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Optimizing Microwave-Assisted Extraction Parameters for Delignification of Water Hyacinth Stems

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Abstract *– Water hyacinth (Eichornia crassipes) is an aquatic plant that grows very quickly in Indonesian waters and is classified as an invasive weed, so it can cause ecological problems. Water hyacinth stems have a high cellulose content of 28-39%. Extraction of cellulose from water hyacinth needs to be done to remove lignin content. The microwave delignification method has the advantage of a rapid increase in temperature so that the extract yield is greater. This study aims to determine the optimal operating conditions in the delignification process of water hyacinth stems using the Microwave Assisted Extraction (MAE) method by examining the process variables: extraction time (5,10,15 minutes) and microwave power (400, 5000 and 600 watts). Based on the results of the study, the optimal operating conditions at a transmission time of 11.5 minutes and a transmission power of 530.5 watts, which were obtained in 20.25% of water hyacinth stems.*

Keywords: *delignification; cellulose; water hyacinth; Green MAE*

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INTRODUCTION

Water hyacinth (Eichhornia crassipes) grows rapidly in several Indonesian waters with the ability to absorb nutrients such as nitrogen, phosphate, potassium, and heavy metals. The rapid growth of water hyacinth causes oxygen levels in the water to decrease because light cannot enter the water (Ratnani *et al*., 2011). Therefore, it is necessary to control or utilize these aquatic plants. Water hyacinth has the highest natural fiber content in its stems, namely 28-39% cellulose, 23-24% hemicellulose, and 16-25% lignin (Chaiwarit *et al*., 2022). Water hyacinth cellulose is generally used as a craft material with a fairly high economic value (Dirga, 2012). Cellulose fibers contained in water hyacinth can not only be used as materials for making crafts but can also be used as basic materials for making bioethanol paper (Nata *et al*., 2013), biopolymers as biofillers such as films, aerogels, sponges, etc. (Yang *et al*., 2023), and biofuels such as biomass from saccharification where polysaccharides in cellulose are fermented to obtain pure ethanol and hydrocarbons (Acharya *et al*., 2021). The composition of natural fiber cellulose can be taken from plants through the extraction process. Extraction is the process of taking compounds from a mixture of liquid or solid phases with the help of solvents (Dirga, 2012). Delignification can be done by several methods, namely the organosol/acetosolv method (Nair *et al*., 2023), the base method (Fitriana *et al*., 2020), the soxhlet method (Wahib *et al*., 2022), the enzymatic method (Squinca *et al*., 2022), and the microwave-assisted extraction (MAE) method (Ndruru *et al*., 2019). Some of the advantages of Microwave Assisted Extraction (MAE) compared to conventional methods include being able to extract fibers contained in plants. Less solvent is used and the extraction time is faster. Sample throughput increases because samples can be extracted simultaneously, analyte results and productivity are better than conventional methods (Thi *et al*., 2017).

The method of extraction involving the microwave energy and a polar or nonpolar solvent

extraction of dissolved materials in samples is known as the microwave-assisted extraction (MAE) (Llompart *et al*., 2019). Microscopically, microwave energy is heated with a frequency of 300-300,000 MHz to be used as solvent heat in the sample, in order to enable recession of the chosen compound from the solvent sample, making the temperature gradient minimal and heating quicker. In fact, the heating process of molecules is owned by the dual mechanism of ionic conduction and dipole rotation. Ionic conduction and dipole rotation are usually simultaneously occurred in both the solvent and the sample, and this phenomenon will be very effective in converting microwave energy to heat. The dissolved ions will migrate and collide with the molecules because the direction of the ions would also change with the changes in the electric field around the sample (Agastha, 2018). According to Akmala & Supriyo (2020), cellulose structure may be altered under microwaves, while decomposition of lignin and hemicellulose in lignocellulose may occur where heat is generated directly from the increase in activity in the molecules, which will aid in the fragmentation of complex structures into simpler compound structures. Thereby with this mechanism, lignocellulose degradation could speed up cellulose hydrolysis.

Cellulose extraction from water hyacinth was carried out by the vacuum microwave-assisted extraction method at a power level of 300 watts. The maximum yield achieved was 61.07% using 96% ethanol concentration in an extraction time of 8 min. Such a good result reflects the presence of active organic compounds in the product (Putri *et al*., 2023). There has also been an investigation into cellulose extraction from water hyacinth by applying the MAE method using deionized water as solvent and power of 350W in 10 min. The result was a good reduction of lignocellulose of about 23% and cellulose recovery of 60.42% (Thi *et al*., 2017). The aim of this current study is to optimize the operating conditions of delignifying the water hyacinth stem with MAE under controlled time (minutes) and power (watts) variables effect on lignin disappearance percentages. Testing parameters include moisture content test, yield test, and cellulose content test.

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MATERIALS AND METHOD

Materials

Water hyacinth as the main raw material taken from the Tembalang river area. 96% v/v ethanol, H2O2, aquades, and Vaseline.

Equipments

MAE (Microwave Assisted Extraction), filter paper, water bath, thermometer, hose, blender, glassware and oven. The water hyacinth delignification process was carried out using the MAE method with 96% ethanol solvent, a feed to solvent ratio of 1:15 g/ml, microwave power of 300, 400 and 500 watts and operating time variables of 5, 10 and 17 minutes.

Experimental Design

The experimental design using Design Of Experiment (DOE), namely Response Surface Methodology (RSM), can be seen in the following table:

Standart Run	Irradiation Time (Minutes)	Irradiation Power (Watt)
1	5	400
2	5	600
3	15	400
4	15	600
5	3	500
6	17	500
7	10	358
8	10	641
9	10	500
10	10	500

Table 1. Response Surface Methodology (RSM)

Raw Material Preparation

Water hyacinth is obtained in the Rawa Mulawarman area, Tembalang City, Semarang. Next, the water hyacinth is cleaned and cut into small pieces to speed up the drying process. The cut water hyacinth is then dried in the sun until dry. After that, the dried stems are smoothed using a blender.

Delignification with MAE (Microwave Assisted Extraction)

The finely ground water hyacinth stems were taken as much as 50 g, weighed with a digital scale, and put into a shell extraction tube. Then 750 ml of 96% ethanol was added with a measuring cup. After that, the shell tube containing the water hyacinth stem sample and 96% ethanol was put into the microwave with the power and minutes set according to the variables.

Filtration

Standar Run	Irradiation Time (Minutes)	Irradiation Power (Watt)	Delignification Percentage (%)
5	3	500	15,50
1	5	400	16,42
2	5	600	18,55
7	10	358	15,42
9	10	500	20,22
10	10	500	20,25
8	10	641	18,25

The MAE process sample was filtered using filter paper. The residue formed on the filter paper was rinsed with sufficient distilled water to neutralize the pH and clean the dirt that was still present in the residue. The residue was dried in an oven for 15 minutes at a temperature of 80°C.

Bleaching

H_2O_2 was mixed into the sample with a ratio of 1:15 using microwave-assisted extraction. Then set the power to 300 watts and the time to 20 minutes. After that, the bleaching results were filtered with filter paper, and enough distilled water was added. The residue obtained was dried again in an oven at a temperature of 80°C for 30 minutes.

Delignification Percentage

Based on SNI 0492:2008, the determination of lignin content can be done using the Klason method, where the initial sediment weight is divided by the dry weight after being ovened. The percentage of lignin can be calculated using eq. (1):

$$\%\ Delignification\ Percentage = \frac{A}{B} \times 100\% \quad (1)$$

RESULTS AND DISCUSSION

In this investigation, the per cent delignification achieved with lignin removal products was reported to be between 15.42%-20.25% from water hyacinth stems with the aid of microwave-assisted extraction (MAE), as seen from Table 2. According to Chaiwarit *et al*. (2022), the water hyacinth stem lignin composition was 25% while this study found that the lignin content fell below that of the original composition from the water hyacinth stems. The highest percentage of delignification was observed at a transmission time of 10 minutes and a transmission power of 500 watts, and this value was 20.25%. This was due to the microwave length that optimally dissolved the lignocellulose bonds within a certain time, which resulted in the best removal of the subject of lignin. The smallest percentage of delignification was recorded at 10 and with a power of 358, the result being 15.42%, which suggests that the microwave length and duration have not been able to ensure optimal lignocellulose-breaking effects.

Table 2. Percentage Delignification Response from the interaction of the Irradiation Time Factor (x1) with the Irradiation Power Factor (X2).

3	15	400	15,59
4	15	600	17,94
6	17	500	19,45

Table 3 shows the results of the p-value analysis of variance (ANOVA) for the percentage of delignification of water hyacinth stems. In the interaction response, the reality is not significant; this is because the resulting p-value is more than 0.05, namely 0.121. Where if the P-value > F is more than 0.05, then statistically the process fails to reject the null hypothesis. The null hypothesis states that there is no significant relationship between the independent variables (Piry *et al*., 2024).

Table 3. ANOVA CCD Optimization In Quadratic Model

Factor	SS	df	MS	F value	p-value Prob > F	
Model	37,2936	4	37,2936	82.873,08	0,01819	*Significant*
Linear	15,1643	2	15,1643	33.697,59	0,009939	*Significant*
X_1	6,1708	1	6,1708	13.712,97	0,005436	
X_2	8,9935	1	8,993	19.985,59	0,004503	
Square	22,1293	2	22,1293	49.175,49	0,008251	*Significant*
X_1^2	8,8007	1	8,8007	19.557,14	0,004552	
X_2^2	13,3286	1	13,3286	29.618,35	0,003699	
Interaction X_1X_2	0,0121	1	0,012	0,0222	0,121282	*Not Significant*
Error	2,17960	4	0,545			
Lack of fit	2,179	3	0,726	1.614,18	0,018204	*significant*
Pure error	0,000045	1				
Cor Total	33,09209	9				

X1 = Irradiation Time (minutes), X2 = Irradiation Power (watts), Y = Delignification Percentage (%), df = Degree of Free, SS = Sum of Squares, p-value ($p<0.05$).

Verification of the relationship between independent variables and the percentage of delignification can be expressed in the form of sample distribution in the regression model in Figure 1. The purpose of each predictive model is to minimize the difference between the observed value and the predicted value. Figure 1. shows the distribution of samples 2, 7, 8, 9, and 10, which are close to the regression line. So it can be said that each sample has a match between the results according to the prediction model and is significant with the existing theory. The addition of transmission power and time will also increase the percentage of lignin removal. Microwaves can absorb the lignin surface so that lignocellulose polymer degradation occurs so that lignin can be extracted, and the process is formed linearly with the transmission time. The greater the transmission power (watts) and the transmission time period (minutes), the greater the percentage of delignification produced. The optimum value of the percentage of delignification was obtained at a power of 500 watts with a time of 10 minutes of 20.25%.

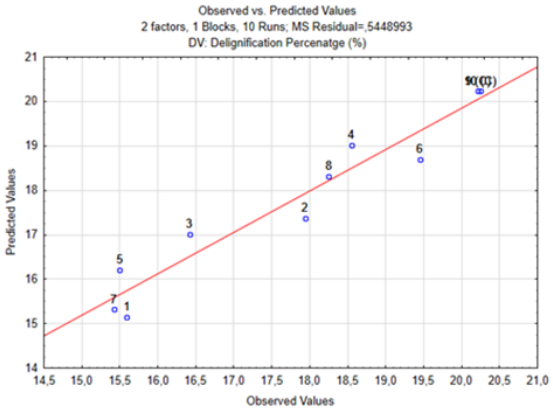

Figure 1. Graph of the relationship between Observation Values - Predictions in Linear Regression

Table 4. Regresseion Equation Responsse

Response	Quadratic Poynomial Model Equation Response	Standar Error	R^2
Y =	35,0604 – 0,5448993 X_1 – 0,0555 X_1^2 + 1,2858 X_1 – 0,0002 X_2^2 + 0,1814 X_2		0,93414

Table 4 shows the model response formed by a 2nd order polynomial equation with the resulting R^2 of 0.93414. The model equation has been modified by ignoring the interaction value between the transmission time (X1) and the transmission power (X2) / 1L by 2L. This is because the F value is greater than the F table, where 0.0221 < 0.1925, so that H0 is accepted, where the relationship between the 2 variable factors does not have a significant average difference.

Figure 2. shows the Pareto diagram of the influence between quadratic, linear, and interaction effects on the results of the delignification percentage, where the main cause is the quadratic emission power with a value of 4.945, with a p-value of 0.5. All independent variables pass the p-value limit other than the interaction relationship variable, so that the interaction variable does not have a significant effect on the model response.

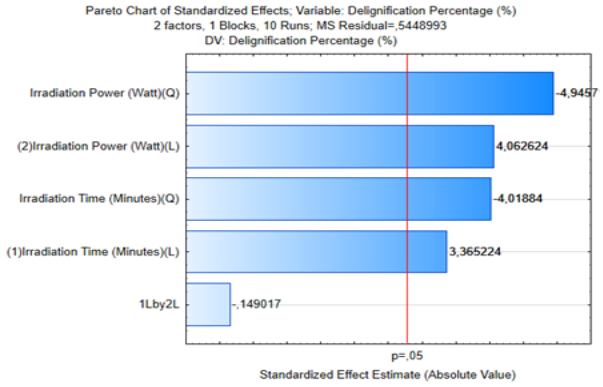

Figure 2. Pareto diagram of the influence of variables

Figure 3. shows a 3D fitted surface graph of the relationship between the dependent variable (percentage delignification) and the independent variables (emission time and emission power). The X and Y axes represent two independent variables, namely emission time and emission power. The Z axis represents the predicted value of the dependent variable, namely percentage delignification. The

sample point that approaches the dark color is the experiment that has a large influence and conformity to the theory that occurs. There is a gradient in the distribution of experimental points, where if there is a decreasing gradient, there is a change in the lignin percentage results.

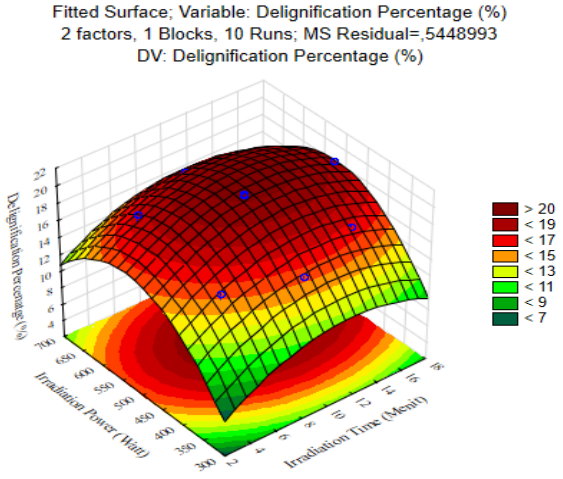

Figure 3. 3D Graph of Process Variable Interaction

Table 4 Verification of Optimum Delignification Process Conditions Using MAE

Factor	Observed Minimum Value	Observed Optimum Value	Observed Maximum Value
Irradiation Time (Minutes)	3	11,5	17
Irradiation Power (Watt)	358,6	530,5	641,4

Model verification and optimum conditions are needed for empirical needs in future research. The determination of the optimum point of the delignification process is found by comparing the interpretation of all influential variables and the response of the analysis results carried out by STATISTICA. Table 4 shows the observed optimum value obtained with the time and transmission power, respectively, of 11.5 minutes and 530.5 watts, so that this value is the largest percentage of delignification.

CONCLUSION

The delignification process with the help of Microwave Assisted Extraction (MAE) was used to obtain cellulose in water hyacinth, where the optimum conditions obtained were at a time of 11.5 minutes and a transmission power of 530.5 watts with a delignification percentage of 20.25% which was seen at the threshold of the model prediction value.

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