OPTIMIZATION OF MICROWAVE-ASSISTED HYDROTROPIC FRACTIONATION OF RAMIE (*Boehmeria nivea* L.) USING RESPONSE SURFACE METHODOLOGY

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The optimization of the fractionation of ramie fiber using microwave assistance and a urea solution as the hydrotropic agent was evaluated in this study through response surface methodology (RSM). A central composite design, a commonly used matrix design in RSM, was implemented. The independent variables for this research included the liquid-to-solid ratio, hydrotrope concentration, and duration of hydrotropic fractionation. At the same time, the yield, lignin content, and cellulose content of the pulp served as the dependent variables. A series of second-degree polynomial equations were developed to establish the relationship between pulp yield, lignin content, and cellulose content with the parameters of the fractionation process, including both the antagonistic and synergistic effects of these parameters. The significance of the process parameters was determined through the analysis of variance, and information regarding the most influential variables on pulp yield, lignin content, and cellulose content was derived from Pareto charts. The highest yield of the fractionation process was achieved with microwave-assisted fractionation of ramie fiber at a liquid-to-solid ratio of 43.40%, a hydrotrope concentration of 36.82%, and a fractionation time of 73.63 minutes. The lowest lignin content in the pulp was obtained during microwave-assisted fractionation at a liquid-to-solid ratio of 25.59%, a hydrotrope concentration of 3.18%, and a fractionation time of 6.36 minutes. Conversely, the maximum cellulose content in the pulp was realized during microwave-assisted fractionation at the same liquid-to-solid ratio of 43.40%, hydrotrope concentration of 36.82%, and fractionation time of 73.63 minutes.

Keywords: hydrotropic fractionation, ramie, RSM, CCD, microwave

INTRODUCTION

The finiteness of fossil resources, the growing global demand for energy, materials, and chemicals, as well as environmental issues related to the utilization of fossil-based products are the driving forces for the development of renewable source-based fuels, materials and chemicals production.[1] Lignocellulosic biomass, which is biologically photosynthesized from carbon dioxide and water by solar energy, is considered a potential renewable resource for fuels, materials, and chemicals industries. Lignocellulose biomass is also prized for its various remarkable characteristics, such as its abundance, degradability, and reactivity. As long as solar energy exists, lignocellulose production will never dry up, thus it is considered as an inexhaustible resource. It is estimated that 181.5 billion tons of lignocellulose is globally produced by nature every year, while only 4.5% of it is already utilized in different application areas.[2]

Polymeric carbohydrates and aromatic polymers are the two major components of lignocellulose biomass. The two polymeric carbohydrates of lignocellulose are cellulose and hemicelluloses, which constitute approximately 35%–50% and 20%–35%, respectively, while lignin as the aromatic polymer constitutes approximately 10%–25%. Upon fractionation, depolymerization, and chemical upgrading, the three major lignocellulose components can be transformed into various biomass-derived platform fuels, chemicals, and materials. Syngas products, hydrocarbons, phenols, oxidized products, and macromolecules are examples of lignin-derived potential products obtained by various processes, such as pyrolysis, combustion, hydrolysis, gasification, hydrogenolysis, thermolysis, and oxidation.[3] Hemicelluloses have also gained growing attention, since they present interest in the fields of bioactive polymers, biocomposites, biofilms, hydrogels, ester derivatives, thermoplastic derivatives, emulsion stabilizers, and possible biomedical applications.[4] Furthermore, versatile cellulose can be directly utilized as pulp, paper, fiber, textile, micro- and nano-cellulose, as well as substrate for cellulase production, which is broadly utilized in food, textile, and paper processing. Various cellulose derivatives, with a wide-ranging spectrum of applications, are obtained through esterification and etherification reactions.[5] Moreover, the sugars in cellulose become the intermediate sustainable feedstock for fuel production as aviation fuel[6] and ethanol,[7] and for various biomass-derived platform chemicals.

The development of the lignocellulose fractionation methods is mainly promoted by the prospective broad spectrum of potential applications of lignocellulose components. Nevertheless, the complexity of the lignocelluloses' hierarchical structure has been considered as a critical challenge in lignocellulose fractionation. Basically, lignocellulose fractionation can be performed by various methods: physical, chemical, physicochemical, and biological processes. Mechanical methods, microwave irradiation, and low-frequency ultrasound irradiation are examples of physical fractionation.[8] Chemical fractionation processes are conducted by utilizing strong acids, alkalis, organic solvents, ozone, ionic liquids, and hydrotropes. Moreover, steam explosion, liquid hot water treatments, ammonia fiber explosion, supercritical fluid treatment, and ammonia recycled percolation are some of the physicochemical processes;[9] while biological processes are commonly conducted by using either enzyme-producing microorganisms or purified enzymes.

Ligninolytic enzymes, such as copper-containing laccase (Lac), lignin peroxidase (LiP), feruloyl esterases, aryl alcohol oxidase (AAO), manganese peroxidase (MnP) and versatile peroxidase (VP) are the major enzyme responsible for the lignocellulose fractionation process.[10,11] *Schizophyllum commune* ENN1, *Ischnoderma resinous* AW04-02, *Trametes versicolor*, *Fomitella fraxinea* AW 04-03, and *Trametes hirsuta* NBRC are fungi of the Basidiomycetes family utilized in biofractionation of lignocelluloses.[10–12] Moreover, Sachez *et al.*[10] and Kumar *et al.*[11] mentioned that bacteria from *Pantoea*, *Pseudomonas*, *Aeromonas*, *Azotobacter*, *Flavobacteria*, *Xanthomonas*, *Bacillus*, and *Cellulomonas* strains are the typical enzyme-producing microorganisms used for lignocellulose fractionation purposes.

Among the existing and applicable methods available, chemical methods are the most common ones applied in lignocellulose fractionation. Some of them utilize harsh chemicals and severe treatment conditions, such as acid and alkali treatment. Those methods involve the use of high amounts of energy and are thus considered harmful for the environment. However, as the concept of an environmentally friendly process stimulates the use of lignocellulose components extracted from renewable raw materials, the implementation of sustainable processing techniques is gaining more interest in recent research. Therefore, in order to develop an economical and ecological process, more recent research within this area has aimed to optimize the so-called green fractionation processes.

Hydrotropic-based lignocellulose fractionation has been considered a green technique in the field of biomass fractionation.[13] Hydrotropic chemistry refers to the use of an approved surfactant to facilitate the dissolution of many sparingly soluble organic compounds, as well as water-insoluble compounds, including lignin, in aqueous solutions.[14] Sodium xylene sulfonate,[15,16] sodium cumene sulfonate,[15,16] p-toluenesulfonic acid (p-TsOH),[13] sodium benzoate,[17] and urea are some of the hydrotropes utilized in the lignocellulose fractionation, for biomasses such as rice straw, wheat straw, sugarcane bagasse and oat hulls.

Hydrotropic-based lignocellulose fractionation is influenced by several process parameters, thus parameter process optimization is an essential step during hydrotropic fractionation. Process optimization can be carried out by several methods. One of them is one factor at a time (OFAT) design, in which one factor is varied, while the other factors are kept fixed. Unfortunately, OFAT is considered inefficient, as it involves a large number of experiments, as well as unreliable, leading to false optimal conditions.[18] Recently, many studies applied statistical experimental designs for process optimization. Response surface methodology (RSM) is one of the statistical-based experimental designs commonly applied. It allows simultaneous effects analysis of multiple independent parameters, while only a small number of experiments are required.

The current research focused on optimizing the microwave-assisted fractionation of ramie fiber using urea solution as a hydrotropic agent. The goal of the fractionation process is to produce ramie fiber pulp with increased cellulose content, compared to the untreated material, while minimizing the presence of lignin in the ramie fiber. For this study, ramie (*Boehmeria nivea* L.) was chosen as raw material since it is an easily cultivated plant,[19] while urea is considered a good solubilizing agent for lignin.[20]

EXPERIMENTAL

Materials

Ramie fiber (RF) used in this study was purchased from a local ramie plantation and ramie fiber processing unit in Wonosobo, Central Java. The ramie was air-dried, chopped, and ground into powder. The dried RF powder was then sieved with a 60-mesh and used as the feedstock for the microwave-assisted hydrotropic fractionation. All the chemicals (sulphuric acid, *etc.*) used during the study were of analytical grade (Merck, Sigma Aldrich).

Design of experiments

RSM has been ascertained as a reliable tool for process parameters optimization purposes.[21,22] The central composite design, one of the matrix designs commonly applied in RSM, other than Box-Behnken design and two-level full factorial design, was applied in the present study.[23] Hydrotrope concentration, reaction time as well as the liquid-to-solid ratio are some of the dominating parameters in the fractionation process.[24] Thus, those variables were set as independent variables, while yield, lignin content, and cellulose content of the pulp were taken as dependent variables. The design and analysis of the experiment were carried out by Statistica 8 software; the factors and their level were tabulated in Table 1. The general expression for a total number of experiments for central composite design is given as follows:

$$\mathrm{N} = 2^{\mathrm{n}} + 2\mathrm{n} + \mathrm{n_c} \tag{1}$$

where n represents the number of independent variables and $\mathrm{n_c}$ represents the number of replicate runs. The first, second, and third terms in Equation (1) represent the factorial, axial, and replicate runs, respectively. The number of independent variables in this study was 3, thus there are 8 factorial points and 6 axial points. Statistica 8 set the number of replicates of 2, hence according to Equation (1), a total of 16 experiments were obtained. The alpha (α) taken in this experimental design was 1.682, which was obtained by means of the following relation:[23]

$$\alpha = 2^{\frac{\mathrm{n}}{4}} \tag{2}$$

Microwave assisted hydrotropic fractionation

10 grams of chopped ramie fiber (dry basis) was inserted into a three-neck glass flask, followed by the addition of urea solution as a hydrotropic agent. The microwave power level was set at a medium level, which represents a power level of 119.7W. The microwave-assisted hydrotropic fractionation was conducted for a certain duration in accordance with the experimental variations listed in Table 1. The solid residue obtained from the hydrotropic fractionation was separated by the applied filtration process. Eight hundred milliliters of hot water was poured into the residue for washing purposes.

The solid residue was then oven-dried at 105 °C and the weight of the pulp was recorded for pulp yield calculation.

Table 1
Levels and codes of factors for central composite design

Factors	Symbol	Unit	Low	High	Value of code
Liquid to solid ratio	x_1	v/w	30	40	-1=30; 0=35; 1=40
Hydrotrope concentration	x_2	%	10	30	-1=10; 0=20; 1=30
Time	x_3	Min	20	60	-1=20; 0=40; 1=60

Composition analysis

The lignin content was evaluated based on the content of acid-insoluble lignin, determined gravimetrically as Klason lignin, following the Indonesian National Standard SNI: 0492:2008. In summary, a 72% sulfuric acid solution was used to hydrolyze the free extractive sample. The hydrolysis took place at 30 °C for 2 hours. After hydrolysis, the sample was diluted with distilled water until the concentration of sulfuric acid reached 3%. The hydrolyzed mixture was then treated in a three-neck glass flask at 100 °C for 4 hours. Prior to filtering the mixture, a filtering crucible was ignited and tarred. The residue captured in the crucible was subsequently dried at 105 °C until a constant weight was achieved.

The cellulose content was measured following SNI: 0444-1989. A one-gram sample of free extractives was treated with 15 mL of a 17.5% sodium hydroxide solution at 20 °C and stirred for 1 minute. Afterward, 10 mL of the 17.5% sodium hydroxide solution was added, and the mixture was agitated for 45 seconds. Another 10 mL of the 17.5% sodium hydroxide solution was introduced, with 10 seconds of agitation following the addition. The mixture was then allowed to settle in a water bath for 3 minutes, followed by the addition of another 10 mL of the sodium hydroxide solution. The mixture was agitated for 10 minutes, and this sequence was repeated at intervals of 2.5, 5, and 7 minutes, after which it underwent 30 minutes of incubation at room temperature. Subsequently, 100 mL of distilled water was added to the solution, which was then incubated for an additional 30 minutes at room temperature. The mixture was filtered, and 400 mL of distilled water was used to wash the solid residue. Finally, it was dried and weighed to determine the cellulose content.

Data analysis

The least square method of multiple regressions was applied in the analysis of the experimental data responses. The correlation of the responses as a function of the independent variables was expressed as a second-order polynomial equation (3):

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i x_i + \sum_{i=1}^{k} \sum_{j=i+1}^{k} \beta_{ij} x_i x_j$$

where y is the predicted response (pulp yield, lignin content, and cellulose content), k is the number of independent variables (factors), x_i and x_j are the coded independent variables, β_0 is the constant coefficient, and β_i, β_{ii}, and β_{ij} are the regression coefficient of linear, quadratic and interaction term, respectively.

Statistica 8 statistical software was used for regression, estimation of the coefficient parameter, and graphical analysis of the experimental data. The fitness of the model was evaluated by considering the value of the coefficients of determination (R^2). The identification of the significant effects and the statistical significance of the model were based on variance analysis (ANOVA). The regression analysis produced a fitted quadratic polynomial equation, which was then used to generate response surfaces and contour plots of the data by using the mentioned software, which also gave a Pareto chart, two-dimensional contour plots of the response models, as well as the observed vs predicted responses.

RESULTS AND DISCUSSION

Chemical composition of untreated ramie fiber

The chemical composition of ramie fiber found in this study is tabulated in Table 2, in comparison with the data found in the literature. The constituents of ramie fiber vary among different samples, with cellulose, hemicelluloses, and lignin percentages ranging from 60.30% to 86.9%, 5% to 18%, and 0.6% to 7.32%, respectively. Factors, such as fiber pretreatment method, like decortication and degumming, as well as the geographical region of the plant growth, contribute to variations in the chemical compositions of ramie fiber. In this study, the cellulose, hemicelluloses, and lignin contents of ramie fiber were found to be 60.30%, 16.80%, and 7.32%, respectively. The cellulose content identified in this research was lower than that reported in various other studies. Conversely, the lignin content in this ramie fiber sample was significantly higher than in others. Nevertheless, since all the components of lignocellulose hold value and can be transformed into a variety of products, the chemical composition of ramie presented here offers insights into its potential uses.

The broad range of possible direct applications for lignocellulosic components, particularly cellulose, and its derivatives, has rendered the fractionation and optimization of ramie fiber an intriguing topic for research. One purpose of lignocellulose fractionation is to enhance the uniformity of the raw material based on its lignocellulosic components and structural characteristics. The pulp obtained through hydrotropic fractionation is anticipated to contain a higher cellulose level compared to untreated ramie fiber.

Table 2
Chemical composition of ramie fiber

References	Components			
	Cellulose (%)	Hemicelluloses (%)	Lignin (%)	Ash (%)
This work	60.30	16.80	7.32	0.27
[19]	68-76	14-18	4-7	NA
[25]	71.09	12.11	1.06^{a}	NA
[26]	72.40	15.89	1.7	NA
[25]	86.9^{b}	14-17.9	0.8-1.5	NA
[27]	72	5-16.7	0.6-0.8	NA

^{a}acid insoluble lignin; ^{b}alfa cellulose

Additionally, lignocellulose fractionation serves to break down and eliminate lignin, meaning that pulp derived from microwave-assisted hydrotropic fractionation is expected to have lower lignin content than that found in the original ramie fiber.

Microwave assisted hydrotropic fractionation of ramie fiber

Ramie fiber fractionation was conducted by applying urea as the hydrotropic compound for lignin disruption. Kunz *et al.*[28] mentioned that urea is considered as a good hydrotrope, and later Wang *et al.*[20] stated that urea has a great ability in lignin dissolution processes. All 16 designed experiments for the microwave-assisted hydrotropic fractionation of ramie fiber were carried out as per the experimental design shown in Table 3. The results of the microwave-assisted hydrotropic fractionation process, which included yield, lignin, and cellulose content of the pulp, are also summarized in Table 3.

Table 3
Experimental data for microwave assisted hydrotropic fractionation of ramie fiber

Run	Coded variables			Decoded variable			Responses		
	x_1	x_2	x_3	Liquid to solid ratio x_1	Hydrotrope concentration (%) x_2	Time (min) x_3	Yield (%)	Lignin (%)	Cellulose (%)
1	-1	-1	-1	30	10	20	90.31	6.30	71.03
2	1	-1	-1	40	10	20	93.97	7.15	71.47
3	-1	1	-1	30	30	20	87.06	8.02	69.40
4	1	1	-1	40	30	20	85.31	9.71	70.80
5	-1	-1	1	30	10	60	81.93	4.65	67.99
6	1	-1	1	40	10	60	86.29	6.31	64.68
7	-1	1	1	30	30	60	85.03	8.42	65.64
8	1	1	1	40	30	60	85.51	7.19	61.07
9	0	0	-α	35	20	6.364	86.57	9.40	69.05
10	0	0	+α	35	30	73.63	81.29	7.14	78.64
11	0	-α	0	35	3.18	40	88.15	3.37	60.78
12	0	+α	0	35	36.82	40	81.27	6.90	56.80
13	-α	0	0	25.59	20	40	80.04	7.74	62.29
14	+α	0	0	43.408	20	40	81.87	8.83	60.43
15	0	0	0	35	20	40	78.60	7.72	64.91
16	0	0	0	35	20	40	78.60	7.72	64.91

Moreover, it was stated that the mode of action of urea as a hydrotropic agent in the fractionation of lignocellulosic biomass relies on the synergistic effect of strong hydrogen bonding with lignin functional groups and the disruption of intermolecular hydrophobic interactions among lignin macromolecules. This dual mechanism facilitates the partial solubilization of lignin without the use of harsh reagents or organic solvents, thereby enhancing the environmental sustainability and selectivity of the process.[29,30]

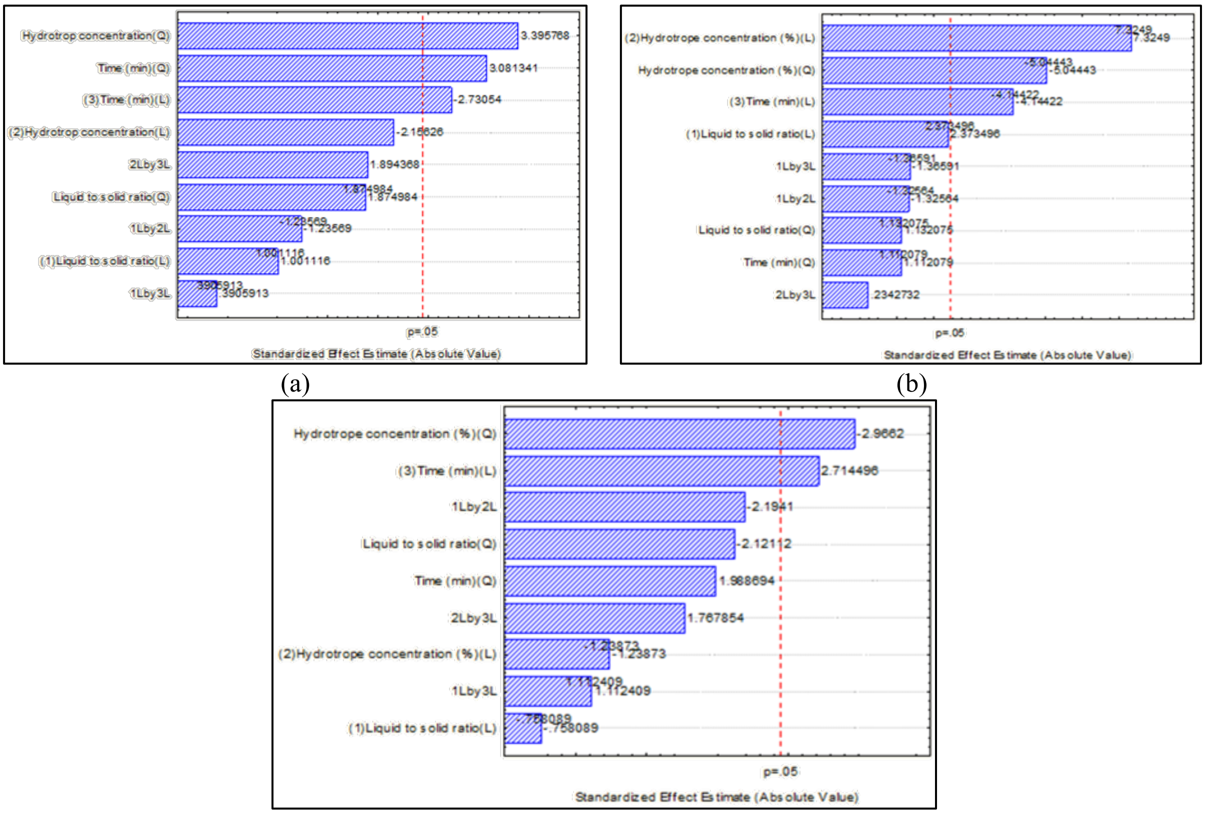
The regression technique of RSM presents an empirical type mathematical relationship between the desired responses and the parameters affecting the process, without taking into account the detailed mechanism involved in the process.[31] As an example, the weight of lignin in the extract was correlated with the fractionating agent concentration, fractionation time and the temperature in soda fractionation of ramie fiber[22] and in bidirectional fractionation of ramie fiber.[31] In this work, pulp yield, lignin content and cellulose content of the pulp, coded as y_1, y_2 and y_3, respectively, are the desired responses, whereas liquid-to-solid ratio, hydrotrope concentration and reaction time are the parameters affecting the fractionation process, coded as x_1, x_2 and x_3, respectively. The general form of the relation between the fractionation process parameters and the pulp yield, lignin content and cellulose content is given by a second degree polynomial equation, as shown through Equations (4), (5) and (6), correspondingly:

$$y_1 = 78.26 + 1.43x_1 + 3.27x_2 - 3.09x_3 + 5.92x_1^2 - 3.92x_2^2 + 5.37x_3^2 - 2.32x_1x_2 + 0.73x_1x_3 + 3.55x_2x_3 \quad (4)$$

The impacts of each process parameter on the responses are shown through the negative and positive signs of the regression coefficients in the polynomial equations, indicating antagonistic and synergistic effects, respectively. The pulp yield, as represented in Equation (4), is positively influenced by the linear term of the liquid-to-solid ratio (x_1) and hydrotrope concentration (x_2), as well as by the quadratic term of the liquid-to-solid ratio (x_1^2) and the quadratic term of fractionation time (x_3^2). The interaction between the liquid-to-solid ratio and fractionation time (x_1x_3), in addition to the interaction between hydrotrope concentration and fractionation time (x_2x_3) also produces a synergistic effect on the pulp yield. Furthermore, the linear term for fractionation time (x_3), the quadratic term for hydrotrope concentration (x_2^2), and the interactive effect of the liquid-to-solid ratio and hydrotrope concentration (x_1x_2) exert an antagonistic influence on the pulp yield. Additionally, the Pareto chart illustrated in Figure 1(a) indicates that the quadratic term for hydrotrope concentration, along with the linear and quadratic terms for fractionation time, are significant factors affecting the yield of the ramie fiber during the hydrotropic fractionation process.

$$y_2 = 15.71 + 0.7x_1 + 0.4x_2 + 2.17x_3 - 1.81x_1^2 - 1.23x_2^2 + 0.40x_3^2 - 0.51x_1x_2 - 0.52x_1x_3 + 0.09x_2x_3 \quad (5)$$

The synergistic effect of the linear term of liquid-to-solid ratio (x_1), hydrotrope concentration (x_2), and fractionation time (x_3) towards the lignin remaining in the pulp is shown in Equation (5). The quadratic term of fractionation time (x_3^2) also gives a positive effect on the lignin content, in addition to the interaction effect of hydrotrope concentration and fractionation time (x_2x_3). Moreover, Equation (5) also shows the antagonistic effect of the quadratic term of liquid-to-solid ratio (x_1^2), the quadratic term of hydrotrope concentration (x_2^2), the interaction effect between liquid-to-solid ratio and hydrotrope concentration (x_1x_2), as well as the interaction effect between liquid-to-solid ratio and fractionation time (x_1x_3) toward the lignin content. The disruption of the original heterogeneous structure of lignocellulose materials, which facilitates subsequent processes,[32] the dismantling process on lignin, and the lignin removal that obstructs the following processes are some of lignocellulose fractionation functions. Thus, it is expected to get a lower pulp lignin content.



(a) (b)

(c)

Figure 1: Pareto chart of standardized effect of: (a) yield, (b) lignin concentration, and (c) cellulose concentration of microwave assisted hydrotropic fractionation of ramie fiber

The Pareto charts for lignin and cellulose content of the pulp shown in Figure 1 (b and c) indicate that the linear and quadratic terms of hydrotrope concentration, as well as the linear term of fractionation time significantly affect the lignin content, while the cellulose content is significantly affected by the quadratic term of hydrotrope concentration and the linear term of fractionation time. Morone *et al.*[33] conducted an OrganoCat for rice straw pretreatment, where time, pressure, and temperature were the process parameters evaluated for their significance. It was found that the lignin removal was significantly affected by all the main effects and their interactions, while the main effect of temperature, time, and the interaction effect of time and pressure were significant for cellulose recovery.

$$y_3 = 45.33 - 1.34x_1 - 4.55x_2 - 2.19x_3 - 6.37x_1^2 + 4.80x_2^2 + 4.27x_3^2 - 5.07x_1x_2 + 2.57x_1x_3 + 4.08x_2x_3 \qquad (6)$$

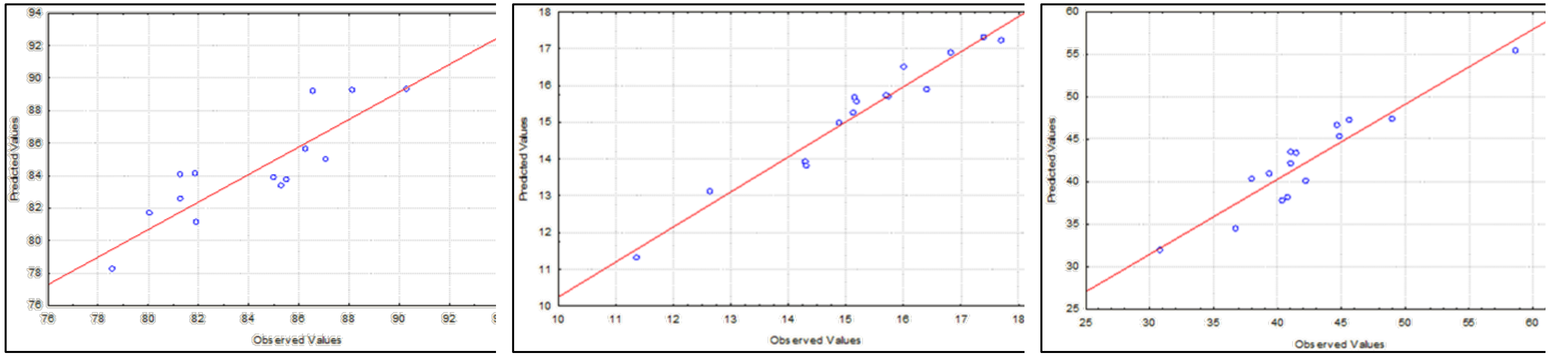
Furthermore, the negative and positive signs of the regression coefficients in the polynomial equation shown in Equation (6) also inform us of the synergistic and antagonistic effects of each process parameter. The synergistic effect is presented by the quadratic term of hydrotrope concentration (x_2^2), the quadratic term of fractionation time (x_3^2), the interaction effect between liquid-to-solid ratio and fractionation time (x_1x_3), and the interaction effect of hydrotrope concentration and fractionation time (x_2x_3). Meanwhile, the other ones act as antagonists. The verification of the impact of each term in the empirical mathematical relationship, as presented by Equations (4) to (6), was carried out by employing variance analysis (ANOVA). The results of the variance analysis were tabulated in Table 4. The variance analysis shows the significance of the model, which is evaluated through its probability value (p-value) and its F-test or Fisher's test. In such cases, it is considered significant if the p-value is lower than 0.05 and if the value of F is larger than its corresponding coefficient. A p-value lower than 0.05 indicates that only a 5% chance that a 'Model F- value' may perhaps ascend due to noise. Meanwhile, the large F value depicts a good prediction of the experimental data that, based on the ratio of the mean square of group variance due to error, measures the variance of data about the mean.[23,34] The quadratic term of hydrotrope concentration, the linear term of fractionation time, and quadratic fractionation time are the three parameters that are observed to be significant for the pulp yield model with p-values less than 0.5. The linear and quadratic terms of hydrotrope concentration, as well as the linear term of fractionation time, are observed as the significant parameters towards the lignin content of pulp, while the quadratic term of hydrotrope concentration and the linear term of fractionation time are the two significant parameters for the cellulose content of pulp.

Lack of fit, the coefficient of determination (R^2), and adjusted-R^2 are model analysis parameters that can be applied to assess the goodness of the model fit. It was stated that the R-squared levels should be higher than 0.75 ($R^2 > 0.75$). The R^2 value is always between 0 and 1, and its magnitude indicates the suitability of the model.[18] In this work, the coefficient of determination (R^2) for the pulp yield, lignin content, and cellulose content model was 0.84, 0.95, and 0.88, correspondingly. This implies that 84%, 95%, and 88% of the experimental data confirm compatibility with the data predicted by the model for pulp yield, lignin content, and cellulose content. These values of the coefficient of determination indicate that the regression model accurately fits the data and can predict future outcomes. This finding was also supported by the illustration of the observed vs predicted response for yield, lignin concentration, and cellulose concentration of microwave assisted hydrotropic fractionation of ramie fiber in Figure 2.

Response surface methodology provides the response surface plot and contour plot, which may serve as visualization tools for model equation prediction. The theoretical three-dimensional plot and graphical representation of the regression equation, which shows the relationship between the response and the independent variables is called the response surface plot. The response surface plot was obtained by using Statistica 8 software. Figures 3-5 present the response surface plots for microwave-assisted hydrotropic fractionation of ramie fiber for the interaction effects between liquid: solid ratio and hydrotrope concentration, and also between liquid: solid ratio and time on pulp yield, lignin content, and cellulose content.The analysis conducted by using Statistica 8 also offered the value of the minimum and the maximum responses. The yield of the fractionation process was found to be maximum at microwave-assisted fractionation of ramie fiber conducted at the liquid-to-solid ratio of 43.40%, hydrotrope concentration of 36.82%, and fractionation time of 73.63 min.

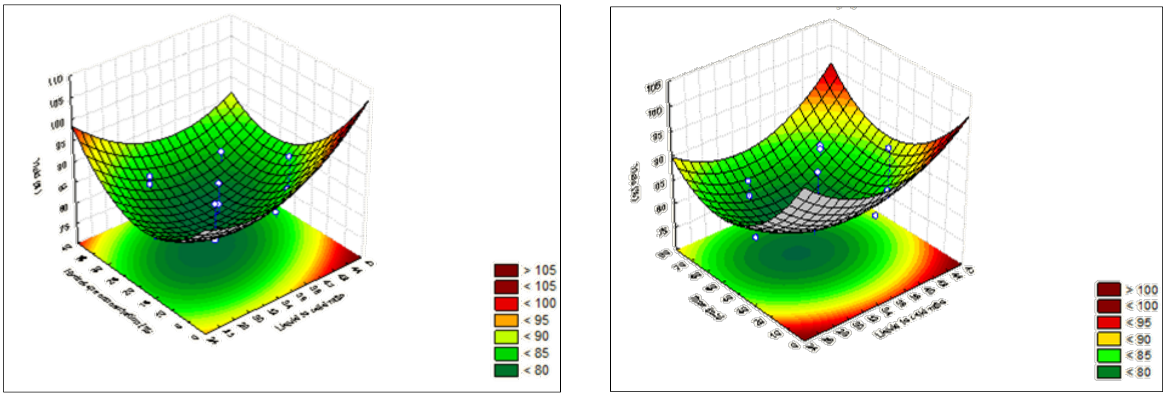
Table 4
Analysis of variance (ANOVA) for response surface quadratic model

Source	Yield					Lignin					Cellulose				
	SS	Df	MS	F	P	SS	df	MS	F	p	SS	df	MS	F	p
x_1 (L)	7.0	1	7.0	1.0	0.3	1.6	1	1.6	5.6	0.06	6.1	1	6.1	0.5	0.5
x_1 (Q)	24.7	1	24.7	3.5	0.1	0.3	1	0.3	1.2	0.30	48.1	1	48.1	4.5	0.08
x_2 (L)	32.7	1	32.7	4.6	0.0	16.1	1	16.1	53.5	0.00	16.4	1	16.4	1.5	0.26
x_2 (Q)	81.2	1	81.2	11.5	0.0	7.6	1	7.6	25.4	0.00	94.0	1	94.0	8.8	0.03
x_3 (L)	52.5	1	52.5	7.4	0.0	5.1	1	5.1	17.1	0.01	78.7	1	78.7	7.3	0.03
x_3 (Q)	66.9	1	66.9	9.4	0.0	0.3	1	0.3	1.2	0.31	42.2	1	42.2	3.9	0.09
x_1x_2	10.7	1	10.7	1.5	0.2	0.5	1	0.5	1.7	0.23	51.4	1	51.4	4.8	0.07
x_1x_3	1.0	1	1.0	0.1	0.7	0.5	1	0.5	1.8	0.22	13.2	1	13.2	1.2	0.31
x_2x_3	25.3	1	25.3	3.5	0.1	0.0	1	0.0	0.0	0.82	33.4	1	33.4	3.1	0.13
Error	42.3	6	7.0			1.8	6	0.3			64.1	6	10.6		
Total SS	276.5	15				39.5	15				541.6	15			



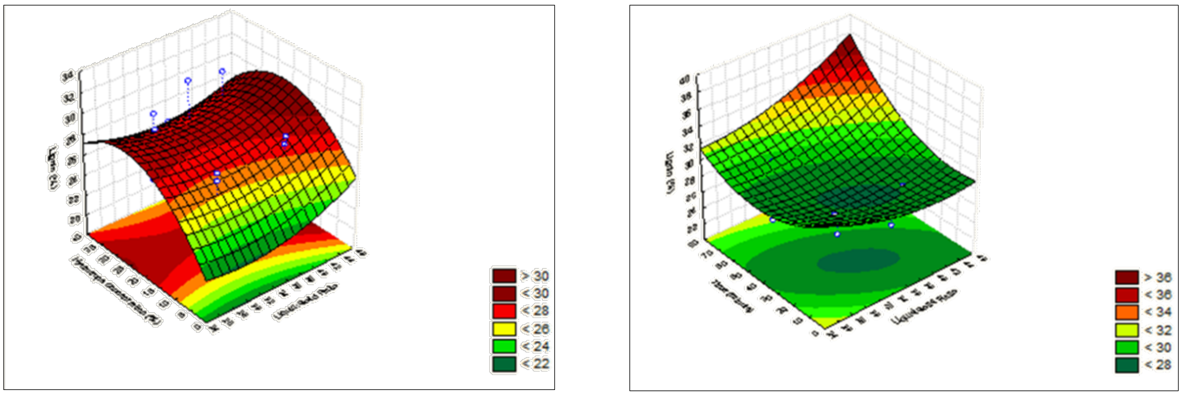
(a) (b) (c)

Figure 2: Observed *vs* predicted response: (a) yield, (b) lignin concentration, and (c) cellulose concentration of microwave assisted hydrotropic fractionation of ramie fiber



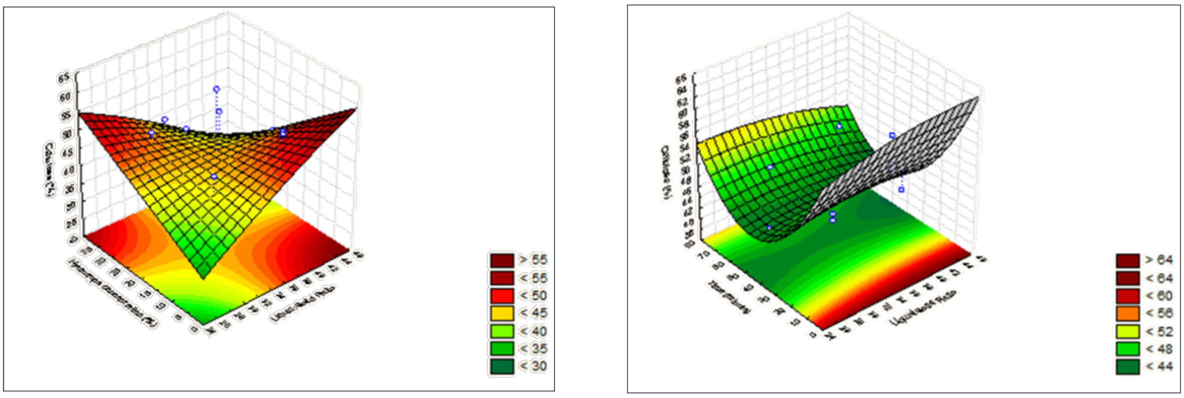
(a) (b)

Figure 3: Response surface plots for interaction effects on fractionation response: (a) liquid:solid ratio and hydrotrope concentration, (b) liquid:solid ratio and time on the pulp yield after microwave assisted hydrotropic fractionation of ramie fiber



(a) (b)

Figure 4: Response surface plots for interaction effects on fractionation response: (a) liquid:solid ratio and hydrotrope concentration, (b) liquid:solid ratio and time on lignin content after microwave assisted hydrotropic fractionation of ramie fiber



(a) (b)

Figure 5: Response surface plots for interaction effects on fractionation response: (a) liquid:solid ratio and hydrotrope concentration, (b) liquid:solid ratio and time on cellulose content after microwave assisted hydrotropic fractionation of ramie fiber

The lignin content of the pulp was found to be minimal after microwave-assisted fractionation of ramie fiber conducted at liquid-to-solid ratio of 25.59%, hydrotrope concentration of 3.18%, and fractionation time of 6.36 min, while the cellulose content of the pulp was found to be maximum when the microwave-assisted fractionation of ramie fiber was conducted at the liquid-to-solid ratio of 43.40%, hydrotrope concentration of 36.82%, and fractionation time of 73.63 min.

CONCLUSION

Response surface methodology allows for the establishment of a set of second-degree polynomial equations that relate responses, specifically pulp yield, lignin content, and cellulose content, to the parameters of the fractionation process. The results from regression analysis provide insights into the antagonistic and synergistic impacts of the process parameters on the fractionation responses. The analysis of variance indicates the significance of the process parameters, while the Pareto chart highlights the most influential variables affecting pulp yield, lignin content, and cellulose content. The fractionation process achieved its highest yield during microwave-assisted fractionation of ramie fiber at a liquid-to-solid ratio of 43.40%, a hydrotrope concentration of 36.82%, and a fractionation time of 73.63 minutes. The lignin content in the pulp was at its lowest during microwave-assisted fractionation of ramie fiber at a liquid-to-solid ratio of 25.59%, a hydrotrope concentration of 3.18%, and a fractionation time of 6.36 minutes. Conversely, the cellulose content of the pulp reached its peak during microwave-assisted fractionation of ramie fiber at a liquid-to-solid ratio of 43.40%, a hydrotrope concentration of 36.82%, and a fractionation time of 73.63 minutes.

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