ISSN (Online): 2455-7838
SJIF Impact Factor (2016): 4.144

EPRA International Journal of

Research & Development

(IJRD)

Monthly Peer Reviewed & Indexed
International Online Journal

Volume:2, Issue:1, January 2017

Published By :
EPRA Journals

SJIF Impact Factor: 4.144 ISSN: 2455-7838(Online)
EPRA International Journal of Research and Development (IJRD)
Volume: 2 | Issue: 1 | January | 2017

SODA-ANTHRAQUINONE PULPING OF OXYTENANTHERA ABYSSINICA

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ABSTRACT

Bamboo is a fast growing non-wood plant with long and thin fibers. Therefore, it has potential as a raw material for pulping and papermaking from lowland bamboo (Oxytenanthera abyssinica). Samples were harvested, sorted, dried and milled using Wiley Mill and chips of greater than 5mm in length was selected. In this work, the effect of soda- Anthraquinone (AQ) pulping conditions on pulp yield and kappa number of lowland bamboo were evaluated. The bamboo chips were pulped using digester pulping unit with 15, 20 and 25% varying alkali charge; 60, 120 and 180 minute cooking time and 150, 175 and 200°C cooking temperature. The maximum pulp yield and minimum kappa number obtained was 52.24% and 12.16, respectively. This was achieved under the optimum conditions: at the active alkali of 20%, cooking temperature of 150°C and cooking time of 180 minute with high value of combined desirability, i.e. 0.98. This study indicated that good pulp yield can be produced from O. abyssinica with more environmentally friendly chemical pulping process. Finally, it can be concluded that O. abyssinica has a great potential for manufacturing of pulp in paper and paper products processing industries.

KEYWORDS: *Bamboo, Kappa number, Pulp yield, Soda-AQ pulping.*

I. INTRODUCTION

Worldwide cellulose pulp is produced from hardwoods, softwoods and agro residues. Hard wood and soft wood species based wood pulping accounts to about 95% of the total worldwide pulp production. The rest 5% pulp comes from non- wood raw materials, mainly agro residues and grasses (Jimenez *et al.*, 2005). Bamboo is an important fibrous raw material used in pulp and paper industries. It is a naturally occurring composite biological plant material which grows abundantly in most of the tropical countries. It is considered a composite material because it consists of cellulose fibers agglomerated in hemicelluloses matrix and lignin as an encrusting substances (Lakkad and Patel, 1981). More than 1,500 species and 90 genera of bamboos are found in the world, covering 36 million hectare (ha) of land which is distributed in the tropical and sub-tropical belt between 46^0 north and 47^0 south latitude at elevations as high as 4000m above sea level (Zhou *et al.*, 2005; FAO, 2007). Bamboo is fast growing species has very high biomass and is well established as an excellent fiber raw material for the production of pulp and paper (INBAR, 2003).

There are about 43 species of bamboo under 11 genera in Africa, covering over 1.5 million hectare. About 93% of Africa bamboo species are found only in Madagascar (FAO, 2007; Seyoum Kelemework, 2008). Bamboo forest in Ethiopia is the largest in Africa and it has two indigenous bamboo species: the highland bamboo *Yushania alpina* (synonym *Arundinaria alpina*) and the lowland bamboo *Oxytenanthera abyssinica*. Out of which the highland bamboo comprises about 130,000 ha and lowland bamboo covers over 850,000 ha (Kelbessa et al., 2000; Embaye Kasahun, 2003). In Ethiopia, the potential of bamboo resources have been used for bamboo-based panel and some hand woven products. Currently, there are some works coming to surface on the physical and mechanical properties of these species (Seyoum kelemework, 2008). However, the selection of bamboo species for various applications is not only related to the physical and the mechanical properties but knowledge on the chemical composition is the determinant one particularly for the pulp production.

The ever growing demand for pulp and paper products has forced countries of the world to look for potential and sustainable raw material for industries. Ethiopia as a developing country with very low paper and paper board consumption per capita (1kg/person/year) is expected to grow significantly. The ongoing economic development and livelihood improvement in Ethiopia inevitably would create huge demand and consumption of pulp and paper products due to increasing economic welfare household consumption, expansions of schools and universities, and increasing of manufacturing sectors that needs packaging and wrapping.

In Ethiopia one of the problems in pulp and paper industry is almost absence of pulp mill but there are over 22 companies involved in paper making and trading businesses, of which only 'Ethiopia Pulp and Paper Share Company' and 'Barguba plc' uses imported pulp for their paper mills, while others import, produced paper rolls for further processing. Ethiopian paper mills were designed to utilize mixture of short and long fiber pulp. Ethiopia imported more than 117,000 tons of paper and pulp in 2013, spending more than 2.6 billion Birr, according to data obtained from Chemical and Construction Input Industry Development Institute.

Furthermore, the ever increase price of raw material (pulp) for the production of paper and the absence of pulp mill in the country compared with the foreign exchange burden is a major motive to develop the pulp industry in the country. The growing demand for paper products has made the issue of paramount importance. Ethiopia is endowed with good agro-ecology to develop its natural resources that enable it produce pulp from both wood and non-wood inputs. Among the non-wood resources, the existence of bamboo forests creates a good opportunity for developing the pulp and paper industry sub-sector.

Bamboo forest in Ethiopia is the largest in Africa and the country can sustainably produce three million cubic meters of dry weight biomass annually from its two indigenous bamboo species namely *Yushania alpina* and *Oxytenanthera abyssinica*. Despite this potential, current uses are primarily limited to construction of traditional houses, low-grade furniture, household utensils, beehives, fences, and handicrafts and laminated bamboo floor boards. Bamboo is preferred because it has an important role to play in the balance of oxygen and carbon dioxide in the atmosphere, a viable replacement for wood as an enduring natural resource (WAALCA, 2000).

Therefore, the rationale of this research work is to investigate the potential of *O. abyssinica* resource in Ethiopia in fiber based products (pulp) for paper production which could be a good source of raw material.

II. MATERIALS AND METHODS

O. abyssinica, the main raw material for pulp production in this work is available in Ethiopia grows in the western part along major river valleys and in the lowlands bordering Sudan. The *O. abyssinica* samples used for this study were collected around Assosa town of Benishangul Gumuz Region.

Soda-AQ Pulping of *Oxytenanthera abyssinica*

The *O. abyssinica* culms were chipped with hammer mill and chips of greater than 5mm in length was selected. Soda cooking liquor was prepared from a standard concentrated solution of sodium hydroxide by serial dilution with distilled water. Using alkaline pulping method (Soda-AQ) the *O. abyssinica* chips were weighed and charged into a reactor with the required amount of chemical solution at liquor to solid ratio of 4:1. The reactor was heated to the operating pulping conditions used to cook bamboo chips are shown in table (1). The resulting pulp was thoroughly washed with tap water and the pulp yield was determined gravimetrically after drying at 103 ±2^{o}C to constant weight in the oven. In each batch of bamboo pulping were added 0.1% AQ in 1:4 bamboo chips to liquor ratio. The pulp was screened through a 250-µm -1mm mesh sieve size and separated for screened yield and kappa number was determined for each runs.

Table1: Experimental conditions setup.

Pulping conditions	Soda (AQ)
Active alkaline percent as oven dry	15, 20, 25
Liquor to wood ratio	4:1
Anthraquinone (AQ) percent	0.1
Time to max. temperature (min)	30, 60, 90
Time at max. temperature (min)	60, 120, 180
Max. temperature (°C)	150, 175, 200

III. RESULTS AND DISCUSSION

a) Effects of pulping conditions on *O. abyssinica* pulp yields and kappa number

The effect of pulping time, cooking temperature and active alkali on pulp yield and kappa number were studied and evaluated for best pulping conditions. Determination of residual lignin in the pulp is the most important in all pulp analyses. It indicates the degree of delignification obtained by the cook and forms the basis of comparison for many of the cooking results, such as yield, screenings, pulp brightness, etc.

The kappa number is an index used by the pulp and paper industry to express the residual lignin content in unbleached pulp after cooking. Lignin is responsible for the brown coloration of paper on service and residual lignin is removed at the subsequent bleaching stage to obtain desired brightness. Lower residual lignin in pulp content required less amount of bleaching chemicals to obtain the same brightness of bleached pulp. Therefore, the lignin content must be well known, so that only a minimum amount of bleaching chemicals will be used. The higher the lignin content in the pulp the more is the kappa number. The pulp having high lignin content is termed as hard cooked pulp and the pulp with low lignin content as soft cooked pulp. The hard cooked pulp requires more bleaching chemicals to attain particular brightness compared to soft cooked pulp.

The collected data were analyzed using Design expert® 7.0.0 software to determine the effects of active alkali concentration, pulping temperature and time. The dependent variables used as a response parameter were the pulp yield and kappa number. All experiments were carried out in a randomized order to minimize the effect of unexpected variability in the observed response due to extraneous factors.

b) Adequacy check for the developed models

The adequacy of the model was checked by analysis of variance (ANOVA) and some diagnostic plots. Analysis of variance (ANOVA) was employed to test the siginificance of the developed models. Table 2 and 3 showed the summary of the analysis of variance (ANOVA) of the two responses pulp yield and kappa number, respectively.

Table 2: Analysis of variance (ANOVA) for pulp yield.

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	605.19	18	33.62	26.35	< 0.0001
A-Temperature	376.55	2	188.28	147.58	< 0.0001
B-Active alkali	84.25	2	42.13	33.02	0.0001
C-cooking time	60.52	2	30.26	23.72	0.0004
A*B	19.21	4	4.80	3.76	0.0523
A*C	43.7	4	10.94	8.57	0.0054
B*C	20.91	4	5.23	4.10	0.0427
Residual	10.21	8	1.28		
Cor Total	615.40	26			

*Interaction between the factors/ the process variables.

In the present study for the response pulp yield the Model F-value of 26.35 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" occur due to noise.Values of "Prob > F" less than 0.0500 shown model terms were significant.

In this study it was realised that the individual factors and their interaction effects were significant model terms. For the response kappa number, the Model F-value of 18.10 implies the model was significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC were significant model terms. Values greater than 0.1000 indicated the model terms were not significant.

Table 3: Analysis of variance (ANOVA) for kappa number.

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	2088.22	18	116.01	18.10	0.0001
A-Temperature	446.25	2	223.12	34.81	0.0001
B-Active alkali	317.67	2	158.83	24.78	0.0004
C-cooking time	943.38	2	471.69	73.58	< 0.0001
AB	125.18	4	31.30	4.88	0.0274
AC	235.37	4	58.84	9.18	0.0044
BC	20.37	4	5.09	0.79	0.5608
Residual	51.28	8	6.41		
Cor Total	2139.50	26			

The quality of the model developed coude be evaluated from their coefficients of correlation, $\mathbf{R^2}$ (coefficent of determination) as showed in Table 4 was high for both responses, a value > 0.75 indicates the aptness of the model. For a good statistical mode, the $\mathbf{R^2}$ value should be close to one. Results ensured a satisfactory adjustment of the 2FI model to the experimental data and indicated that approximately 98.34% and 97.60% of the variability in the dependent variables of pulp yield and kappa number, respectively could be explained by these models and only 1.66% and 2.4% of the total variance could not explained by these models for pulp yield and kappa number, respectively. The value of $\mathbf{R^2}$ for both responses was very high and close to one which indicates a good agreement between experimental and predicted values. The predicted $\mathbf{R^2}$ was in a reasonable agreement with the adjusted $\mathbf{R^2}$ for both responses.

Table 4: Model adequacy measures for pulp yield and kappa number

	Pulp yield	Kappa number		Pulp yield	Kappa number
Std. Dev	1.13	2.53	R-Squared	0.9834	0.9760
Mean	47.05	22.03	Adj R-Squared	0.9461	0.9221
C.V. %	2.40	11.49	Pred R-Squared	0.8111	0.7270
RESS	116.25	584.16	Adeq Precision	20.374	16.181

In this case model for pulp yield the "Pred R-Squared" of 0.8111 was in reasonable agreement with the "Adj R-Squared" of 0.9461. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. In this case, ratio of 20.374 indicates an adequate signal as shown in Table 5 for pulp yield. So, this model can be used to navigate the design space.

In modeling the Kappa numner adequecy, the "Pred R-Squared" of 0.7270 was in reasonable agreement with the "Adj R-Squared" of 0.9221. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Ratio of 16.181 indicated an adequate signal as shown in Table 5 for kappa number. So, this model can be used to navigate the design space.

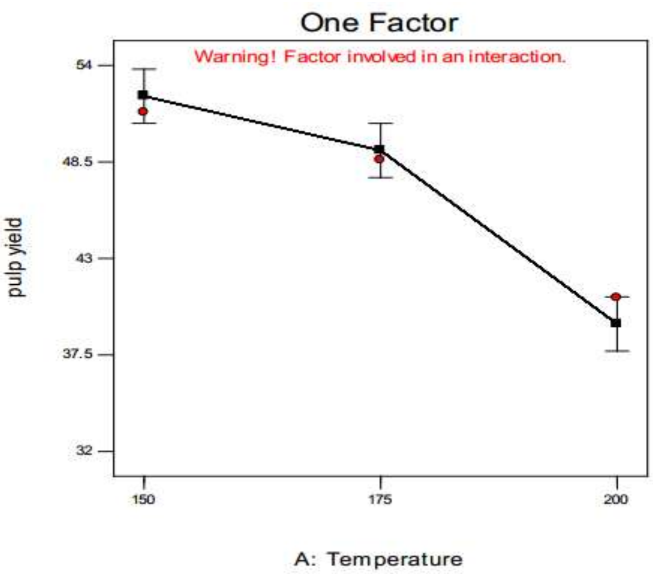
c) Effect of Pulping Process Variables

The overall screened yield for soda-AQ pulping conducted in this study. The effects of pulping time, cooking temperature and active alkali concentration on pulp yield and kappa number were studied and evaluated for best pulping conditions. Based on the analysis of variance, pulping was significantly affected by various interactions between the process variables. In addition to the interaction effect, significant individual process variables that affect the pulp yield and kappa number were cooking temperature, active alkali concentration, and reaction time. There was a general decrease in the pulp yield and kappa number (residual lignin) due to an increase in the temperature, active alkali concentration and cooking time taking uniformly the AQ concentration to 0.1% and 4:1 liquor to solid ratio.

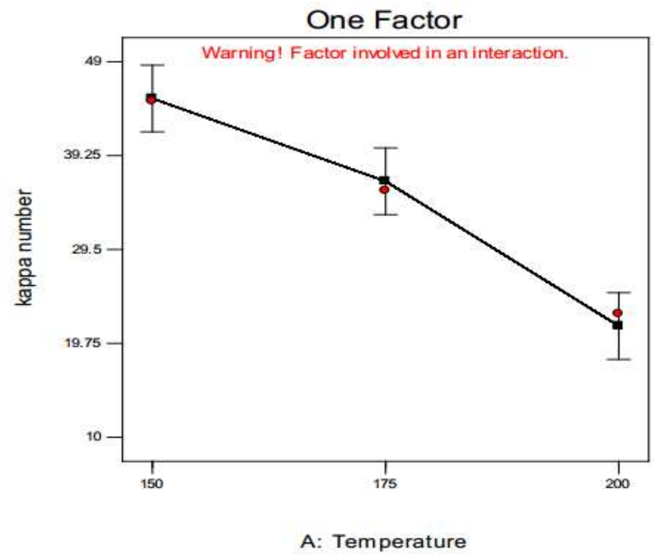
i) Effect of Individual Process Variables

As shown in Figure 1 (a) and (b) below the pulp yield and kappa number were significantly affected by reaction temperature. It can be seen from the figure that with increasing reaction temperature from 150°C to 200°C the pulp yield and kappa number generally decreased.

The decreasing in pulp yield and kappa number at higher reaction temperature was due to higher rate of reaction. This might be due to lignin, hemicelluloses and cellulose were dissolved out at different rates during cooking and these rates were much accelerated by increasing the temperature, and thus decreasing the pulp yield and residual lignin. Higher temperature resulted in higher reaction rate constant which will lead to higher rate of reaction and eventually decreased pulp yield and kappa number (residual lignin)



. (a)

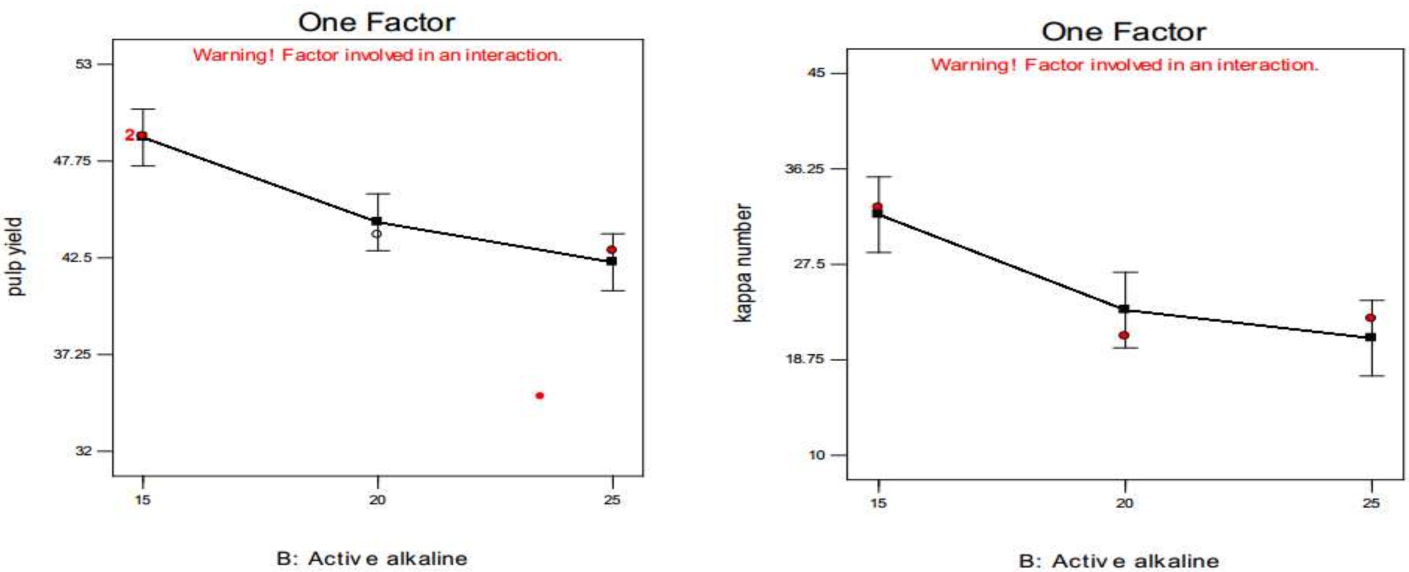


(b)

Figure 1: Plot of temperature versus ; (a) pulp yield at active alkali 20% and cooking time 180minutes and (b) Kappa number at active alkali 15% and cooking time 60 minutes.

Figure 2 (a) and (b) showed that the effects of active alkali concentration on pulp yield and kappa number (residual lignin), respectively. Increasing the amount of active alkali concentration decreased the pulp yield and kappa number significantly.

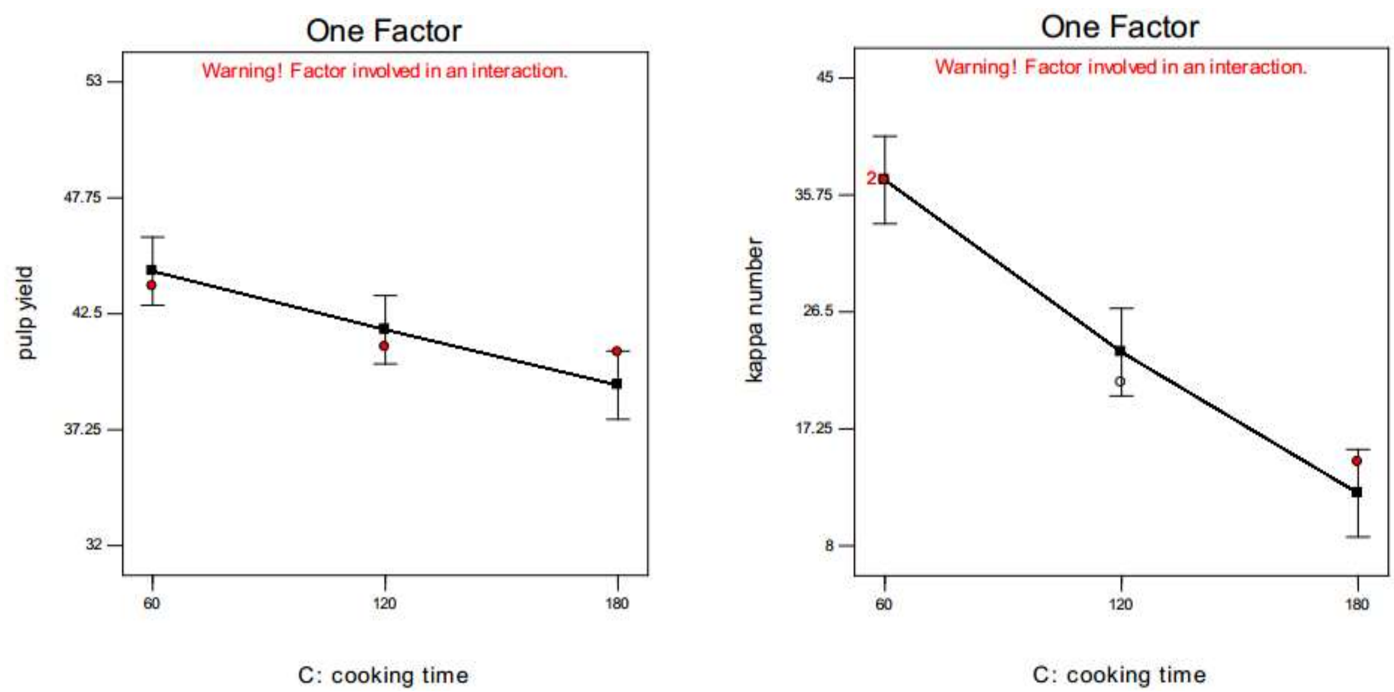
The concentration of active alkali was one of the important factors that affect the pulp yield, pulp quality and kappa number. The presence of AQ reduced the degradation effect of alkali while enhancing the dissolution of lignin.



(a) (b)

Figure 2: Plot of active alkali versus; (a) pulp yield at temperature of 200°C and cooking time of 60 minutes and (b) Kappa number at temperature of 150°C and cooking time 120minutes

Figure 3 (a) and (b) below illustrates the effect of pulping time on pulp yield and kappa number, respectively. When the pulping time increased the pulp yield decreased slightly while the kappa number decreased highly, this was because of pulping reaction got enough time for effective delignification and dissolution of alkali soluble hemicelluloses components.



(a) (b)

Figure 3: Plot of cooking time at active alkali 20% versus; (a) pulp yield at 200°C and (b) Kappa number at 150°C.

Generally, the longer the period of pulping, the higher the degree of delignification and the lower the pulp yield as a result of partial depolymerisation of cellulose chain as well as the removal of the hemicelluloses components. At a particular charge of effective alkali, pulping for 180 minutes at 200°C resulted in the lowest yield, while the highest yield was recorded for the pulp made for 60 minutes at 150°C.

ii) Interaction Effect between Process Variables

Interaction between Process Variables can affect the performance of delignification. But, as it already seen, the interaction effects of the process variables are significant. The interactions are between temperature, cooking time and active alkali concentration on pulp yield and kappa number.

Generally, an increase in reaction temperature was found to decrease the pulp yield and kappa number. This was due to similar explanation given in the previous section.

From the three interaction effects shown in the figures, there was a general decrease in the pulp yield and residual lignin due to an increase in the concentration of active alkali, temperature and cooking time at a uniform amount of 0.1% AQ and 4:1 liquor to solid ratio. It was noticed that nearly half of the original material was dissolved after 60 minutes of pulping and this attested the dissolving power of caustic soda as pulping liquor. The high dissolution of the initial material may also be attributed to the presence of highly soluble certain cell wall components such as fats, fatty acids, fatty alcohols, phenols, terpenes, resin acids and waxes. As observed from this study, increasing the alkali charge resulted in a decrease in the pulp yield as well as kappa number. The lignin content and pulp yield decreased slightly for the same period and markedly after high cooking time.

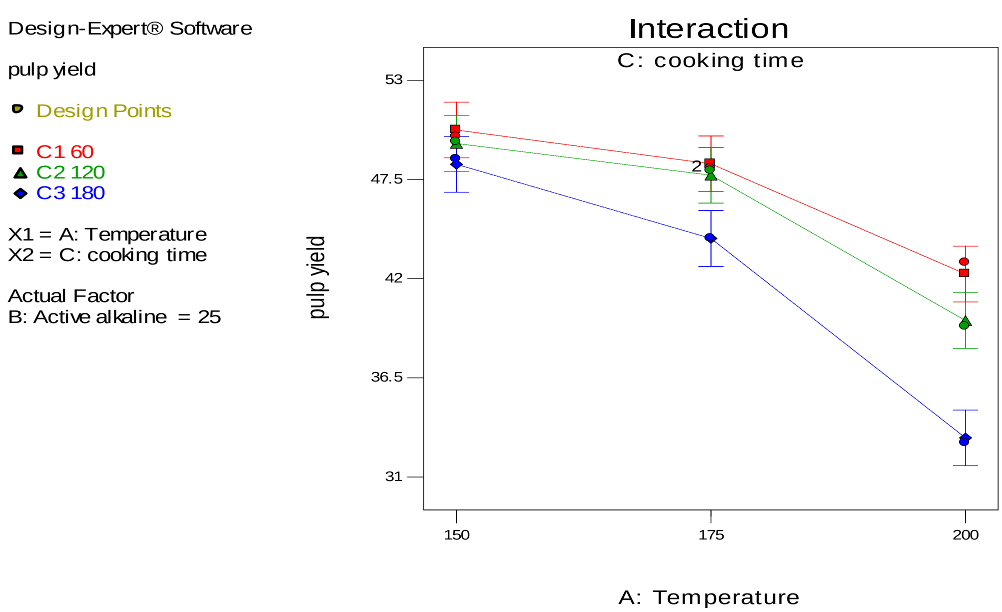


Figure 4: Interaction effect of temperature and cooking time versus pulp yield when active alkali concentration was 25%.

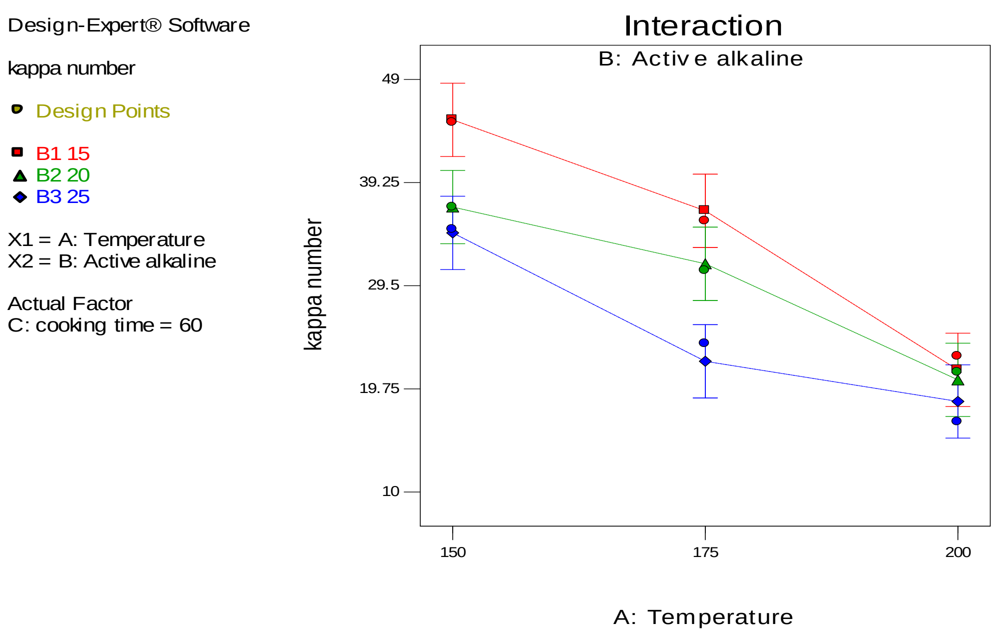


Figure 5: Interaction effect of temperature and active alkali concentration versus kappa number when cooking time was 60 minutes.

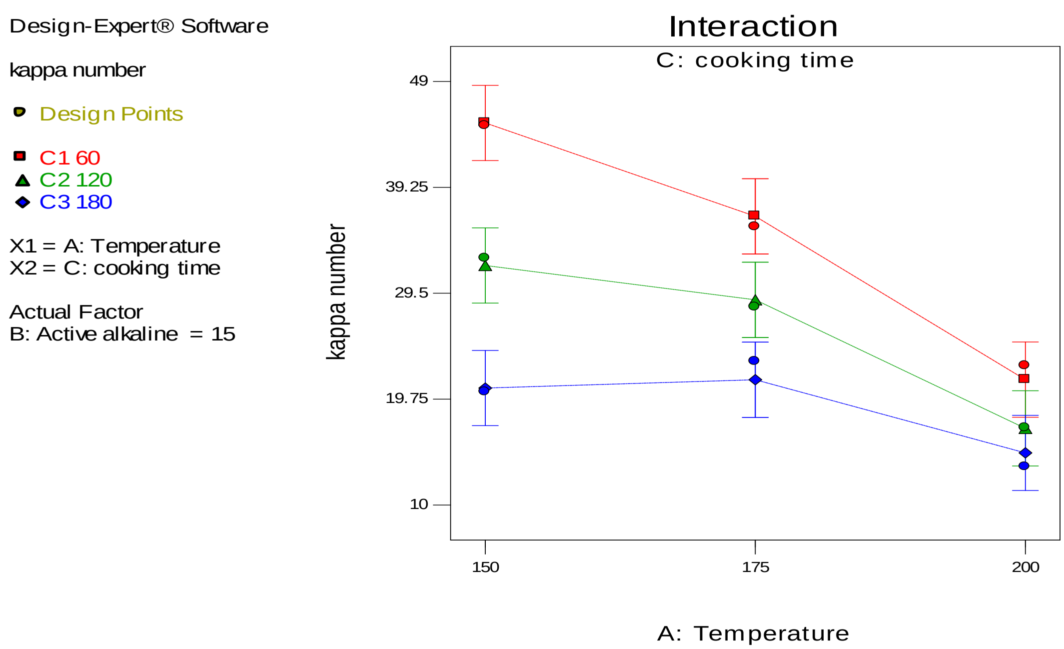


Figure 6: Interaction effect of temperature and cooking time versus kappa number when active alkali concentration was15%.

iii) *Optimization*

One of the objectives of the present study was to find the optimal process parameters for better pulp yield at minimum kappa number. The process variables such as cooking temperature, concentration of active alkali and pulping time were optimized. In optimizing the pulping process, cooking temperature, concentration of active alkali and pulping time were set of process parameters held to be "in range" while the pulp yield and kappa number were the two responses that need to be "maximized" for pulp yield and "minimized" for kappa number. Table 6 showed the summary of factors/responses and goals and the corresponding set of specific objectives that were optimize the process condition to have the highest responses. Numerical optimization was used to optimize any single or combination of goals.

Table 5: Constraints applied for optimization.

Factors/responses	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Temperature	in range	150	200	1	1	3
Active alkali	in range	15	25	1	1	3
cooking time	in range	60	180	1	1	3
pulp yield	Maximize	32.88	52.29	1	1	3
kappa number	Minimize	10.9	44.9	1	1	3

The model capable of predicting the maximum pulp yield and the minimum kappa number value showed that the optimum values of the process variables were temperature of 150°C, active alkali of 20% and cooking time of 180 minute. Under these conditions, the predicted pulp yield and kappa number were 52.24% and 12.16, respectively. Desirability function was used to identify the optimum levles of factors and to get maximum desirable responces. The optimized batch was selected with maximum combined desirability value i.e. 0.980 and the graph of the optimized solution for the pulping condition is shown in figure 7.

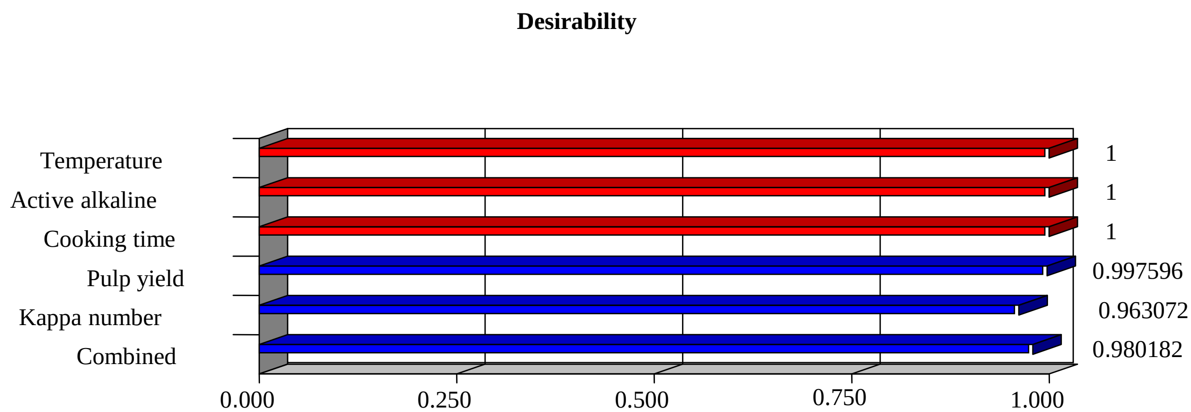


Figure 7: Desirability plot of optimization solution for the responses.

This was good in terms of cost efficiency for commercial scale, as only small amount of energy power and moderate chemicals were needed for the quality pulp production. This might be due to the low dissolvability of holocellulose parts and more lignin dissolved in higher alkaline percentage and higher temperature, and thus decreasing the pulping yield and kappa number.

IV. CONCLUSIONS AND RECOMMENDATIONS

This work was intended to study the influence of pulping parameters (active alkali (soda), cooking temperature, and time) on the pulp yield and kappa number of *O. abyssinica* pulp.

Variability of these operating conditions was the pre-dominant factors for the pulp yield and quality of *O. abyssinica* pulp.

There are different methods of pulping from *O. abyssinica* culms. In this paper, soda-anthraquinone pulping method was used. In this pulping method the liquor used for the pulping was NaOH-AQ solution. In this study based on the analysis of experimental results, it is realized that the individual factors and their interaction effects are significant model terms on pulp yield and kappa number. This shows that the capability of the design of the experimental analysis is successfully capturing these effects. The maximum pulp yield and minimum kappa number obtained was 52.24% and 12.16

respectively were achieved under the optimum conditions; at the active alkali of (20%), cooking temperature of 150°C and the cooking time of 180 min with high value of combined desirability, i.e. 0.98. This is good in terms of cost efficiency for commercial scale, as only small amount of energy power and moderate chemicals are needed for the quality pulp production.

The observed quantitative difference in the quantity and quality of the pulp was due to active alkali, cooking temperature and time variability. Thus, determination of the appropriate amount of the active alkali, optimal temperature and time for the recommended particle size needs to have a consideration to get the maximum amount of the required product.

It is advisable to use *O. abyssinica* for pulp production in Ethiopia since it is a fast growing plant, with short harvesting time and has good pulping characteristics which make bamboo a preferred raw material for pulping.

Finally, it can be concluded that *O. abyssinica* has a great potential for manufacturing of pulp in paper and paper products processing industry.

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