NH_3	5.7 : 1	1.1	6.3	135		100	125	54.1	13.41	51.7
VIII	Straw (undepithed)	NH_3	5.7 : 1	2.03	11.6	135		100	125	48.0	20.1	44.4
IX	Straw (undepithed)	NH_3	5.7 : 1	2.81	16	135		100	125	52.1	22.0	44.4
X	Straw (undepithed)	NH_3	5.7 : 1	1.4	7.98	135		90	120	64.5	7.5	45.3
XI	Straw (depithed)	NH_3	5.7 : 1	2.45	13.96	135		90	120	58.9	18.3	42.2
XII	Straw (depithed)	NH_3	5.7 : 1	3.5	19.94	135		90	120	58.2	18.9	46.2
XIV	Straw (depithed)	NH_3	5.7 : 1	1.24	7	125		85	45	72.3	59.57	58.9
XIII	Straw (depithed)	NH_3	5.7 : 1		10	125		85	90	64.2	55.07	61.4
XV	Straw (depithed)	NaOH	5.7 : 1		5	120		90	90	62.6	61.08	40.3
XVI	Straw (depithed)	NH_3	5.7 : 1	2.74	24.4	125		90	90	63.7	56	39.9
XXVI	Straw (undepithed)	NH_3	9.7 : 1	4.1	35.4	125		75	30	50.7	41.6	37.8
XXXIII°°	Straw (undepithed)	NH_3	8.8 : 1	4.02	34.7	125		75	70	51.4	38.7	26.3
XXVII	Straw (undepithed)	NH_3	8.8 : 1	4.1	35.4	125		75	90	53.6	46.3	29.4
XVII	Straw (depithed)	NH_3	10.6 : 1	4.32	45.8	130		75	45	46.8	45.1	34.6
XXXVII	Straw (undepithed)	NaOH	10.6 : 1	4		room temp			120	56.2		68.7
XXXVI	Straw (undepithed)	NH_3	10.6 : 1	4.01		room temp			120	61.7		65.1

° Digestion in rotating digesters of 3 liters capacity, heated by hot air

°° The average of three digestions.

NH_3 based on straw was used at 125°C for 70 minutes. A shorter time seem not to be adequate (no. XXVI) while a longer time could also affect the pulping adversely.

Ammonia pulping at room temperature which was attemped on cook no. XXXVI seem to be inpracticable since the pulp obtained were not defiberized but ground into dust.

Bagasse

The pulping experiments on bagasse is shown on table 3.The effect of time temperature and chemicals used could be seen from this table.It seems that the ammonia process could not give any acceptable bleachable pulps, even if very high amounts of chemicals were used during the digestion.Cooks at high and lower liquor ratios gave similar results,contradicting the claims of the inventor. The kappa numbers of the ammonia cooks were extremely high.

The soda cooks made for comparison using 15% NaOH gave kappa numbers in the range of 22 - 26 and were treated as a chemical pulp. Even if a higher amount of chemicals and a higher temperature was needed than when digesting straw to obtain a satisfactory bleachable pulp, here again the soda process proofs to be superior in its delignifying action than the ammonia process.

Bamboo

Experiments on straw and bagasse by the ammonia process showed that this process delignify with great difficulty plant material of open structure and accessibility. It would therefor be inacceptable to use it for bamboo. Cook no 18 support these findings. Treatment of this pulp as a semichemical one for defiberization similar to straw and bagasse gave a very low screened yield (13.7 %) at a high kappa number (151).For comparison purposes the soda process was applied on bamboo using 18% NaOH,at 168°C digestion temperature (no.XXXII). A kappa number of 41 was obtained.

3. Bleaching Experiments

The pulps obtained by the ammonia process were mostly dark in colour.Some of the pulps were bleached in a one stage hypochlorite bleach and a few in

Table 3.

Digestion conditions on bagasse and bamboo

Cook no.	Raw material	Process	Liquor ratio	Liquor conc. %	Chemicals on bd raw material %	Max. Temp °C	Max. Press. kg/cm²	Time to max. temp. minute	Time at max. temp. minute	Pulp Yield: Total yield %	Pulp Yield: Screened yield %	Kappa number on screened pulp
14	Bagasse (depithed)	NH_3	6.5 : 1	2.33	15.1	135	4.2	77	120	84.6	30.2	88.3
15	Bagasse (depithed)	NH_3	6.5 : 1	3.38	21.8	135	5.9	90	120	71.1	64.4	125
16	Bagasse (depithed)	NH_3	6.5 : 1	4.06	26.2	145	10	90	120	69.6	60.3	131.4
XVIII	Bagasse (depithed)	NH_3	10.6 : 1	4.32	45.4	130		75	120	71.3	46.8	112.9
XXI	Bagasse (depithed)	NH_3	10.6 : 1	4.06	43	140		90	120	77.8	34.3	107.1
XXV	Bagasse (depithed)	NH_3	8 : 1	3.30	26.4	130		75	45	77.4	55.4	116.6
XXIV	Bagasse (depithed)	NH_3	8 : 1	3.30	26.4	130		75	90	73.6	48.9	119.3
XXII	Bagasse (depithed)	NH_3	5.6 : 1	4.06	22.7	140		90	120	76.7	40.5	119.1
XIX	Bagasse (depithed)	NaOH	5.7 : 1		15	130		75	120	55.9	32.7	26.2
XX	Bagasse (depithed)	NaOH	5.7 : 1		15	140	-	90	120	56.5	38.9	22.8
XXIII	Bagasse (depithed)	NaOH	5.7 : 1		15	130		75	90	54.6	31.5	26.6
XXIX	Bagasse (depithed)	NH_3	8.7 : 1	4.04	32.3	130		85	45	[illegible]	57.8	131
XXXI	Bagasse (depithed)	NH_3	7.1 : 1	4.04	28.7	130		85	45	71.3	54.3	120.7
XXX	Bagasse (depithed)	NH_3	5.7 : 1	4.04	23.0	130		85	45	74.4	59.9	119.0
18	Bamboo	NH_3	9.5 : 1	3.98	36.8	130			180	80.4	13.7	151
XXXII	Bamboo	NaOH	3.5 : 1	5.72	18	165			150	45.4	45.0	41

three stages by chlorination, extraction and a hypochlorite treatment. The bleaching experiments were all performed on the accepted fraction of the pulps after screening.

The one stage bleaching trials were done at 5% consistancy and 40°C. The time for bleaching varies from 30 minutes to 300 minutes depending on the rawness of the pulps and the amount of chlorine applied as hypochlorite.

In the three stage bleaching experiments, the chlorination stage was carried out at 25°C for 120 minutes, the alkali extraction stage at 70°C for 120 minutes and the hypochlorite stage at 40°C.

The results of the bleaching trials are shown in table 4. Brightnesses between 70 - 80 % GE could be obtained in a one stage bleach by using 4% chlorine on the bleachable soda pulp (no.I) and 27% chlorine on ammonia pulp no.13. The best ammonia pulp with the lowest kappa number no. XXXIII were bleached with 10% chlorine based on pulp to reach 71% GE brightness. When the three stage bleaching was applied on this pulp a brightness of 85.5 % GE could be reached.

4. Pulp Evaluation

The result of the physical evaluation of the straw pulps are given in table 5. The pulps are listed in groups having kappa numbers of the same range. The physical characteristics of pulps unbeaten and beaten to 40°-60°SR listed, shows that for ammonia pulps these were on the low side.To reach a freeness of 40°SR some of the ammonia pulps have to be beaten for 40 minutes (no.10, 13_{II}) while for no. 8 soda pulp this was reached in 10 minutes.

For pulps with kappa numbers in the range of 40, cook no.1 has the lowest physical strength as well as the lowest brightness. This cook was carried out at a high liquor ratio (11 : 1) with 44% NH_3 based on straw.The screened yield without further refining (treated as a chemical pulp) was actually on the high side. Even if this pulp was not well delignified some degradative action by ammonia solutions or vapours must have taken place. Pulp no. XV is comparable with pulp no. XVI not only in yield and kappa numbers but also in physical strength. The brightness of pulp XV, which is a soda cook (5% NaOH),however is higher by more than 10 points compared to pulp XVI (24.4 % NH_3).

Table 4

Bleaching experiments on straw and bagasse pulps

Pulp from cook no.	Type of raw material	b.d. yield raw pulp %	Kappa no.	Bleaching chemicals consumed, on finished bleached pulp at 10% moisture: Cl_2 %	NaOH %	Cl_2 (as hypo) %	Time minutes	Bleaching losses % on b.d. pulp	Final bleached yield % (10% moisture)	Brightness % GE
3	Straw (undepithed)	48.52	43.2			12.37	75	8,93	49.09	58.75
3	Straw (undepithed)	48.52	43.2			21.90	150	13.72	46.51	68.00
XXVIII	Straw (undepithed)	53.69	27.04			13.42	130	9.48	54.00	78.00
I/NaOH	Straw (undepithed)	48.37	14.26			3.95	60	8.94	48.93	72.30
B°	Straw (undepithed)	51.40	25.00			9.90		9.02	51.96	71.10
B°	Straw (undepithed)	51.40	25.00	4.92	2.02	5.04		10.72	50.99	85.50
13	Straw (depithed)	59.00	66.00			9.61	30	6.40	61.36	51.48
13	Straw (depithed)	59.00	66.00			18.54	120	15.07	55.67	67.85
13	Straw (depithed)	59.00	66.00			27.27	300	17.52	54.07	73.53
17	Straw (depithed)	55.00	55.00			21.88	180	17.74	50.27	63.30
XI	Straw (depithed)	55.50	46.00	7.49	2.75	6.43		18.38	50.33	82.30
XXV °°	Bagasse (depithed)	75.37	116.60			26.81	120	16.90	70.26	60,10
XXI °°°	Bagasse (depithed	77.76	107.00	15.34	3.04	8.29		23.87	65.94	

° mixture of pulps XXXIII + XXXIV + XXXV

°° bleaching carried out on a fraction corresponding to 73 % of total yield,(accepted screened pulp)

°°° Bleaching carried out on a fraction corresponding to 43 % of total yield,(accepted screened pulp)

Table 5

Physical characteris of straw pulps unbeaten and beaten to 40°-60° SR*

Pulp from cook no.	Kappa number	° SR	Tear factor	Burst factor	Breaking length m	Double folds	Brightness G.E
2	39	26	25	19	1700	1	29
		46	21	30	2600	2	
		57	22	31	3100	5	
IX	44.4	22	45	19	2100	2	40
		38	40	27	3300	2	
		68	33	33	3900	2	
XV	40	24	47	20	2700	2	55
		40	37	27	4100	7	
		62	38	35	4300	9	
XVI	40	25	43	25	2400	2	41
		42	32	33	4100	5	
		59	28	35	3700		
7	62	29	39	24	2000	2	24
		41	38	38	3600	5	
		61	28	41	3630	3	
10	67	24	46	24	2700	3	27
		41	41	35	4600	10	
		64	29	38	4400	11	
XIII	61	22	31	17	1900	1	30
		43	29	26	3400	4	
		60	28	33	3500	5	
13 I	66	34	34	23	2300	3	23
		59	30	33	4200	6	
13 II	57	17	41	20	1800	1	23
		40	44	34	3800	6	
		60	39	39	4300	14	

(continued)

Pulp from cook no.	Kappa number	° SR	Tear factor	Burst factor	Breaking length m	Double folds	Brightness G.E
XXVII) A XXVIII)	29	23	31	28	2200	1	38
		42	34	38	3600	5	
		61	26	37	3900	9	
" * " Bleached			87	26	3693	5	
XXXIII)	26	30	42	29	3000	3	40
XXXIV) B		41	36	36	4200	4	
XXXV)		60	32	44	4700	7	
" * " Bleached (3 stages)		36	34	26	3400	5	85.5
		61	37	41	5000	13	
" * " Bleached (1 stage)		33	38	29	3400	5	71
8	11.9	28	61	36	4600	87	44
		39	52	50	6100	192	
		63	38	54	7300	532	
I	14.3	37	40	32	3900	6	50
		63	35	55	5800	46	
IV	22.5	30	47	31	3500	5	42
		56	49	53	5900	95	
XXXVII	68.7	8	10	15	300		39
		42	19	17	500		
		55	14	18	800		
XI Bleached (3 stages)			32	35	3500		

° The pulps were beaten in a laboratory niagara beater and in a PFI mill

Ammonia pulps with kappa numbers in the range of 60 - 70 were rather dark as shown in group 2 of table 5. Brightness values were between 23-30 % GE. The best strength characteristics are shown by pulp no. 10. A breaking length of 4600 m was obtained at 40°SR with 41 tear factor and 35 burst factor.

The pulp mixture A (XXVII + XXVIII) and B (XXXIII + XXXIV + XXXV) belongs to the group with low kappa numbers. Pulp mixture B with the lowest kappa number of all the ammonia pulps obtained so far has also relatively good physical properties in the unbeaten as well as the beaten state.Breaking length of 4700 meters and 44 burst factor was reached at 60°SR. All the ammonia pulps show, however, very low folding endurance.

The soda pulps (8, I, IV) have superior physical properties compared with the ammonia cooks as shown in group 4 of table 5. Despite the fact that straw fibers are short, in the unbeaten state sheets of 4600 m breaking length, 61 tear factor and 36 burst factor could be obtained, with 87 double folds. By beating to 63°SR, these would become 7300 m, 38 and 61 respectively. Double folds amounts to 532 at this freeness.

Sheets of very low strength were obtained from cold soda pulping.No sheets could be made from the cold ammonia pulping. Evaluation of bleached pulps showed that the strength properties were not adversely affected.

Table 6 shows the results of pulp evaluation tests on bagasse and bamboo pulps. Bagasse and bamboo are better pulp raw material than straw. By applying the ammonia process, however, pulps of similar physical characteristics as ammonia straw pulp were obtained. The superior qualities of the soda pulps are shown on pulp no. XX and XXXII. High breaking lengths (8000 m) and burst factors (65) were obtained from bagasse and bamboo soda pulps. The high tear factors of which were obtained on soda pulps, were completely absent on ammonia bamboo pulp.

4. <u>Chemical Analysis</u>

The pulping experiments made by the ammonia process seem to behave differently from conventional processes as well as from claims made by the inventor. To have a better understanding of the process and also of the pulp

Table 6

Physical characteristics of bagasse and bamboo pulps

Pulp from cook no.	Kappa number	° SR	Tear factor	Burst factor	Breaking length m	Double folds	Brightness G.E
Bagasse							
14	86	9	23	18	700	-	22
		45	23	25	2600	2	
		66	21	30	3600	7	
15	125	10	47	17	1800	1	27
		40	51	33	5200	25	
		62	36	39	5800	33	
XXX	119	13	27	13	445	-	30
		36	39	19	2800	1	
		60	37	25	3300		
XXIX	130	14	31	14	551	-	
		40	47	23	3000	3	
		59	44	27	4100	3	
XX (soda)	22.8	9	76	31	3600	9	44
		40	62	61	7900	342	
		63	41	65	8200	689	
Bamboo							
18		9	32	18	5200	-	26
		27	53	21	2200	1	
		49	60	20	2800	2	
XXXII (soda)		7	169	28	2000	-	35
		42	148	67	8100	472	
		65	109	72	8500	524	

behavior, a chemical analysis was made on some straw pulps as shown in table 7. The raw material used for pulping was also analyzed.

The analysis shows that the ammonia pulps with kappa numbers in the range of 40 - 50 contains 5% lignin. The soda chemical pulps on the other hand have low lignin contents but high pentosans content. The pentosans content of the ammonia pulps were relatively low, compared with the soda pulps. This explains the pulp behavior during beating. The ammonia pulps were rather hard to beat and the strength developemnt was slow.

Pulp XV and XVI which were very similar in behavior during pulping as well as during beating have also very similar chemical composition. Pulp XXXIII mixture having the lowest kappa number among the ammonia pulps have also a low lignin content. The kappa numbers and the lignin contents show good correllation. The adverse effects of high temperatures on delignification is shown on pulp no. 5. A lignin condensation is probably taking place. Similar behavior was observed on former soda pulping experiments on grasses when alkali concentration used were on the low side. High temperatures during ammonia pulping would convert the ammonia into the vapour phase, making it inaccesible for delignification.

The ash and silica content of the ammonia pulps were twice as high as the chemical soda pulps. This explains again its inferior strength properties developed during beating.

5. Ammonia Recovery and Black Liquor Analysis

One of the advantages of the ammonia process claimed was its simplified chemical recovery. The posibilities of ammonia recovery was examined on all digestion experiments. This was carried out by gas release as well as by black liquor distillation. The gas could be released from cooks 1-19 carried out in the electrically heated digesters, equipped with release valves. The black liquors remaining which might still contain ammonia were then distilled for further ammonia recovery.

The rotating air-heated digesters were not equipped with release valves. Ammonia recovery was carried out by straight distillation by black liquor,

Table 7

Chemical analysis of straw pulps and raw material
(% based on bone dry material)

Sample	Ash	SiO_2	Lignin	Alc-benz. extract	Alpha* cellulose	Pentosans	Solubilities	
							Hot water	1% NaOH
Straw pulp								
2	23.08	21.16	5.10	1.92	81.59	16.32		
5	21.96	20.35	9.97	3.21	80.27	13.95		
8 (soda)	11.76	9.89	1.99	1.32	78.86	23.87		
13_I	22.43	20.80	8.16	2.76	82.13	14.93		
13_{II}	24.17	23.05	8.80	2.16	85.16	16.40		
7	23.42	21.48	7.47	2.75	83.02	11.40		
I (soda)	10.10	8.93	0.81	1.49	76.66	23.72		
IV (soda)	20.94	18.53	1.69	1.68	78.05	21.99		
VII	23.29	20.76	5.85	3.43	78.90	10.68		
VIII	23.11	20.91	4.85	3.19	80.03	11.53		
IX	23.89	21.54	5.23	2.96	81.95	11.89		
XV	20.48	19.02	5.08	2.11	78.06	17.61		
XVI	20.71	19.62	5.56	1.99	78.15	18.11		
XXXIII mixture	23.00	21.50	2.29	2.08	76.27	16.34		
Raw material								
Straw (undepithed)	21.69	15.85	11.94	5.35	28.78	19.77	14.46	50.09
Straw (depithed)	17.95	16.07	13.47	2.84	52.02	22.14	7.55	47.68
Bagasse (depithed)	1.59	1.07	20.38	1.69	45.82	26.06	1.00	28.29

* not yet corrected for ash.

after cooling down and opening the digestion apparatus.The black liquors were also analyzed for its solid and ash contents. The results are given in table 8.

When the gas was released immediately after digestion, the ammonia recovered was between 50 - 78 % and a further 4 to 26 % obtained from black liquor distillation. To improve ammonia recovery an experiment was made in which air was injected after the gas release. After injecting air for 60 minutes an additional 14% of the ammonia was recovered, sothat around 90% could be obtained without distillation. The ammonia obtained from the black liquor distillation was another 2.5 %.

Straight black liquor distillations from cooks carried out in the rotating digesters (no. II-XXVVI) could recover 60 to 95 % ammonia based on feed. Losses or consumption were therefore 5 to 40 %. When these losses or consumption was calculated based on the amount of raw material digested, consumption was between 1 - 8 %.

The total solids of ammonia black liquors, were lower than those of the soda process. This is expected since delignification during the ammonia process was markedly lower. The ammonia lignin precipitated by acidification consisted of very fine particles which went through the filtering medium, sothat no determinations could be made. Compared with soda black liquors the ash contents of ammonia liquors were also low. This is in agreement with chemical analysis made on the pulps. The ammonia pulps of straw have very high ash contents compared with the soda pulps. It seems that the soda process was able to put the inorganic part of the straw into solution.

Even if the solids content of the ammonia black liquors were relatively low, visually the liquors from straw cooks were very dark in colour, darker than the soda black liquors. A lighter shade was observed on liquors from bagasse cooks.

From ammonia recovery studies it is shown that a large part of the ammonia was not consumed during digestion, sothat black liquors could be reused without any additional treatment. This is especially true when digesting with high percentages of ammonia. This was experimented also, by reusing the black liquor of a cook in which 20% NH_3 on raw material was used.In the first stage

Table 8

Ammonia recovery and black liquor analysis

No.	Cook no.	NH3 Feed gram	NH3 on material %	Recovery Gas release %	Black liquor %	Total %	Losses %	%[a]	Black liquor analysis Total solid %	Ash %
1.	1	88.5267	17.55		66.5	66.5	33.5	5.9	5.91	1.14
2.	2[b]	148	44	21.4	72.2	93.6	6.4	2.8	2.59	0.45
3.	3	139	41.5	11.28	53.9	65.18	34.8	14.4	5.89	1.96
4.	4[c]	174.6	42	24.2	26.3	50.5	49.5	20.6	3.68	0.26
5.	5	49.26	9.77	49	20.26	69.26	30.73	3	4.82	1.13
6.	6	73.132	14.5	56.48	4.3	60.78	39.22	5.69	5.82	1.43
7.	7	103.43	20.5	58.45	3.99	62.44	37.56	7.7	4.41	1.03
8.	9[c]	35.875	13.7	17.63	2.5	20.13	79.9	10.9	4.6	0.34
9.	10	38.53	7.34	57.35	11.5	68.85	31.15	2.29	4.24	0.25
10.	11	37.75	7.2	58.28	7.2	65.48	34.54	2.48	3.76	0.26
11.	12	37	7	59.7	6.1	65.8	34.2	2.4		
12.	13	305.66	17.44	64.4	15	79.4	20.5	3.58	5.09	0.31
13.	14	74.56	18.6	78.34	7.91	86.25	13.75	2.56	1.95	0.13
14.	15	108.16	27	61.8	26.73	88.53	11.47	3.1	1.29	0.06
15.	16	104.93	26	66.77	12.87	79.64	20.36	5.3	3.69	0.11
16.	17	107.48	27	76.06	8.43	84.49	15.51	4.17	3.43	0.27
17.	18	138.902	37		89.53	89.53	10.45	3.85	1.4	0.25
18.	19	137.241	36.4		61.7	61.7	38.3	13.93		
19.	II	66.38	17.38		91.6	91.6	8.4	1.48	3.5	0.61
20.	III	29.98	7.93		68.88	68.88	31.12	2.5	3.72	0.74
21.	V	52.17	52.17		90.8	90.9	9.1	1.26	3.11	0.3
22.	VI	40.94	13.92		79.3	79.3	20.7	2.9	2.06	0.25
23.	VII	23.71	6.3		84.6	84.6	15.4	0.97	5.0	0.89
24.	VIII	43.76	11.6		94.6	94.6	5.4	0.63	5.69	0.86
25.	IX	60.58	16		78.3	78.3	21.7	3.48	5.24	0.93
26.	X	31.41	7.98		57.2	57.2	42.8	3.4	3.15	0.26
27.	XI	54.97	13.96		62.1	62.1	37.9	5.3	3.22	0.29
28.	XII	78.524	19.94		66.03	66.03	33.96	6.77	3.12	0.23
29.	XVI	68.3	24.4		72.2	72.2	27.8	6.77		
30.	XVIII	113.62	45.4		84.4	84.4	15.6	7.1	1.76	0.04
31.	XXI	108.12	43		81.17	81.17	18.23	7.9	2.08	0.03
32.	XXII	57.855	23.1		67	67	33	7.6	2.1	0.08
33.	XXIV	66	26.4		80	80	20	5.28	2.14	0.1

(continued)

No.	Cook no.	NH_3 Feed gram	NH_3 on material %	Recovery Gas release %	Black liquor %	Total %	Losses %	%[a]	Black liquor analysis Total solid %	Ash %
34.	XXV	66	26.4		79	79	21	5.52	1.9	0.1
35.	XXVI	99.22	35.4		92.78	92.78	7.22	2.56	2.22	0.5
36.	XXVII	99.22	35.4		81.45	81.45	18.55	6.6	2.89	0.65
37.	XXVIII	99.22	35.4		76.21	76.21	23.79	8.43	3.19	0.72
38.	XXIX	95.22	31.74		82.4	82.4	17.6	5.58	1.7	0.11
39.	XXX	67.35	22.45		70	70	30	6.66	2.43	0.11
40.	XXXI	67.5	28.13		57.3	57.3	42.7	12	2.91	0.12
41.	XXXIII	97.284	35		85.36	85.36	14.64	5.09	3.5	0.91
42.	XXXIV	97.284	35		87.64	87.64	12.36	4.29	3.41	0.67
43.	XXXV	97.284	35		86.62	86.62	13.38	4.65	3.46	0.76
44.	XXXVI	97.284	35		92.5	92.5	7.5	2.6	1.14	0.45

a. based on b.d. raw material
b. cooled down before releasing
c. leak

Soda pulping

Cook no.	Total solid %	Ash %
8	13.62	5.31
I	6.16	2.32
XV	8.90	3.05
XIX	10.25	3.50
XX	10.31	3.44
XXIII	9.73	3.29

3.44 % ammonia was consumed and in the second stage 5.72 %. The total solids in the first black liquor was 3.10 % and the second 5.39 %. An increase in kappa number is observed also.

Black liquor XXIII (bagasse soda) and XXIV (bagasse ammonia) were analyzed for their BOD and COD values. For liquor XXIII BOD_5 was 17130 ppm and COD 89056 ppm, while for liquor XXIV these were 8970 ppm and 27222 ppm respectively.

6. Microscopical Studies

Evaluation of the ammonia process was not only carried out by studying physical and chemical properties but also by examining the pulps under the microscope. Photomicrographs were made and shown in the following figures. These photomicrographs should however be studied with reservations since it could only show a very small part of the sample and could give the wrong impression. It is safe to say,however, that most of the pulps were undercooked, except for the chemical soda pulps and showed a large amount of broken fibers and fiber bundels. The inability of ammonia solutions at room temperature to soften the middle lamelia and act as a lubricant during refining is shown by the photomicrograph of pulp XXXVI (pulping by soaking at room temperature). No defiberization occurred. In contrast photomicrograph of pulp mixture B (XXXIII - XXXV) showed a well defiberized sample. This pulp was the most delignified of the ammonia pulps.

CONCLUSIONS

Compared to the soda process the ammonia process is at a disadvantage, even when appled to rice straw, since it delignifies with difficulty, but attacks the carbohydrate fraction, resulting in pulps with lower sheet strength properties. Application of the ammonia process on bagasse and bamboo is inpracticable.

High quality equipment is needed in ammonia pulping, since high pressures are developed during the process, which means that high capital investment has to be made.

Recoverability of the chemicals is a must since high amounts of chemicals has to be applied to have reasonable results, even if only a small amount of ammonia is consumed. This requires additional equipment and arrangements which need further studies, especially from the point of view of economics and practicability.

Last but not least it is rather unpleasant to work with ammonia even on a laboratory scale. Operation in closed systems should therefore be considered.

Due to the points mentioned above it would therefore be unacceptable to recommend the ammonia process as an alternative to the soda process, especially for developing countries.

Bandung, 27th August 1981
For Cellulose Research
Institute

DR. Roehjati Joedodibroto

A P P E N D I X

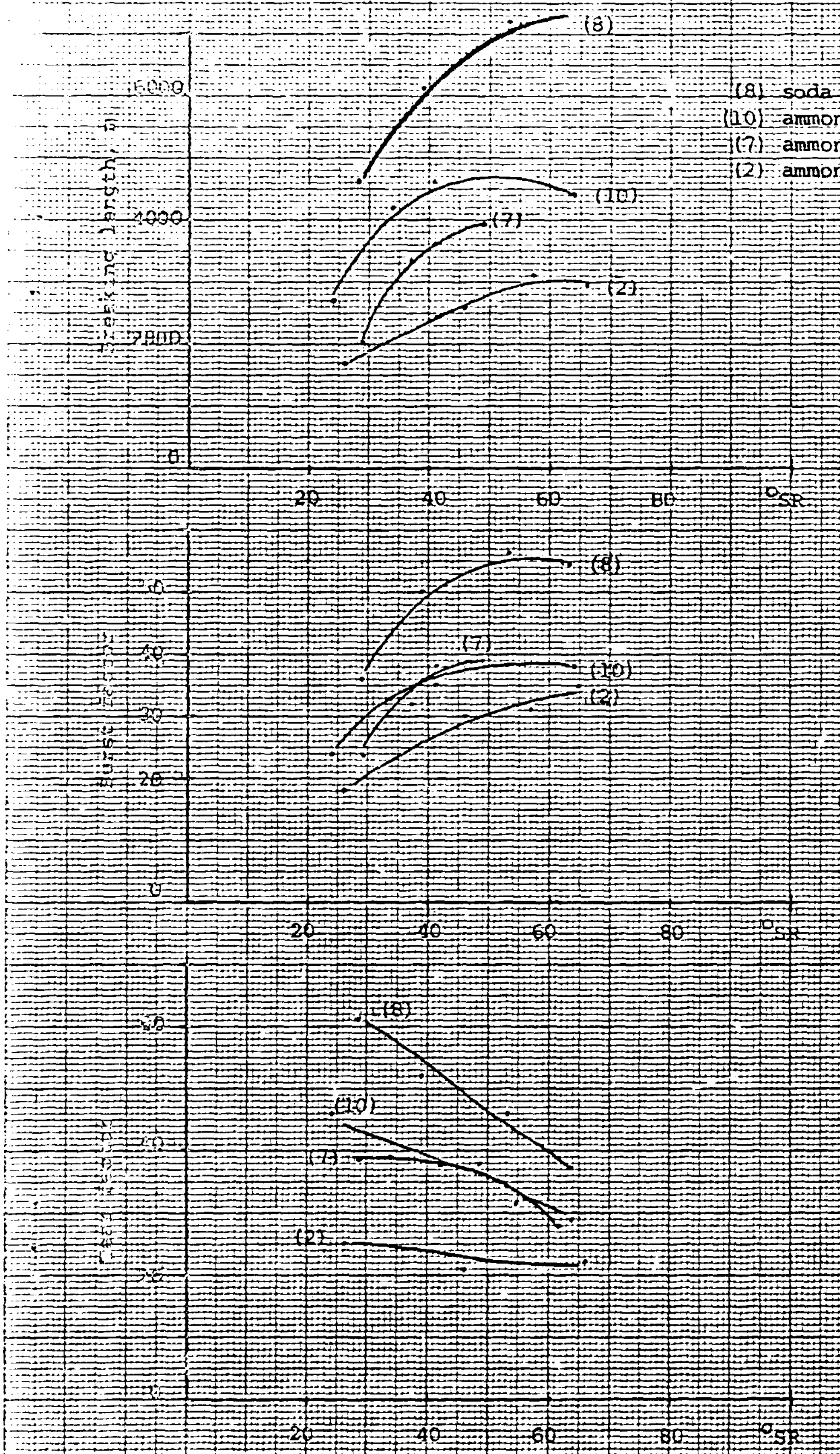


Fig. 1. Strength development of straw pulps.

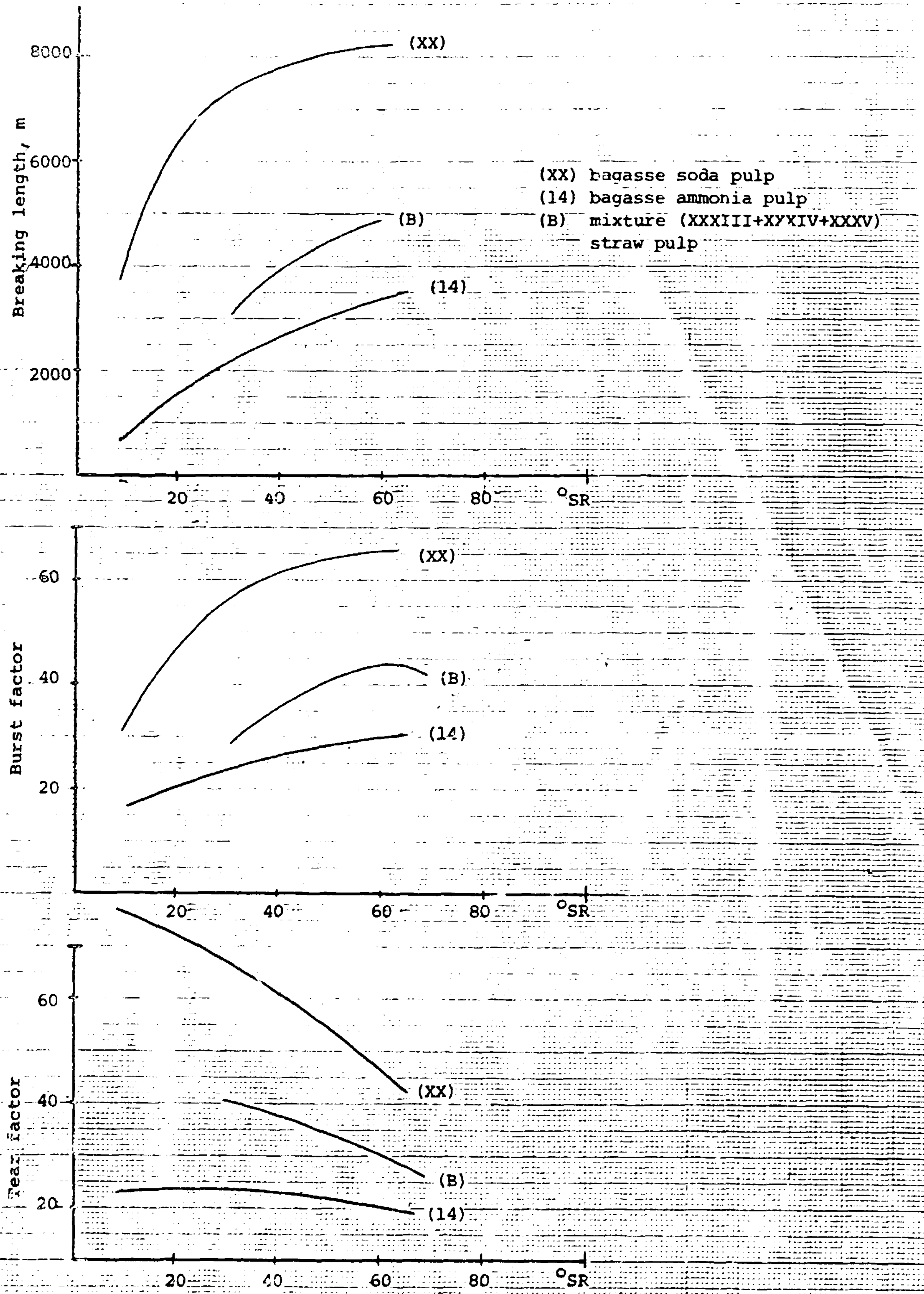


Fig. 2. Pulp strength development.

Fig. 3. Photomicrographs of screened straw pulps

a. Pulp I (soda) d. Pulp 10

b. Pulp 2 e. Pulp 13_{I}

c. Pulp 7 f. Pulp 13_{II}

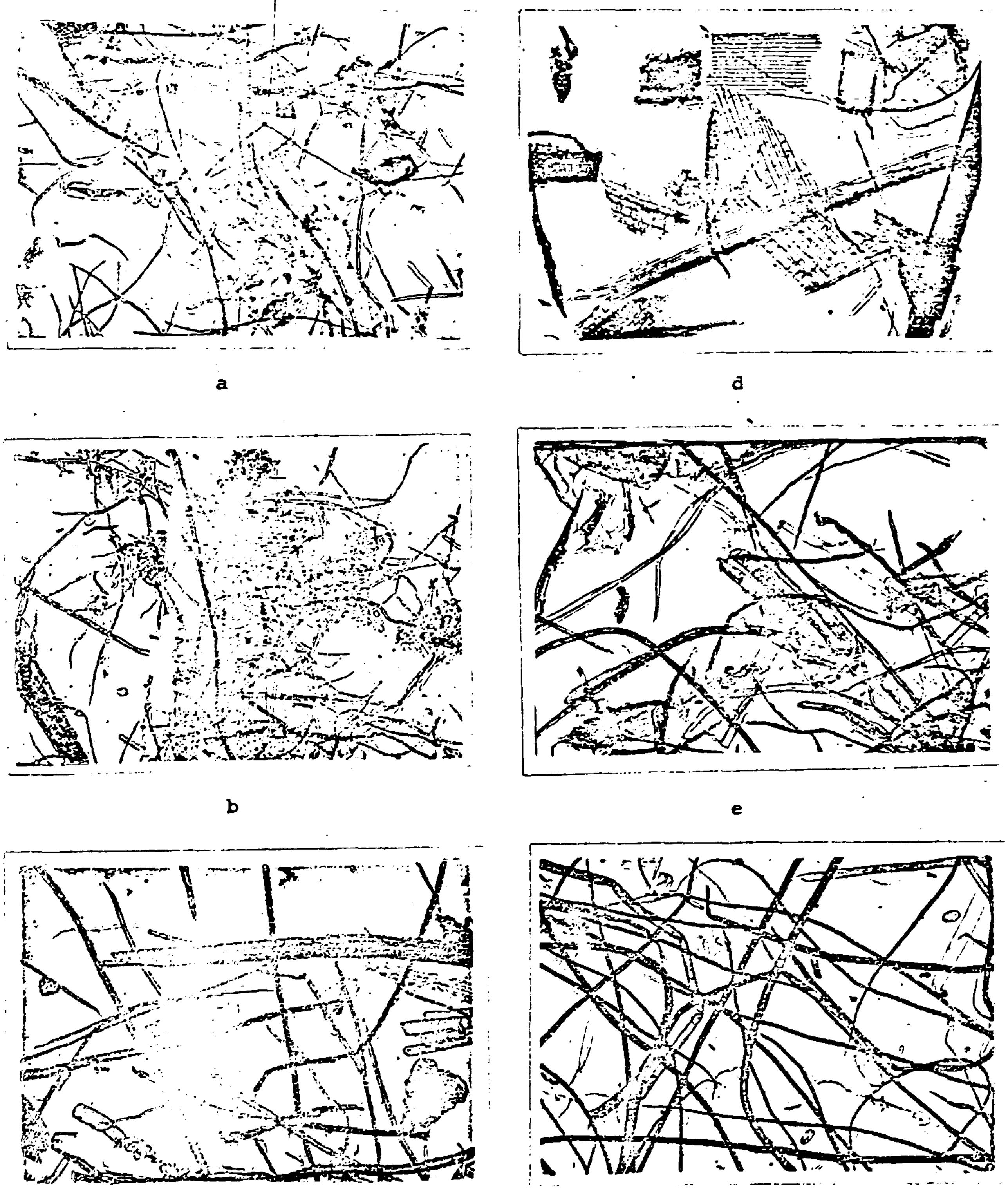
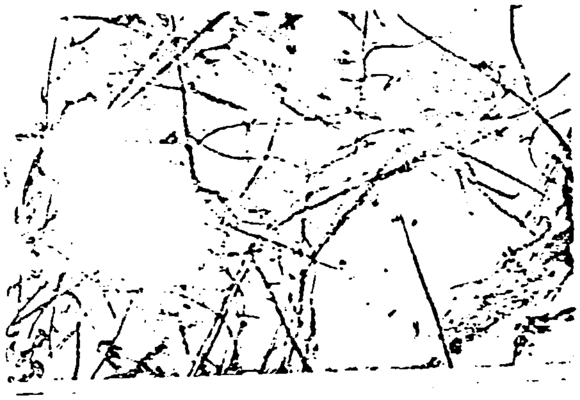


Fig. 4. Straw, Bagasse and Bamboo screened pulps.

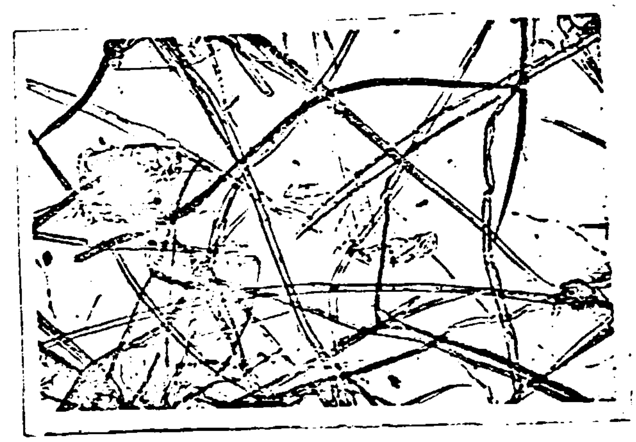
a. Pulp B (mixture straw)
b. Pulp XXVIII (straw)
c. Pulp 18 (bamboo)
d. Pulp XXXVI (cold ammonia)
e. Pulp 15 (bagasse)
f. Pulp XXXII (bamboo soda)



a

c

b



d

Fig. 5.

a. Pulp XXVII (straw ammonia pulp)
b. Pulp XVI (straw ammonia pulp)
c. Pulp XXVIII (straw ammonia pulp)
d. Pulp XX (bagasse soda pulp)