

Optimization of Palm Frond Pulping Using a Soda-Anthraquinone Process in a Circulating Digester: A Sustainable Approach

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Article History:

Submitted 9 November 2024
revision 14 December 2024
Accepted 17 December 2024
Online 24 December 2024

ABSTRACT: Oil palm fronds, typically discarded after pruning, have potential as a raw material due to their lignocellulosic content. This study optimizes the soda-anthraquinone pulping process using a circulating digester. It investigates the effects of cooking temperatures (140, 150, and 160°C), cooking times (120, 180, and 240 minutes), and NaOH concentrations (10%, 15%, and 20%) with 0.1% anthraquinone, employing Response Surface Methodology (RSM) based on Central Composite Design (CCD). Analysis with Design Expert 13 software revealed significant impacts on yield (19.01-31.00%), kappa number (9.24-15.69), and viscosity (2.91-34.45 cP). Optimal conditions were 140°C, 120 minutes, and 10% NaOH, yielding 30.57% pulp, kappa number of 13.87, and viscosity of 24.03 cP. This research underscores the environmental benefits of utilizing palm fronds, contributing to waste reduction and circular economy practices, and demonstrates the potential for industrial scalability, offering a sustainable alternative to traditional pulping methods.

Keywords: anthraquinone; circulated digester; oil palm fronds; pulp; soda process

1. Introduction

Pulp and paper products are essential for daily use, including writing, tissue, printing, and packaging. The pulp and paper industry serves nearly 5 billion people globally, making it one of the largest industries in the world (Bajpai, 2018). However, there is a growing deficit of raw wood as the main material for the paper industry which consumes over 10% of global wood production highlighting the need for sustainable practices and alternative raw materials (Małachowska et al., 2020).

Crude Palm Oil (CPO) derived from palm fruit is a versatile and widely used vegetable oil that is a crucial income source for millions of farmers significantly contributing to foreign exchange, generating employment, and advancing palm oil-based industries in Indonesia (Aisyah & Trihernawati, 2023; Ariyanto et al., 2024). In 2022, Indonesia produced 46.82 million tons of palm oil from a plantation area of 15.34 million hectares (Riau Province Central Bureau of Statistics, 2024). However, oil palm plantations generate significant waste including fronds from the pruning process during harvesting (Fahmi, 2015; Sunarno, 2014). The production of palm oil is proportional to the amount of waste produced including oil palm fronds (Safrizal et al., 2022).

The pruning process generates 40-50 fronds per tree annually with each frond weighing 4.5 kg (dry weight) (Andrian et al., 2018). Oil palm fronds are harvested throughout the tree's life cycle, yet they often end up as underutilized agricultural waste (Hongsriphan et al., 2022). Oil palm fronds contain 58% cellulose, 24% hemicellulose, 8% lignin, and 5% extractives. The soda pulping process is environmentally friendly and economical (Harsini & Susilowati, 2016; Miati et al., 2015). The Soda-anthraquinone pulping which replaced traditional soda pulping offers comparable yields to the Kraft process, improves delignification, reduces kappa numbers, and increases yield, brightness, and viscosity (Bajpai, 2021). Previous research on palm frond pulping using rotating cylindrical batch digesters has shown promising results (Chibudike et al., 2021; Nurul Husna et al., 2018; Wang et al., 2012). In Addition, Soda-AQ cooking is regarded as more eco-friendly because it does not generate harmful gases like hydrogen sulfide and sulfur dioxide making it ideal for processing non-wood materials (Yusnimar et al., 2022).

Despite these advancements, limited research on using oil palm fronds in an M/K circulation digester exists. Circulation digesters provide better liquor distribution and temperature control, potentially improving the soda-

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anthraquinone process's efficiency. This study investigates the combined effects of cooking temperature, NaOH concentration, and cooking time on oil palm fronds pulped with anthraquinone in an M/K circulation digester. Fresh palm fronds from the pruning process were used with the experimental design based on Central Composite Design. The anthraquinone soda pulping procedure involved cooking times of 120, 180, and 240 minutes, cooking temperatures of 140, 150, and 160°C, NaOH concentrations of 10%, 15%, and 20% (oven-dry basis), and 0.1% anthraquinone. This research provides insights into optimizing these parameters for industrial applications, emphasizing palm fronds' potential as a sustainable and economically viable raw material for pulp production.

2. Materials and Methods

2.1 Materials

Fresh oil palm fronds of the Tenera variety (Dura x Psifera cross) were collected from a local plantation. The fronds were first cleaned to remove dirt and dust, then cut into uniform chips with dimensions of 20 mm in length, 20 mm in width, and 4 mm in thickness. A screening process was performed to ensure uniform particle size. The chips were subjected to morphological analysis to determine their initial chemical composition. These pretreatment steps ensured consistent and reliable results in subsequent pulping experiments.

2.2 Chemicals

The primary reagents used in the study included NaOH at concentrations of 10%, 15%, and 20% as well as anthraquinone 0.1% which served as a delignification catalyst. Additional chemicals used for analysis included Dichloromethane, Methanol, KMnO_4 (0.1 N), H_2SO_4 (4N), KI (1N), $\text{Na}_2\text{S}_2\text{O}_3$ (0.1N), and Cupriethylenediamine (1M). A starch indicator was used in titration procedures. All chemicals were of analytical grade to ensure accuracy and reliability in the analysis.

2.3 Equipment and Apparatus

The primary apparatus for this study was the M/K circulation digester, which promotes uniform cooking by ensuring effective liquor circulation, which leads to consistent delignification and minimal fiber degradation. The M/K circulating digester is particularly noted for its adaptability to different feedstocks and its ability to enhance the pulping process while maintaining environmental standards. This technology has been implemented widely due to its reliability and alignment with modern demands for sustainable and cost-effective pulp production processes.

Auxiliary equipment included an oven for drying, a furnace for high-temperature treatments, a Soxhlet extractor for solvent extraction, a vacuum pump, and a disintegrator for fiber separation. Laboratory essentials such as magnetic stirrers, screening buckets, desiccators, scales, thermometers, and a range of glassware (beakers, burettes, separators, etc.) were also employed to support the experimental procedures.

2.4 Cooking Treatment

The cooking process was carried out in the M/K circulation digester. Palm frond chips were cooked using varying conditions based on the experimental design. The variables included cooking temperatures of 140°C, 150°C, and 160°C; cooking times of 120, 180, and 240 minutes; and NaOH concentrations of 10%, 15%, and 20%. A fixed concentration of 0.1% anthraquinone was added to enhance delignification. The liquor-to-solid ratio was maintained at 4:1 for all experiments. After cooking, the pulp was washed thoroughly with distilled water to remove residual chemicals and impurities.

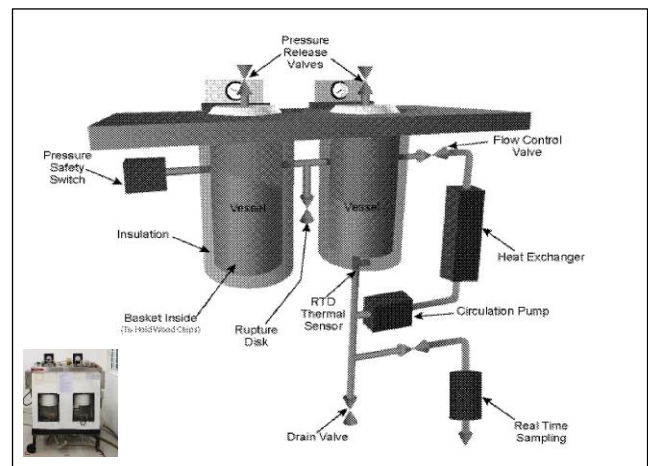


Figure 1. M/K circulating digester (Brochure *M/K Digester Systems Inc.*, 2021 & Diego, 2015)

2.5 Pulp Analysis

The cooked pulp was analyzed for yield, kappa number, and viscosity. Additionally, the chemical composition of the raw material and pulp was determined. Holocellulose content was measured using the Wise Method, while α -cellulose and lignin contents were analyzed based on ASTM D 1103 and SNI 8429 standards, respectively. Moisture and ash contents were measured following SNI 08-7070 and SNI 14-1031 protocols. The results were used to evaluate the impact of cooking conditions and optimize the pulping process.

This study used palm fronds from the Dura x Psifera cross variety (Tenera). The samples were first chipped to dimensions of 20 mm in length, 20 mm in width, and 4 mm in thickness, then screened to remove dirt and dust. Afterward, the samples underwent morphological analysis to determine the characteristics of the raw material. Following the analysis, the samples were cooked in an M/K circulation digester with a chip-to-NaOH ratio of 1:4, varying NaOH concentrations (10%, 15%, 20%), temperatures (140°C, 150°C, 160°C), cooking times (120, 180, 240 minutes), and the addition of 0.1% SAQ. All variables with three-level variation consisted of 17 processes, each of which followed the central composite design (Table 1) obtained from the Design Expert application version 13. The cooked samples were then disintegrated to separate the clumped pulp into individual fibers

The pulp was filtered, washed to remove residual chemicals, and dried. Subsequently, the pulp was analyzed for holocellulose (Wise Method), alpha-cellulose (ASTM D 1103), lignin content (SNI 8429), and moisture content (SNI 08-7070). Finally, the pulp was evaluated for yield (Bajpai, 2018), viscosity (SNI 0936-2008), and kappa number (SNI 0494-2008).

3. Results and Discussion

In this study, three independent variables were used, namely the comparison of cooking temperature, cooking time, and NaOH concentration with the resulting responses observed screened yield, kappa number, and viscosity. The data was then processed using Design Expert version 13 software to determine the optimum conditions.

Table 1 presents the response data obtained from the Central Composite Design (CCD) experiments focused on the pulping process of oil palm fronds. The table includes variations in cooking temperature, cooking time, NaOH concentration, and the resulting responses: screened yield, kappa number, and viscosity.

The response data from the RSM CCD illustrates the relationships between pulping parameters and the resulting pulp properties and guides future experiments and industrial applications (Reningtyas et al., 2023). The ability to identify optimal conditions for pulp production using RSM CCD underscores the value of these methodologies in enhancing both the efficiency and quality of industrial processes, particularly in sustainable raw material utilization such as oil palm fronds.

Table 1. Response data from the Central Composite Design (CCD) of Soda Anthraquinone Pulping

No.	Experimental variables			Responses		
	Cooking Temperature	Cooking time	NaOH Concentration	Screened Yield	Kappa Number	Viscosity
	A (°C)	B (minutes)	C (%)	Y ₁ (%)	Y ₂	Y ₃ (cP)
1	140	120	10	28.81	15.69	27.83
2	160	120	10	27.79	13.94	18.09
3	140	240	10	31.00	13.22	21.62
4	160	240	10	27.29	11.36	13.94
5	140	120	20	23.31	14.95	15.41
6	160	120	20	21.05	11.14	5.32
7	140	240	20	22.47	11.47	9.46
8	160	240	20	19.01	12.63	2.96
9	133	180	15	29.52	12.35	21.20
10	167	180	15	22.18	12.65	3.78
11	150	79	15	25.89	12.66	30.36
12	150	281	15	23.60	11.54	10.07
13	150	180	7	28.73	13.32	4.35
14	150	180	23	21.14	9.24	34.60
15	150	180	15	27.01	11.36	15.81
16	150	180	15	26.56	11.07	15.40
17	150	180	15	25.19	11.56	15.04

3.1. Statistical Analysis

The relationships between experimental variables (cooking temperature, cooking time, and NaOH concentration) and responses (screened yield, kappa number, and viscosity) were analyzed using statistical tools, including Analysis of Variance (ANOVA) and regression modeling. The statistical analysis aimed to identify significant factors, evaluate their interactions, and develop predictive models for optimizing the pulping process.

3.2. Yield Response

To determine the interaction of the screened yield response to the three experimental variables, cooking temperature, cooking time, and NaOH concentration, an analysis of the suggested model, namely the linear model, was carried out

using Analysis of Variance (ANOVA). To be declared to have a significant effect, the P-value must be less than 0.05 (Soeswanto et al., 2023). Table 2 shows that the model has a P-value <0.0001 or less than 0.05 so that the model can be declared significant. Specifically, the temperature and concentration factors are the factors that are stated to be the most significant to the model because they have a P-value of less than 0.05 namely 0.0003 and <0.0001 respectively. In addition, it is known that the F-value in the lack of fit row of 1.95 states that the inaccuracy is not significant to the pure error. The P-value of Lack of fit of 0.39 indicates that this model is not significant. A not significant lack of fit indicates that the model is suitable for use (Rengga et al., 2019).

Table 2. ANOVA result of screened yield response with a linear model

Source	Sum of squares	df	Mean square	F-value	P-value	Remark
Model	167.91	3	55.97	34.57	< 0.0001	Significant
A-Cooking temperature	38.04	1	38.04	23.5	0.0003	Significant
B-Cooking time	1.86	1	1.86	1.15	0.30	Not significant
C- NaOH Concentration	128	1	128	79.06	< 0.0001	Significant
Residual	21.05	13	1.62			
Lack of fit	19.25	11	1.75	1.95	0.39	Not significant
Pure error	1.8	2	0.9			
Cor total	188.95	16				

The cooking time factor is not significant because the P-value is 0.30 or greater than 0.05. This not significant prediction states that the cooking time factor does not significantly affect the screened yield response. A model

streamlining was carried out to obtain a better equation model by eliminating the cooking time factor. The results of the ANOVA analysis of the screened yield response after model streamlining can be seen in Table 3.

Table 3. ANOVA results of screened yield responses after model streamlining

Source	Sum of squares	df	Mean square	F-value	P-value	Remark
Model	166.04	2	83.02	50.74	< 0.0001	Significant
A- Cooking temperature	38.04	1	38.04	23.25	0.0003	Significant
C- NaOH concentration	128	1	128	78.23	< 0.0001	Significant
Residual	22.91	14	1.64			
Lack of fit	21.11	12	1.76	1.96	0.39	Not significant
Pure error	1.8	2	0.9			
Cor total	188.95	16				

Table 3 shows that the remaining two factors, the cooking temperature and NaOH concentration factors, have the most influence on the significance of the model. Cooking temperature and NaOH concentration show that the P-values are 0.0003 and < 0.0001 respectively. Both P-values are less than 0.05 so the model can be declared significant. In addition, it is known that the F-value in the lack of fit 1.96 states that the inaccuracy is not significant to the pure error. The P-value of Lack of fit of 0.39 indicates that this model is not significant and suitable for use. Equation (1) shows the linear model regression of these two factors on the yield response.

$$Y_1 = 59.54 - (0.17 \times A) - (0.61 \times C) \quad (1)$$

Remarks:

- Y_1 is screened yield (%)
- A is the cooking temperature (°C)
- C is NaOH concentration (%)

The effect of cooking operating conditions on the resulting screened yield value may not be seen if the analysis is only carried out using ANOVA data and the best-fit model. A 3D graph in Figure 2 can be used to explain the situation more quickly. However, a 3D graph can only show the effects of two factors and a response simultaneously. The highest yield was 31.00% in pulping with a NaOH concentration of 10%, a cooking temperature

of 140°C, and a time of 240 minutes. The lowest yield was in pulping with a NaOH concentration of 20%, a cooking temperature of 160°C, and a cooking time of 240 minutes. The lowest yield was noted at higher temperatures and concentrations, demonstrating a potential degradation of cellulose due to excessive alkaline conditions leading to the depolymerization and fragmentation of the cellulose molecules (Yusnimar et al., 2022).

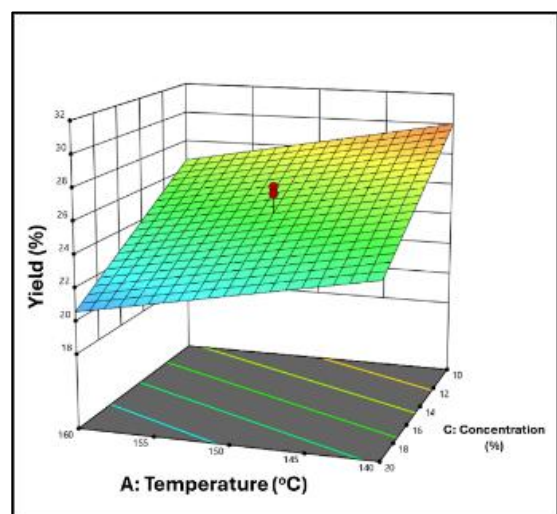


Figure 2. The 3D plot of the effect of the screened yield

3.3. Kappa Number Response

The kappa number, an indicator of lignin content and, thus, the quality of the pulp, varied inversely with both temperature and NaOH concentration. For example, the lowest kappa number (9.24) when processed at higher concentrations and intermediate temperatures, aligning with the expectation that reduced lignin content correlates with improved pulp quality. This trend underlines the importance of carefully balancing the pulping conditions to maintain a lower kappa number, essential for achieving higher quality pulp suitable for paper production (Putri et al., 2023).

To determine the interaction of the kappa number response to the three experimental variables, temperature, time, and NaOH concentration, an analysis of the

suggested model, namely the linear model, was carried out using Analysis of Variance (ANOVA). To be declared to have a significant effect, the P-value must be less than 0.05 (Soeswanto et al., 2023). Table 4 shows that the model has a P-value of 0.05 so the model can be declared significant. Specifically, NaOH concentration factors are the factors that are stated to be the only factors that are significant to the model because they have a P-value of 0.04 or less than 0.05. In addition, it is known that the F-value in the lack of fit 32.87 states that this model is significant to the pure error. The P-value of Lack of fit of 0.09 indicates that this model is significant. A significant lack of fit in a model suggests that it is not appropriate or adequate for the given data (Rahman et al., 2021).

Table 4. Results of ANOVA of kappa number response with linear model

Source	Sum of squares	df	Mean square	F-value	P-value	Remark
Model	16.95	3	5.65	3.3	0.05	Significant
A-Cooking temperature	2.44	1	2.44	1.43	0.25	Not significant
B-Cooking time	5.82	1	5.82	3.4	0.09	Not significant
C- NaOH Concentration	8.69	1	8.69	5.07	0.04	Significant
Residual	22.27	13	1.71			
Lack of fit	22.15	11	2.01	32.87	0.03	Significant
Pure error	0.12	2	0.06			
Cor total	39.22	16				

Since the model of the three variables is not significant, the model needs to be streamlined by eliminating the cooking temperature factor since it is the most not significant factor to the kappa number response. Table 5 shows that the remaining two factors, the cooking time and NaOH concentration factors are the most influence on the significance of the model. Cooking time and NaOH

concentration show that the P-values are 0.08 and 0.04 respectively. These factors help the P-value of the model become less than 0.05 so that the model can be declared significant. In addition, it is known that the F-value in the lack of fit 7.24 states that the inaccuracy is not significant to the pure error. The P-value of Lack of fit 0.13 also indicates that this model is not significant and suitable for use.

Table 5. Results of the kappa number response ANOVA with the Linear model after streamlining

Source	Sum of squares	df	Mean square	F-value	P-value	Remark
Model	14.51	2	7.25	4.35	0.03	Significant
B-Cooking time	5.82	1	5.82	3.49	0.08	Not significant
C- NaOH Concentration	8.69	1	8.69	5.21	0.04	Significant
Residual	23.35	14	1.67			
Lack of fit	22.82	12	1.9	7.24	0.13	Not significant
Pure error	0.53	2	0.26			
Cor total	37.86	16				

The linear model regression of these two factors on the kappa response is represented in Equation 2.

$$Y_2 = 16,77 - (0,01x B) - (0,16 x C) \quad (2)$$

Remarks: Y_2 is Kappa number, B is Cooking time (minutes), C is NaOH concentration (%).

The effect of cooking operating conditions on the resulting kappa number may not be seen if the analysis is

only carried out using ANOVA data and best-fit models. For a more straightforward explanation, a 3D graph can be used. However, a 3D graph can only show the effects of two factors and a response simultaneously. The interaction between cooking time and NaOH concentration further emphasized the importance of optimizing these parameters for effective delignification. The use of a 3D graph provides a clearer understanding of these trends, highlighting the combined effect of these factors on the kappa number.

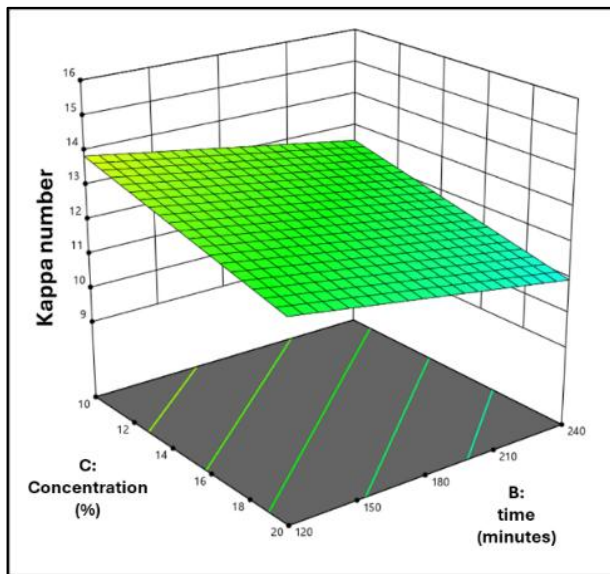


Figure 3. Relationship between cooking time and concentration on kappa number response

As seen in Figure 3, the kappa number was significantly influenced by both cooking time and NaOH concentration. The lowest kappa number of 9.24 was achieved at a cooking time of 180 minutes and a NaOH concentration of 23% at 150°C, indicating enhanced delignification with longer cooking times and higher NaOH concentrations. On the other hand, the highest kappa number of 15.69 was observed at a cooking time of 120 minutes and NaOH concentration of 10% at 140°C, showing that shorter cooking times and lower NaOH concentrations resulted in less effective lignin removal. These findings highlight the critical role of cooking time and NaOH concentration in controlling the kappa number, with the optimal balance required for efficient delignification. An excessively high kappa number can lead to problems such as yellow spots in the pulp, whereas lower values help protect the cellulose fibers from degradation (Putri et al., 2023).

3.4. Viscosity Response

Viscosity is an important measure of the molecular weight of cellulose (Mughtar et al., 2022). Higher viscosity typically indicates longer cellulose chains, which enhance the strength

and quality of the final product (Rostamitabar et al., 2021). The observed viscosity values, ranging from 2.96 to 34.60cP, emphasize the need to control the cooking conditions to optimize the molecular integrity of the cellulose while achieving the desired pulp characteristics.

Pulp viscosity indicates the average value of cellulose polymerization. This analysis shows a relative indication of the reduction in cellulose molecular weight caused by the pulping process. The average degree of cellulose polymerization (DP) is carried out by measuring the viscosity of a cellulose solution with a known concentration in Cupriethylenediamine (CED) solvent because CED can dissolve cellulose faster and has good chemical stability (Smook, 1992).

The magnitude of the influence of the three experimental variables, cooking temperature, cooking time, and NaOH concentration, on the viscosity response was analyzed using Analysis of Variance (ANOVA). To be declared to have a significant effect, the P-value must be less than 0.05 (Soeswanto et al., 2023). Table 6 shows that the model has a P-value of 0.12 so the model can be declared not significant. In addition, all three factors cooking temperature, cooking time, and NaOH concentration have P-values over 0.05 (0.06, 0.11, and 0.94 respectively). Moreover, the F-value in the lack of fit is 16.91 states that this model is not significant to the pure error. The P-value of Lack of fit of 0.06 indicates that this model is not significant. A not significant lack of fit indicates that the model is suitable for use (Rengga et al., 2019).

Since the model of the three variables is not significant, the model needs to be streamlined by eliminating the NaOH concentration factor since it is the most not significant factor in viscosity response. Table 7 shows that the remaining two factors, the cooking temperature and cooking time factors are the most influence on the significance of the model.

Cooking temperature and cooking time show that the P-values are 0.05 and 0.10 respectively. These factor help the P-value of the model become equal to 0.05 so that the model can be declared significant. In addition, it is known that the F-value in the lack of fit 15.51 states that the inaccuracy is not significant to the pure error. The P-value of Lack of fit 0.06 also indicates that this model is not significant and suitable for use.

Table 6. ANOVA results of viscosity response with a linear model

Source	Sum of squares	df	Mean square	F-value	P-value	Description
Model	499.16	3	166.39	2.38	0.12	Not significant
A-Cooking temperature	294.02	1	294.02	4.20	0.06	Not significant
B-Cooking time	204.78	1	204.78	2.92	0.11	Not significant
C- NaOH Concentration	0.3589	1	0.36	0.01	0.94	Not significant
Residual	910.55	13	70.04			
Lack of fit	900.86	11	81.90	16.91	0.06	not significant
Pure error	9.69	2	4.84			
Cor total	1409.71	16				

Table 7. ANOVA results of viscosity response with linear model after streamlining

Source	Sum of squares	df	Mean square	F-value	P-value	Remark
Model	498.81	2	249.40	3.83	0.05	Significant
A-Cooking temperature	294.02	1	294.02	4.52	0.05	Significant
B-Cooking time	204.78	1	204.78	3.15	0.10	Not significant
Residual	910.91	14	65.06			
Lack of Fit	901.22	12	75.10	15.51	0.06	Not significant
Pure Error	9.69	2	4.84			
Cor Total	1409.71	16				

The linear model regression for viscosity is represented in Equation 3.

$$Y_3 = 96.73 - (0.46 \times A) - (0.06 \times B) \quad (3)$$

Remarks: Y_3 is Viscosity (cP), A is the cooking temperature ($^{\circ}\text{C}$), B is Cooking time (minutes)

The effect of cooking operating conditions on the effect on viscosity may not be seen if the analysis is only carried out using ANOVA data and best-fit models. For a more straightforward explanation, a 3D graph can be used. However, a 3D graph can only show the effects of two factors and a response simultaneously. The interaction between cooking temperature and cooking time further emphasized the importance of optimizing these parameters for effective delignification. The use of a 3D graph provides a clearer understanding of these trends, highlighting the combined effect of these factors on the viscosity.

As seen in Figure 4, The viscosity of the pulp was primarily affected by cooking time and temperature. The lowest viscosity (2.96) occurred at the longest cooking time of 240 minutes and the highest cooking temperature of 160°C , suggesting that prolonged cooking at high temperatures leads to excessive breakdown of cellulose, resulting in lower viscosity. Conversely, the highest viscosity (34.6) was observed at a cooking time of 180 minutes and a cooking temperature of 150°C , indicating that a moderate cooking time and temperature maintain the integrity of the cellulose, yielding a higher viscosity. This is because the longer cellulose chain has stronger internal interactions and is easily tangled, making it thicker. As a result, it will take longer to pass through the viscometer capillary tube (Nurul Husna et al., 2018). These results

highlight the importance of optimizing both time and temperature to achieve desired pulp viscosity.

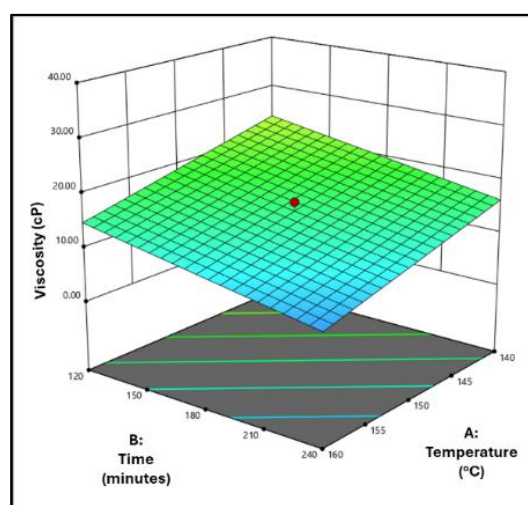


Figure 4. Relationship between temperature and time on viscosity response

3.5. Optimization of Pulping

The optimization of pulp yield from oil palm fronds was carried out using Design Expert 13 software. This optimization aimed to determine the best combination of pulping variables: temperature, cooking time, and NaOH concentration. A balance between these variables is essential to achieve the optimum yield. The desired pulp yield was set to its maximum value, while the kappa number and viscosity were targeted to remain within the range of experimental results. The details of these findings can be seen in Table 8.

Table 8. Desired response values for pulping process optimization

Variable	Target	Minimum	Maximum
Cooking temperature ($^{\circ}\text{C}$)	in range	140	160
Cooking time (minutes)	in range	120	240
NaOH concentration (%)	in range	10	20
Screened yield (%)	maximum	19.10	31.00
Kappa number	in range	9.24	15.69
Viscosity (cP)	in range	2.91	34.45

The optimal solution suggested by the RSM model occurred at a cooking temperature of 140°C, a cooking time of 120 minutes, and a NaOH concentration of 10%. This solution predicts a yield of 30.57%, a kappa number of 13.87, and a viscosity of 24.03cP. The results of this optimization process are presented in Table 9.

Table 9. Solution results of the optimization process

Variable	Value
Cooking temperature (°C)	140
Cooking time (minutes)	120
NaOH concentration (%)	10
Screened yield (%)	30.57
Kappa number	13.87
Viscosity	24.03
Desirability	0.99

The desirability function is a commonly employed method for optimizing multiple responses simultaneously (Thorisingam & Mustafa, 2022). The desirability value in this study is 0.99, which can be seen in Table 9 of the optimization process solution results. A desirability value closer to one indicates that each response value is precise and optimal in the solution results (Fitria, 2022).

3.6. Chemical Analysis of Oil Palm Fronds and Oil Palm Frond Pulp From Cooking With Optimum Condition

The chemical analysis of raw palm fronds and palm frond pulp, shown in Table 10, reveals significant changes in composition following the pulping process. Holocellulose, which represents the total polysaccharide content (cellulose and hemicellulose), increased from 72.02% in the raw material to 90.12% in the pulp, indicating the effectiveness of the pulping process in removing non-cellulosic materials like lignin and extractives. This increase is desirable as holocellulose contributes to fiber strength and paper quality.

The α -cellulose content, which refers to the purest form of cellulose, showed a remarkable increase from 38.90% in raw material to 78.67% in the pulp. This suggests that a significant portion of non-cellulosic polysaccharides and impurities was removed during the pulping process, leaving behind a higher cellulose concentration essential for high-

quality paper production. The ASTM D 1103 method confirmed this substantial purification of the cellulose content.



Figure 5. Comparison Chip from palm oil fronds and the pulp after cooking

Lignin content negatively impacts paper strength and quality. Using SNI 8429 as the test method, it was significantly reduced from 16.34% in raw material to 3.57% in pulp. This reduction in lignin is essential for improving the pulp's bleaching potential and overall paper quality. Lower lignin content is associated with easier pulp bleaching and better fiber bonding, contributing to more robust, durable paper products.

Additionally, the moisture content decreased slightly from 11.40% in raw palm fronds to 8.07% in the pulp, as measured by SNI 08-7070. This reduction is beneficial as lower moisture content is preferable for storage and further pulp processing. Ash content, measured at 4.28% in the raw material, was effectively reduced in the pulp, aligning with the desirable goal of minimizing non-fibrous inorganic content in the final product.

The extractive content in raw palm fronds was measured at 7.82% (SNI 14-7197), consisting of various non-structural components such as waxes, fats, and phenolic compounds. These substances are primarily removed during the pulping process, contributing to the overall increase in the holocellulose and α -cellulose content.

Table 10. Chemical analysis of palm fronds and palm frond pulp. *Modification of Wise method (Wise et al., 1946)

No.	Content	Unit	Palm Frond	Palm Frond Pulp	Test Method
1	holo cellulose	%	72.02	90.12	Wise Method*
2	Alfa cellulose	%	38.90	78.67	ASTM D 1103
3	Lignin	%	16.34	3.57	SNI 8429
4	Moisture	%	11.40	8.07	SNI 08-7070
5	Ash Content	%	4.28	-	SNI 14-1031
6	Extractive content	%	7.82	-	SNI 14-7197

4. Conclusions

This study demonstrates the potential of oil palm fronds as a sustainable raw material for high-quality pulp production using the Soda-anthraquinone process in a circulating digester, achieving optimal yield and quality under specific conditions. By reducing reliance on wood-based raw materials, this research contributes to environmental sustainability and highlights the economic advantages of utilizing agricultural waste. Beyond its application in the pulp and paper industry, the findings provide a foundation for exploring non-wood biomass processing methods for other agricultural residues. Future work could investigate scaling this process to industrial levels, integrating advanced delignification technologies, and exploring diverse biomass feedstocks for broader applications in sustainable material production.

Acknowledgements

The authors thank the University of Riau, the Institute of Technology of Bandung, and The Centre for Standardization and Service of Cellulose Industry for their support in the present work.

Statement

I acknowledge that I used ChatGPT and Grammarly to assist in improving the flow, coherence, and language quality of this document. These tools were used to refine the clarity and structure of my writing while ensuring the content remains my original work

CRedit authorship contribution statement

Suhendri: Writing – original draft, Data curation.

Evelyn: Resources, Writing – review & editing.

Tjandra Setiadi: Formal analysis, Supervision.

Hendro Risdianto: Conceptualization, Methodology, Validation.

Declaration of competing interest

The authors declare they have no conflict of interest or financial conflicts to disclose. This article does not contain studies with human or animal subjects performed by the authors.

Data availability

The data that has been used is confidential.

References

- Aisyah, S. A., & Trihernawati, T. (2023). Pembuatan Kertas Berbahan Baku Pelepah Kelapa Sawit (*Elaeis guineensis* Jacq.). *Jurnal Keteknik Pertanian*, 11(2), 165–174. <https://doi.org/10.19028/jtep.011.2.165-174>
- Andrian, I. R., Sylvia, M., & Indriana, L. (2018). Uji sifat fisik dan sifat kimia pulp dari limbah pelepah kelapa sawit (*Elaeis guineensis* Jacq.). *Jurnal Universitas Muhammadiyah Jakarta. Seminar Nasional Sains Dan Teknologi*.
- Ariyanto, E., Alqorni, M. F., & Mardwita, M. (2024). Efisiensi Thermal Alat Economizer pada Pre-Treatment Section dalam Pengolahan Crude Palm Oil (CPO). *Eksergi*, 21(1), 48. <https://doi.org/10.31315/e.v21i1.9974>
- Bajpai, P. (2018). *Biermann's Handbook of Pulp and Paper*. In *Journal of Chemical Information and Modeling* (3 ed.). Elsevier Inc.
- Bajpai, P. (2021). *Nonwood Plant Fiber for Pulp and Paper*. Elsevier Inc.
- Chibudike, H. O., Mgbachiuzo, E., Ndukwe, N. A., Obi, N. I., Adeyoju, O. A., & Chibudike, E. C. (2021). Fiber studies and pulping experiments on Empty Fruit Bunch (EFB) of oil palm (*Elaeis Guineensis*) using soda-AQ process. *International Journal of Scientific & Engineering Research*, 12(1).
- Fahmi, M. (2015). *Manajemen Penunasan Kelapa Sawit (Elaeis guineensis Jacq) di Sungai Bahaur Estate, PT Windu Nabatindo Abadi, Kalimantan Tengah [Skripsi]* (p. Institut Pertanian Bogor.). Institut Pertanian Bogor.
- Fitria, S. (2022). *Multi-Response Optimization of Skewing Carton Using Taguchi Method with Desirability Function Approach*.
- Harsini, T., & Susilowati. (2016). Pemanfaatan Kulit Buah Kakao dari Limbah Perkebunan Kakao sebagai Bahan Baku Pulp dengan Proses Organosolv. *Jurnal Ilmiah Teknik Lingkungan*, 2.
- Hongsriphan, N., Subsanga, J., Suebsai, P., Sitthipong, S., & Patanathabut, P. (2022). Use of oil palm frond waste to reinforce poly(lactic acid) based composites with the improvement of interfacial adhesion by alkali treatment. *Journal of Metals, Materials and Minerals*, 32(1), 134–143. <https://doi.org/10.55713/jmmm.v32i1.1244>
- Małachowska, E., Dubowik, M., Lipkiewicz, A., Przybysz, K., & Przybysz, P. (2020). Analysis of cellulose pulp characteristics and processing parameters for efficient paper production. *Sustainability (Switzerland)*, 12(17), 1–12. <https://doi.org/10.3390/su12177219>
- Miati, Jemi, R., & Cipta, E. (2015). Kajian pulping soda berbahan baku pelepah pisang (*Musa Paradisiaca*). *Jurnal Hutan Tropika*, X(2), 24–30.
- Nurul Husna, M. H., Ibrahim, M., & Mohd Yunus, N. Y. (2018). Response surface methodology on the effects of soda – anthraquinone pulping conditions on oil palm empty fruit bunches pulp properties. *Malaysian Journal of Analytical Sciences*, 22(2), 203–218.
- Putri, R. W., Rahmatullah, R., & Siagian, F. (2023). The Effect of H-Factor on Kappa Number and Viscosity in Continuous Digester. *CHEESA: Chemical Engineering Research Articles*, 6(1), 26. <https://doi.org/10.25273/cheesa.v6i1.15681.26-33>
- Rahman, M. L., Akter, E., Hossain, Z., Sultana, A., & Das,

- K. (2021). Modeling gestational age and lack of fit. *Journal of South Asian Federation of Obstetrics and Gynaecology*, 13(3), 81–85.
<https://doi.org/10.5005/jp-journals-10006-1902>
- Rengga, W. D. P., Hartanto, D., Wibowo, B. T., & Setiawan, M. (2019). Ekstraksi Minyak Kulit Biji Mete dari Limbah Kulit Biji Mete (*Anacardium occidentale*) dengan Bantuan Ultrasonik. *Jurnal Eksergi*, 16(1), 1–6.
- Reiningtyas, R., Rahayuningsih, E., Kusumastuti, Y., & Kartini, I. (2023). Optimasi Aplikasi Pelapisan Nanopartikel Seng Oksida pada Kain Katun-Indigo dengan Response Surface Methodology. *Eksergi*, 20(2), 100. <https://doi.org/10.31315/e.v20i2.9877>
- Riau Province Central Bureau of Statistics. (2024). *Statistik Kelapa Sawit Provinsi Riau 2022*. BPS Provinsi Riau.
- Safrizal, D., Herry, M., Rahmadhan, N. C., & Satriananda. (2022). Pembuatan kertas komposit dari tandan kosong kelapa sawit (*Elaeis guineensis*) dan limbah kertas HVS. *Jurnal Reaksi (Journal of Science and Technology) Jurusan Teknik Kimia Politeknik Negeri Lhokseumawe*, 20(1), 1–8.
- Smook, G. A. (1992). *Handbook for pulp & paper technologist* (2nd ed.). Angus Wilde Publications.
- Soeswanto, B., Yusuf, Y., Sihombing, R. P., Suryadi, J., Luviana, A., Asyari, R. A. A., & Adhitasari, A. (2023). Signifikansi Kadar Kafein pada Kopi Kerinci Robusta dalam Berbagai Interval Waktu. *Eksergi*, 20(3), 168. <https://doi.org/10.31315/e.v20i3.10353>
- Sunarno, S. (2014). Sintesis Katalis Ni/ZSM-5 untuk Pirolisis Cangkang Sawit Menjadi Bio-Oil. *Eksergi*, 11(1), 23. <https://doi.org/10.31315/e.v11i1.326>
- Thorisingam, Y., & Mustafa, M. S. (2022). Augmented Approach to Desirability Function Based on Principal Component Analysis. *International Journal of Academic Research in Progressive Education and Development*, 11(2), 526–533.
<https://doi.org/10.6007/ijarped/v11-i2/13270>
- Wang, X., Hu, J., Liang, Y., & Zeng, J. (2012). TCF bleaching character of soda-anthraquinone pulp from oil palm frond. *BioResources*, 7(1), 275–282.
- Wise et al. (1946), L. E., Murphy, M., D'Addieco, A. A. "Chlorite holocellulose, its fractionation and bearing on summativewood analysis and studies on the hemicelluloses", *Pap. Trade J.*, 122(2):35-43,
- Yusnimar, Evelyn, Aman, A., Chairul, Rahmadahana, S., & Amri, A. (2022). Manufacturing of high brightness dissolving pulp from sansevieria-trifasciata fiber by effective sequences processes. *Communications in Science and Technology*, 7(1), 45–49.
<https://doi.org/10.21924/cst.7.1.2022.681>