# SODA-AQ PULPING OF COTTON STALKS

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Presented at 2001 TAPPI Fall Technical Conference.

## ABSTRACT

Egyptian and US cotton stalks were pulped using a soda-anthraquinone process. The effect of pulping process variables was investigated. Pulps were bleached using oxygen and hydrogen peroxide. Bleached and unbleached pulps were refined and tested for strength. Egyptian stalks produced better unbleached pulps, with lower Kappa numbers, higher yields, and better refined strength. The American stalk pulps had better bleachability. The strength of the bleached pulps from both stalk types was the same, with values somewhat inferior to mixed hardwood pulp.

## INTRODUCTION

Nonwood raw materials account for 5-7% of the worldwide production of paper pulp (1). The production of this type of pulp has increased more rapidly than that of pulp from wood in the last two decades, by a factor of about two in Latin America and three in Africa and the Middle East (2). In countries such as Egypt which have no forests, cotton stalk is one of the agricultural residues available for pulping and papermaking. Cotton stalks are available in large quantities in several parts of the world. In Egypt, 1.9 million tons are produced annually. The stalks contain a substantial percentage of pith cells which, together with the dark-colored outer bark, create problems in both pulping and papermaking processes.

Two major problems have hindered commercial utilization of cotton stalks for production of pulp and paper. The first problem is that of transportation of the raw materials, which are bulky in nature. This problem can be solved to some degree with densification techniques. The second problem is that of debarking, which is made difficult due to the fact that cotton stalks are thinly-branched, bushy plants. In the last few years, many efforts have been directed towards the exploitation of lignocellulosic materials by new pulping systems with two main objectives in mind: (1) to avoid or reduce the use of chemical agents which, alone or interacting with the raw materials, could produce a serious environmental impact, such as in the case of sulfate (kraft) and sulfite methods; 2) to achieve a selective separation of the main components (cellulose, hemicellulose, and lignin) in a non-degraded form, with subsequent processing according to different methods (3).

The use of soda pulping, with or without additives, can constitute an alternative method for producing chemical or semichemical pulps from cotton stalks without the above-mentioned problems (4-6). Cotton stalks, like many nonwoods, tend to respond better to soda pulping than does wood.

The object of this work was to investigate the utilization of whole, undebarked cotton stalks for preparation of chemical pulps by the soda-anthraquinone (soda-AQ) method and to evaluate the strength of the resulting pulps.

## METHODS AND MATERIALS

### Raw Material

The cotton stalks used in this work were from both Egyptian and US plants. For both sources, the stems were cut into pieces about 2.5-4.0 cm in length by hand. No debarking was used. Samples of both stalks were milled in a Wiley mill, using a 0.4 mm screen. The milled samples were subjected to standard compositional analysis, according to TAPPI standards.

**Pulping and Bleaching**

Pulping of the cotton stalk was carried out in 3-liter pressure bombs placed on a rotating rack in a heated air oven. The following pulping conditions were used:

NaOH Charge: 12, 15, 18 % on OD fiber
AQ Charge: 0.075 % on OD fiber
Liquor-to-Fiber Ratio: 4:1
Maximum temperature: 150 and 165 C
Time to maximum temperature: approx. 30 minutes
H-factor: 200,400 800

After pulping, a sample of black liquor was obtained an analyzed for pH and residual alkali content.

For each trial, the pulped material was defibrated by passing it through a Sprout-Waldron single-disk refiner equipped with 8-inch grooved plates rotating at 3600 rpm. An initial pass was made with a disc clearance of 0.30 mm, followed by another pass with a clearance of 0.15 mm. The resulting pulp was washed and passed through a Voith laboratory flat screen equipped with 0.25-mm slots. Rejects were oven-dried and weighed. Accepts were centrifuged, fluffed, weighed, tested for consistency, and used for subsequent processing and analysis. All indicated analyses were carried out according to TAPPI standards. Viscosity was tested after acid chlorite delignification.

TCF bleaching was carried out using an O-Q-PO-PO sequence, with the following designations:

O – oxygen delignification
Q – acid chelation with EDTA
PO – atmospheric peroxide bleaching with oxygen enrichment

The oxygen and peroxide stages were performed in 3-liter, stainless pressure bombs placed on a rotating rack in a heated air oven. The chelating stage was carried out in sealed polyethylene bags placed in a hot-water bath. The bleaching conditions are presented in Table 1.

**Table 1. Bleaching conditions**

| Stage | Chemicals | Consistency, % | Temperature, C | Time, hour |
|---|---|---|---|---|
| O | NaOH – 2 % on OD<br>Oxygen – 40 psig | 10 | 100 | 1 |
| Q | H2SO4 – ph 4<br>EDTA – 0.2 % on OD | 10 | 70 | 1 |
| PO1 | H2O2 – 2 % on OD<br>Oxygen – 40 psig | 10 | 90 | 1 |
| PO2 | H2O2 – 2 % on OD<br>Oxygen – 40 psig | 10 | 90 | 1 |

After each stage, the pulp was washed thoroughly with distilled water. The indicated analyses were carried out according to TAPPI standards.

**Refining and Papermaking**

Refining of both unbleached and bleached pulp samples was carried out in a PFI mill, to decreasing freeness levels. Standard, 1.2-gram (60 g/m$^2$) handsheets were made and tested according to TAPPI standards.

## RESULT AND DISCUSSION

### Raw Material Analysis

The results of chemical analysis of both types of cotton stalk are shown in Table 2, along with some values from the literature.

**Table 2. Chemical analysis of raw materials (OD basis)**

| | American Stalks | Egyptian Stalks | Literature (7-9) |
|---|---|---|---|
| Ash, % | 1.12 | 1.84 | 3.0 – 4.36 |
| Silica, g/kg | --- | 4.92 | 0.6 |
| Hot water solubility, % | 14.23 | 10.77 | 6.6 – 13.7 |
| Alcohol-benzene solubility, % | 3.03 | 2.93 | 5.5 – 7.85 |
| 1 % NaOH solubility, % | 40.64 | 39.60 | 34.85 – 44.6 |
| Alpha cellulose, % | 42.22 | 48.83 | 43.7 |
| Pentosans, % | --- | 17.45 | 12.4 – 20.1 |
| Lignin % | 26.94 | 22.50 | 19.5 – 21.0 |
| Cellulose/lignin ratio | 1.57 | 2.17 | --- |
| Pith, % | 4.09 | 4.17 | --- |
| Bark, % | 33.53 | 30.81 | --- |

Given the wide range of values found in the literature (7-9), the values obtained for both stalk types were considered reasonable.

The hot water and alcohol-benzene solubility values for both types of stalk indicated the presence of a considerable content of lightweight aliphatic and aromatic compounds. The high solubility of both in 1 % NaOH indicated easy access and degradation of the cell wall material, implying that pulping chemical charges should be moderate. Overall comparison of the data revealed a moderately lower content of non-cellulosic materials and a potential for greater yield and pulp quality for the Egyptian stalks.

### Pulping of American Cotton Stalks

Table 3 contains the data from the soda-AQ pulping of the American cotton stalks under a combination of H-factors and alkali charges.

**Table 3. Data for soda-AQ pulping of American cotton stalks**

| Trial | H-factor | % NaOH | Total Yield, % | Screen Yield, % | Rejects, % | Kappa | Viscosity, cps | Liquor pH | Residual Alkali, gpl Na2O |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 200 | 12 | 42.49 | 26.09 | 16.40 | 96.8 | 92.2 | 10.9 | 2.35 |
| 2 | 200 | 15 | 43.83 | 28.23 | 15.60 | 80.0 | 65.0 | 12.0 | 3.20 |
| 3 | 200 | 18 | 34.99 | 28.59 | 6.40 | 66.4 | 63.8 | 12.8 | 7.83 |
| 4 | 500 | 12 | 40.93 | 25.23 | 15.70 | 88.8 | 85.7 | 10.8 | 1.36 |
| 5 | 500 | 15 | 34.28 | 28.86 | 5.42 | 54.7 | 49.6 | 12.0 | 3.24 |
| 6 | 500 | 18 | 32.81 | 29.91 | 2.90 | 43.1 | 49.6 | 12.7 | 7.81 |
| 7 | 800 | 12 | 46.14 | 26.52 | 19.62 | 86.1 | 78.3 | 11.9 | 2.48 |
| 8 | 800 | 15 | 35.11 | 29.91 | 5.20 | 53.7 | 47.3 | 12.0 | 3.28 |
| 9 | 800 | 18 | 30.64 | 28.74 | 1.90 | 33.1 | 31.0 | 12.6 | 7.19 |

Overall, the low values for total and screened yield confirmed the significant presence of extraneous and non-cellulosic materials, as indicated by the analytical data. The total yield values were in close agreement to those found in the literature for soda-AQ cooks on cotton stalks (10). At a given H-factor, increasing the alkali charge

resulted in the expected behavior, in general; lower total yield, higher yield of screened accepts (due to improved fiber liberation), lower rejects, lower Kappa number, lower viscosity, and higher alkali residual. In general, the same effects were noted when the H-factor was increased for a given charge of alkali, although the residual alkali decreased. Since it may be considered that a bleachable-grade pulp should have an unbleached Kappa number in the range of 25-40, it was apparent that the higher alkali charge and H-factor would be required in order to produce a pulp suitable for subsequent bleaching.

Each of the unbleached pulps was refined in a PFI mill to a freeness of approximately 300 CSF. The mechanical properties of the resulting handsheets are shown in Table 4.

**Table 4. Paper properties for unbleached pulps from American cotton stalks**

| Trial | PFI Revs | Freeness, CSF | Tear Index, mN*m2/g | Burst Index, kPa*m2/g | Breaking Length, km |
|---|---|---|---|---|---|
| 1 | 1000 | 333 | 8.45 | 1.65 | 4.70 |
| 2 | 1000 | 293 | 8.25 | 1.62 | 4.75 |
| 3 | 1000 | 315 | 6.85 | 1.35 | 5.06 |
| 4 | 1000 | 292 | 8.45 | 1.65 | 5.01 |
| 5 | 1000 | 306 | 7.80 | 1.53 | 5.61 |
| 6 | 850 | 300 | 5.25 | 1.23 | 5.50 |
| 7 | 1000 | 300 | 8.1 | 1.59 | 5.15 |
| 8 | 750 | 300 | 5.9 | 1.08 | 5.50 |
| 9 | 850 | 300 | 7.11 | 0.70 | 5.30 |

The tensile and tear strength values obtained were comparable to those in the literature for soda and soda-AQ pulps from cotton stalk (7, 9-10). However, some discrepancy was noted in the sources for burst strength; while one reference contained values close to those obtained above (9), two others showed values 3-4 times higher (7, 10). The reason for this discrepancy is not known.

**Pulping Comparison Between American and Egyptian Cotton Stalks**

To compare the pulping potential of both types of stalks, a sample of each was pulped under identical conditions (12 and 18 % NaOH, 0.075 % AQ, 4:1, 165 C, 800 H-factor). The results are shown in Table 5.

**Table 5. Results of pulping of both American and Egyptian cotton stalks**

| | NaOH % | Kappa | Total Yield, % | Screened Yield, % | Rejects, % | Residual Alkali, gpl Na2O |
|---|---|---|---|---|---|---|
| American | 12 | 95.4 | 37.4 | 30.9 | 7.00 | 1.40 |
| | 18 | 22.5 | 34.0 | 32.9 | 1.10 | 6.94 |
| Egyptian | 12 | 89.7 | 44.5 | 34.9 | 10.3 | 1.20 |
| | 18 | 21.5 | 39.4 | 39.0 | 0.45 | 8.10 |

As predicted by the analytical data, the Egyptian stalks resulted in higher yields and a lower Kappa number than the American stalks. This performance advantage was further evidenced when the pulps from the 12 % NaOH cooks were beaten in a PFI mill to decreasing freeness levels. As shown in Table 6, the pulp from the Egyptian stalks, while having a lower Kappa number, required more refining to reach a given level of freeness. The strength properties of the Egyptian stalk pulp were better than for the American stalk pulps, over the range of refining studied. Overall, the pulping and papermaking results indicated that the Egyptian cotton stalk had better inherent fiber properties when compared to the American stalks.

**Table 6. Comparison of refining response and sheet properties for American and Egyptian cotton stalks, unbleached**

| Stalk Type | PFI Revs | Freeness CSF | Basis Wt, g/m2 | Density g/cc | Tear Index, mN*m2/g | Burst Index, kPa*m2/g | Breaking Length, km |
|---|---|---|---|---|---|---|---|
| American | 100 | 547 | 65.5 | 0.369 | 8.50 | 1.51 | 3.25 |
| | 300 | 480 | 66.2 | 0.382 | 7.79 | 1.96 | 3.97 |
| | 500 | 362 | 65.7 | 0.527 | 7.32 | 3.37 | 5.91 |
| | 900 | 290 | 62.9 | 0.535 | 6.18 | 4.06 | 7.01 |
| | | | | | | | |
| Egyptian | 100 | 580 | 65.7 | 0.439 | 8.97 | 1.76 | 4.07 |
| | 300 | 514 | 65.1 | 0.475 | 8.86 | 2.71 | 5.24 |
| | 500 | 483 | 66.3 | 0.527 | 8.71 | 3.32 | 6.20 |
| | 800 | 395 | 65.8 | 0.578 | 7.35 | 4.79 | 7.73 |
| | 1100 | 301 | 65.1 | 0.628 | 7.25 | 5.51 | 8.09 |

**Bleaching**

The response of pulps from both raw material sources (pulped with 18 % NaOH) to bleaching with an O-Q-PO-PO sequence is shown in Table 7.

**Table 7. Results from bleaching of pulps from American and Egyptian cotton stalks (18 % NaOH in pulping)**

| | Stage | Residual H2O2 g/l | Kappa | Viscosity, cps | Brightness, % ISO |
|---|---|---|---|---|---|
| American Stalks | | | | | |
| | O | | 17.7 | | |
| | Q | | | | |
| | PO | 0.42 | 11.0 | 22.5 | 70.1 |
| | PO | 4.29 | 8.6 | 20.4 | 74.0 |
| | | | | | |
| Egyptian Stalks | | | | | |
| | O | | 12.9 | | |
| | Q | | | | |
| | PO | trace | 10.2 | 15.7 | 63.8 |
| | PO | 0.01 | 9.4 | 14.8 | 70.9 |

While the unbleached Kappa numbers of the two pulps were similar (22.5 and 21.5), the Egyptian stalk pulp was delignified more during the oxygen stage. During the peroxide stages, however, this advantage disappeared, resulting in pulps with again a very similar Kappa number. The brightness and viscosity of the American stalk pulp after each stage of peroxide bleaching was significantly higher compared to the Egyptian stalk pulp. This result, coupled with the significantly higher peroxide residual (lower consumption), showed that the American stalk pulp had better bleachability than the Egyptian stalk pulp.

**Strength Evaluation of the Bleached Pulps**

Each of the bleached pulps was refined in a PFI mill to decreasing freeness levels. The data are shown in Table 8, along with literature data for bleached soda and kraft pulps from whole cotton stalks. Both the American and Egyptian stalk pulps had strength properties comparable to those obtained in the literature.

**Table 8. Refining response and mechanical properties for handsheets from bleached pulps made from American and Egyptian cotton stalks (literature data shown for comparison)**

| Stalk Type | PFI Revs | Freeness CSF | Basis Wt, g/m2 | Density g/cc | Tear Index, mN*m2/g | Burst Index, kPa*m2/g | Breaking Length, km |
|---|---|---|---|---|---|---|---|
| American | 100 | 359 | 64.1 | 0.79 | 7.80 | 5.11 | 7.17 |
| | 300 | 300 | 62.6 | 0.80 | 7.55 | 5.70 | 7.51 |
| | 500 | 259 | 64.7 | 0.82 | 7.45 | 6.24 | 7.77 |
| | 1000 | 142 | 64.2 | 0.88 | 7.15 | 6.73 | 8.01 |
| | | | | | | | |
| Egyptian | 100 | 419 | 63.7 | 0.67 | 7.80 | 4.43 | 6.80 |
| | 300 | 350 | 63.1 | 0.69 | 7.55 | 5.10 | 7.41 |
| | 500 | 290 | 64.3 | 0.71 | 7.40 | 6.01 | 8.07 |
| | 1000 | 214 | 64.2 | 0.79 | 7.35 | 6.28 | 8.20 |
| | | | | | | | |
| Soda (7) | --- | 220 | --- | 0.780-0.897 | 2.30-6.88 | 1.97-5.84 | 4.63-9.10 |
| | | | | | | | |
| Kraft (7) | --- | 220 | --- | 0.751-0.897 | 6.89-8.05 | 5.17-5.84 | 7.67-9.10 |

Tensile, tear, and bursting strength are plotted versus freeness in Figures 1, 2, and 3, respectively. Data for a common mixed southern hardwood pulp are included for comparison (11). The American and Egyptian stalk pulps were almost identical with respect to tear, burst, and tensile strength at a given freeness. The hardwood pulp had a significantly higher tear and tensile strength and a slightly higher burst strength than for the two pulps tested.

## SUMMARY

Using soda-AQ pulping, it was possible to produce unbleached and bleached pulps from both American and Egyptian whole cotton stalks with acceptable properties, comparable to values found in the literature. The higher quality of the Egyptian stalks (lower extractables, lower lignin content, higher cellulose content) resulted in pulps with higher yields and lower Kappa numbers when compared to the American stalks pulped under the same conditions. However, the American stalk pulps had better bleachability. After refining, the strength properties of both bleached pulps were almost identical.

## CONCLUSIONS

**1.** Compared to American cotton stalks, the Egyptian cotton stalks had a lower content of extractables and lignin and a higher content of cellulose.

**2.** For the soda-AQ pulping of American cotton stalks, an increase in alkali charge at a given H-factor resulted in a lower total yield, a higher screened yield, fewer rejects, a lower Kappa number, lower viscosity, and a higher alkali residual.

**3.** For the soda-AQ pulping of American cotton stalks, an increase in H-factor at a given alkali charge generally resulted in a lower total yield, a higher screened yield, fewer rejects, a lower Kappa number, lower viscosity, and a lower alkali residual.

**4.** Under the soda-AQ conditions studied, the Egyptian stalks produced pulps with lower Kappa numbers and higher total yields compared to those from the American stalks.

**5.** The unbleached pulps from the Egyptian stalks required more refining to reach a given level of freeness than did the unbleached pulps from American stalks. At a given level of freeness, the Egyptian stalk pulps had better tear, burst, and tensile strength than did the American stalk pulps.

**6.** Although both pulps had a similar Kappa number after soda-AQ pulping with an alkali charge of 18 % NaOH on OD fiber, the pulp from the Egyptian stalks was delignified significantly more in the standard

oxygen stage than the pulp from the American stalks. During the two stages of peroxide bleaching, however, the pulp from the American stalks consumed less alkali and had a higher brightness and viscosity than did the pulp from the Egyptian stalks. Overall, the American stalk pulp had better bleachability.

7. Both bleached pulps demonstrated similar tear, burst, and tensile strength when refined over a range of freeness values. The tear and tensile values obtained were significantly lower than for mixed southern hardwood pulp, while the burst values obtained were slightly lower.

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**Figure 1. Tearing strength of bleached cotton stalk pulps versus southern hardwood**
**● = American cotton ▲= Egyptian cotton ■ = hardwood**

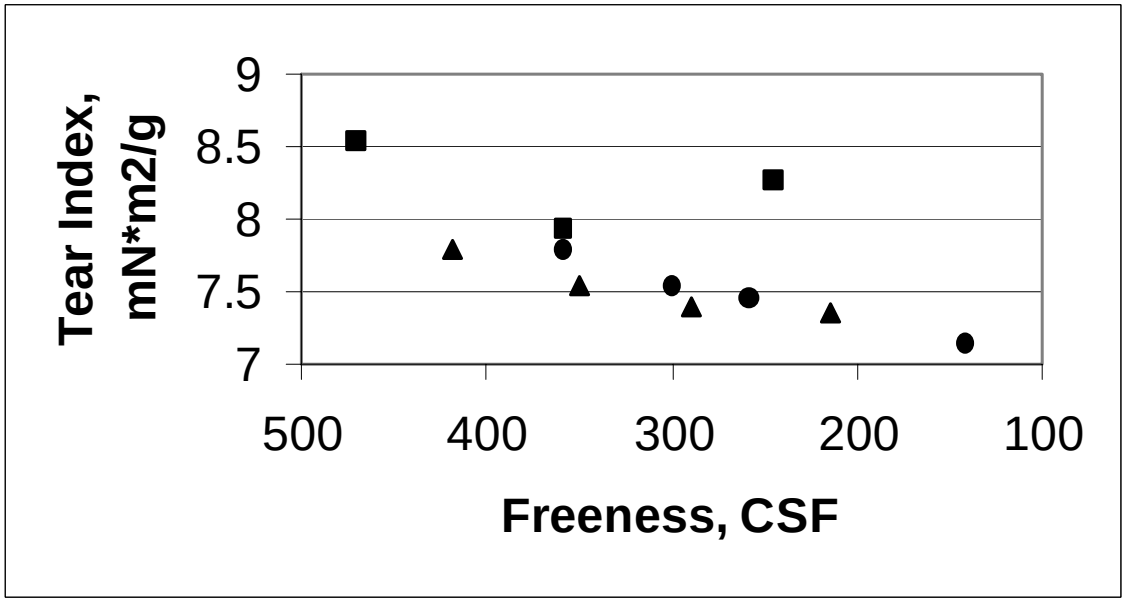


**Figure 2. Bursting strength of bleached cotton stalk pulps versus southern hardwood**
**● = American cotton ▲= Egyptian cotton ■ = hardwood**

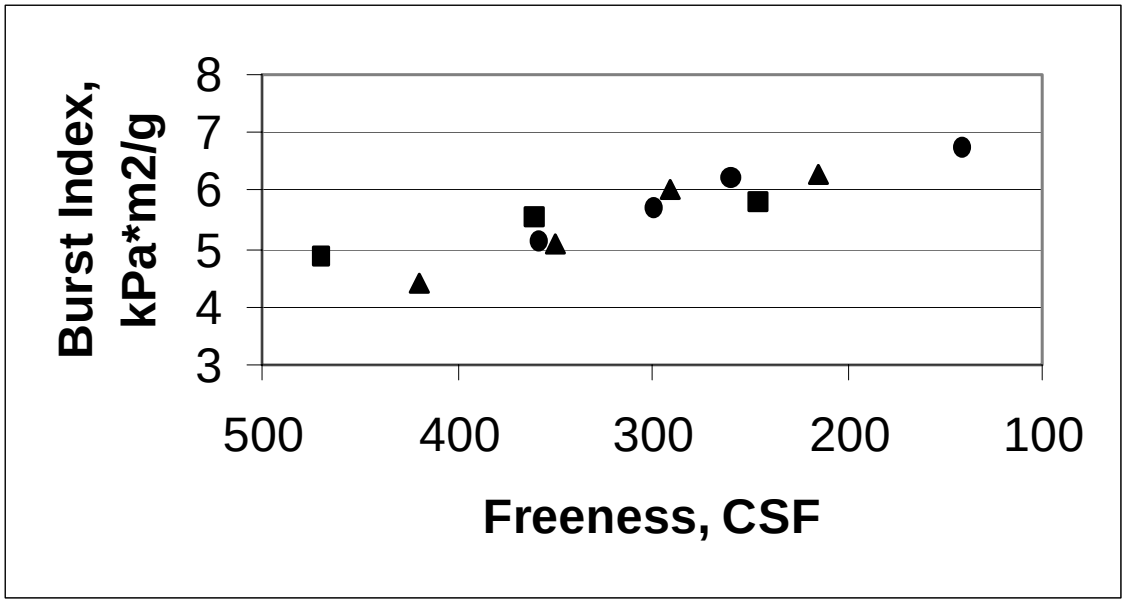

**Figure 3. Tensile strength of bleached cotton stalk pulps versus southern hardwood**
**● = American cotton ▲= Egyptian cotton ■ = hardwood**