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Pulp Production from Cotton Stalks

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Abstract
Deforestation due to the use of wood products for the pulp and paper industry, as things stand right now, poses a serious environmentally unsustainable problem. This study is commenced to study pulp production from the cotton stalk using both Kraft and soda pulping. The produced pulp Yield and Kappa numbers were determined as per the TAPPI standard method. The analytical and research laboratory certified reagents with proper specifications used were sodium hydroxide, distilled water, sodium sulphite, sodium carbonate for both soda and kraft pulping, hydrogen peroxide to bleach the produced pulp, Potassium permanganate, Sulfuric acid, Potassium iodide, Sodium thiosulfate, and starch indicator to determine the kappa number. The maximum pulp yield of 32% - 34.2% for both Kraft and soda pulping was obtained. The Kraft pulping method was found to yield more pulp, and operating parameters of temperature, active alkali and cooking time had a great effect on both kraft and soda pulping. The soda and kraft pulping successfully and efficiently pulped cotton stalks and pulp are developed from the stalks.

Key words: Cotton Stalk, Pulp, Pulp Bleaching, Kappa Number

Introduction
Pulp/paper is a web of fibres derived from wood and non-wood celluloses from which non-cellulose components are separated by chemical or mechanical pulping [1, 2]. At present, the production of pulp is entirely dependent on wood celluloses. 90-92% of world pulp production relies on wood [3, 4]. The consumption of celluloses for pulp production is anticipated to increase from 325 million tons in 2000 to 570 million tons by 2020 with an annual growth rate of 2.8% [5]. This demand is most widely satisfied with the deforestation of forests. Research demand is reverting to the use of cellulose from agricultural residues due to the environmentally unsustainable problem emanating from deforestation and for the abundance and economic value of agricultural biproducts [6].

Cotton is currently the leading plant fibbers crop worldwide Smith and Cothren providing income to millions of farmers worldwide being grown for commercial use in more than 80 countries including Australia, China, Egypt, India, Pakistan, the USA and Uzbekistan [7, 8]. Cotton leaves several residues such as stalks, side branches, leaves, bolls, and seeds with adhering cotton lint in the field during harvest Cotton stalks are a promising material for paper production having a cellulose content of 45.5% which is equitable to the cellulose content of the main raw material coniferous tree which is 42-51% [9]. This chemistry of the material fits it for paper and pulp production [10]. Restudied Cotton Plant Stalk as an alternative raw material to the board industry [11]. They reported 23 million tonnes of cotton stalks are generated and Most of the stalk produced is treated as a waste though a small part of it is used as domestic fuel. The bulk of the stalk is burnt off in the fields after the harvest of the cotton crop. They characterized the cotton stalks as they have fiber dimensions comparable to the most commonly available species of hardwood which can be used for the manufacture of particleboards, preparation of pulp and paper, hardboard, corrugated boards, and boxes, and as a source of cellulose. Studied The characteristics of lignocellulosic agro wastes such as sugarcane bagasse, cereal straw, cotton stalks, rice husks, etc [12] for the manufacture of binderless panel boards and confirmed the use such lignocellulosic materials for the production of panels can replace expensive, synthetic, petrochemical-based resin adhesives commonly used. They also eliminate formaldehyde emission resulted from the adhesives which make the panels particularly appropriate for indoor use.

The Conversion of the materials into paper or paperboard involves various processes such as material preparation, pulping, bleaching, chemical recovery, pulp drying which is included for non-integrated mills only, and papermaking. In some cases, mills may also include converting operations like coating, box making, etc. However, these operations are usually performed at separate facilities. Some composite pulp and paper mills perform multiple operations simultaneously involving chemical pulping, bleaching, and papermaking, pulping and unbleached papermaking, etc [2]. Non composite mills perform either pulping with or without bleaching or papermaking with or without bleaching at a time

[13]. The three main processes of Pulping, mechanical process, chemical process, and mechanochemical process. The most commonly used pulping method in paper industries is the Chemical process and its classifications are Kraft, soda, and soda AQ. Kraft pulping is the category that can produce high strength paper but the associated environmental pollution requires a recovery system [14]. Soda pulping is less popular and is an obsolete method that uses NaOH. Kraft process overtook the soda pulping because the soda pulping paper loses strength due to the chemical soda. Although the soda pulping diminishes paper strength it does not involve sulfur compounds. Therefore, another process based on Soda pulping the so-called Soda AQ pulping which is soda pulping with additive anthraquinone can yield higher pulping comparing to Kraft pulping and the obsolete soda one [15].

Therefore, utilizing agricultural biproduct materials for pulp and paper production is vital in many ways. In this study to produce pulp from one of the agricultural residues, cotton stalks were used due to their free availability and most abundant and are highly cultivated for the future especially in developing countries as the expansion of the textile factory and increases the demand for cotton.

Materials and Methods

Materials

Cotton stalks were collected from the agricultural site of Matama cotton farming land. The chemicals and reagents used for this research work were in analytical grade and certified by research boratories with the proper specification. Sodium hydroxide, sodium sulphite, and sodium carbonate were obtained from the postgraduate research laboratory of chemistry, Wollo University. Hydrogen peroxide and the chemicals and reagent used to determine the kappa number I.e. Potassium permanganate, Sulphuric acid, Potassium iodide, Sodium thiosulfate, and starch indicator were obtained from a research laboratory of textile industry development institute, Ethiopia Deionized water was used during all experiments.

Methods

Cotton Stalk Preparation

Figure 1 below shows manual smashing cotton stalks to smaller sizes, sieving to remove particle sizes from the powder, and drying of the powders. Cotton stalks were mechanically decorticated, grounded cleaned, and dried under shade for one week. The dried chips were dried in an oven at 105oC and analyzed the moisture content every 2 hours using the equation (1) below:

Moisture content (%)=(W1-W2)/W1……………………. (1)

Where, W1 = mass of chipped cotton stalks before drying W2 = mass of chipped cotton stalks after drying. Moisture content determination continued until a constant weight was obtained.

Figure 1: A Photo showing cotton stalk preparation

Pulping

Carried out Kraft and soda pulping. In Kraft pulping the dried cotton stalks were pulped using the mixture of 12.5% by weight solution combined from 8.6% NaOH, 27.1% Na2SO4, and 14.3% Na2CO3 and Calculated the individual weights of solid chemicals required by wt % as indicated below:

Total weight of the mixture of NaOH, Na2SO4, and Na2CO3 = 12.5(%)*1000ml = 125 g, accordingly;

$NaOH = 0.586 * 125 = 73.25g$
$Na_2SO_4 = 0.271 * 125 = 33.875g$
$Na_2CO_3 = 0.143 * 125 = 17.875g$

The same calculation was followed to prepare more white liquors of 200ml, 300ml, and 500ml.

For soda pulping, 20%, 15%, and 10% by weight solution of NaOH were used in cooking liquor. For instance, taking 20% NaOH by weight, dissolved 20g (0.2 *100ml) of NaOH in diionized water to make up 100ml to maintain the required concentration of the. Different cooking liquors with 15%, 10%, and 20% of alkali concentrations were used to determine the effect of alkali on pulp yield. Both Kraft pulping and soda pulping were incubated at boiling temperature with the prepared 5:1 ratio of cotton stalks powder to Liquor ratio for 2hrs, 3hrs, and 4hours to determine the effect of cooking time on kraft pulping and for 3hrs in the case of soda pulping.

Figure 2: A Photo showing cotton stalk pulping

Washing and Filtration

Figure 3 below shows black liquor collected from the cooked mixtures of cotton stalks, chemicals, and de-ionized water, and the washing of the black liquor to remove unwanted substances. The cooked mixtures containing cooking chemicals that can be recovered were filtered to obtain black liquor. Filtration was carried out several times to remove the lignin traces completely. Washed the filtered black liquor in water to remove the reminder lignins and chemicals associated with the brown stock followed by filtration. Repeated the washing with 1000ml water and the filtrations several times until the black liquor was removed to take the pH to neutral and to reduce the lignin content.

Figure 3: A Photo showing black liquor and its washing

Bleaching

Figure 4 below shows the bleaching of black liquor, filtration, and bleached pulp. As can be seen in figure 3 above the collected black liquor from both kraft and soda pulping was black which is not convenient for paper/pulp production. Bleaching was carried out in 200ml of water with 10ml of the bleaching agent H2O2 to completely remove the brown color and got white pulp. The whitened pulp was incubated in a hot plate at 80℃ for 1 hr followed by drying at 105ºC for 2 hours in a hot air oven to remove moisture from the bleached pulp.

Figure 4: A photo showing Pulp bleaching, bleached pulp, and filtrated bleached pulp

Pulp Yield and Kappa Number Determination

The pulp yield was determined by the method as per the TAPPI standard using equation (2) below.

$$\text{Pulp yield (\%)} = \frac{\textit{Wt.of pulp produced}}{\textit{Wt.of original cotton stalk}} * 100 \ldots\ldots\ldots(2)$$

To determine kappa number One gram of Oven dried pulp sample dissolved in 700ml distilled water to disintegrate the pulp so that no fiber bundles exist. To the dissolved pulp; 30 ml of 0.1N potassium permanganate solution was added and waited for 5 minutes until it saturates the disintegrate pulp sample and followed the addition of 30ml of 4N standardized sulfuric acid solution to the saturated pulp to make a strong acidic reaction solution. The prepared mixtures' strong acidic reaction solution was heated at 25oc for 10minutes with continuous stirring using a magnetic stirrer. At the end of the reaction time, 5ml of potassium iodide was added to stop the reaction. Finally; as shown in figure 5 below 100 ml of the mixture was titrated against 0.1N sodium thiosulfate to pale color, two drops of starch indicator solution were added and the titration was continued to a colourless solution. The Kappa number determined by using equation (3) and lignin % by using equation (4) below.

Figure 5: A photo showing Kappa number determination by titration

$$K = \frac{p\,*}{0.1}\ FW \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

$$p = \frac{(b-a)\,N}{0.1} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

Where; K = kappa number, f = factor for correction to a 50% permanganate consumption, dependent on the value of p w = weight of moisture-free pulp in the specimen, g, p = amount of 0.1 N permanganate consumed by the test specimen, mL b = amount of the thiosulfate consumed in the blank determination, mL, a = amount of the thiosulfate consumed by the test specimen, mL and N = normality of the thiosulfate

Results And Discussions

Mass Loss During Size Reduction and Debarking

To prepare raw materials for pulping, size reduction, and debarking was conducted on cotton stalks and the following mass loss was observed.

Table 1: Cotton stalk mass loss during size reduction and debarking

Run	Cotton stalks mass before size reduction and debarking (kg)	Cotton stalks mass after size reduction and debarking (kg)	Mass loss (kg)
1	100gram	78.4gram	21.6gram
2	100 gram	80.5gram	19.5 gram
3	100 gram	79.5gram	20.5gram

The average mass loss is 20.53 grams from 100 grams of cotton stalks.

Moisture Content Determination of Cotton Stalks

The moisture content of the cotton stalk was determined by using equation (1) and the results are as follows:

Table 2: Moisture Content of Cotton Stalks

Run	Mass before drying(kg)	Mass after drying (kg)			Moisture content (%)
		2hr	4hr	6hr	
1	0.9	0.854	0.834	0.833	7.44
2	0.9	0.843	0.829	0.828	8.00
3	0.9	0.839	0.832	0.830	7.78

From this tabulated data the average moisture content of cotton stalk was 7.74% which in the range of recommended level of moisture content. This suits the cotton stalks for the application.

Cotton Stalk Pulp Yield

As per equation, (2) Yield of cotton stalk pulp for Soda pulping results was presented in the table below.

Table 3: Result of soda pulping

Run	Active alkali (%)	Temperature (°C)	Time (hr.)	Pulp yield %
1	10	100	3	24.7
2	15	100	3	29.3
3	20	100	3	32

The maximum pulp yield was 32% obtained when 20% soda concentration was used at 100°C and 3hr (Run3), and the minimum pulp yield obtained when the soda concentration was 10% at constant temperature and time. The result indicates that active alkali has a positive effect on pulp yield at constant temperature and cooking time. Active alkali increases the lignification rate of the cotton stalk and more cellulose was extracted as the active alkali increase. With experimentation at elevated temperature and selection of more suitable types of cotton stalks much more pulp yield can be obtained.

Effects of Active Alkali On Soda Pulp Yield

Pulp yield is affected by cooking temperature, cooking time, and active alkaline of the liquor solution. Figure 5 displays the effect of active alkali on pulp. It is evident from figure 5 that the yield of pulp increases with an increase in soda concentration.

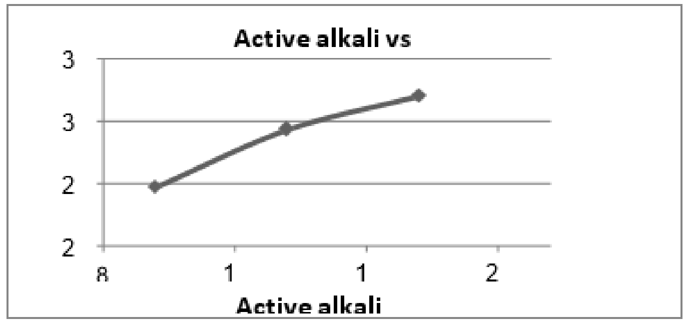

Figure 6: Effect of soda concentration on pulp yield

Active alkali influenced the pulp yield as observed from the graph, as active alkali increases the lignification rate increase since sodium hydroxide act as white liquor, react and solubilization of lignin to break down the bond between cellulose and lignin so higher the active alkali means more solubilization can occur and more cellulose was extracted.

Kraft Pulping

The yield of cotton stalk pulp was calculated by equation (2) and the results were presented in the table below:

Table 4: Kraft Pulping Yield

Run	Active alkali	Pulping temp. (°C)	Time (hr.)	Pulp yield %
1	15	100	4	36.5
2	20	100	2	33.7
3	15	100	2	34.2
4	10	100	2	31.8
5	15	100	3	35.4

The maximum pulp yield obtained from the experiment was 36.5% at the cooking conditions of 15% active alkali, temperature and time were at 100°C and 4hr respectively. The minimum pulp yield was obtained 31.8% at the cooking condition of 10%active alkalies, 100°C temperature, and 2hr of holding time. Kraft pulping method was better than that of soda pulping depending on the yield of pulp produced. The maximum pulp obtained by the kraft method was 34.2%, and 32% was recorded on the soda pulping method. Since the kraft pulping method use sodium sulphate and sodium carbonate to facilitate the lignification of cellulose it was easy to bleach, it gives white and bright white color after bleached with hydrogen peroxide than that of soda pulping because in Kraft pulping method sodium sulphate and sodium carbonate were used instead of sodium sulphide. The result of the experiments suggests kraft pulping than soda pulping.

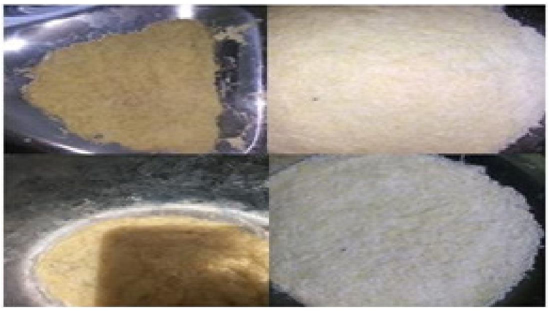

Figure 7: Bleached Soda and Kraft Pulp Respectively

Effects Of Active Alkali On Pulp Yield Based On Kraft Pulping

The effect of active alkali on pulp yield was expressed at constant temperature and time in figure 6 below:

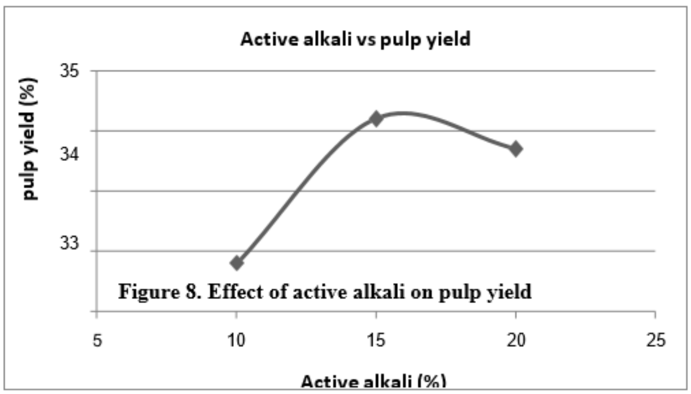

Figure 8. Effect of active alkali on pulp yield

Active alkali influenced pulp yield as observed from the above graph. Active alkali increase so does the pulp yield up to the maximum point and the pulp decrease as the active alkali increase and increase beyond the maximum point. Active alkali increase means the lignification rate also increases more cellulose can be extracted but as it increases beyond the maximum it was affecting the pulp yield. The maximum pulp yield was recorded at 34.2% on cooking condition of 15% active alkali at constant temperature and cooking time. In the kraft pulping method, sodium sulfate was used as cooking chemicals so the lignification rate can be affected by active alkali and sulfidity.

Effect of Cooking Time On Pulp Yield Based On Kraft Pulping

Effect of cooking time on pulp yield based on Kraft puling was expressed in figure 7 below:

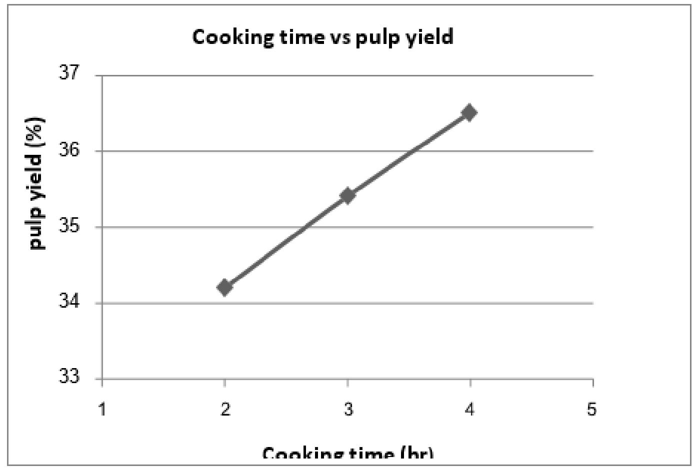

Figure 9: Effect of cooking time on pulp yield

From the above graph as time increase pulp yield also increase which indicates that as time increase lignification rate also increases so more cellulose was extracted than short time cooking. Here the experiment was conducted at a minimum temperature and high cooking time which was somewhat not good on the yield of pulp. Cooking at a lower temperature for a long time is not good as it is energy-intensive, so it is better to cook at a short time at an elevated temperature for utilizing energy and time efficiently.

Kappa Number Determination

Kappa number of pulp and lignin (%) were determined based on equation (3 and 4) and the results were tabulated in Table 5 below Kappa Number, Pulp Yield, And Lignin (%)

Run	Active alkali	Yield (%)	Kappa no	Lignin (%)
1	10	31.8	42.3	5.58
2	15	34.2	31.5	4.16
3	20	33.7	36.7	4.84

According to table 5, the pulp yield from run #1 had the highest kappa number. In contrast, the pulp yield from run #2 had the lowest kappa number. This indicated that yield and kappa number had an inverse proportion relationship, more yield means less lignin present, and cellulose was extracted effectively. A higher kappa number indicates the higher lignin content on the pulp and needs more hydrogen peroxide to bleach. The kappa number at maximum pulp yield was 31.5 (run #2) at constant temperature and cooking time, and it was acceptable since it was easily bleachable.

By kraft pulping; higher pulp yield and easily bleachable pulp were obtained than the soda pulping method. Poor extraction of cellulose was observed in soda pulping and also higher kappa number and lignin obtained from this pulping. Higher kappa number and lignin (%) indicate low quality, yield, and require more hydrogen peroxide for bleaching. Kraft pulping method reduced the negative effects of soda pulping. Kraft pulping carried out at the optimum condition of 15% soda concentration at 100oC and for 2hr results good quality pulp than soda pulping.

Effect of Active Alkali On Kappa Number

figure 8 shows the effects of soda concentration on kappa number during the Kraft pulping of cotton stalk.

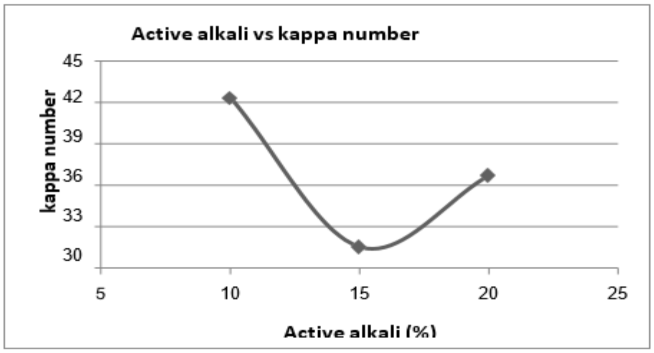

Figure 10. Effect of Soda Concentration On Kappa Number

It can be seen that soda concentration had a significant influence on the kappa number. An increase in soda concentration resulted in a clear reduction in kappa number. The effect of soda concentration was estimated at constant temperature and cooking time, but as the concentration of soda increase beyond the maximum point kappa number was increased it indicates the extraction of cellulose at higher soda concentration was not effective. Since the pulping method was Kraft so the percentage of sulfidity had an influence on kappa number like that of active alkali, in this study sulfidity was not properly considered, the variation of the result may be affected by sulphide. As a recommendation to analyse the effect of soda concentration on kappa number, consider the percentage of sulfidity becomes better to analyse the effect properly.

Conclusion

The cotton stalk can be successfully pulped with soda and Kraft pulping process in different conditions. The maximum pulp yield was determined for both Kraft and soda pulping were 34.2% at 15% alkali concentration, 100oC, and 2 hr pulping conditions, 32% at 10% alkai concentration, 100oc and 3 hr pulping condition respectively. From the two pulping methods Kraft pulping method was selected and it was better than that of soda depending on pulp yield and bleachable. Also, Kraft pulping method was better in energy consumption and a short time of cooking. Kappa number is the indication of residual lignin in the pulp and it was determined at different conditions. Kappa number was determined based on Kraft pulping method, from the results of the experiment, it can be seen that as the soda concentration increases the kappa number decreases. At the maximum yield, the kappa number was determined and it was 31.5. Active Alkali and cooking time have a great effect on pulp yield. active alkali has to increase and then decreasing effects on pulp yield and effect of cooking time on pulp yield at maximum temperature has an increasing effect. Generally, the result shows the Kraft pulping method was the best pulping method, and operating parameters of temperature, active alkali, and cooking time had a great effect on both Kraft and soda pulping. From the results of this work, it can be concluded that cotton stalk can be used as a raw material for pulp and paper production.

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