

Parameter Optimization for Pulp from Rice Straw and used Paper with a Box-Behnken Design

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Abstract

In pulping process, cooking time, ethanol concentration and temperature were reported to influence pulp yield, cellulose content and acid-insoluble lignin content. In this study, the effects of the three parameters on the optimum pulp yield, cellulose content and acid-insoluble lignin content were further examined with the Box-Behnken design. This optimization aimed to discover the optimal condition for a pulping process using straw and recycled paper with ethanol concentrations of 30, 45 and 60%. As paper material, the biomass was processed with an organosolv process using ethanol as the cooking liquid to benefit almost all constituent components to obtain pulp. A quadratic model was used to assess the obtained research data. The results showed that the optimal condition comprised a cooking time of 100 min, an ethanol concentration of 60% and a temperature of 90 °C. The condition resulted in 57.59% cellulose content, 91.46% pulp yield and 41.28% acid-insoluble lignin content.

Keywords: ethanol, mixed fiber paper, organosolv, straw, used paper

1. Introduction

In the pulp industry, alternative materials such as bagasse, bamboo and cereal straw are used to guarantee the sustainability of pulping. The principal fibers of these raw materials are renewable making them a good addition to a sustainable pulp and paper industry. In 2016, the production of fiber pulp for the world's paper and board was around 6,317 metric tons per year with 2,465 metric tons, or 39.02% coming from straw pulp (Food and Agriculture Organization of the United Nations, 2017). Rice straw is a renewable energy source and an agricultural waste that can be used as a pulping material. Depending on the location and paddy plant varieties, rice straw production can achieve 10-15 tons per hectare for each harvest. In Indonesia, one of the largest

rice producers in the world, the recorded rice production was approximately equivalent to 59.2 million tons in 2018 (Statistic Indonesia, 2018). The ratio of paddy and straw of the variety used was 2:3; thus, the rice straw produced in 2018 was 88.8 tons. As biomass waste, rice straw is mainly used as a type of feedstuff, fuel fertilizer and industrial raw material. As a raw material for pulp manufacturing, rice straw is relatively low-cost and easy to handle (Liu *et al.*, 2018). Biomass waste has high potential because it does not compete with food. Using rice straw can result in reduced deforestation, wood utilization, increased cellulose imports and eco-friendly paper production. Comparing other agricultural residues with rice straw revealed that rice straw is an effective alternative cellulose-containing material for producing pulp (Rodriguez *et al.*, 2008). The process of changing lignocellulose compounds (degradation) in rice straw can be carried out chemically via the acid hydrolysis of pure enzyme (Kumar and Sharma, 2017). However, research on the benefits of rice straw for pulp materials is still limited.

There are many factors influencing the feasibility of using raw materials in papermaking like their composition. The chemical structure of rice straw varied from season to season; thus, it depends on the class (El-Sayed *et al.*, 2020). To determine cellulose, lignin and halo cellulose, Shao *et al.* (2017) pulped straw with soda. It was then processed with soda-anthraquinone for small industrial production because of the lower associated investment costs, the high value of the residue and high throughput, as well as the short cooking time to turn the straw into pulp.

The pulp-making process is affected by pH, chemical composition and consistency (Faisal and Baehaqi, 2017). The concentration, temperature and cooking time have a positive influence on the Kappa number and pulp yield (Akpakpan *et al.*, 2011). For pulping wheat straw using 2% sodium hydroxide and glycerol as a delignifying solvent, a relatively short cooking time (approximately 30 min) is suggested with a delignification temperature of approximately 195-205 °C (Saberikhah *et al.*, 2011).

With a cooking liquor comprising 10.5% sodium hydroxide and 3.5% sodium sulfite, the wheat straw pulp yield was 55% (Mah-e-Kamil *et al.*, 2009). Based on the investigation of paper pulp made of soda-anthraquinone, the best characteristics were provided by 15% w/w sodium hydroxide and 1% w/w anthraquinone (Rodriguez *et al.*, 2008). The optimum condition for pulping bagasse was achieved at the 10% sodium hydroxide, cooking temperature of 80 °C and cooking time of 90 min (Suseno *et al.*, 2017). Soda pulping of

Opuntia ficus-indica waste was carried out at cooking temperatures of 160 and 175 °C with 20 and 28% sodium hydroxide, 60- and 120-min cooking period, and a ratio of fiber to liquor 1:9 (Colin-Chavez *et al.*, 2020). Most of the ash and 76% of the lignin were separated from rice straw in the KOH pre-extraction step (Popy *et al.*, 2020). Alkaline extraction of the pulp delignified with the acetic acid/formic acid ratio 9:1 reduced cellulose, hemicellulose, lignin contents and pulp yield (Hidayati *et al.* 2019) because silica was retained on the fiber. An analysis of the chemical components of rice straw showed that it had cellulose, hemicellulose and lignin contents of 32.1, 24 and 18%, respectively (Anwar *et al.*, 2014). Meanwhile, the paper used had a cellulose content of 85 to 99% and a lignin content of 0 to 5% without hemicellulose (Anindyawati, 2010).

Different material compositions can influence tear resistance and tensile strength. One way to handle scarcity and the high cost of paper materials from virgin pulp is to reuse paper and straw as paper materials. The recycled fiber is less hydrophilic compared with virgin fiber. Increasing short fiber and ash content and decreasing long fiber reduce the mechanical strength of the recycled paper, and additives are necessary to achieve the mechanical strength required. The recycled paper, resulting from secondary cellulose fibers, meets the tensile strength, density and burst strength as required by the Technical Association of the Pulp and Paper Industry (TAPPI) standards (Ahmed and Mohammed, 2014). The chemical treatment has various effects on the fiber characteristics but it does not significantly influence the strength.

The strength of the pulp sheet is related to pulp kinking and bleaching. Enzyme treatments and pressurized peroxide bleaching can improve the pulp brightness and stability of fibers in the recycled paper to obtain qualified pulp. Used paper waste can create an environmental problem because of the greenhouse gasses released in landfill sites. Therefore, in this research, the pulp was made through organosolv process by combining used paper and rice straw. The organosolv process was selected because it is considered an environmentally friendly pulping process used on more easily degraded materials. This study employed a chemical method using ethanol solution to separate the fibers from their mixture.

So far, no research has been conducted using rice straw and paper pulp with optimization of the process condition although the process response is influenced by independent variables. Thus, determining the relationship between independent variables and response and producing the best response

was carried out using the response surface method (RSM). The present work emphasized the optimization by RSM with Box-Behnken design (BBD) to investigate the effect of the organosolv process condition of rice straw and used paper pulp. To this end, the study aimed to determine the optimum condition of pulp synthesis from both materials with BBD.

2. Methodology

2.1 Materials

The materials required for pulp-making were hydrochloric acid (38%) (Merck, Germany), phloroglucinol (99.0%) (Merck, Germany), ethanol (99.9%) (Merck, Germany), sodium hydroxide (98%) (Merck, Germany) and sodium hypochlorite (6%) (Magnificent Cosmo Cosmoceuticals, India). The post-consumer recycled paper (A4 office paper) was collected from Universitas Syiah Kuala and the rice straw was obtained from Blang Jaro field, Indrapuri District, Aceh Besar, Indonesia.

2.2 Content Analyses

The dry weight analyses were performed on an oven (Mettler UL40, Netherlands) until a constant weight was obtained according to Standar Nasional Indonesia (SNI) 1971:2011 (Badan Standardisasi Nasional [BSN], 2011). The rice straw was analyzed for water and ash contents according to SNI 08-7070-2005 (BSN, 2005) and SNI ISO 2144:2010 (BSN, 2010), respectively. Ash content was analyzed on a furnace (Nabertherm, United States) for 30 min at 900 °C. The hemicellulose content was determined using a boiling flask by placing 1 g of the rice straw inside. Hemicellulose and cellulose contents and acid-insoluble lignin were analyzed based on SNI 8430:2017 (BSN, 2017b) and SNI 8429: 2017 (BSN, 2017a), respectively. The procedure was similar to the procedure of TAPPI (1999, 2006) with two samples of 1.5 g + 0.1 g for cellulose and 2.0 g + 0.1 g for lignin weighed to the nearest 0.0001 g. The pulp was weighed on a digital balance (PA214, Ohaus Pioneer, United States) to determine the pulp yield as a percentage of dry material. Although Kappa number or delignification selectivity is more appropriate in evaluating pulping parameters, they were not analyzed because of their instabilities.

2.3 Synthesis of Pulp

The pulp was made of rice straw and used paper cut around 1 x 1 cm to ease the mixing with water and repulping process. The rice straw was placed in the oven at 70 to 80 °C until the material mass was constant. Before becoming fibrous, the straw was ground to optimize the acid-insoluble lignin degradation to influence the pulp bleaching. Higher acid-insoluble lignin resulted in harder bleaching and worse pulp. The mixture was immersed in NaOCl solution to make the color stable without turning yellow or losing its strength and brightness during storage. These hypochlorite ions (nucleophile) experienced an addition reaction in positive sites of lignin and colorful materials with double bond compounds so that they turned pale in color. The materials were oxidized into single bond compounds to result in brighter fibers. The pulping stages in the synthesis of pulp were as follows.

2.3.1 Cooking

The A4 office paper (56.3 g) was pulped to homogenize it in a mixture with rice straw (68.8 g). The pulping process was performed in a hydropulper (Pnshar, China) of 3.7-L effective volume fitted with a condenser. This organosolv process used organic matter in the form of ethanol as a cooking solution. Sodium hydroxide solution 5% w/w was used as a catalyst in an ethanol mixture having volume fractions of 30, 45 and 60% v/v. The liquor-to-solid (o.d) ratio was 10. Conditions for impregnation were 20, 43 and 55 min to raise its temperature to 90, 125 and 160 °C, respectively. The cooking process was carried out in at temperatures of 90, 125 and 160 °C by electrical heating. The cooking time was 80, 100 and 120 min with a maximum pressure of 1.1 MPa.

Pulping time was measured from the temperatures. Therefore, the acid-insoluble pulping time was 100-175 min. At the end of the cooking, its pressure was reduced by venting before opening the hydropulper. The result was then filtered to separate the remaining cooking result in the form of black liquor.

2.3.2 Washing and Screening

The pulp was washed, immersed in NaOCl 5%, washed again and dried to ease the weighing. After cooling, the mixture was blended (Philips, Indonesia) to form a pulp. After 10 min, the pulp was filtered, and the pulp yield, cellulose

and lignin contents were analyzed. To make paper, the remaining fiber was rinsed with distilled water (2 L) to separate it from the solution. The pulp was distributed with water in the hydropulper. After screening, the pulp was squeezed and dried at 22 °C in a drying cabinet for two days to remove excess water, and placed in a bag for determination of its weight.

2.4 Optimization Process

Optimization was performed with the Design-Expert 10.0.3 software with the BBD. It was successfully implemented in various optimization studies like processing conditions for the development of tomato foam (Balasubramanian *et al.*, 2012), fermentation conditions for rapid and efficient accumulation of macrolactin A (He *et al.*, 2013) and cellulose extraction from an olive tree (Landolsi, 2021). BBD optimizes the process because it presents more acceptable research design that was not carried out in a research. It results in some options to compute the maximum, minimum, or target value range for the output of the optimization process. The experimental design was processed with BBD because it facilitated the achievement of an optimal condition (Lubis, 2019) by analyzing several responses and variables. RSM with BBD was used as a method based on optimizing the process parameters of pulp synthesis to predict the effect of dependent variables on response variables.

The dependent variables were cooking time (80 to 120 min), ethanol concentration (30 to 60%) and cooking temperature (90 to 160 °C). In BBD, the cooking time was A variable (min), the ethanol concentration was B variable (%) and the temperature was C variable (°C). This study limited the BBD matrix to three response variables, namely pulp yield, cellulose and acid-insoluble lignin contents. The predicted response was obtained with a statistical model from BBD in the Design-Expert software. Each response variable was then analyzed one by one using analysis of variance (ANOVA).

ANOVA is a technique for analyzing the statistical association of the second order-polynomial model and the effects of individual correlation of the responses. The ANOVA was used to determine the significant interaction between the factor variables and the response variables (Table 3). The ANOVA distinguished the average value of data by comparing their variances to find responses among variables. This research used ANOVA for assessing the average variance among groups compared with that within groups. The ANOVA model used can be selected as suggested by the software, namely the model with the highest level and resulted in a significant ANOVA value.

Models that delivered significance on ANOVA and no significance on lack of fit were chosen to analyze the variables. Coefficients of the model were statistically analyzed by ANOVA on a 95% significance level. The software also provided a facility for the normal plot of residual to indicate whether the difference between actual responses with the predicted response value follows the straight line. Data points getting closer to the normal line showed the data that were normally distributed, which meant actual results were close to predicted pulp yield, cellulose and acid-insoluble lignin contents.

3. Results and Discussion

3.1 Dry Mass, Water and Organic Matter Contents

The content analysis indicated that the fresh rice straw contained 23.9% dry mass, whereas dry rice straw contained 90% dry mass, or 10% water content. Dry rice straw contained components of fiber fraction, namely 32.59% cellulose, 27.27% hemicellulose, 20% lignin and the rest was 20% ash. The content of this untreated rice straw and rice straw-paper pulp in the research is presented in Table 1. The degradation of lignin caused the cellulose that was previously bound by lignin to be released again; thus, the pulp obtained had a higher content of cellulose. As a result, the cellulose and acid-insoluble lignin contents in rice straw-paper pulp were still higher than in rice straw. This was probably because the content analysis was carried out immediately after filtering and cellulose and lignin were added from the use of recycled paper.

Table 1. Organic matter content of rice straw

Component	Content (%)	
	Rice straw	Straw/paper pulp
Cellulose	32.59	51.07-61.56
Hemicellulose	27.27	-
Lignin	20.00	31.31-48.56
Ash	20.00	19.53

3.2 Analysis with BBD

Table 2 shows the highest value of pulp cellulose yield with ethanol concentration of 60%.

Table 2. BBD matrix for experimental design of pulping

Run	Cooking time (min)	Ethanol concentration (%)	Cooking temperature (°C)	Pulp yield (%)	Cellulose of pulp (%)	Acid-insoluble lignin of pulp (%)
1	80	45	160	91.94	53.71	44.60
2	120	45	160	79.02	60.56	31.32
3	100	45	125	89.94	59.08	38.32
4	100	45	125	89.94	59.08	38.32
5	120	30	125	82.41	59.35	32.49
6	80	30	125	93.79	51.07	48.34
7	80	45	90	96.24	51.07	48.56
8	100	45	125	89.94	59.08	38.32
9	100	60	160	87.46	58.55	36.97
10	100	30	160	90.09	56.49	40.34
11	100	45	125	89.94	59.08	38.32
12	100	60	90	91.20	57.42	41.16
13	120	45	90	94.04	52.94	46.14
14	100	45	125	89.94	59.08	38.32
15	100	30	90	95.94	54.66	44.60
16	80	60	125	89.00	60.41	35.96
17	120	60	125	79.55	61.56	31.31

Table 3 predicted the yield, cellulose and acid-insoluble lignin contents to approach the model of more than 90%. The coefficient of determination, R^2 , represented the fitness of pulp yield and cellulose and acid-insoluble lignin contents and their relationship with the mathematical model.

This coefficient value showed that the predicted result was close to the actual result (Alexander *et al.*, 2015) and significant with a second-order polynomial model. A high coefficient showed a close relationship between the data because of the conformity of the experimental data and predicted data. The probability P-values less than 0.05 showed significant inter-variable interaction, whereas P-values more than 0.1 showed insignificant interaction. Any P-value smaller than 0.05 indicated that the model did not fit the actual data (Hidalgo *et al.*, 2018) of the yield, cellulose and acid-insoluble lignin contents.

Table 3. ANOVA of quadratic model for response variables

Source	Yield of pulp	P-value		Remark
		Cellulose of pulp	Acid-insoluble lignin of pulp	
Model	0.0004	0.0028	0.0007	Significant
A – Cooking time	< 0.0001	0.0019	0.0002	
B – Ethanol concentration	0.0097	0.0034	0.0048	
C – Temperature	0.0003	0.0098	0.0010	
AB	0.5412	0.0317	0.0158	
AC	0.0092	0.1038	0.0180	
BC	0.5086	0.8006	0.9850	
A ²	0.0163	0.0435	0.7419	
B ²	0.0904	0.3751	0.1073	
C ²	0.082	0.0028	0.0022	
R ²	0.9602	0.9300	0.9533	
Adjusted R ²	0.9090	0.8400	0.8932	

3.3 Effect of Time and Ethanol on Pulp Yield

Yield reflected the amount of pulp from the material. It was influenced by alpha-cellulose. The higher yield showed a more effective pulping process (Rahman *et al.*, 2017). Variables used in this pulping process were cooking time and ethanol content to obtain the interaction of one variable with the other. The conditions of the pulping process, such as temperature, were shown in Table 2. The highest pulp yield was 96.24% obtained at the 80-min cooking time, 45% ethanol concentration and 90 °C. The lowest pulp yield was 79.02% at the 120-min cooking time, 45% ethanol concentration and 160 °C. An optimum condition with the 92.39% yield was at the 100-min cooking time, 60% ethanol concentration and 90 °C. The yield in Figure 1 was higher than the yield of 61.77% in a research with a cooking time of 150 min (Navaee-Ardeh *et al.*, 2005). This predicted yield (92.39%) was relatively higher than the actual yield (91.20% in Table 2) probably because the effect of cooking time on the pulp yield was relatively more dominant than the actual cooking time. The predicted yield from the Design-Expert 10.0.3 software utilized Equation 1, which is a polynomial used to predict pulp yield resulting from the BBD.

$$\text{Yield} = 59.42 + 1.34A + 0.16B - 0.31C + 0.0016AB - 0.0038AC + 0.001BC - 0.006A^2 - 0.006B^2 - 0.002C^2 \quad (1)$$

3.4 Effect of Ethanol on Cellulose Content

Cellulose could increase tensile strength but the higher temperature could degrade cellulose and polymerize dissolved lignin. Figure 1b shows the effect of the cooking time and ethanol on the cellulose content at an optimum temperature (90 °C). The present work yielded 51.07 to 60.43% cellulose. Over time, the highest content was obtained. In the first 80 min, the content was 48.50%. At 120 min, it became 54.11%. Longer time and greater ethanol enhanced the content. At 120 min and 60% ethanol, the content was 54.99 to 56.51%. This content was lower than 60.57%, which was the content of cellulose pulp before treatment with dilute acids in another study (Bouri and Amraini, 2010).

However, this content decreased at a temperature of 90 to 120 °C because the acid-insoluble lignin content increased. This process used ethanol to control the degradation towards greater lignin. Ethanol was utilized to dissolve the lignin fiber and other products from the rice straw and used paper. Treatment in ethanol could improve the dissolution of lignin, and thus avoid cellulose dissolution. The conversion was endothermic and affected by heat depending on the temperature. Reduction of cellulose could improve bleaching ability. Equation 2 predicted the cellulose content as a function of the actual values of A, B, and C, and their interactions.

$$\text{Cellulose} = -42.23 + 0.96A + 0.5B + 0.48C - 0.006AB + 0.002AC - 0.0003BC - 0.004A^2 + 0.003B^2 - 0.002C^2 \quad (2)$$

3.5 Effect of Cooking Time and Ethanol Content on Acid-insoluble Lignin Content

Increasing the time to achieve the cooking temperature caused the cooking time to degrade the lignin to be shorter. Therefore, an important factor in this process was the cooking time. Increasing cooking time caused the acid-insoluble lignin content to increase because ethanol had enough time to break more lignin and create spaces on the raw materials (Figure 1c). However, longer cooking could cause the acid-insoluble lignin content in the pulp to decrease again.

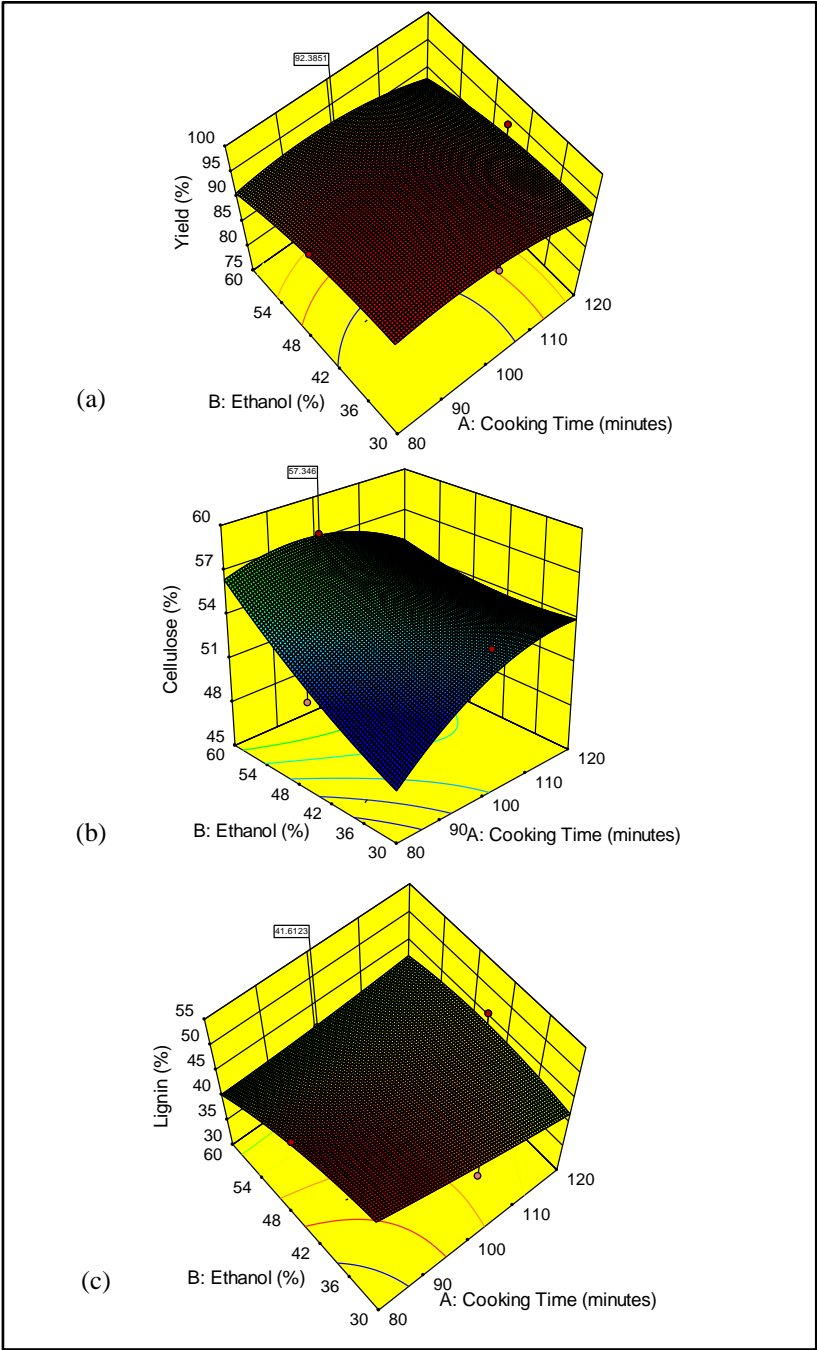


Figure 1. The effect of cooking time on ethanol content

With the decrease in the amount of ethanol during cooking, the acid-insoluble lignin that was removed from the raw pulp will re-unite with the raw pulp and it was hard to separate them again. In turn, the lignin content might decrease (Rayhan *et al.*, 2020). The figure was obtained from the optimization of the variables in Table 2 by Design-Expert software using the BBD. The lignin content was plotted based on the interaction of cooking time and ethanol content as independent variables whereas the pulp yield, cellulose and acid-insoluble lignin content as the dependent variables. Optimization of these dependent variables also provided better estimation for the regression coefficients by using a quadratic interaction term.

Based on the study, the acid-insoluble lignin content in the optimization was changed over the ranges of ethanol 30-60%, the cooking time of 80-120 min, and cooking temperatures of 90-160 °C.

Based on the optimization result, it was obvious that an optimum condition at 100 min, 60% ethanol and 90 °C with acid-insoluble lignin of 41.61% supported the effectiveness of the pulping process. The ethanol effect on the acid-insoluble lignin content was significant (Table 3).

More ethanol resulted in more acid-insoluble lignin. Ethanol separated lignin from the fibers (Hochegger *et al.*, 2019) at a certain temperature and increased hydroxyl ions in the pulp. Ethanol was the best solvent to improve the selectivity of the lignin reaction, make the pulp brighter and shorten the cooking time. In addition, ethanol caused better delignification, which can be increased because of the increasing content of ethanol and insoluble lignin. The ethanol content of 5 to 70% was often used as the solvent. At ethanol of 30 to 45%, the delignification strength increased (Equation 3). The ethanol of more than 45% gave less acid-insoluble lignin. This process was less effective at a higher content (60%). The unbleached pulp of rice straw was still in cream color; thus, it was suitable for manila paper.

$$\begin{aligned} \text{Lignin} = & 119.01 - 0.31A - 0.47B - 0.54C + 0.01AB - 0.004AC \\ & + 0.00003BC + 0.0007A^2 - 0.007B^2 + 0.003C^2 \end{aligned} \quad (3)$$

3.6 Interaction between Cooking Time and Ethanol Concentration

The effect of the cooking time and ethanol concentration (AB) was not significant on the responses (Figure 1 and Table 3 with $p = 0.5412$, 0.0317 and 0.0158). Longer time and more lignin fragments that were released underwent a condensation reaction and re-polymerization causing the lignin

to be insoluble in ethanol (Rambe *et al.*, 2013). Pulping at higher ethanol content showed an almost linear increase of the acid-insoluble lignin. In Figure 1c at ethanol concentrations of 30 to 45%, the acid-insoluble lignin content improved (6.2%) at 45% ethanol; thus, the content was eligible as mentioned previously.

3.7 Interaction between Cooking Time and Temperature

The cooking time and its interaction with temperature affected the pulp yield and cellulose and acid-insoluble lignin contents (Figure 2). At an optimum temperature (90 °C) and 100-min cooking time, the responses were optimum: cellulose content – 57.34%; yield – 92.39%; and acid-insoluble lignin – 41.61%. Long time and high temperature could cause cellulose degradation resulting in a lower yield than the optimum yield. At lower temperatures and longer cooking time, lignin was more easily degraded than cellulose; thus, the acid-insoluble lignin content was higher. Based on this study, a longer time resulted in more acid-insoluble lignin in the pulp at 90 °C.

The lower solubility of lignin at a lower temperature (Jansson and Branvall, 2014) caused lignin to combine with lignin in fiber at lower ethanol content. Cooking time, cooking temperature and ethanol concentration had an important effect on the yield of pulp, cellulose and acid-insoluble lignin with a minimum confidence level of 93% (Equations 1-3). However, compared with the cooking time, the cooking temperature affected the non-wood pulp cellulose damage more.

3.8 Interaction between Ethanol and Temperature

Figure 3a exhibits the interaction of ethanol content and cooking temperature (BC). Pulp yield would be low ($p < 0.05$). At low ethanol content, the yield response was quite sensitive to temperature. The highest yield was 96.24% at 45% ethanol. The lowest yield was 79.55% at 60% ethanol. This yield caused changes in high temperature and ethanol content to be insensitive. The change was not significant at 60% ethanol when the temperature was varied so that the 60% ethanol content was considered the optimum level. The yield associated with low ethanol content and temperature was negligible. As a result, the interaction of these two variables could be neglected.

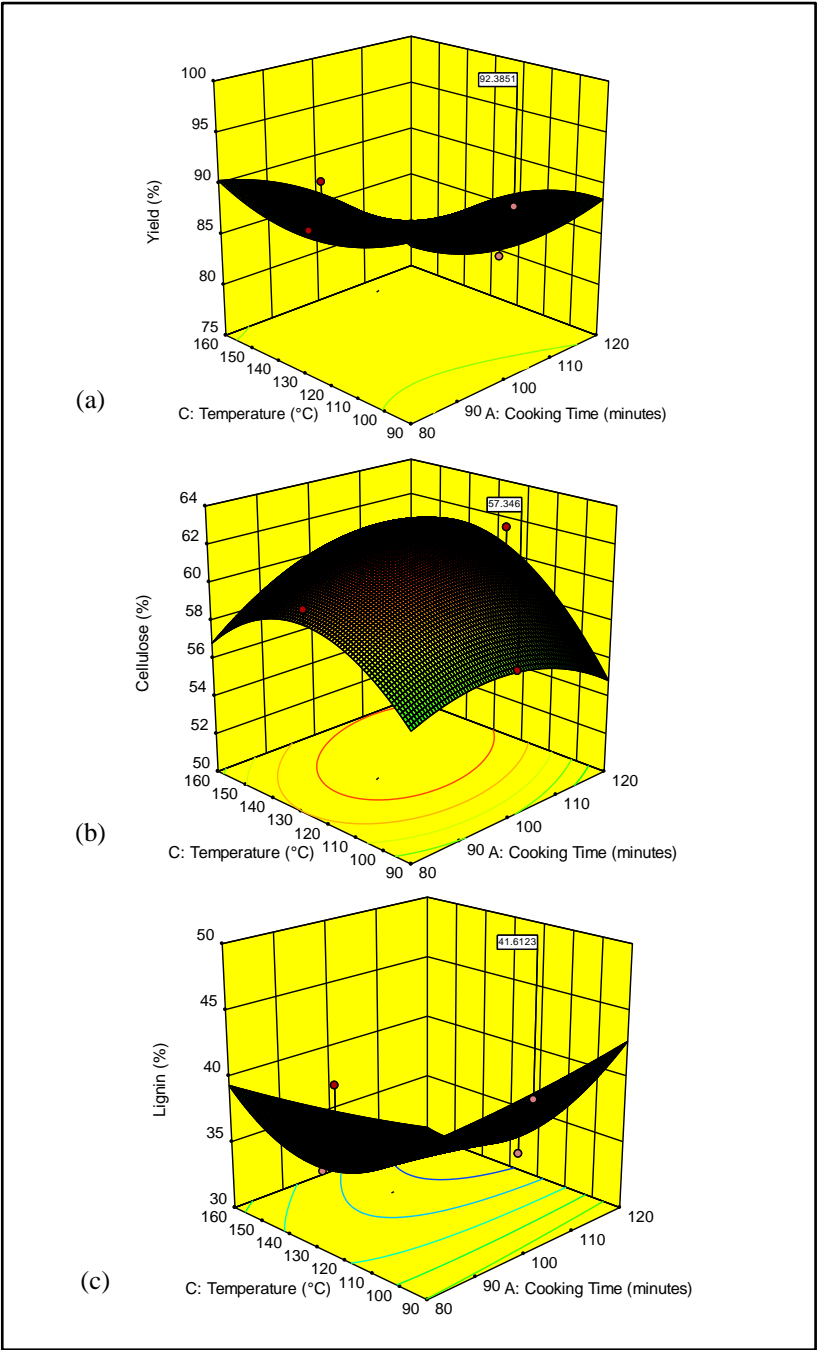


Figure 2. Plots expressed the interaction of temperature and cooking time on pulping variables

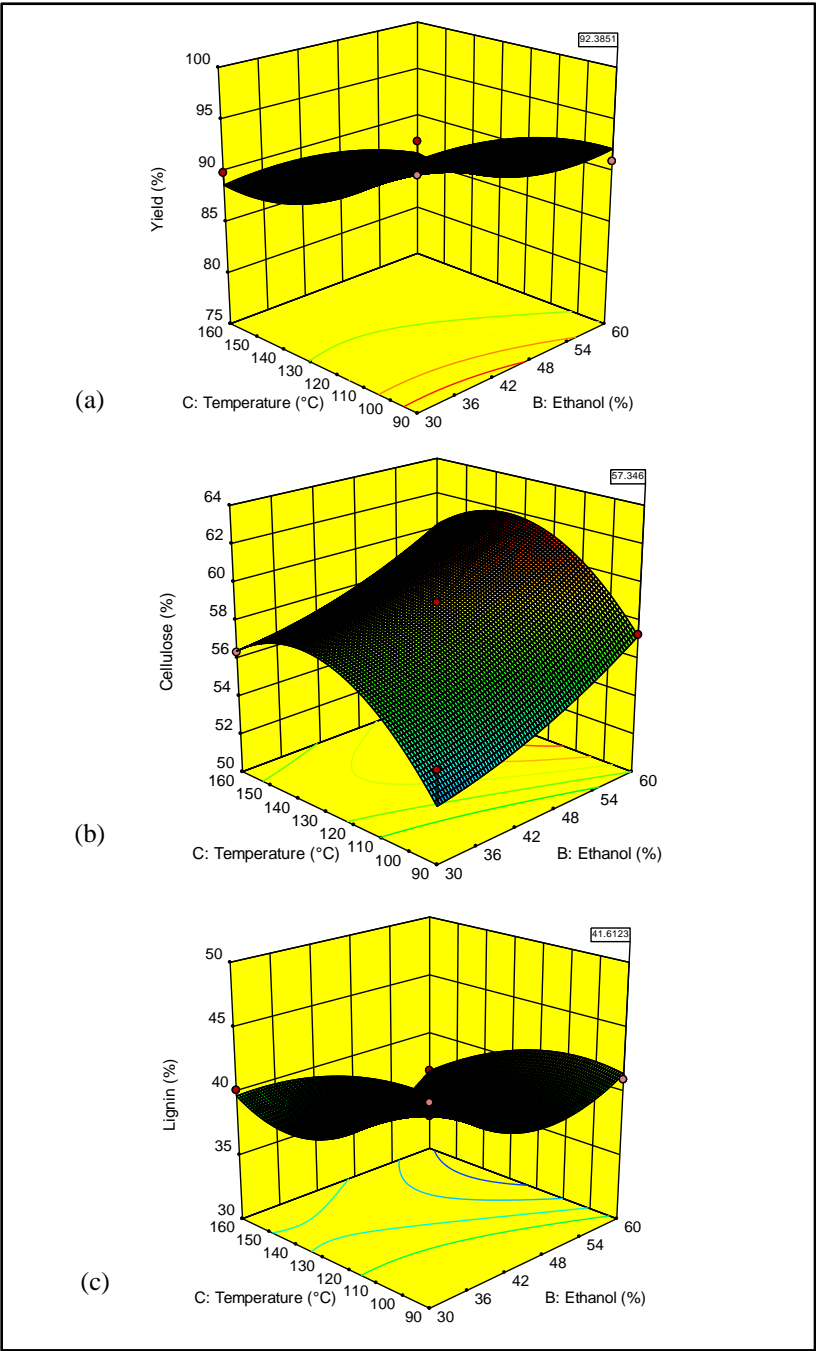


Figure 3. Plots expressed the interaction of ethanol and temperature on pulping variables

Cellulose content continued to increase at a low temperature (90 °C) as the interaction of ethanol and cooking temperature (Figure 3b). A significant increase in cellulose content occurred at an increase in temperature with the highest content of 61.56% and high ethanol content (60%). Higher ethanol content could control degradation more towards lignin. Cellulose degradation resulted in higher ethanol content and temperature but then stopped and moved in the opposite direction at 160 °C.

As the ethanol content increased, the acid-insoluble lignin decreased at the initial temperature increase, and the delignification strength might become stronger (Figure 3c). The lowest lignin content was 31.31% in 60% ethanol.

3.9 Optimization of Variables for Pulp Synthesis

Variation of the data was estimated by the BBD matrix using the response variables (Table 2). ANOVA showed that the model p-values were 0.0004, 0.0028 and 0.0007 (Table 3). The model was declared significant ($\alpha = 0.05$) if the P-value was less than 0.05. The optimum pulp yield based on this optimization result was 91.20%, 57.42% cellulose content and 41.16% lignin content at 100-min cooking, 60% ethanol and 90 °C. An investigation to validate the data was carried out by selecting the most optimal conditions in the study to achieve the maximum amount of pulp yield and cellulose content, and minimum acid-insoluble lignin contents. A subsequent investigation was carried out with a hydropulper to optimize the pulping conditions, which resulted in 91.46% pulp yield, 57.59% cellulose content and 41.28% acid-insoluble lignin content. All models predicted results that were nearly identical to the measured values. These results validated the model by confirming the optimal system conditions to achieve the targeted pulp yield, cellulose and acid-insoluble lignin contents.

4. Conclusion

A combination of rice straw and used paper is a potential agribusiness waste as pulp material. Optimization of the pulp yield together with cellulose and acid-insoluble lignin contents was determined with BBD. Higher cooking temperature, until it passed the optimum temperature, led to decreased pulp yield and increased acid-insoluble lignin. At various ethanol concentrations (30, 45 and 60%) at three cooking times (80, 100, and 120 min), organosolv pulps of rice straw with used paper were obtained. In pulp making these

materials, the 100-min cooking time, 60% ethanol concentration and 90 °C were the optimum process condition because they result in the optimum yield, cellulose and acid-insoluble lignin contents of 91.46, 57.59 and 41.28%, respectively.

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