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# Optimization of Alkaline Pulp Extraction from Napier Grass Using Response Surface Methodology

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**Abstract.** Napier grass which has several types of them has been studied by researchers for few applications such as biofuel, sugar and pulp production. This research focused on specific type of Napier grass which is the Dwarf Napier due to high cellulose content to produce pulp. Delignification of Dwarf Napier grass was carried out by soda anthraquinone pulping under varying conditions selected according to experimental design software with three factors. The influence of these three factors including concentration of sodium hydroxide (15%, 17.5% and 20%), concentration of anthraquinone (0.05% and 0.1%) and duration of cooking time (30, 60 and 90 min) on pulp yield was studied. 18 runs of experiment were conducted and the result obtained was used to analyse the optimized condition for pulp extraction by using RSM. Results indicated that the optimized response obtained while Napier grass stem is treated with 17.5% sodium hydroxide and 0.05% AQ with 66 mins of cooking time meanwhile the predicted yield is 34.87 %. However, the actual pulp yield obtained (36.38%) is higher than the predicted yield. The pulp which extracted with optimized condition is then bleached with hydroxide peroxide.. Fourier transform infrared spectroscopy (FTIR) was used to characterize the raw grass fiber and pulp obtained.. Napier grass is successfully demonstrated to be an effective alternative source for producing paper pulp. In the this study Dwarf Napier grass fibres were successfully pulped applying soda AQ process although the obtained pulp is not as high as the studied done before due to the limitations. FTIR analysis also confirmed the removal of lignin as well as most of the hemicellulose during the pulping process. Therefore, the abundance and fast growth of Napier grass are an added advantage as an alternative non-wood source for papermaking

## 1. Introduction

Today, paper is one of the most important elements in our life and society. Due to the rising demand of paper based product, the consumption of paper and pulp and paper industries have been steadily increased over the world as well [4]. As a result massive deforestation and replantation have been occurred all around the world as the virgin pulp is the main source and raw material for pulp and paper industries. It should be noticed that those consequences has contributed to climate change and altered the ecological balance [4].

Due to the environment concern, researchers has been identifying new non wood materials that can deliver the standard of paper (which are high yield, low cost and high quality) to minimize the deforestation as the quality of the biomass raw material is very significant for pulp and paper industry [12]. There are many perennial plants which have a short life cycle including switch, reed grasses and



bamboo that have been determined, planted and researched for their applicability for pulp and paper industry [7]. Therefore, the rapid growth, abundance, high productivity and easy cultivation of Napier grass make it a potential supply to meet the current needs [7]. It is a well-known forage crop because of its high biomass production rate and excellent agronomic characteristics. Napier Grass is also known as elephant grass and its scientific name is *Pennisetum Purpureum*. The concept of paper making is to remove the cellulose in the wood or any plant-based raw material from the non-cellulosic compound by using several type of pulping process such as chemical and mechanical pulping. Soda process is one of the standard pulping methods that will be used for non-wood raw material [6]. However, soda process is suffered from low pulp yield of inferior which is caused by the higher temperature, long cooking time and caustic charges needed to produce bleachable grade pulps [3]. Therefore, additive which can improve the soda process is developed. As a result, soda pulping with anthraquinone as additive was introduced to decrease the carbohydrate degradation while pulping.

In this research, Napier grass fibres was used for pulp extraction with alkaline hydrolysis and anthraquinone as the additive to determine the most suitable parameter for pulp production. The main objectives of this research were to determine the optimum extraction condition with highest yield of dry pulp using Response Surface Methodology (RSM) method, to analyze the properties of the pulp produced and to identify the functional group in Napier grass pulp using FTIR.

## 2. Material and Methods

### 2.1. Material

Dwarf Napier Grass was obtained from one of the farm in Malaysia. Sodium hydroxide, acetic acid, anthraquinone, hydrogen peroxide were used in the pulping process. Meanwhile, FTIR spectrophotometer was used to identify the functional group in pulp produced. .

### 2.2 RSM model

In order to investigate the optimum condition for alkali pulping, Design Expert software version 10.0.1 was used as the RSM tool to design the experiment. Percentage of sodium hydroxide, percentage of anthraquinone and duration of cooking time controlled were the factors while pulp yield was the response as shown in Table 1.

**Table 1.** Factor listed in Design Expert

Name	Units	Type	Levels	L[1]	L[2]	L[3]
<b>NaOH</b>	%	Discrete	3	15	17.5	20
<b>AQ</b>	%	Discrete	2	0.05	0.01	
<b>Cooking time</b>	min	Discrete	3	30	60	90

### 2.3 Fibre Extraction

Napier grass roots and leaf were removed and left only the stems. The stems were then soaked in distilled water for 48 hours (retting process). The grass fibres were then washed before it was dried in oven for 2 days at 80°C [7]. The dried fibre was then blended to reduce the particle size.

### 2.4 Pulping

Soda-AQ pulping of Napier grass fibre was conducted using a beaker and a stirrer hot plate with cooking condition as stated in Table 1. 150 ml sodium hydroxide solutions of various concentration was heated to 110°C on a stirrer hot plate then 15g of dried fibres was weighted and mixed with the sodium hydroxide solution while the solution was continuously heated and stirred at 1100 rpm. Pulp was then filtered out from the black liquor and washed with 10% acetic acid and distilled water to neutralize the reaction. The pulp was then dried in the oven for 24 hr at 70°C. The result was recorded in Table 2.

### 2.5 Bleaching of pulp

The Dwarf Napier grass pulp obtained was then cooked in the mixture of 0.01M sodium acetate buffer solution, 2% hydrogen peroxide and distilled water with a ratio of 1:1:1. The mixture solution was first heated up to 90°C before the pulp was cooked for 2 hours. Then, the bleached pulp was filtered and washed with distilled water before drying process.

### 2.6 Functional Group Analysis

Fourier transform infrared spectroscopy (FTIR) was used to identify the functional groups existed in the dried raw Dwarf Napier grass, dried pulp and dried bleached pulp. All the spectra were recorded in the range of 350 to 7800 $\text{cm}^{-1}$  region with 20 scans in each case at a resolution of 4 $\text{cm}^{-1}$ .

## 3. Results and Discussion

### 3.1 Pulp Yield

From **Table 2**, 17.5% of sodium hydroxide and 0.1% of anthraquinone with a 60mins cooking time showed the best pulp yield of 35.91% among other conditions while 20% of sodium hydroxide and 0.1% of anthraquinone with a 90 mins cooking time had the lowest pulp yield of 30.87%. The result obtained was used as an input for Design Expert software to analyse the optimized condition.

**Table 2.** Result Summary for each run

Run	% of NaOH	% of AQ	Cooking time (min)	Pulp yield
1	15	0.05	30	31.57%
2	15	0.05	60	33.37%
3	15	0.05	90	32.98%
4	15	0.1	30	31.59%
5	15	0.1	60	32.73%
6	15	0.1	90	33.24%
7	17.5	0.05	30	32.68%
8	17.5	0.05	60	35.09%
9	17.5	0.05	90	33.24%
10	17.5	0.1	30	31.92%
11	17.5	0.1	60	35.91%
12	17.5	0.1	90	33.33%
13	20	0.05	30	32.15%
14	20	0.05	60	32.84%
15	20	0.05	90	34.46%
16	20	0.1	30	31.91%
17	20	0.1	60	32.15%
18	20	0.1	90	30.87%

### 3.2 Response surface model

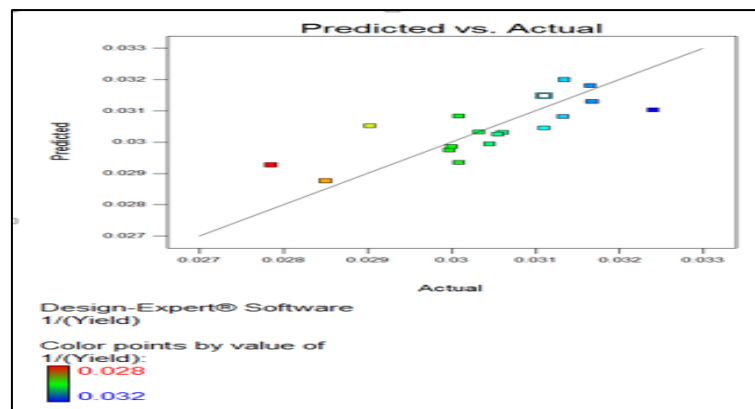
**Figure 1** represents predicted value versus actual value. It can be observed that it has a few divergence of point from the diagonal indicating that these response surface model equations is inadequately to represent the interaction of the three factors which might be caused by the sampling error which is discussed in limitations. **Table 3** has showed the model summary statistics. It showed

that linear model has a relatively small standard deviation of 0.001148 which is suggested to be used in response surface method. As shown in **Table 4**, analysis of variance table has shown the response surface model for pulp yield has F-value of 3.44 which indicate the model is significant as the value of “Prob>F” is less than 0.5. In this case,  $A^2$  and  $C^2$  are significant model terms which has a value of “Prob>F” of 0.0303 and 0.0319 respectively while A, B, C and  $B^2$  is not significant. The higher the value of F, the more significant effect on the response. Therefore, the model term having most significant effect on the response is  $A^2$  followed by  $C^2$  which have F value of 6.02 and 5.89 respectively

**Figure 2** showed the 3D graph of the results from the three factors. Optimized condition obtained for the best yield of Dwarf Napier grass stem is 17.5% sodium hydroxide and 0.05% AQ with 66.5 mins of cooking time. Pulp yield from the optimized condition was 36.378%.

**Table 3.** Model Summary Statistics

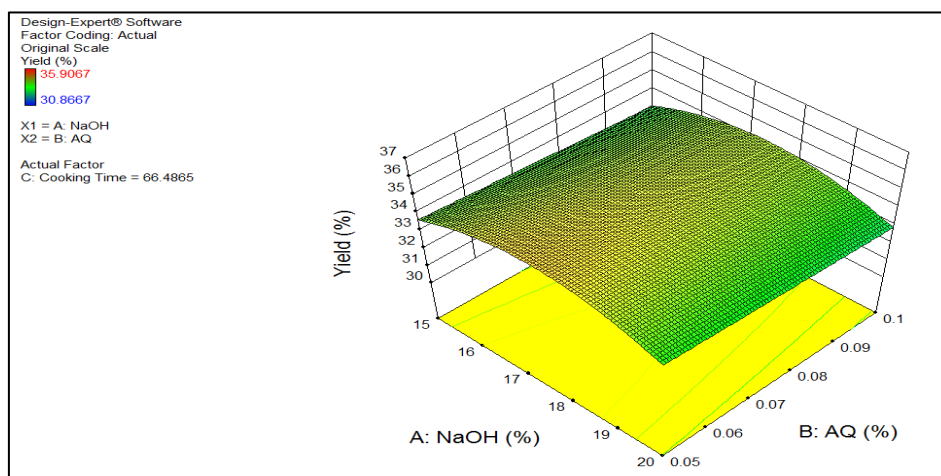
Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	1.148E-003	0.1811	0.0056	-0.2461	2.806E-005	Suggested
2FI	1.218E-003	0.2753	-0.1200	-0.7178	3.869E-005	
Quadratic	8.903E-004	0.6832	0.4017	-0.3326	3.001E-005	Aliased



**Figure 1.** Predicted response to actual response graph

**Table 4.** Result Summary for each run

Analysis of variance table [Partial sum of squares - Type III]						
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	1.327E-005	5	2.653E-006	3.44	0.0369	significant
A-NaOH	1.111E-007	1	1.111E-007	0.14	0.7109	
B-AQ	1.167E-006	1	1.167E-006	1.51	0.2422	
C-Cooking Time	2.800E-006	1	2.800E-006	3.63	0.0810	
$A^2$	4.646E-006	1	4.646E-006	6.02	0.0303	
$B^2$	0.000	0				
$C^2$	4.540E-006	1	4.540E-006	5.89	0.0319	
Residual	9.255E-006	12	7.712E-007			
Cor Total	2.252E-005	17				



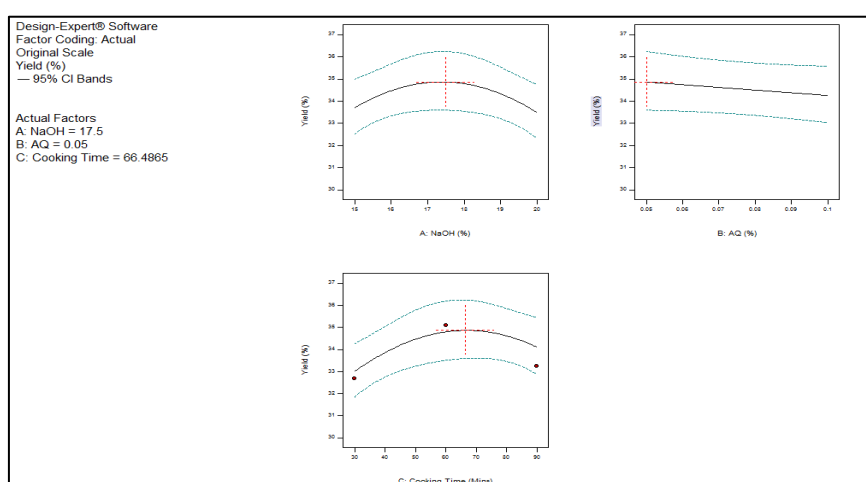
**Figure 2.** Response surface methodology result of three factors

### 3.3 Effect of variables

**Figure 3** showed the yield to each factor accordingly and it can be observed that the yield increased as the concentration of sodium hydroxide increased. Then it started to decrease after it reached its peak from 17.5% to 20% of sodium hydroxide. Therefore, 17.5% of sodium hydroxide is the most suitable concentration for Napier grass.

From **Figure 3** which showed the relation between yield percentage to AQ%, a slope is obtained from effect of AQ% which had no highest peak in the response. From the result obtained from **Table 2**, it can be concluded that the effect of AQ on the pulp yield is not consistent which might cause by the sampling error and limitation which is discussed in section. However, 0.05% of AQ show a higher effect on the response.

As result showed in **Table 2**, both 60 mins and 90 mins cooking times have obtained highest pulp yield which means the optimized condition is lay in between. From **Figure 3**, pulp yield to cooking time showed the higher the cooking time the higher the pulp yield but the pulp yield was dropped after it passed 66.5 mins, therefore the optimized cooking time is 66.5 mins.



**Figure 3.** Yield to factors graph from Design Expert

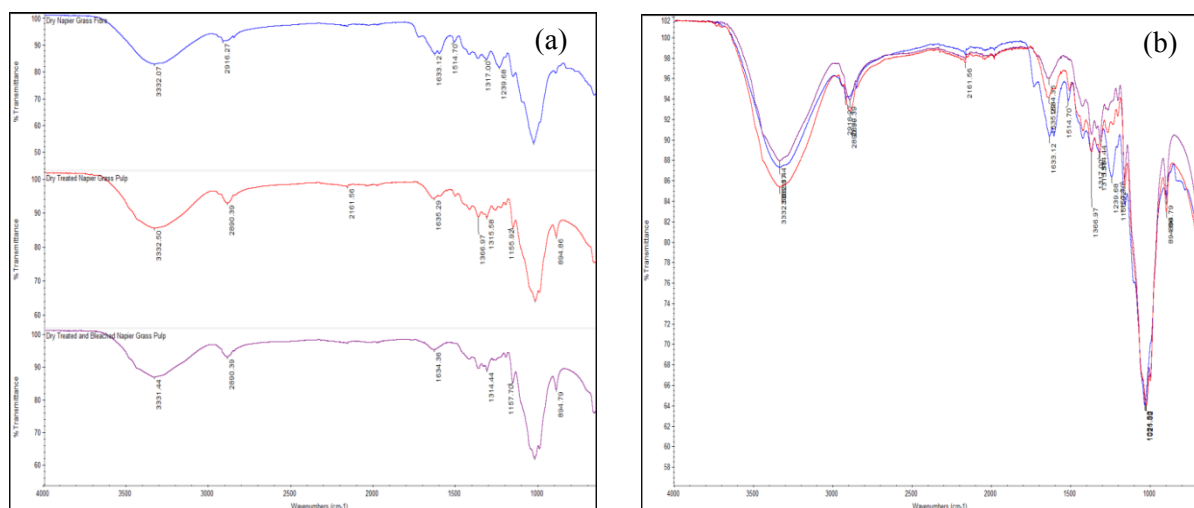
### 3.4 Functional Group Analysis

FTIR spectra for dry Napier grass, dry treated Napier Grass pulp and dry treated and bleached Napier grass pulp was shown separately in **Figure 4(a)** while **Figure 4(b)** shown the FTIR spectra of three

samples were combined. In comparison of **Figure 4(a)** and **Figure 4(b)**, it can be concluded that all three samples showed a strong absorption band at  $3331\text{cm}^{-1}$  which corresponds to OH- bond. Presence of hydroxyl groups are proved by the existence of intense and broad spectrum [1]. Besides, the dry raw Dwarf Napier grass has showed a higher transmittance than dry Dwarf Napier Grass pulp and dry bleached Dwarf Napier grass pulp at absorption bands of  $2916$  and  $2890\text{ cm}^{-1}$  which corresponds to the asymmetric and symmetric stretching vibrations of methylene group of all the three (cellulose, hemicelluloses, and lignin) constituents [7]. Apart from that, a difference of transmittance occurred at  $1727\text{ cm}^{-1}$  absorption band which corresponds to C=O stretching of the carbonyl and acetyl groups in hemicelluloses due to the removal of hemicellulose. Due to the removal of hemicellulose during the pulping process, the intensity of the carbonyl group decreased in both dry pulp and dry bleached pulp [7].

An absorption band at  $1634\text{cm}^{-1}$  was attributed to bending mode of the absorbed water. Others than that, both dry treated Dwarf Napier Grass pulp and dry bleached Dwarf Napier grass pulp has a relatively higher transmittance compared to raw Dwarf Napier grass at absorption bands of  $1605$ ,  $1514$ , and  $1428\text{ cm}^{-1}$  which corresponds to aromatic ring vibrations [10]. The intensity of this band significantly decreased in obtained pulp due to the removal of lignin in the process. Furthermore, both dry treated pulp and dry bleached pulp has showed a significant difference at  $1455\text{ cm}^{-1}$  absorption band which corresponds to the C-H deformation methyl, methylene, and methoxyl groups combined with the aromatic ring vibration of lignin [13]. The absorption bands at  $1377$  and  $1319\text{ cm}^{-1}$  correspond to C-H asymmetric deformation and -OH bending vibration of cellulose [10]. Moreover, the absorption bands at  $1239\text{ cm}^{-1}$  correspond to -COO vibration of acetyl groups in hemicelluloses [9]. The intensity of this band significantly decreased in both dry treated pulp and dry t bleached pulp due to the removal of hemicellulose in the process.

The absorption band at  $1164\text{ cm}^{-1}$  corresponds to C-O antisymmetric bridge stretching of cellulose while absorption band at  $1110\text{ cm}^{-1}$  belongs to C-C and C-O asymmetric in phase ring stretching of cellulose. A strong absorption band at  $1054\text{ cm}^{-1}$  corresponds to C-O-C symmetric stretching dialkyl ether linkages and C-O stretching vibration in cellulose [8]. Besides, it can be observed that both obtained pulp showed a strong absorption band at around  $894\text{cm}^{-1}$  which corresponds to C-H rocking vibrations of cellulose.



**Figure 4.** FTIR spectra of dry grass, dry pulp and dry bleached pulp (a) separated (b) combined

#### 4. Conclusion

The perennial Dwarf Napier grass has shown a promising result to be an effective alternative source for producing pulp for paper. In this study the soda-AQ pulping process of Dwarf Napier grass fibres were optimized by using RSM method to produce higher yield. The pulp obtained from the optimized condition was 36.378%. The removal of lignin and hemicellulose during the pulping process has been proved by the functional group analysis using FTIR. Therefore, Dwarf Napier grass which can be found abundantly and fast growth should be considered as an alternative source for papermaking industry.

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