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Chemical pulp from kenaf  
by two-stage bisulphite

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APPLIED SCIENTIFIC RESEARCH CORPORATION OF THAILAND

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BLEACHED CHEMICAL PULP FROM KENAF

REPORT NO. 15  
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# CHEMICAL PULP FROM KENAF BY TWO-STAGE BISULPHITE PROCESS

By Chien Chu<sup>\*</sup>, Naiyana Niyorwan<sup>\*</sup>, and Anchalee Puangvichit<sup>\*</sup>

## SUMMARY

Thai kenaf (Hibiscus sabdariffa) was pulped by a two-stage bisulphite process using sodium, magnesium, and ammonium bases. Compared with the single stage bisulphite process, the two-stage bisulphite process gives higher yield, better strength, and improved bleachability. The sodium base two-stage bisulphite process gives better quality of pulp than the magnesium base and ammonium base two-stage bisulphite process. The ammonium base showed lower yield and lower strength than the other two bases.

## INTRODUCTION

Bisulphite process of various bases for pulping kenaf was reported in this series of studies. The process has good yield of pulp with strength properties lower than that by alkaline processes.

In recent years, two-stage bisulphite process has been developed for wood pulp to get higher yield and better quality of pulp (Lightfoot and Sepall 1965). The Sivola Process is a two-stage sodium bisulphite process. It is widely used for wood pulp. A special chemical recovery system has been developed in the United States by the Combustion Engineering, Inc.

The Sivola Process involves a chemical recovery system more complicated than the conventional kraft recovery system.

The magnesium base two-stage bisulphite process was developed by the Weyerhaeuser Co., Everett, Washington, U.S.A. about 1960. The recovery system is quite straight forward as the standard magnesite recovery system.

The ammonium base is less popular for chemical pulp than the other two bases due to lower yield and lower strength.

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## EXPERIMENTAL PROCEDURES AND RESULTS

The experimental procedures for bisulphite pulping of kenaf were reported in the earlier series of this study. For two-stage bisulphite pulping, only slight modification for one additional stage was involved. Air-dry kenaf chips were cooked at liquid ratio of 1:3 with bisulphite solution at total  $\text{SO}_2$  12-16% on raw material, with pH 3.5-4.0 at 145-155°C for 2-2½ hours.

The second stage cooking solution containing alkali as 6% caustic soda or magnesia or ammonium hydroxide on raw material was added to the digester after cooling by venting the digester and open the digester cover. In several cases cooling overnight was resorted due to the short working hours available in a day. The second stage cooking under alkaline condition was usually conducted at high temperature of 160-170°C for 1-1½ hours. Then the digester was cooled and vented and the crude pulp was washed, then refined in a laboratory disc refiner and centrifuged. The wet pulp cake was bleached with about 8-10% available chlorine in C/E/H bleaching sequence with final acid washing.

The bleached pulp was pressed to remove excess water and air-dried. The dried pulp was converted into handsheets with usual sizing. The handsheets were conditioned overnight under 55% humidity and 25°C. Then physical tests were made for breaking length, bursting strength, tearing strength, brightness, folding strength and sheet density. The results are tabulated in Table 1.

Table 2 shows the quality of selected pulp prepared by the two-stage bisulphite process in comparison to the quality of other pulps to demonstrate the distinct features of the two-stage bisulphite pulps. The significant point is the improved bleachability with only about 8% available chlorine for brightness above 80, whereas the usual kraft pulp requires about 9% available chlorine. The other point of the two-stage bisulphite pulp is the higher bleached yield at about 45% on raw material, which is about 10% higher than that of bleached kraft pulp.

The pulp strength of the two-stage bisulphite pulp is very close to that of bleached kraft pulp except the tearing strength and bursting strength due to the higher yield of the bisulphite pulp.

TABLE 1. PULPING AND BLEACHING CONDITIONS IN THE TWO-STAGE BISULPHITE PULPING OF KENAF STALKS

| Cook no.  | 800                | 803                | 796                | 809                    | 792                    | 792                    | 798                    | 815                  | 774                  | 768                             | 823                          | 879                  | 760                                |
|---|--------------------|--------------------|--------------------|------------------------|------------------------|------------------------|------------------------|----------------------|----------------------|---------------------------------|------------------------------|----------------------|------------------------------------|
| Raw material  | Thai kenaf air-dry | Thai kenaf air-dry | Thai kenaf air-dry | Thai kenaf air-dry     | Thai kenaf air-dry     | Thai kenaf air-dry     | Thai kenaf air-dry     | Thai kenaf air-dry   | Thai kenaf air-dry   | Thai kenaf, castor stalks (50%) | Cuban kenaf, yang wood (50%) | Thai kenaf air-dry   | Thai kenaf fresh (small stem)      |
| <b>First stage bisulphite pulping</b>                   | Mg base            | Mg base            | Mg base            | 20% NaHSO <sub>3</sub> | 20% NaHSO <sub>3</sub> | 20% NaHSO <sub>3</sub> | 20% NaHSO <sub>3</sub> | NH <sub>3</sub> base | NH <sub>3</sub> base | NH <sub>3</sub> base            | NH <sub>3</sub> base         | NH <sub>3</sub> base | NH <sub>3</sub> base               |
| Stock solution, pH value                                | 3.55               | 3.50               | 3.55               | 4.0                    | 4.0                    | 4.0                    | 4.0                    | 3.95                 | -                    | 3.6                             | 4.0                          | 3.9                  | 3.4                                |
| Total SO <sub>2</sub> , % concentration                 | 6.32               | 5.00               | 6.32               | -                      | -                      | -                      | -                      | 5.34                 | 6.08                 | 5.76                            | 5.34                         | 7.06                 | 4.86                               |
| Free SO <sub>2</sub> , % concentration                  | 3.32               | 2.66               | 3.32               | -                      | -                      | -                      | -                      | 2.80                 | 3.20                 | 3.02                            | 2.80                         | 3.66                 | 2.56                               |
| Total SO <sub>2</sub> , on oven-dry chips               | 12.0               | 16.0               | 12.0               | 12.8                   | 12.8                   | 12.8                   | 12.8                   | 15.0                 | 16.0                 | 16.0                            | 16.0                         | 16.0                 | 18.0                               |
| <b>Base:</b>  |                    |                    |                    |                        |                        |                        |                        |                      |                      |                                 |                              |                      |                                    |
| NaOH, % concentration                                   | -                  | -                  | -                  | -                      | -                      | -                      | -                      | -                    | -                    | -                               | -                            | -                    | -                                  |
| MgO, % concentration                                    | 2.0                | 2.0                | 2.2                | -                      | -                      | -                      | -                      | -                    | -                    | -                               | -                            | -                    | -                                  |
| NH <sub>3</sub> , % concentration                       | -                  | -                  | -                  | -                      | -                      | -                      | -                      | 2.0                  | 2.0                  | 1.6                             | 2.0                          | 2.1                  | -                                  |
| Liquor to chip ratio ml/g (add water to stock solution) | 2.5:1              | 3:1                | 2.5:1              | 3:1                    | 5:1                    | 5:1                    | 3:1                    | 3:1                  | 3:1                  | 3:1                             | 3:1                          | 2.5:1                | 4:1                                |
| Max. temp., °C  | 150                | 155                | 155                | 155                    | 155                    | 155                    | 150                    | 155                  | 150                  | 150                             | 155                          | 155                  | 150                                |
| Time to max. temp., hr                                  | 2                  | 2                  | 2                  | 2                      | 2                      | 2                      | 2                      | 2                    | 2                    | 2½                              | 2½                           | 2                    | 2½                                 |
| Time at max. temp., hr                                  | 3                  | 2½                 | 2½                 | 2½                     | 3                      | 3                      | 2½                     | 2½                   | 3½                   | 4                               | 2½                           | 2                    | 3½                                 |
| <b>Second stage alkali pulping</b>                      | Caustic soda       | Magnesia           | Caustic soda       | Soda ash               | Caustic soda           | Caustic soda           | Caustic soda           | Caustic soda         | No 2nd stage         | No 2nd stage                    | Caustic soda                 | Ammonia              | No 2nd stage                       |
| <b>Cooking solution added</b>                           |                    |                    |                    |                        |                        |                        |                        |                      |                      |                                 |                              |                      |                                    |
| NaOH, % on oven-dry chips                               | 5                  | -                  | 5                  | -                      | 6                      | 6                      | 5                      | 6                    | -                    | -                               | 7                            | -                    | -                                  |
| MgO, % on oven-dry chips                                | -                  | 6.0                | -                  | -                      | -                      | -                      | -                      | -                    | -                    | -                               | -                            | -                    | -                                  |
| NH <sub>3</sub> , % on oven-dry chips                   | -                  | -                  | -                  | -                      | -                      | -                      | -                      | -                    | -                    | -                               | -                            | 6                    | -                                  |
| Na <sub>2</sub> CO <sub>3</sub> , % on oven-dry chips   | -                  | -                  | -                  | 8                      | -                      | -                      | -                      | -                    | -                    | -                               | -                            | -                    | -                                  |
| Water added to chip ratio                               | 3:1                | 1:1                | 1.6:1              | 1.5:1                  | 2.5:1                  | 2.5:1                  | 1:1                    | 1:1                  | -                    | -                               | 1:1                          | 1:1                  | -                                  |
| Max. temp., °C  | 95                 | 170                | 170                | 170                    | 170                    | 170                    | 160                    | 170                  | -                    | -                               | 170                          | 170                  | -                                  |
| Time to max. temp., hr                                  | ½                  | ½                  | ½                  | ½                      | 1                      | 1                      | ½                      | ½                    | -                    | -                               | ½                            | 1                    | -                                  |
| Time at max. temp., hr                                  | ½                  | 1                  | 1                  | 1                      | 1                      | 1                      | 1                      | 1                    | -                    | -                               | 1                            | 1½                   | -                                  |
| Pulp yield, % on oven-dry chips                         | 42.0               | 50.9               | 55.2               | 63.0                   | 56.4                   | 56.4                   | 51.4                   | 48.1                 | 51.7                 | 56.1                            | 77.3                         | 46.8                 | 57.9                               |
| Bleaching sequence                                      | C/E/H              | C/E/H              | H                  | C/E/H                  | C/E/H                  | -                      | C/E/H                  | C/E/H/D              | C/E/H                | DC/E/HD                         | -                            | H                    | C/E/H                              |
| Chlorination stage (c), ½ hr                            |                    |                    |                    |                        |                        |                        |                        |                      |                      |                                 |                              |                      |                                    |
| Cl <sub>2</sub> , %                                     | 4                  | 6                  | -                  | 5                      | 6                      | -                      | 6                      | 6                    | 7                    | 6(D3, C3)                       | -                            | -                    | C <sub>1</sub> 4, C <sub>2</sub> 3 |
| Alkali extraction stage (E), ½ hr                       |                    |                    |                    |                        |                        |                        |                        |                      |                      |                                 |                              |                      |                                    |
| NaOH, %   | 2                  | 2                  | -                  | 2                      | 2                      | -                      | 2                      | 2                    | 2                    | 2                               | -                            | -                    | 2                                  |

TABLE 1. Continued.

| Cook no.   | 800                                | 803                | 796                | 809                | 792   | 792                | 798                | 815                | 774   | 768                             | 823                          | 879                | 760                           |  |
|--|------------------------------------|--------------------|--------------------|--------------------|---|--------------------|--------------------|--------------------|---|---------------------------------|------------------------------|--------------------|-------------------------------|--|
| Raw material   | Thai kenaf air-dry                 | Thai kenaf air-dry | Thai kenaf air-dry | Thai kenaf air-dry | Thai kenaf air-dry  | Thai kenaf air-dry | Thai kenaf air-dry | Thai kenaf air-dry | Thai kenaf air-dry                                    | Thai kenaf, castor stalks (50%) | Cuban kenaf, yang wood (50%) | Thai kenaf air-dry | Thai kenaf fresh (small stem) |  |
| <u>Second stage alkali pulping (continued)</u>                       | Caustic soda                       | Magnesia           | Caustic soda       | Soda ash           | Caustic soda  | Caustic soda       | Caustic soda       | Caustic soda       | No 2nd stage  | No 2nd stage                    | Caustic soda                 | Ammonia            | No 2nd stage                  |  |
| Hypochlorite bleaching stage (H), pH 11, 3 hr, available chlorine, % | 2                                  | 2                  | 5                  | 2                  | 2   | -                  | 2                  | 2% + 2% (D)        | 2   | 3(H2, D1)                       | -                            | 5                  | 2                             |  |
| Total chlorine applied, %  | 6                                  | 8                  | 5                  | 7                  | 8   | -                  | 8                  | 10                 | 9   | 9                               | -                            | 5                  | 9                             |  |
| Bleached yield   |                                    |                    |                    |                    |   |                    |                    |                    |   |                                 |                              |                    |                               |  |
| % on oven-dry unbleached pulp  | 86.5                               | 87.4               | 90.0               | -                  | 80  | -                  | 85.5               | 84.0               | 84.0  | 87.2                            | -                            | 92.1               | 95                            |  |
| % on oven-dry chips  | 36.3                               | 44.4               | 50.0               | -                  | 45  | -                  | 44.0               | 40.5               | 43.5  | 49.0                            | -                            | 43.2               | 55                            |  |
| Paper no.  | 546                                | 541                | 486                | 494                | 485   | 478                | 550                | 498                | 488   | 461                             | 509                          | 562                | 454                           |  |
| <u>Physical properties of handsheets</u>                             |                                    |                    |                    |                    |   |                    |                    |                    |   |                                 |                              |                    |                               |  |
| (A) Freeness   |                                    |                    |                    |                    |   |                    |                    |                    |   |                                 |                              |                    |                               |  |
| Initial freeness, S.R. ml  | 600                                | 770                | 890                | 880                | 860   | 880                | 700                | 880                | 720   | 720                             | 890                          | 700                | 750                           |  |
| Final freeness, S.R. ml  | 400                                | 415                | 430                | 385                | 375   | 435                | 410                | 430                | 450   | 440                             | 400                          | 350                | 460                           |  |
| Time of beating, min   | 2                                  | 3½                 | 2½                 | 4                  | 4   | 5                  | 2½                 | 4                  | 2½  | 3                               | 3½                           | 3                  | 3                             |  |
| (B) Test data (50% R.H., 23°C)                                       |                                    |                    |                    |                    |   |                    |                    |                    |   |                                 |                              |                    |                               |  |
| Basis weight, g/m <sup>2</sup>                                       | 62.5                               | 59.3               | 53.1               | 62.2               | 64.3  | 126                | 64.8               | 65.9               | 62.1  | 51.8                            | 127.4                        | 50.3               | 51.6                          |  |
| Bulk, ml/g   | 1.47                               | 1.37               | 1.43               | 1.25               | 1.23  | 1.29               | 1.36               | 1.29               | 1.30  | 1.41                            | 1.74                         | 1.39               | 1.44                          |  |
| Burst factor   | 21                                 | 30                 | 31                 | 35                 | 35  | 49                 | 29                 | 25                 | 26  | 25                              | 27                           | 21                 | 17                            |  |
| Tear factor  | 90                                 | 97                 | 113                | 94                 | 95  | 118                | 117                | 88                 | 74  | 65                              | 112                          | 69                 | 69                            |  |
| Breaking length, m   | 4,192                              | 5,270              | 5,925              | 6,468              | 6,174   | 6,723              | 5,647              | 4,834              | 5,337   | 4,904                           | 5,102                        | 5,661              | 4,641                         |  |
| Brightness, %  | 90                                 | 90                 | 58                 | 81                 | 86  | 42                 | 70                 | 94                 | 76  | 88                              | 22                           | 44                 | 72                            |  |
| Folding endurance  | 24                                 | 141                | 271                | 454                | 400   | 19                 | 167                | 112                | 55  | 21                              | 2                            | 88                 | 16                            |  |
| Remarks:   | 2nd stage Mechano-chemical pulping |                    |                    |                    | After 1st stage cooking, cool overnight, start 2nd stage cold |                    |                    |                    | D is ClO <sub>2</sub> stage in equiv. Cl <sub>2</sub> |                                 |                              |                    | Opacity low                   |  |

TABLE 2. PHYSICAL PROPERTIES OF HANDSHEETS OF BISULPHITE KENAF PULPS

| Process                         | Single stage<br>magnefite | Two stage<br>magnefite | Sodium<br>bisulphate | Two-stage<br>Sodium<br>bisulphite | Ammonium<br>bisulphite | Two stage<br>Ammonium<br>bisulphite |
|---------------------------------|---------------------------|------------------------|----------------------|-----------------------------------|------------------------|-------------------------------------|
| Cook no.                        | 276                       | 303                    | 462                  | 792                               | 774                    | 815                                 |
| Paper no.                       | 136                       | 541                    | 218                  | 485                               | 488                    | 498                                 |
| Raw material                    | Thai kenaf                | Thai kenaf             | Cuban kenaf          | Thai kenaf                        | Thai kenaf             | Thai kenaf                          |
| Freeness, S.R., ml              | 310                       | 415                    | 400                  | 375                               | 450                    | 430                                 |
| Brightness, %                   | 77                        | 90                     | 85                   | 86                                | 76                     | 94                                  |
| Chlorine applied, %             | 9                         | 8                      | 12                   | 8                                 | 9                      | 10                                  |
| Bleached yield (on dry chip), % | 41.7                      | 44.4                   | 42.7                 | 45.0                              | 43.5                   | 40.5                                |
| Burst factor                    | 24                        | 30                     | 53                   | 35                                | 26                     | 25                                  |
| Breaking length, m              | 5,182                     | 5,270                  | 5,204                | 6,174                             | 5,337                  | 4,834                               |
| Tear factor                     | 57                        | 97                     | 68                   | 95                                | 74                     | 88                                  |
| Folding endurance               | 80                        | 141                    | 274                  | 400                               | 55                     | 122                                 |

Of the three bases for two-stage bisulphite pulping, sodium base is the best. Next is the magnesium base. The ammonium base is the weakest in strength with lower yield than the other two bases. The use of soda ash instead of caustic soda in the second stage gives higher unbleached yield over 60%. This advantage of higher yield is further enhanced by the lower cost of soda ash comparing to the expensive caustic soda in the market.

#### DISCUSSION

The two-stage bisulphite process is distinguished for its good bleachability and high yield with pulp strength comparable to that of kraft pulp. These advantages must be weighed against more complicated pulping procedure.

In view of the smaller and less cost of cooking chemicals without a recovery system in comparison to the total alkali required in kraft pulping, the two-stage bisulphite process can afford the operation in small- and medium-sized pulp mills without a recovery system in the early stage of the project.

In the case of two-stage sodium bisulphite process, the cost of cooking and bleaching chemicals based on 45% bleached yield would be as follows:

|   |                  |                          |
|---|------------------|--------------------------|
| Soda ash for 1st stage bisulphite cooking with 20% $\text{NaHSO}_3$ | 230 kg @ 5.00    | 1,150 baht               |
| Caustic soda for 2nd stage 6% on raw material                       | 133 kg           |                          |
| Caustic soda for alkali extraction in bleaching 2% on bleached pulp | <u>20</u> kg     |                          |
|   | <u>153</u> kg    |                          |
|   | 100 kg @ 3.00    | 300 baht                 |
|   | from cell liquor |                          |
|   | 53 kg @ 6.00     | 318 baht                 |
|   | purchased        |                          |
| Chlorine for bleaching, 9% on bleached pulp                         | 90 kg @ 3.00     | 270 baht                 |
|   | from cells       |                          |
| Line for hypochlorite solution, 4% on bleached                      | 40 kg @ 2.00     | 80 baht                  |
|   |                  | <hr/>                    |
|   | Total            | <u><u>2,118</u></u> baht |



The above cost of chlorine and equivalent part of caustic soda is based on own generation of chlorine by electrolysis of salt in the pulp mill. The total cost of chemicals at about 2,118 baht per tonne of bleached pulp is relatively reasonable comparing to the market value of bleached pulp at 8,000 baht per tonne. The cost of raw material per tonne of bleached pulp would be about 2.23 tonnes kenaf @ 500 baht or 1,115 baht. The total direct material cost would be about 3,233 baht per tonne of bleached pulp.

In case of the two-stage magnesite process with 6% magnesia in the 2nd stage, the conventional magnesite recovery system can be applied. The system is much simpler than the kraft system, thus the alkali-chlorine electrolysis is not necessary; as the alkali demand is very small, being used only as alkali extraction in the C/E/H bleaching sequence, ordinary chlorine can be purchased from the market.

The waste liquor in two-stage ammonia base bisulphite process could be heated with lime for recovery of ammonia, thus only the cost of sulphur and bleaching chemicals would be involved. The disposal of calcium base waste liquor may be done in irrigation of crops.

For unbleached pulp, the two-stage sodium bisulphite process gives a yield over 56% with fair brightness. The yield is about 25% higher than that of the kraft process. Although the tearing strength and bursting strength are lower than those of kraft pulp, the pulp has far better brightness, and can be brightened to 69% brightness by a single stage hypochlorite bleaching with 5% available chlorine. This level of brightness affords its use for newsprint furnished and magazine paper.

#### CONCLUSIONS

- 1) The two-stage bisulphite process for kenaf chemical pulp is apparently better than single stage bisulphite process in respect to pulp yield, pulp strength, and bleachability.
- 2) The two-stage sodium bisulphite process is most efficient, requiring less chemicals than the single stage bisulphite pulping.
- 3) The two-stage magnesium bisulphite process affords a good yield of bleached pulp with good quality.

4) The two-stage ammonium bisulphite process gives lower pulp yield and lower strength than that by sodium base and magnesium base. However, it might produce a less expensive pulp than the other two bases, if ammonia is recovered simply by lime distillation.

5) In the second stage pulping, soda ash gives higher yield of pulp than caustic soda. But magnesia in the second stage is better than caustic soda with the magnesium base bisulphite pulping in the first stage. This magnesia affords combined recovery with the waste magnesia pulping liquor.

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