

Chemical pulp from kneaf by neutral sulphite

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# APPLIED SCIENTIFIC RESEARCH CORPORATION OF THAILAND

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# REPORT NO. 5 CHEMICAL PULP FROM KENAF BY NEUTRAL SULPHITE PROCESS

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By Chien Chu, \* Naiyana Niyomwan, \* and Anchalee Puangvichit\*

#### SUMMARY

Kenaf responded well to neutral sulphite pulping featuring high yield of pulp with good strength but bleach demand was high and amount of chemicals applied for preparing bleachable pulp was high requiring a chemical recovery system.

Fresh Cuban kenaf (Hibiscus cannabinus) chips yielded 63% pulp by heating in a sodium sulphite solution at liquor ratio 4:1 for 3 hours to 170°C and cooking at 170°C for 3 hours. The sodium sulphite solution contained 18 per cent sodium sulphite on ovendry chip weight. The solution was buffered with 2 per cent sodium bicarbonate. The pulp was bleached to brightness 70 per cent with DC/E/D sequence, which had a total of 12 per cent equivalent chlorine. The bleached yield was 79 per cent on pulp weight or 50 per cent on ovendry chips. The bleached pulp had good quality comparable to bleached kenaf kraft pulp except for lower opacity.

Air dry Cuban kenaf gave 56% unbleached pulp by cooking at liquor ratio 3:1 with 16.4 per cent sodium sulphite. The cooking condition was heating in 2 hours to 170°C and cooking at 170°C for 2 hours. The crude pulp was refined and washed. The pulp has good strength and light colour.

Chemical pulp with bleached yield about 50 per cent and brightness 80 per cent was obtained from air dry Thai kenaf (H. sabdariffa). The kenaf chips were cooked with 21 per cent sodium sulphite buffered with 2.5 per cent sodium bicarbonate at liquid ratio 3:1. The cooking condition was heating to  $170^{\circ}$ C in 2.5 hours and cooking at  $170^{\circ}$ C for 3 hours. The crude pulp after refining and washing gave a yield of 62 per cent.

The strength of the pulp from Thai kenaf was developed by bleaching in C/E/H/D sequence with a total of 13.5% equivalent chlorine for a brightness about 80 per cent. The pulp has good strength superior to softwood sulphite pulp except for opacity and tearing strength.

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## INTRODUCTION

Neutral sulphite process has been widely used for pulping hardwood to produce high yield semi-chemical pulp, which is usually unbleachable. Hence this process is known as NSSC process i.e. neutral sulphite semi-chemical process.

Pulping dry Cuban kenaf (<u>Hibiscus cannabinus</u>) by neutral sulphite process was distinguished for its high yield of strong pulp but requiring more bleaching chemicals when compared with pulps by soda and kraft processes as reported by Clark <u>et al</u>. (1962).\* For unbleached pulp the neutral sulphite process has apparent advantages of light colour, good strength, and high yield over most other processes for pulping kenaf.

Also pulp yields and bleachability vary with alkaline buffer chemicals in the neutral sulphite pulping. Fresh and dry kenaf respond to this pulping process with different yields and strength characteristics. All these variables warrant experimental study to appraise the viability of this process for bleached pulp from kenaf.

#### EXPERIMENTAL

Kenaf chips were cooked in a laboratory tumbling digester with a cooking solution containing 16-18% sodium sulphite and 2-5% alkali buffer such as soda ash, or caustic soda, or sodium bicarbonate. The liquor to chips ratio varied from 3:1 up to 4:1. The cooking conditions were heating to a maximum temperature  $170^{\circ}$ C in 2 to 3 hours and cooking at  $170^{\circ}$ C for 2 to  $3\frac{1}{2}$  hours. At the end of cooking, the digester was vented for 15 minutes to cool down to about  $120^{\circ}$ C before opening the digester. The crude pulp was washed and passed through a disc refiner with a stream of water to open the fibre bundles. The pulp was washed and dewatered in a centrifuge. The wet cake was used in bleaching test.

<sup>\*</sup>CLARK, T.F., NELSON, G.H., NIESCHLAG; H.J., and WOLFF, I.A. (1962).—A search for new fiber crops. V. Pulping studies on kenaf. TAPPI 45(10): 780-786.

Multi-stage bleaching was applied to the refined kenaf pulp without screening. The first stage was chlorination for 0.5 hour at room temperature and 3% pulp consistency in a polyethylene bottle with cover. The chlorine was generated by soaking the pulp in hypochlorite solution acidified to pH 1.8 with dilute hydrochloric acid.

For better quality, the chlorination stage was done with a combination of chlorine dioxide gas and chlorine gas. This stage was carried out in a same way as the usual chlorination stage, i.e. at room temperature and 3% consistency. However, the pulp was first soaked in a sodium chlorite solution in a closed polyethylene bottle and then the solution was acidified with dilute hydrochloric acid to pH 3.0 to generate the chlorine dioxide gas. The pulp slurry was shaken for five minutes. The sodium hypochlorite solution was added and followed by dilute hydrochloric acid to bring the pH value to 1.8 for generation of free chlorine gas. The bottle was shaken every five minutes. After half an hour retention time, the bottle was opened and the pulp was washed.

The washed pulp entered directly into the second stage of caustic extraction in which the pulp slurry was extracted in a beaker with 1.5-2.0% caustic soda for half an hour at 10% consistency. The extract had a dark brown colour. The pulp was washed and bleached by the usual bleaching stages.

The hypochlorite bleaching stage was done in sodium hypochlorite solution with available chlorine at about 1-2 per cent on the basis of ovendry pulp. The bleaching condition was  $40-45^{\circ}$ C for 2-3 hours at 10% pulp consistency.

The chlorine dioxide bleaching stage usually came as the last stage of bleaching. It was done at about 10% pulp consistency in a closed polyethylene bottle soaked in a water bath to maintain the temperature at  $70^{\circ}$ C for 4 hours.

The chlorine dioxide gas was generated by soaking the pulp in sodium chlorite solution acidified with dilute hydrochloric acid to pH 3-4.

Acid washing with  $SO_2$  solution was the final stage of the bleaching sequence. The  $SO_2$  solution was prepared by acidifying a sodium bisulphite solution with dilute hydrochloric acid to pl 3.0. The bleached pulp was ovendried to determine the bleached yield.

Handsheets were prepared from the bleached pulp. The pulp slurry was dispersed in a disintegrator and beaten in a laboratory Hollander beater at 0.3% consistency for 3-5 minutes. The Schopper-Riegler (S.-R.) freeness before beating and after beating was measured. Sizing materials consisting of 2% rosin size, 4% alum, and 2% tamarind kernel powder were added to the beaten pulp before sheet formation. The handsheets were dried and conditioned for 24 hours in a room maintained at 23°C and 50% relative humidity.

Physical tests were made in accordance to TAPPI methods in respect to basis weight, bulk, tearing strength, bursting strength and breaking length. The results are shown in Table 1.

## RESULTS AND DISCUSSION

The data shown in Table 1 are conditions of cooking and bleaching and results of physical testings of pulp and handsheets. The neutral sulphite process for pulping fresh Cuban kenaf gave strong pulp with moderate bleach demand. Dry kenaf required more pulping chemicals and higher bleach demand and gave lower yield and lower quality.

When different buffering agents in neutral sulphite pulping are compared, the sodium bicarbonate buffer gave the highest unbleached yield and bleached yield, although the bleach demand was slightly higher than with the other two buffers. Sodium carbonate buffer and caustic soda buffer gave lower cooking yields and bleached yields. Difference of yields between these two buffers was not significant.

For unbleached pulp to make wrapping paper, the neutral sulphite process without buffer as indicated in sample No. 1 for air dry Cuban kenaf appeared good for its light colour and good strength. The chemical consumption at 16.4 per cent on raw material was the lowest. The production of this grade of pulp would be economical even without recovery of chemicals.

For semi-bleached pulp from air dry Thai kenaf to make newsprint and printing paper, sample No. 7 with a single stage hypochlorite bleaching holds promise for its good strength, high bleached yield and fair brightness at 66 per cent.

TABLE 1
PULPING AND BLEACHING CON TIONS IN NEUTRAL SULPHITE PULPING OF KENAF STALKS

| Somming Mo  |                        | 2                                  | 3                     | 7                    | 5                     | 9                                  | 7                     | 8                     |
|---|------------------------|------------------------------------|-----------------------|----------------------|-----------------------|------------------------------------|-----------------------|-----------------------|
| Cook No.  | 244                    | 265                                | 270                   | 273                  | 515                   | 305                                | 265                   | 270                   |
| rial  | Air dry<br>Cuban kenaf | Air dry<br>Thai kenaf              | Air dry<br>Thai kenaf | Fresh Cuban<br>kenaf | Air dry<br>Thai kenaf | Air dry<br>Thai kenaf              | Air dry<br>Thai kenaf | Air dry<br>Thai kenaf |
| Total $\mathrm{Na_2^{SO}_5}$ on ovendry chips, %                | 16.4                   | 18.0                               | 16.0                  | 17.5                 | 0.19                  | 27.0                               | 18.0                  | 16.0                  |
| Na <sub>2</sub> CO <sub>2</sub> , %                             | i                      | 3.0                                | i                     | i                    | 1                     | ı                                  | 3.0                   | ì                     |
| NaOH, %   | 1                      | 1                                  | 0.4                   | ı                    | 1                     | 1                                  | ı                     | 4.0                   |
| NaHCO <sub>5</sub> , %  | ı                      | i                                  | 1                     | 2.2                  | 2.5                   | 2.5                                | ı                     | ı                     |
| Total chemicals applied on ovendry chips, %                     | 16.4                   | 21.0                               | 20.0                  | 19.7                 | 23.5                  | 23.5                               | 21.0                  | 20.0                  |
| Total cooking time, hour  | 4                      | 5                                  | 5.5                   | 0.9                  | 5.5                   | 0.9                                | 5.0                   | ري.<br>د              |
| Liquor to chips ratio, ml/g (dry basis)                         | 3:1                    | 3:1                                | 7:5                   | ۲:4                  | 5:1                   | <b>4</b>                           | т<br>Г                | 3:1                   |
| Max. temp., °C  | 170                    | 170                                | 170                   | 170                  | 170                   | 170                                | 170                   | 170                   |
| Time to max. temp., hour  | α                      | N                                  | 2                     | 8                    | 2.2                   | 3.0                                | 2.0                   | αı                    |
| Time at max. temp., hour  | N                      | ح                                  | 3%                    | К                    | К                     | К                                  | М                     | 3%                    |
| Pulp yield, % on ovendry chips                                  | 55.5                   | 52.8                               | 54.8                  | 4.59                 | 62.2                  | 60.5                               | 52.8                  | 54.8                  |
| Bleaching sequence  | i                      | c/E/H <sub>1</sub> /H <sub>2</sub> | DC/E/D                | DC/E/D               | C/E/H/D               | DC/E/H <sub>1</sub> H <sub>2</sub> | н                     | ı                     |
| Total equivalent chlorine applied, % on ovendry unbleached pulp | ι                      | 11.0                               | 12.0                  | 7<br>0.0             | 13.5                  | 14.0                               | 10.01                 | ı                     |
| Chlorination stage (DC) ${ m ClO}_{2}$ %                        |                        | ı                                  | 3.0                   | 3.0                  | 11.0                  | 12.0                               | i                     | .1                    |
| c1 <sub>2</sub> , %   | ı                      | 0 8                                | 2.0                   | 2.0                  | i                     | ı                                  | ı                     | ì                     |

Table 1 (continued)

| Sample No.   | _       | 2      | 3     | 47    | 5         | 9                  | 7     | 8     |
|--|---------|--------|-------|-------|-----------|--------------------|-------|-------|
| Alkali extraction stage (E)  | ŧ       | 2.0    | 2.0   | 2.0   | , 0°      | 2.5                | 1     | ı     |
| Hypochlorite bleaching stage (H) Available chlorine, %                   | ı       | H 2.0  | 1     | ı     | ۲.        | H <sub>1</sub> 1.5 | 0     | ı     |
| Chlorine dioxide bleaching stage $(\mathfrak{D})$ $\mathtt{ClO}_{2},~\%$ | ا<br>غو | ı      | 92.0  | 92.0  | 0.38      | 1                  | 1     | 1     |
| Bleached yield % on ovendry unbleached pulp                              | ı       | 77.0   | 78.4  | 78.5  | .9.62     | 76.9               | 88    | 1     |
| % on ovendry chips   | 1       | 40.5   | 42.7  | 50.0  | 5.64      | 46.4               | 46.4  | ı     |
| Paper No.  | 112     | 125    | 127   | 129   | 193       | 140                | 122   | 128   |
| Physical properties of handsheets  | sts     |        |       |       |           |                    |       |       |
| Initial freeness SR., ml   | 800     | 200    | 049   | 069   | 840       | 720                | 200   | 610   |
| Final freeness, SR., ml  | 300     | 320    | 290   | 295   | 200       | 280                | 200   | 560   |
| Time of beating, minute  | К       | 8      | 5     | 8     | 9         | K                  | ح     | К     |
| Basis weight, $g/m$  | 55.3    | 55.3   | 54.6  | 53.9  | 87.6      | 74.1               | 55.7  | 56.3  |
| Bulk ml/g  | 1.31    | 1.23   | 1.10  | 1.13  | 1.15      | 1.05               | 1.31  | 1.38  |
| Burst factor   | 42.7    | 45.4   | 46.5  | 53.3  | 52.9      | 9.64               | 45.0  | 25.2  |
| Tear factor  | 79.3    | 95.6   | 70.2  | 71.2  | 58.5      | 74.1               | 101.0 | 62.5  |
| Breaking length, m   | 8,263   | 8, 196 | 8,572 | 9,738 | 8,022     | 8,776              | 8,202 | 7,020 |
| Folding endurance  | 580     | 902    | 772   | 096   | 560       | i                  | 536   | 158   |
| Brightness, %  | 45      | 75     | 73    | 70    | 80        | 7.4                | 99    | 30    |
| Sizing of handsheets:-Rosin size, %                                      | a       | CU     | 8     | Q     | ر.<br>تر. | 2.0                | 0.0   | α     |

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|                               |    |     | •   |        |             | ,   |     |    |
|-------------------------------|----|-----|-----|--------|-------------|-----|-----|----|
| Sample No.                    |    | 2   | 5   | 7      | 5           | 9   | 7   | 8  |
| Alum, %                       | 4  | 4   | 7   | 7      | 2.0         | 4.0 | 0.4 | 7  |
| Kaolin, %                     | ΓV | īΛ  | ίΛ  | 5      | ı           | 1   | ı   | ſΛ |
| Tamarind kernel powder, %     | 7  | CJ  | 2   | 2 gyps | gypsum 10.0 | •   | 5.0 | 2  |
| Sodium tripolyphosphate, %    | 1  | ı   | 1   | •      | 0.0         | 2.0 | 2.0 | i  |
| Whiten (brightening agent), % | t  | 6.0 | 0.3 | 0.3    | 0.3         | 0.3 | 0.3 | i  |
|                               |    |     |     |        |             |     |     |    |

When the neutral sulphite process for Thai kenaf is compared with polysulphide kraft process as shown in Table 2 for bleached pulp, the polysulphide process has advantages of lower bleach consumption, higher strength, and comparable bleached yield.

The crucial problem of neutral sulphite pulping for bleached pulp from kenaf is the chemical recovery system. Although many NSSC recovery systems have been advocated for wood pulp, they are all very complicated and seldom applied except for unbleached semi-chemical pulp. This is due to the presence of thiosulphate complication in these NSSC recovery systems. The thiosulphate in cooking liquor at high temperature makes unbleached pulp which is difficult to bleach.

In the wood pulp industry cross recovery of NSSC spent liquor in kraft pulp mill has been successfully applied for its simplicity. The MSSC waste liquor can be used for dissolving smelt from kraft recovery boiler or mixed with the kraft black liquor as make-up for sodium and sulphur losses in the recovery cycle. Thus NSSC waste liquor can substitute salt cake as make-up. The disadvantage of cross recovery for MSSC process is the limited size of NSSC production in a kraft pulp mill, with a usual ratio of kraft to NSSC pulp production at about 4:1, i.e. a 100 t/d kraft pulp mill can accommodate a 25 t/d NSSC pulp production by cross recovery with a kraft recovery system. On the other hand, a kraft pulp mill can extent its production by 25 per cent by adding a MSSC production unit. This is the advantage of combination for production of chemical pulp from kenaf.

Lower opacity of MSSC pulp was noted when compared with kraft pulp.

## CONCLUSION

- 1. Production of chemical pulp from kenaf stalks by neutral sulphite process affords high yield and good strength. But high bleach demand and lower opacity make the neutral sulphite process less attractive for fully bleached chemical pulp when compared with kraft process.
- 2. Sodium bicarbonate as buffer in neutral sulphite pulping of kenaf gives higher yield than sodium carbonate buffer and caustic soda buffer.

TABLE 2
PHYSICAL PROPERTIES OF HANDSHJETS FROW BLEACHED KENAF PULPS BY NSSC AND KRAFT PROCESS

| Raw material                                 | Fresh C | Fresh Cuban kenaf    | Dry Ih | Dry Thai kenaf       |
|--|---------|----------------------|--------|----------------------|
| Process of pulping                           | NSSC    | Polysulfide<br>kraft | NSSC   | Polysulfide<br>kraft |
| Cook yield, %                                | 63.4    | 52.0                 | 54.8   | 51.9                 |
| Total chlorine applied, % on unbleached pulp | 12.0    | 8.0                  | 13.3   | 10.0                 |
| Bleached yield, % on chips                   | 50.0    | 44.2                 | 42.7   | 42.8                 |
| Freeness, SR., ml                            | 295     | 350                  | 290    | 250                  |
| Burst factor                                 | 53.3    | 63.9                 | 46.5   | 43.6                 |
| Breaking length, m                           | 9,738   | 10,346               | 8,372  | 8,894                |
| Tear factor                                  | 71.2    | 103                  | 70.2   | 80.8                 |
| Folding endurance                            | 096     | 226                  | 772    | 733                  |
| Brightness, %                                | 20      | 80                   | 73     | 80                   |
| Remarks: .                                   |         |                      |        |                      |
| Cook No.                                     | 273     | 407                  | 270    | 379                  |
| Paper No.                                    | 129     | 183                  | 127    | 165                  |
|  |         |                      |        |                      |

- 3. For unbleached pulp, the neutral sulphite process without buffer applied to air dry kenaf is distinguished for its low chemical demand, high yield and good strength with light colour.
- 4. For semi-bleached pulp, the neutral sulphite process has advantages of good bleached yield, high strength and fair brightness.
- 5. Fresh kenaf yields better pulp with less bleach demand and higher strength by the neutral sulphite process than dry kenaf does.

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