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Development Production Process Cellulose from Rice Straw

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Abstract

Biomass The largest in Indonesia is rice straw, where content the largest is cellulose. Cellulose the beneficial in industry biomedicine, energy, and electronics. In this research, we develop a production process for cellulose with a novelty is multistage method are first-stage crusher mechanical 5000 rpm, stage 2 hydrolysis NaOH and stage 3 delignification with NaClO2. The aim is get maximize the mass of cellulose and investigate the interaction between 3 factors design that is NaOH concentration, temperature reaction and reaction time its effect on cellulose mass. This research provides results that by using the method multistage, cellulose was successfully obtained from rice straw. Mass model cellulose in the production process cellulose to be used in the optimization stage is quadratic. Optimization of mass cellulose from rice straw using RSM (response surface methodology), algorithm genetics, and PSO method. Optimum mass cellulose at NaOH content = 7%, temperature = 80 ºC, and reaction time = 5 hours. Characterization morphology through scanning microscope electron (SEM) shows the formation cellulose shaped stem. FTIR shows group its function is group hydroxyl, CH group, band on CO group and characteristics of β-glycosidic bonds. Diffraction X-rays (XRD) reveal that cellulose has a crystallinity of 63.2% and analysis thermogravimetry (TGA) shows that % heavy experiences degradation as it rises temperature, where cellulose experiences degradation at temperature around 250ºC and after 350ºC, the remaining residue decomposes at a slow degradation rate.

Keywords: *Straw Rice, Cellulose, Multistage, Modeling, Optimization.*

Introduction

Indonesia is the 5th producer biomass largest in the world. In Indonesia, potential biomass the biggest is rice straw (Energy Outlook 2021) . Rice straw in its use does not affect the food chain, because rice straw cannot be eaten. (Londoño-Pulgarin et al. 2021) Rice straw management in Indonesia is carried out conventionally, namely by landfilling, composting, incineration, animal feed and open-field burning. These things have a negative impact on the environment or require a long time, so sustainable management is needed.

Rice straw consists of cellulose (40–50%), hemicellulose (20–30%) and lignin (10–18%) (Guo et al. 2020) , cellulose is trapped in a hemicellulose-lignin matrix, which makes its separation very difficult. (Rocha et al. 2020) (Guo et al. 2020) . Cellulose has a complex hydrogen bonding network between cellulose molecules, cellulose is insoluble in common solvents. Currently, the extraction of cellulose from rice straw uses inorganic acid-base (Guo et al. 2020) and organic solvent extraction. The resulting cellulose extraction has a cellulose content that is not optimal. The cellulose content can be increased by modification, are mechanically by crushing at 5000 rpm, followed by alkaline hydrolysis is NaOH.

Cellulose is polymer the most common natural on earth. Cellulose nature renewable, cheap and available in a way wide. Cellulose used in industry biomedicine, packaging food, pharmacy (Guo et al. 2020) (Masłowski, Miedzianowska, and Strzelec 2019) (Masłowski, Miedzianowska, and Strzelec 2019) , electronics, energy (Melikoğlu, Bilek, and Cesur 2019) , source material burn alternative (Ganguly et al. 2020) and nutrition like glucose to use man (Prakash et al. 2018) .

In the production process cellulose from straw rice, there are many variables that need to be analyzed. These variables are not few and varied. Direct experiments for all of these variables are expensive. They are also time-consuming and difficult. These are the reasons why an accurate model is needed.

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Many alternative parameter values in the cellulose production process from rice straw, causing the need for the best solution, which can be obtained by optimization methods. Therefore, optimization is needed in the cellulose production process from rice straw. This model and optimization are expected to be used to predict the mass of cellulose from rice straw and optimum operating conditions. Based on the explanation above, this study conducted the development of the cellulose production process from rice straw with multistage method are first stage crusher mechanical 5000 rpm, stage 2 base hydrolysis is NaOH and stage 3 delignification with $NaClO_2$ equipped with modeling and optimization of the process, with the aim of maximizing the mass of cellulose, and to investigate the interaction between 3 design factors are NaOH content, temperature and reaction time to reveal their effects on cellulose mass.

Methodology

Materials

In this study, biomass was used is rice straw. This rice straw comes from Rambutan District, Banyuasin Regency South Sumatra Indonesia. The NaOH used is NaOH ≥ 99.0% (E-Merck). The H_2SO_4 used for testing cellulose content is H_2SO_4 98% (E-Merck). $NaClO_2$ is used for delignification is $NaClO_2$ 80% Sigma-Aldrich.

Problem Formulation

The problem formulation in this research is objective function, constraints and optimization variables. The objective function in this research is maximum mass cellulose. The constraints in this study are temperature 80 - 140°C, NaOH content 3 - 7% and reaction time 2 - 5 hours. Optimization variables in this research are NaOH content, temperature and reaction time

Experimental Design

In this research, the influencing factors mass cellulose are NaOH content, temperature and reaction time. Each factor has 3 levels, for NaOH levels are 3, 6 and 7 %wt, temperatures are 80, 100 and 140°C and reaction times are 2.4 and 5 hours. The experimental design in this research begins by using Taguchi method. Degrees total freedom in this study with 3 factors and 3 levels is 6. The orthogonal matrix used has degrees the same or more freedom rather than total degrees freedom factor main, which in this study is 6. The orthogonal matrix is L9 (3^3).

Taguchi orthogonal arrangement gives the lowest beta power level of 23.6% and an average of 56.4% which indicates that the probability of the design being able to detect the desired signal from the existing noise is the lowest at 23.6% and an average of 56.4%. This value means that failure to detect has a high probability of 100% - 23.6% = 76.4% or an average failure to detect of 100 - 56.4 = 43.6%. To overcome this, this study conducted additional measurements. Additional measurements were carried out with a factorial design. This study uses 3 factors and 3 levels, so the factorial design is 33 = 27 runs. Each run is carried out 3 replicas.

Production Process Cellulose

Preparation cellulose from rice straw through 3 stages that is 1st stage mechanical, 2nd stage hydrolysis base and the 3rd stage of delignification. At 1st stage, rice straw crushing in a way mechanical with 5000 rpm. 1st stage results followed by the second stage, soaked in water for 6 hours, then mixed with NaOH (3%, 6% and 7% wt), next refluxed at 80°C, 100°C and 140°C for 2, 4 and 5 hours. This is done to remove lignin and hemicellulose from rice straw as well as to purify cellulose. The third stage is delignification. The delignification stage includes the results of the second stage being filtered and the residue washed with 300 ml hot water, then residue dried in the oven, then 5 grams of residue dry 40 ml of 5.25% $NaClO_2$ solution was added, then refluxed at 110 ° C for 4 hours. The result waiting until room temperature, then filtered

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and residue washed with water until obtained solids white cellulose. Solids white (cellulose) is dried in an oven and weighed as a mass cellulose.

Modeling

A mathematical model describing the mass of cellulose is needed to follow the effects of NaOH content, reaction temperature and reaction time on the cellulose production process. The unavailability of rice straw properties in batches and the lack of understanding of the effects of NaOH content, reaction temperature and reaction time on cellulose mass require an empirical model. An empirical model involving a number of parameters can be made with experimental data. The empirical model uses a dependent variable is cellulose mass. The empirical model has 3 independent variables are NaOH content, temperature and reaction time.

Optimization

Optimization of cellulose mass in the cellulose production process from rice straw contains 3 factors are NaOH content, reaction temperature and reaction time. In this study, optimization was carried out using 3 methods are RSM, GA and PSO. RSM is a statistical instrument for experimental design, empirical modeling, and factor impact assessment. RSM can reduce the number of experimental tests required to assess various parameters and their interactions (Thakur et al. 2020) . Three factors of this study were examined through a central composite design. Each factor was varied at three levels: NaOH content of 3, 6 and 7 %wt, reaction temperature of 80,100 and 140^{o}C, reaction time of 2,4 and 5 hours. The design resulted in a total of 27 experiments. Using the reaction surface model and point prediction from specialist design software, the ideal expected values of the variables were obtained. Every experiment was carried out in 3 replicates. The measured reactions were given with the predicted responses to validate the corresponding values.

Genetic Algorithm (GA) is a promising technique for solving nonlinear and complex problems, and has been used for the optimization of several chemical engineering problems (Biyanto 2013) . In general, GA is the most efficient optimization technique in terms of function evaluation. Solving nonlinear problems using GA has been proven to be a valid approach, where computation time is not the most important thing (L. Costa and P. Oliveira 2001) . In this research, generation population and parameters as follows:

Nvar = 3; % number of design variables optimized (factor)

Nbit = 20; % number of bits

Npop = 100; % of population (Population x Generation)

Maxit = 200; % iteration, generation

Pc = 0.85; % crossover probability

Pm = 0.005; % probability mutation

El = 0.05; % elitism

Particle Swarm Optimization (PSO) (Biyanto 2013) (Sateria, Dwi Saputra, and Dharta 2018) is an optimization method that can be used to determine process parameters that produce optimum response values. PSO imitates the social behavior of a flock of birds or fish in a natural habitat. Each individual or particle behaves by using its own intelligence and is also influenced by the behavior of its collective group. When one particle or a bird finds the right or short path to a food source, the rest of the group will also be able to immediately follow the path even though their location is far from the group. Each individual or particle is treated like a point in a certain spatial dimension. There are two factors that characterize the particle status in the search space, namely the particle position and the particle velocity. The following is a mathematical formulation that describes the position and velocity of particles in a certain spatial dimension:

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$X_j(i) = X_1(1), X_2(1), \ldots, X_{jN}(i)$

$V_j(i) = V_1(1), V_2(1), \ldots, V_{jN}(i)$
[1]

With:

T = position particle

R = speed particle

E = E-th iteration

F = index particle

0 = amount particle

Equations for mechanisms particle status updates are as follows:

$V_j(i) = V_j(i-1) + c_1 r_1 (P_{best,j} - X_j(i-1)) + c_2 r_2 (G_{best,j} - X_j(i-1))$

$X_j(i) = V_j(i) + X_j(i-1)$
[2]

With

J = 1,2, …, N represents amount particle

Obj_i = response from experiment

$P_{best,j}$ = *personal best* from jth particle

$G_{best,j}$ = *global best* from all over flock

c_1, c_2 = *learning factors*

Equation 2 is used to calculate the new particle velocity based on the previous velocity, the distance between the current position and the best particle position (personal best) and the distance between the current position and the best swarm position (global best). The particle then moves to the new position. This PSO algorithm is run with a certain number of iterations until it reaches the stopping criteria, so that a solution will be obtained that lies in the global best. This equation will be simulated in a space with a certain dimension with a number of iterations, so that in each iteration the particle position will increasingly lead to the intended target (minimization or maximization of the function value). This is done until the maximum iteration is reached or another stopping criterion is reached.

Characterization

After obtaining the optimal process parameters, the resulting cellulose products were characteristic analysis using SEM, FTIR, XRD and TGA.

Scanning Electron Microscopy (Sem)

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SEM-EDX was used to examine the microscopic structure and surface morphology of cellulose fibers. SEM was performed at an acceleration voltage of 20kV (Fischer et al. 2014) . Before scanning coated with gold.

Fourier Transform Infrared (Ftir) Spectroscopy

FT-IR is used to determine group function (Kunusa et al. 2018) . FT-IR is performed on a spectrometer running on a range transmission 500–4000 cm^{-1} (Khan et al. 2020) . The FT-IR test was carried out to see exists content cellulose. FTIR spectrum for all test objects were recorded on several part of the wave band that ranges between 500–4000 cm^{-1} when using ATR method (Lei et al. 2018) .

X-Ray Diffraction (XRD)

Analysis Diffraction X- rays (XRD) for find out percentage content crystalline and amorphous in cellulose. Crystallinity tall obtained by using diffractometer X- rays equipped with Cu Kα (α=0.154nm) in the 2θ range of 5–90, (setting energy battery: 40kV and 30mA). Empirical method used to get index crystallinity, Xc sample as shown in Equation 3 (Teixeira et al. 2011)

$$Xc = \frac{I_{002} - I_{am}}{I_{002}} X\ 100 \quad [3]$$

where I_{002} and I_{am} are the respective intensities peak material crystalline and amorphous. Equation 4 is Scherrer's equation is used to calculate size crystal

$$\tau = \frac{K\lambda}{\beta \cos\theta} \quad [4]$$

Where,

τ is the dimension upright crystal straight field diffraction with the Miller Index hkl

λ is length wave radiation X- rays (λ=0.154Å)

β is the half full width maximum (FWHM) of peak diffraction (Bhattacharya, Germinario, and Winter 2008)

Thermo Gravimetric Analysis (TGA)

TGA measurements used a heating rate 10°C min^{-1} below N_2 atmosphere (20 ml min^{-1}). Sample weight 4.1023 mg was taken and stored in a desiccator until weighed. TGA was carried out to observe the characteristics of degradation sample cellulose. Samples were held for 1 min at 40 °C and then heated at a rate of 10 °C min^{-1} from 40 °C to 1000 ⁰ C. During the period of heating, fraction weight and difference temperature are recorded as a function temperature.

Results and Discussion

Experimental Results

The effect of temperature on cellulose mass with temperature variations 80 ⁰C, 100 ⁰C and 140 ⁰C, can be seen in Figure 1, which gives the results that the higher the temperature, the lower the cellulose mass. This also applies with NaOH 3 %wt, 6 %wt and 7 %wt, for 2 hours, 4 hours, and 5 hours. Cellulose mass decreased because higher temperature makes more cellulose is dissolved. This is in accordance with Sambuti

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and Khozin's research that cellulose can dissolve at higher temperatures. ((Sambusiti, C., Ficara, E., Malpei, F., Steyer, JP, and Carrère 2013) and (Khozin 2017)). Based on Pareto chart, temperature give negative effects. This means higher temperatures make mass cellulose decrease. Pareto chart can see in Figure 2. In the Pareto chart, the temperature is above the Bonferroni red line, showing that temperature is factors that have an effect significant to mass cellulose. Temperature has more T-*value* than NaOH. That matter interpreted that temperature has more effect significant to mass cellulose than NaOH. From the ANOVA analysis that can be seen in Figure 3, temperature has *p-value* < 0.0001. This shows that temperature is factors that have an effect significant to mass cellulose.

Effect of content NaOH on mass cellulose can see in Figure 4. Figure 4 shows that at operating temperatures 80 ⁰C, 100 ⁰C and 140 ⁰C for 2 hours, 4 hours and 5 hours with increasing NaOH content up to 7 % the cellulose mass increases. On the Pareto chart, NaOH content give effect positive. This means that the more NaOH content then the more mass cellulose. This is because the reaction of NaOH with water is exothermic reaction, can dissolve the more hemicellulose and lignin, so cellulose easier obtained. That matter impact mass cellulose increases. On the Pareto chart, the NaOH content is above the Bonferroni red line, indicating that NaOH content are factors that have an effect significant to mass cellulose. From the ANOVA analysis, NaOH has *p-value* < 0.0001. This shows that NaOH are factors that have an effect significant to mass cellulose.

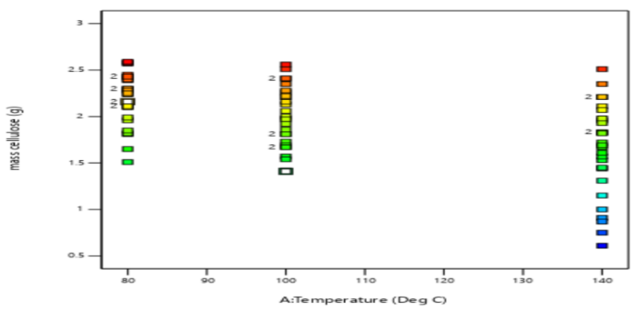

Figure 1. Effect Of Temperature

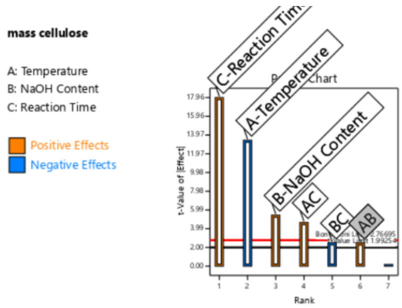

Figure 2. Pareto Chart

ANOVA for Quadratic model

Response 2: mass cellulose

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	14.08	9	1.56	63.38	< 0.0001	significant
A-Temperature	4.70	1	4.70	190.50	< 0.0001	
B-NaOH Content	0.9041	1	0.9041	36.63	< 0.0001	
C-Reaction Time	8.63	1	8.63	349.64	< 0.0001	

Figure 3. ANOVA

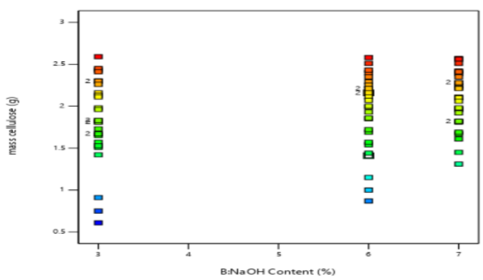

Figure 4. Effect of NaOH

The effect of reaction time on mass cellulose with variations in reaction time is 2 hours, 4 hours and 5 hours, can be seen in Figure 5, which gives the results that the longer the reaction, the greater mass cellulose. This applies NaOH 3% wt, 6% wt and 7% wt, temperature operation 80 ⁰C, 100 ⁰C and 140 ⁰C. This fenomena because the longer reaction, the more lignin is dissolved. This is in accordance with Rizki and Sari's research that heating NaOH can increase reaction lignin degradation in NaOH solution. This is related to effects heat generated during the reaction time. The longer exposure time, effect heat also increases and

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is capable degrade bonds in lignin, so lignin decrease at more exposure (Ramadhani 2019) (Poppy Diana Sari1), Wuwuh Asrining Puri1) 2018) . Lots of lignin dissolved the impact cellulose easier obtained. That matter can make mass cellulose increases. Based on Pareto chart, reaction time give effect positive. This means that longer reaction, the greater mass cellulose. On the Pareto chart, the reaction time is above the Bonferroni line, indicating that the reaction time is factors have an effect significant to mass cellulose. The reaction time has a T-*value* bigger than NaOH content and temperature. That matter interpret that the reaction time has the most significant effect to mass cellulose than NaOH content and temperature. From the ANOVA analysis, the reaction time has a value *p-value* < 0.0001. This shows that the reaction time is factors have an effect significant to mass cellulose.

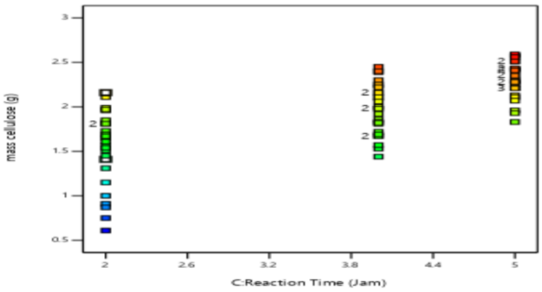

Figure 5 Effect of Reaction Time

Sequential Model Sum of Squares [Type I]

Response 2: mass cellulose

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Mean vs Total	303.73	1	303.73			
Linear vs Mean	12.95	3	4.32	115.59	< 0.0001	
2FI vs Linear	0.9015	3	0.3005	11.26	< 0.0001	
Quadratic vs 2FI	**0.2226**	**3**	**0.0742**	**3.01**	**0.0359**	**Suggested**
Cubic vs Quadratic	0.2885	7	0.0412	1.80	0.1022	Aliased
Residual	1.46	64	0.0229			
Total	319.56	81	3.95			

Figure 6. Sequential Model Sum of Square

Modeling

Joneset al. stated two main goals of modeling, the first to obtain a better understanding of the cause-effect relationships *in* a system, and to provide a better qualitative and quantitative interpretation of the system. The second goal of modeling is more applied or problem-oriented, to obtain better predictions of the behavior of the system which is used immediately in improving control or management of the system. (Jones and Palmer 1987) . The modeling of the cellulose production process in rice straw uses experimental data presented in point 3.1 by drawing a relationship between the influence of NaOH content, temperature and reaction time which results in mass cellulose.

Methodology response surface use composite center design used to determine the variables that influence the results. Relevant factors are NaOH content, temperature operation and reaction time. This analysis aims to determine the mathematical model that will be used to describe phenomenon from the research results. Determination the type of mathematical model that will be used sequential sum of summary (description amount quadratic), lack of fit test (model imprecision test), and model summary statistics (summary statistical model).

Sequential sum of summary analysis can see in Figure 6, that the model can accepted if p-value below 5% (0.05), which shows mark it has influence real to response, meanwhile p-value more than 5%, then the model has inaccuracy or error exceeding 5%, and is considered no accurate to response. Figure 6 shows that the linear model and 2 FI (interaction between two factor) has p-value <0.001, which is significant that has inaccuracies to response was <0.1%. The quadratic model has p-value 0.0359 (3.59%), which is significant that the model inaccuracies shown are below 5% limit . Cubic model shows a p-value of 0.1022 (10.22%), the model has a value above 5%, so that the model said aliased or not suggested as a mathematical model of the response.

Determination second type of mathematical model is using lack of fit test or model inaccuracy test. Testing this type assumes appropriate a model if the deviation test from each model does not significant at a certain ∝ value determined in this study, where ∝ value used is 0.05 or 5%. Model selection according to lack of fit test is based on the p-value that has value more of 0.05, then it will considered that model no influential real and already according to the response. Test results lack of fit can see in Figure 7. Based on Figure 7, linear model obtains p-value of <0.0001 (<0.01%), 2FI model obtained p-value of 0.0330 (3.3%), quadratic model get p-value of 0.0926 (9.26%), cubic model obtained p-value is 0.2034 (20.34%). Based on p-value,

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quadratic model suggested (suggested). Modeling cubic has p-value above 5%, however no recommended because p-value of analysis sequential sum of summary more from 5 %.

Lack of Fit Tests

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Linear	1.71	23	0.0746	3.47	< 0.0001	
2FI	0.8133	20	0.0407	1.89	0.0330	
Quadratic	**0.5906**	**17**	**0.0347**	**1.62**	**0.0926**	**Suggested**
Cubic	0.3022	10	0.0302	1.40	0.2034	Aliased
Pure Error	1.16	54	0.0215			

Figure 7. Lack of Fit Test

Model Summary Statistics

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	0.1933	0.8183	0.8112	0.7967	3.22	
2FI	0.1634	0.8752	0.8651	0.8495	2.38	
Quadratic	**0.1571**	**0.8893**	**0.8753**	**0.8549**	**2.30**	**Suggested**
Cubic	0.1512	0.9075	0.8844	0.8502	2.37	Aliased

Figure 8. Model Summary Statistics

Determination third type of mathematical model is summary statistics model. Model summary statistics analysis can see in Figure 8. Model summary statistics analysis, the parameters used in selecting the appropriate model are standard deviation lowest, R-square (R^2) highest, adjusted R^2 highest, predicted R^2 highest and lowest PRESS. R^2 is coefficient determination proportion or percentage the total variation in the dependent variable explained by the independent variable from equality regression. R^2 between 0 to 1. Smaller R^2 show connection between variables also smaller, likewise larger R^2 (closer to 1) larger relationship between the variables. In the R^2 test, a good model is a model has R^2 approaching 1. Standard deviation in the linear model was 0.1913, in the 2FI model was 0.1634, in the quadratic model is 0.1571 and this model is recommended, while the cubic model is 0.1512 and value the is mark lowest from other models, but this model not recommended because p-value of analysis sequential sum of summary more than 5 %. The R^2 value in the linear model is 0.8183, the 2FI model is 0.8752, quadratic model is 0.8893 and this model is recommended, while the cubic model is 0.9075 and highest from other models, but this model not recommended because p-value of analysis sequential sum of summary more than 5 %. Analysis adjusted R^2 in the linear model is 0.8112, in the 2FI model is 0.8651, in the quadratic model is 0.8753 and this model is recommended, while the cubic model is 0.8844 and highest from other models, but not recommended because p-value of analysis sequential sum of summary more than 5 %. Analysis prediction R^2, in the linear model is 0.7967, in the 2FI model is 0.8495, in the quadratic model is 0.8549 and this model is recommended, while the cubic model is 0.8502. Testing the summary statistics model also takes into account PRESS (Prediction Error of Sum of Squares) value. The PRESS value is used to indicate predictions error amount square from the model. The PRESS value in the linear model is 3.22, in the 2FI model is 2.38, in the quadratic model is 2.3 and this model is recommended, while the cubic model is 2.37.

Conclusion of determination the types of mathematical models above, is sequential sum of summary, lack of fit test and model summary statistics, explained that connection between NaOH content, temperature and reaction time to mass cellulose is a quadratic model. The results of the influence of NaOH content, temperature and reaction time which have an impact on mass cellulose can be seen in Equation 5, which is quadratic equation.

Mass cellulose = 4,21547 - (0,0301506 X temperature) – (0,270343 X NaOH content) –(0,165442 X reaction time) + (0,00105418 X temperature X NaOH content) + (0,00270975 X temperature X reaction time) - (0,0212302 X NaOH content X reaction time) + (2,40741e-05 X temperature ^{2}) + (0,0294444 X NaOH content^2) + (0.0353086 X reaction time^2) [5]

Model Verification

Model verification is carried out using sequential sum of summary, lack of fit test, model summary statistics, sum of square test, normal plot of residuals, residuals-predicted plot, residuals-order plot, and normality

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test. Sequential sum of summary test gives the results that Equation 5 has a p value of 0.0359 (3.59%), which means inaccuracies model below 5%, so the model is appropriate.

The lack of fit test, the hypothesis for testing the model is:

H_0: no lack of fit on the model

H_1: have lack of fit on the model

Significance level $\alpha = 0.05$

Testing to hypothesis use analysis variance (ANOVA). Rejection area is rejected H_0 if p value < 0.05 which is significant nonconformity between the predicted model and the actual model. Reception area is accepted H_0 if p value > 0.05 which is no lack of fit in the model, there are suitability between the predicted model and the actual model. The p value for Equation 5 is 0.0926, which is no lack of fit in the model, there are suitability between the predicted model and the actual model.

The summary statistics model test provides the R^2 value is 0.8893. R^2 close to 1 then standard deviation smaller and more models good for predicting response. This shows that the Equation 5 model can state in accordance.

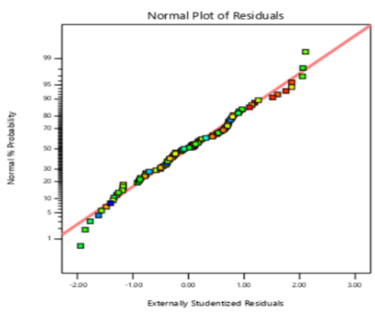
In the sum of square test, a model is state in accordance if mark adjusted R^2 and predicted R^2 have a difference mark smaller of 0.2. The adjusted R^2 value in this study was 0.8753 and the value predicted R^2 of 0.8549. This research has differences mark adjusted R^2 and predicted R^2 more small of 0.2, so the model Equation 5 can state in accordance.

Normal plot of residual in this study can be seen in Figure 9. Figure 9 shows that all the points (experimental data) are around the red line and are not form pattern, which indicates normally distributed residuals. Distributed residue normally required to estimate error accurate standards for estimating model parameters. The appropriate model is one that has a normal or close to normal residual data distribution. This research has normally distributed residuals, so that the Equation 5 model can be state in accordance.

Residuals - predicted plot in this study can be seen in Figure 10. Figure 10 shows all residual data in the study is between the red lines. This shows haven't outliers. Outliers are observations that are not according to the model. With no outlier, the Equation 5 model can state in accordance.

Residuals - order plot this research can see in Figure 11. Residuals - order plot gives inspection to possible hidden variables influence response during the study. Figure 11 shows spread in a way random and not form pattern. With this random distribution, the model Equation 5 can state in accordance.

This research has normally distributed data. The results of the normality test with Kolmogorov Smirnov provide KS value is 0.088. that value more of 0.05, then the variable is stated normally distributed. Normally distributed, this study does not apply cox box transformation



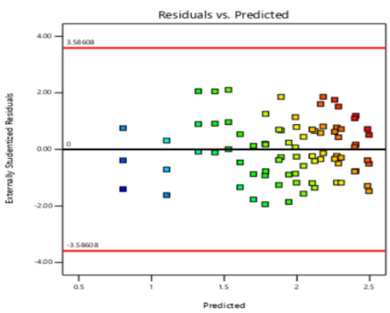


Figure 9. Normal Plot of Residuals **Figure 10. Residuals - Predicted Plot**

Journal of Ecohumanism
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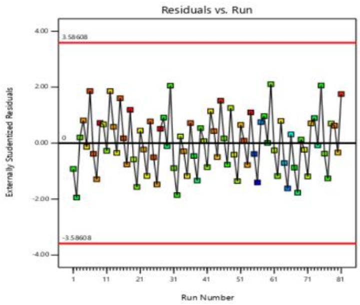

Figure 11. Residuals - Order Plot

Model Validation

Prediction error to actual for the mass cellulose is less than 5 %, at condition NaOH content 3% wt, 6% wt and 7% wt, temperature operation 80 ⁰C, 100 ⁰C and 140 ⁰C for 2 hours, 4 hours and 5 hours. Prediction for the mass cellulose can see in Table 1, which shows mass cellulose error predictions to actual maximum 4.3%. Apart from the explanation above, model validation is also carried out by calculating RMSE and normalized RMSE values. RMSE values for the mass model cellulose is 0.005 and the normalized RMSE is 0.26. With normalized RMSE and RMSE values then the Equation 5 model is capable adapt a data set that shows the model fits.

Table 1. Validation of the Research Model

No	Temperature (⁰ C)	NaOH Content (%)	Reaction Time (hours)	Mass of cellulose (g)	Mass cellulose prediction (g)	Errors (%)
1	80	3	2	1.81	1,781	-1.6
2	80	3	5	2.43	2,485	2,3
3	80	6	4	2.11	2,162	2.5
4	80	6	5	2.43	2,404	-1.1
5	80	7	2	1.96	2,045	4.3
6	80	7	4	2.24	2,274	1.5
7	80	7	5	2.42	2,495	3.1
8	100	3	2	1.42	1,436	1.1
9	100	3	4	1.98	1,944	-1.8
10	100	3	5	2.26	2,303	1.9
11	100	6	4	2	1,989	-0.5
12	100	6	5	2.35	2,285	-2.8
13	100	7	2	1.81	1,785	-1.4
14	100	7	4	2.06	2,122	3.0
15	100	7	5	2.41	2,397	-0.5
16	140	3	5	1.96	1,997	1.9
17	140	6	5	2.07	2	1.7
18	140	7	2	1.31	1	0.9
19	140	7	4	1.82	2	3.1

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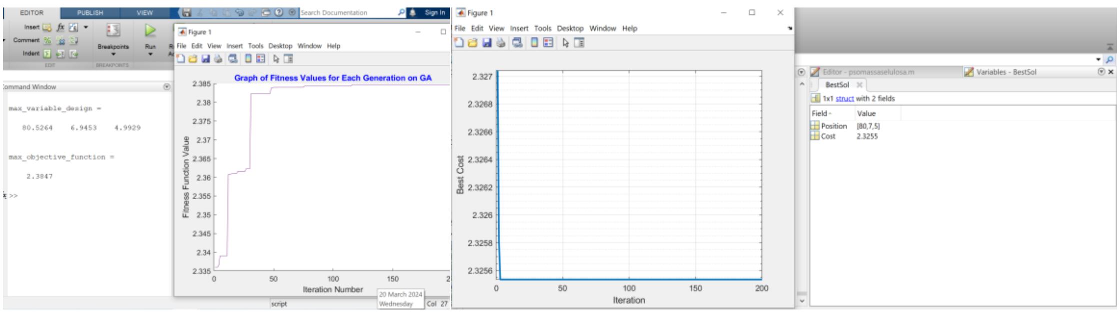
Optimization

Optimization is the process of searching for one or more solutions related to the values of one or more objective functions in a problem so that an optimal value is obtained (Berlianty, I., & Arifin 2010) . In general, optimization means finding the best value (minimum or maximum) of several functions given in a context. Optimization can also mean efforts to improve performance so that it has good quality and high work results. Mathematically, optimization is a way to get extreme values, either maximum or minimum, of a certain function with limiting factors. If the problem to be solved is sought for the maximum value, then the decision is in the form of a maxim (Sari 2014) . Optimization is a discipline in mathematics that focuses on systematically obtaining minimum or maximum values from a function, opportunity, or finding other values in various cases. Optimization can almost be used in various fields to achieve the effectiveness and efficiency of the desired targets. One of the objectives of optimization is determining the minimum, so the objective in the mathematical model is minimization (Maharani 2015) .

In this research, optimization is obtained mass cellulose maximum with minimum NaOH content, minimum temperature and minimum reaction time. As for objective function in this research is maximum mass cellulose. Decision variables are NaOH content, temperature operation and reaction time. Optimization was 3 ways method optimization is RSM (response surface methodology), method algorithm genetics and PSO methods. Optimization results mass cellulose by method RSM can see in Figure 12 (a), method algorithm genetics in Figure 12 (b), the PSO method can see in Figure 12 (c). Figure 12 shows mass cellulose maximum either with the RSM method, algorithm genetics and PSO gave consistent results that is maximum under conditions NaOH content 7 % wt, temperature 80 ºC and reaction time 5 hours.

Number	Temperature	NaOH content	Reaction Time	Mass cellulose	Desirability	
1	80.000	7.000	5.000	2.495	0.803	Selected

(a)



(b) (c)

Figure 12. Optimization of Cellulose Mass by Using Method (a) RSM, (b). Genetic Algorithms, (e) PSO.

SEM Characteristic Results

Cellulose obtained at optimum mass cellulose with condition NaOH content 7% wt, temperature 80ºC and reaction time 5 hours was SEM analysis. The SEM analysis results are shown in Figure 13 (a). Figure 13 (a) shows structure microscopic and morphological surface fiber cellulose. Cellulose from rice straw shaped stem small. That matter in accordance with the results of Davis's research (Davis et al. 2013) .

FTIR Characteristic Results

FT-IR is used to determine group function (Kunusa et al. 2018) Cellulose obtained at optimum mass cellulose with condition NaOH content 7% wt, temperature 80ºC and reaction time 5 hours was FTIR analysis. The results of FTIR analysis are shown in Figure 13 (b). Figure 13 (b) shows the cellulose *spectral*

Journal of Ecohumanism
2024
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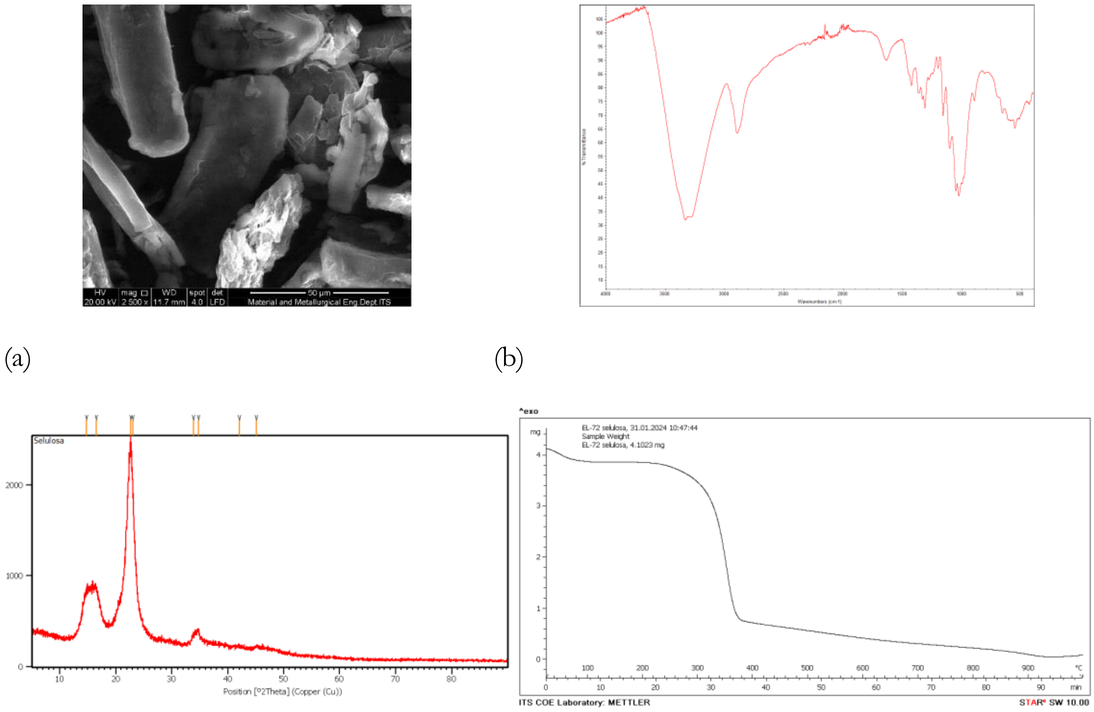
band 3332 cm $^{-1}$ is group hydroxyl, band at 2897 cm $^{-1}$ is C-H group, band at 1029 cm $^{-1}$ is C-O group and band at 896 cm $^{-1}$ is characteristics β- glycosidic bond. That matter in accordance with the results of Abderrahim's research (Abderrahim et al. 2015) .



(a) (b)

(c) (d)

Figure 13 (a). SEM (b) FTIR (c) XRD (d) TGA

XRD Characteristic Results

Cellulose obtained at optimum mass cellulose with condition NaOH content 7% wt, temperature 80 ^{0}C and reaction time 5 hours was XRD analysis. The results of the XRD analysis are shown in Figure 13 (c). XRD is used to study behavior crystals and to evaluate connection between structure and characteristics crystal. Due to cellulose in the structure the molecule part shaped crystals and parts amorphous, this means chain cellulose will held tightly by ties whereno H bonds in amorphous areas (no regular) on the chain cellulose. Size from crystallites and crystallinity cellulose influenced by treatment chemistry and mechanics. From Figure 13 (c), pattern XRD diffraction of cellulose show peak at 22.67 which corresponds to the top characteristics structure cellulose -Iβ (Mohan, D.; Teong, ZK; Sajab, MS; Kamarudin, NHN; Kaco 2021) . During the extraction process progresses , a peak at 16.53° appears , which is indexed to the peak characteristics lattice typical cellulose Iα (Zhao et al. 2017) . This is especially so Because component amorphous such as lignin and hemicellulose removed , and crystal areas cellulose more open (Dehghani et al. 2015) . Index crystalline calculated with Equation 3 and with *under software,* which provides index results crystalline of 63.2%. The results of XRD analysis for cellulose in this study are in accordance with the XRD results for cellulose in this study Suanto (Suanto et al. 2022) .

TGA Characteristic Results

Cellulose obtained at optimum conditions of cellulose mass with condition NaOH content 7% wt, temperature 80 ^{0}C and reaction time of 5 hours was TGA analysis. The results of TGA analysis are shown in Figure 13 (d). The TGA results for cellulose in this study are in accordance with the TGA results for cellulose in Sokker's study.(Sokker et al. 2005) . Figure 13 (d) shows that the weight % undergoes degradation (mass reduction) as the temperature increases. Figure 13 (d) also shows that cellulose undergoes

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degradation at temperatures around 250⁰C and after 350⁰C, the remaining residue decomposes at a slow degradation rate (insignificant decrease in the curve).

Conclusion

The conclusion of this study is based on the results of SEM, FTIR, XRD and TGA showed that by using multistage method are first stage crushing mechanical 5000 rpm, stage 2 base hydrolysis NaOH and stage 3 delignification with $NaClO_2$, cellulose was successfully obtained from rice straw. The model mass cellulose from rice straw to be used in the optimization stage is in the form of quadratic. Optimization mass cellulose from rice straw can be using the RSM, genetic algorithm and PSO. The optimum cellulose mass at NaOH content = 7%, temperature = 80 ⁰C and reaction time = 5 hours.

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