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Optimization of microwave-assisted extraction of natural dyes from jackfruit wood (*Artocarpus heterophyllus* Lamk) by response surface methodology

Selfina Gala, Sumarno Sumarno and Mahfud Mahfud*

Department of Chemical Engineering, Institut Teknologi Sepuluh Nopember, Surabaya 60111, Indonesia

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Abstract

The microwave-assisted extraction method for extraction of natural dyes from plants is more effective than the conventional methods because of shorter processing time and less solvents use. The objective of this research is to optimize the effects of extraction time, microwave power and feed/solvent (F/S) ratio on yields of microwave-assisted extraction using Response Surface Methodology to obtain conditions optimal operation through a Face-centered Composite Design (FCCD). The Face-Centered Composite Design was carried out to minimize the number of experiments. The results showed that the maximum yield of the extraction using FCCD design are microwave power of 600 W, F/S ratio of 0.0564 g/mL and an extraction time of 50 minutes is 3.5747%. And the p-value of model and lack of fit are significant and not significant, respectively. Thus, the selected model can be used to represent the results of experiments of natural dye extraction using microwave-assisted extraction method. In addition, microwave-assisted extraction method has been proven effective in producing natural dyes with a relatively higher yield and in a shorter time.

Keywords: Jackfruit wood, Microwave-assisted extraction, Natural dyes, Optimization

1. Introduction

The use of synthetic colors increases with the development of the textile industry or the batik industry in Indonesia, which requires increasingly diverse dyes and results in an increase in the chemical content in industrial wastewater. Thus, industrial wastewater that contains dyes can be hazardous to health and the environment, because it involves toxins and carcinogens. Therefore, people begin to think negatively of artificial coloring using natural dyes that are non-objectionable, renewable, no carcinogenic, and environmentally friendly sources. The weakness of natural dyes is thinner than coloring and less practical use, but lately some sources of natural dyes have been able to produce dyes that are very beautiful color and economical price than using synthetic dyes [1]. Furthermore, dye textiles sourced from plants function as antimicrobials because they contain tannins that function as antimicrobials [2]. Indonesia as a tropical country has a lot of potential plants to be used as a source of raw materials for natural dyes. One of the plants that contain natural dyes (pigments) is jackfruit that is found in various regions in Indonesia, especially East Java. Jackfruit wood is widely used as material for furniture and buildings. In its processing, wood waste produces wood dust. Jackfruit wood waste is generally not utilized optimally, even though jackfruit wood contains several types, especially the yellow color. Inside jackfruit wood there is a content of tannins with morin type which can cause yellowing of textile materials [3]. The extraction method for producing natural dyes from plants can be obtained by maceration method (soaking). This method is still used in several industries from ancient times until now, especially the batik textile industry which is obtained from boiled raw materials with solvent of water (conventional methods) [4, 5]. In addition, in the laboratory, the extraction process of dyes using conventional methods is usually carried out with Reflux and Soxhlet. Even though, this method requires a relatively large number of solvents with low yields and very long time [3, 6-10]. Therefore, we need to encourage technological innovation in the extraction methods by using microwave as an main energy source with more optimal results in a relatively short time. In addition, this method is also referred to as "green technique" in the process of extracting natural dyes. Basically studies for extraction by microwave or so-called Microwave-Assisted Extraction (MAE) methods occurs when microwave heat up the material and solvent directly which results in local heating which encourages the breaking of walls plant cells and triggers the release of more coloring agents into the solvents [1]. Hence, MAE is an alternative methods that has great potential, especially when the material is extracted from plants [11]. Previous studies have described the need to develop a natural dye extraction process with a variety of plant species which are sources of renewable raw materials, good for health and the environment. Therefore, the extraction process that utilizes microwave as an alternative energy source and is an environmentally friendly technology, this research will be conducted to find the source of natural dyes by microwave-assisted extraction (MAE) method and to optimize the parameters that affect product yields such as time extraction, microwave power and feed to solvent (F/S) ratio. The surface response methodology (RSM) is a combined technique that describes statistical and mathematical methods to verify how certain variables will affect a process, and finally optimize it. The RSM method can reduce the number of experimental variables needed and to assess interactions between several variables [11]. Therefore, the objective of this research is to study the effect of extraction time, microwave power and feed to solvent (F/S) ratio to the yield on extraction of natural dyes from jackfruit wood waste by microwave-assisted

*Corresponding author. Tel.: +6281 5522 3477
Email address: mahfud@chem-eng.its.ac.id
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extraction (MAE) and optimize using Faced-centered Central Composited Design (FCCD) which is one of several Response Surface Methodology (RSM) models that is quite common to be used in several previous studies.

2. Materials and methods

2.1 Material

Jackfruit wood powder was used as the raw material to obtain natural dyes in this study. Firstly, the raw material is dried until the water content of it approximately 2%. Moreover, jackfruit wood that has been dried and then carried out the process of pounding and blending to the size of 35 mesh to 60 mesh and carried out sieving.

2.2 Microwave apparatus

The main equipment used in this study was an Electrolux microwave EMM2007X model with a frequency of 2.45 GHz, a maximum power of 800 Watts, the voltage of 220 V, the dimensions of the microwave with a height of 37.3 cm, width of 28 cm and the length of 46.1 cm. The extraction process was carried out in a three-neck flask glass reactor connected with a Reflux condenser.

2.3 Experimental procedure

The extraction was carried out using a commercial microwave (EMM2007X). The three-neck flask is mounted in the microwave and in the center of the flask is connected to a Reflux condenser that is mounted vertically (Figure 1). On the other side of the flask hole, it is placed a thermocouple to indicate temperature. The power and time control on the microwave has been installed. Firstly, the raw material (jackfruit wood powder) is weighed according to predetermined variables before being added with a constant volume of aquadest as solvent (200 mL) and put into a three-neck flask (extractor tank). Furthermore, the power and extraction time are set according to predetermined variables. After the extraction process is complete, the extracted results are put into a beaker for further filtering using a vacuum pump (vacuum filtering). The filtrate obtained is then concentrated by heating it to a hotplate with a temperature of 80°C to dry and become powder. Next weighing the resulting dye to calculate yield (%). The dyes are analyzed to determine yield (%) and compared with other extraction techniques.

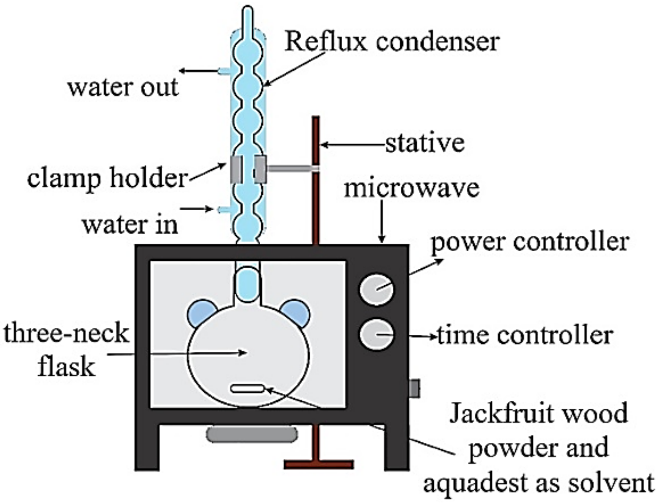

Figure 1 A schematic representation to extraction of natural dyes from jackfruit wood waste using microwave-assisted extraction

2.4 Experimental design

The experiment for extraction of natural dyes from jackfruit wood waste by microwave-assisted extraction was designed using Design-Expert software version 12.0 (Stat-Ease Inc., Minneapolis, MN, USA). This aims to minimize the amount of experimental data in the process of extracting natural dyes from jackfruit wood waste, and in order to obtain optimum conditions that can produce maximum yield. In the optimization using Face-centered Central Composite design (FCCD) for extraction of natural dyes from jackfruit wood waste using microwave-assisted extraction method, three factors are used: extraction time (minutes), microwave power (W) and F/S ratio (g/mL) which can be seen in Table 1. Where the microwave power ranges from 400 to 800 W, the F/S ratio ranges from 0.04 to 0.08 g/mL and the extraction time ranges from 10 to 50 minutes. In this study, the determination of the range of values for each factor to be optimized was carried out by conducting preliminary research. This is necessary in order to obtain the desired optimum value and the determination of the range of values for each factor to be optimized does not seem like trial and error.

Table 1 Factors in the experimental design

Name	Units	Coded			Actual		
		Low	Medium	High	Low	Medium	High
Extraction Time (A)	min	-1	0	1	10	30	50
Microwave Power (B)	W	-1	0	1	400	600	800
F/S Ratio (C)	g/mL	-1	0	1	0.04	0.06	0.08

After entering each factor value in the Design-Expert application version 12 (Stat-Ease Inc., Minneapolis, MN, USA) by selecting face-centered central composite design (FCCD), it was obtained 20 runs for optimization of natural dye extraction from jackfruit wood powder by microwave-assisted extraction method (Table 2). In addition, Table 2 also shows actual and predicted yield of natural dyes obtained from jackfruit wood waste extracted by microwave-assisted extraction method.

Table 2 Summary of experiment design by face-centered central composite design (FCCD)

Run	Coded-Factors			Responses (yield, %)		
	Extraction Time (min)	Microwave Power (W)	F/S Ratio (g/mL)	Observed	Predicted	Residual
1	0	0	0	3.0433	3.3845	-0.3412
2	1	0	0	3.3292	3.5269	-0.2349
3	-1	-1	1	2.1406	2.2363	-0.0957
4	0	0	0	4.0225	3.3845	0.6380
5	-1	0	0	3.3158	3.2617	0.0541
6	0	0	1	2.9619	2.9514	0.0105
7	0	0	0	3.0875	3.3845	-0.2970
8	0	0	0	4.0517	3.3845	0.6672
9	0	0	0	3.2423	3.3845	-0.1422
10	1	-1	1	2.7013	2.6515	0.0498
11	-1	-1	-1	2.3363	2.3084	0.0279
12	0	1	0	2.6700	2.8417	-0.1717
13	0	0	-1	3.0000	3.1913	-0.1913
14	-1	1	1	2.2619	2.2437	0.0182
15	1	1	1	2.4700	2.4527	0.0173
16	-1	1	-1	2.6663	2.6708	-0.0045
17	1	1	-1	3.0013	2.8604	0.1409
18	1	-1	-1	2.7313	2.7042	0.0271
19	0	0	0	3.2215	3.3845	-0.1630
20	0	-1	0	2.7508	2.7599	-0.0091

Coded value was finished to know the description of the individual independent variables effect on the response. The dependency of each experimental response is symbolized by Y and the model as follows:

$$y = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n} \beta_{ii} x_i^2 + \sum_{i=1}^{n} \sum_{j=i+1}^{n} \beta_{ij} x_i x_j + \varepsilon \quad (1)$$

Where β_0 is a term constant, β_i, β_{ii} β_{ij} are coefficients, ε is error factor, x_i and x_j is variable (A, B and C) and n is amount of variables. The coefficient is determined by multiple linier [12].

3. Results and discussion

3.1 Development of regression model

In the extraction of natural dyes from jackfruit wood waste using microwave-assisted extraction method, ANOVA analysis is carried out to identify the factors and important interactions that affect the yield of natural dyes extracted using microwave-assisted extraction method. ANOVA analysis results can be seen in Table 3. Important factors affecting the yield of natural dyes extracted using microwave-assisted extraction method are shown by p-values of less than 0.05. Table 3 shows that the quadratic parameter in the form of microwave power (B) has a significant effect on the yield of natural dyes extracted using microwave-assisted extraction method (p-value < 0.05).

Table 3 Analysis of variance (ANOVA) of quadratic model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3.62	9	0.4018	3.18	0.0427	significant
A-Time	0.2287	1	0.2287	1.81	0.2079	
B-Microwave power	0.0167	1	0.0167	0.1327	0.7232	
C-F/S ratio	0.1439	1	0.1439	1.14	0.3107	
AB	0.0213	1	0.0213	0.1687	0.6900	
AC	0.0002	1	0.0002	0.0015	0.9700	
BC	0.0630	1	0.0630	0.4995	0.4959	
A^2	0.0022	1	0.0022	0.0176	0.8972	
B^2	0.9370	1	0.9370	7.43	0.0214	
C^2	0.2697	1	0.2697	2.14	0.1744	
Residual	1.26	10	0.1262			
Lack of Fit	0.1798	5	0.0360	0.1662	0.9645	not significant
Pure Error	1.08	5	0.2164			
Cor Total	4.88	19				

Std. Dev., 0.3552; R^2, 0.7413; Adjusted R^2, 0.5085; Pred. R^2, 0.4494; Adeq. Precision, 5.2870 (Suggested).

The analysis of variance (ANOVA) results in Table 3 using the quadratic model are generally significant. This is shown in the analysis of variance in Table 3 for the quadratic model in general giving the model F-value of 3.18 that implies the model is significant. The lack of fit F-value of 0.1662 implies the lack of fit is not significant. There is a 96.45% chance that a lack of fit F-value is large could occur due to noise. An insignificant lack of fit is good. The coefficient of determination (R^2), adjusted coefficient of determination (Adj-R^2) and adequacy precision can be used to evaluate the suitability of the model. The model is usually adequate when the p-value of model <0.05; lack of fit >0.05; and R^2 >0.9 [13, 14]. Predicted R^2 is 0.4494 is in a reasonable rate with adjusted R^2 adjusted to 0.5085, where the value does not have a difference of 0.1 which means the model is appropriate. Adeq precision >4 which indicates an adequate signal and model and can be used to navigate the next design. The standard deviation of the model is 0.3552. A small value of the standard deviation indicates a good correlation that has a close value between the experimental data and the prediction model [15]. In addition, adeq. precision value is adequate (5.2870), which means the model can be used for optimization [16]. In addition, the optimization of natural dyes extraction using microwave-assisted extraction method with face-centered central composite design (FCCD) can also be obtained equations that can be used to predict the natural dyes yield obtained. Based on the experimental design that has been done, the yield of natural dyes extracted using microwave-assisted extraction method can be seen in the quadratic equation as follows:

$$\text{Yield (\%)} = 3.38 + 0.1512 * A + 0.0409 * B - 0.1200 * C - 0.0516 * AB + 0.0049 * AC - 0.0887 * BC + 0.0284A^2 - 0.5837B^2 - 0.3132 * C^2 \quad (2)$$

These equation can be used to make predictions about the yield of natural dyes from jackfruit wood waste obtained by microwave-assisted extraction method. The coefficients for Equation (2) are represented by constants, linear coefficients for independent variables, interactive term coefficients and coefficients of quadratic terms [17].

The optimum conditions for obtaining maximum yield in natural dyes extraction using microwave-assisted extraction method with face-centered central composite design (FCCD) are: microwave power of 600 W, F/S ratio of 0.0564 g/mL and extraction time for 50 minutes. From these optimum conditions, the maximum yield obtained from natural dyes extraction using microwave-assisted extraction method is 3.5747%. In general, it can be said that the model obtained can be used to represent experimental results or determine yields in natural dyes extraction using microwave-assisted extraction method [15].

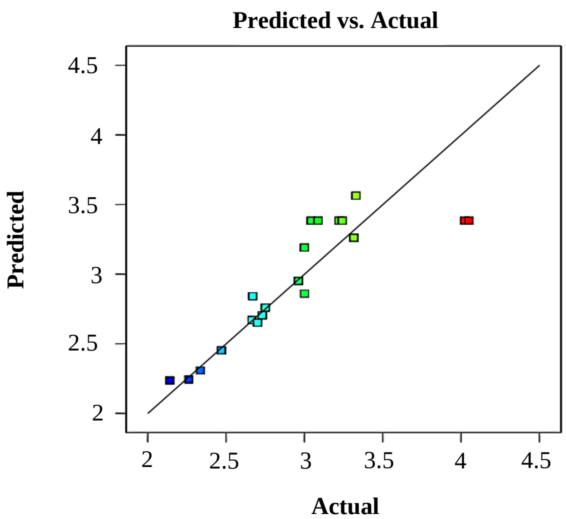

Figure 2 Experimental yield of extraction of natural dyes from jackfruit wood vs predicted from the model

Figure 2 shows the plot between experimental yield of extraction of natural dyes from jackfruit wood and those predicted from the model. From Figure 2, it can be seen that there is an interaction between actual and predicted values that is approaching a straight line. This shows the accuracy of the model in correlating natural dyes yield with independent reaction variables. Thus, the quadratic model chosen can represent the experimental data well [18].

3.2 Effect of parameters and optimization

In this study, to examine the effect of several factors on the response in the form of natural dyes yield extracted using microwave-assisted extraction method, it can be seen from a graphical representation known as a contour plot. Where the contour plot is obtained from the regression model in the quadratic model can be seen in Figure 3.

The extraction time is one of the important factors that affect the acquisition of the extraction. The extraction time is closely related to the length of time the solvent contact with the material. In general, the length of time of extraction will increase yield. The longer the extraction time means that the yield obtained will increase until it reaches the maximum point. After reaching the maximum point, the yield of product will slowly decrease due to the content of the dyestuff in the material that is increasingly reduced and the ability of the solvent which has limitations in dissolving the material [19]. In addition, a longer period of extraction time can degrade the compounds of the dyes to be extracted, so that it can cause the yield to be less. This must be avoided to obtain maximum extraction yields [20]. In Figure 3 it can be seen that there is an increase in yield which corresponds to the higher microwave power used (between 400 and 600 W) and the decrease in the yield for microwave power between 600 and 800 W for extraction time of 10 minutes and microwave power between 400 and 600 W for extraction time of 50 minutes. This can happen because the higher the microwave power used can cause the solvent to reach boiling point in a faster time. So that the high power microwave is not effective for use in a long time, although for some points the trend is constant [21-23]. In addition, based on Figure 3, it can also be seen that there are optimum conditions obtained in the interaction between microwave power and F/S ratio. The use of high F/S ratio causes the yield of natural dyes to be less. This is because the higher amount of solvent used (in this case, water) can cause swelling (excessive swelling) of the raw material used and cause excessive thermal pressure. This is caused by thermal absorbed by solvents from high microwave power so that the generation of thermal to the material will be too fast. Excess thermal absorbed by the material will adversely affect phytochemical compounds [24]. Meanwhile, a sufficient volume of solvent can produce more dye than using a large amount of solvent. This is because the heat transferred by the solvent to the material will cause the material to absorb energy more optimally so that the absorption of energy will go directly to the matrix material. This will cause the cell wall to break and release the dye into the solvent which can easily occur [22].

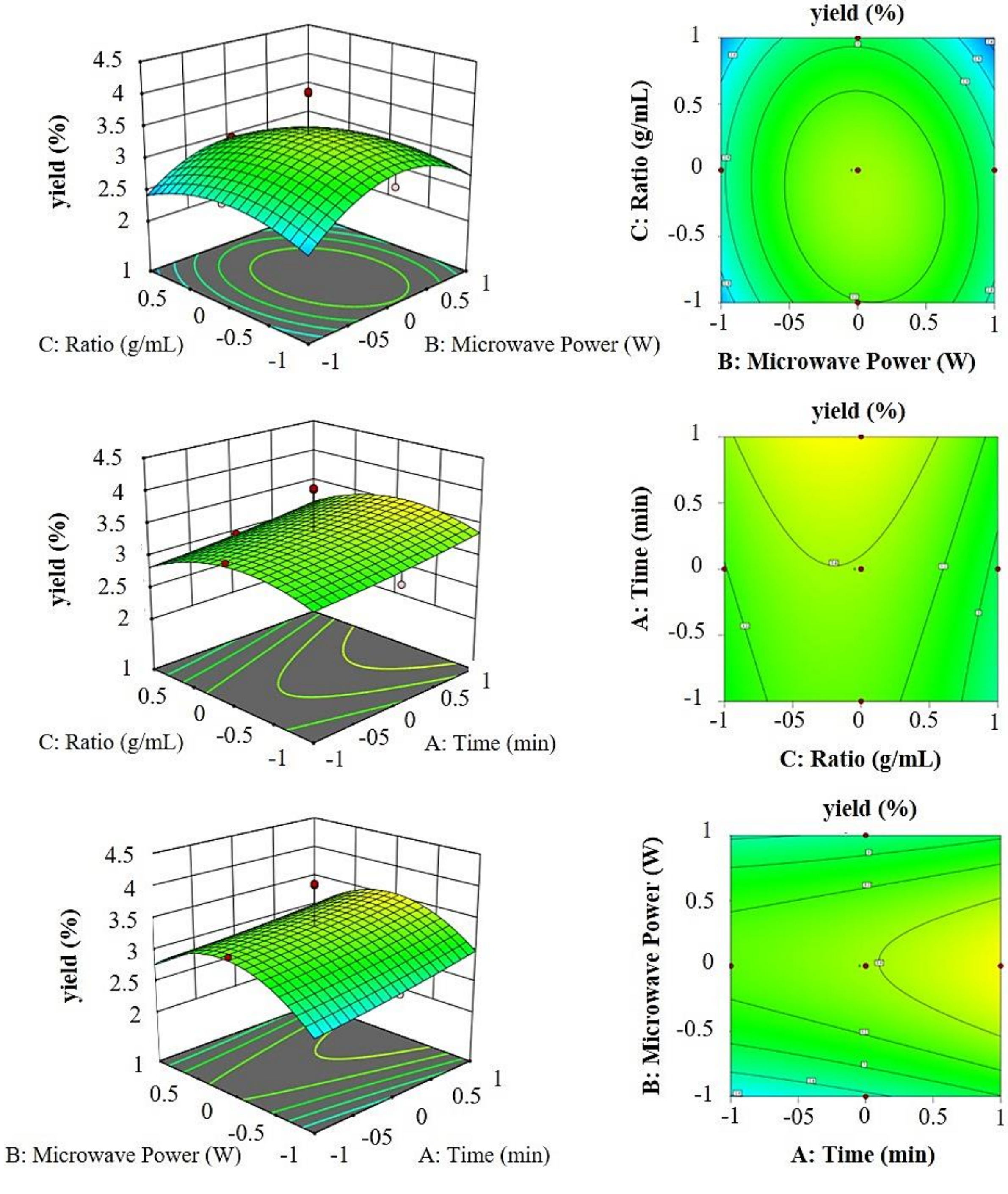

Figure 3 2D and 3D Contour plots that show the interaction between extraction time (A), microwave power (B) and F/S ratio (C) on the extraction of natural dyes using microwave-assisted extraction method

3.3 Comparison by other techniques

In reviewing the effectiveness of microwave-assisted extraction method in producing natural dyes from jackfruit wood, this study also compares with several methods that have been carried out. As shown in Table 4.

Table 4 Comparison of the optimum yield of natural dyes extracted by several methods

Method of extraction	Extraction time (min)	Yield (%)	Reference
Soxhlet Extraction (SE)	360	2.60	[25]
Ultrasonic-assisted Extraction (UAE)	50	3.67	[21]
Heat-Relux Extraction (HRE)	180	3.50	[20]
Microwave-assisted Extraction (MAE)	30	3.57	Present work

In Table 4, it can be seen the comparison of natural dye extraction from jackfruit wood between microwave-assisted extraction method and several other methods that have been carried out in several previous studies. In addition, in Table 4, it can also be seen that the yield for ultrasonic-assisted extraction, heat-reflux extraction and microwave-assisted extraction methods is almost the same. However, this does not apply to the extraction of natural dyes using Soxhlet extraction. This is because the extraction using Soxhlet extraction, which takes a longer time when compared to ultrasonic-assisted extraction, heat-reflux extraction and microwave-assisted extraction methods, actually produces a lower yield when compared to ultrasonic-assisted extraction, heat-reflux extraction and microwave-assisted extraction methods. Based on the length of extraction time, it can be said that Soxhlet extraction and heat-reflux extraction methods requires a relatively long extraction time (≥ 3 hours). Meanwhile, ultrasonic-assisted extraction and microwave-assisted extraction methods requires a relatively short extraction time (≤ 50 minutes). So it can be concluded that the extraction of natural dyes with microwave-assisted extraction method can produce relatively high yields with a relatively short extraction time.

Natural dyes that have been extracted from jackfruit wood or other biomass by microwave-assisted extraction method have the potential to be applied in various fields, such as energy (in the form of solar cells) [26], textiles (dyeing fabrics) [27-30] and several other fields. To be able to be applied in these various fields, it is necessary to have a deeper and detailed study before it can finally be applied on a higher scale or produced in the form of certain products.

4. Conclusions

Optimization of microwave-assisted extraction (MAE) of natural dyes from jackfruit wood (*Artocarpus heterophyllus* Lamk) have been successfully performed using response surface methodology by Face-centered central composite design (FCCD) type. The maximum yield from this design is 3.5747% obtained under operating conditions: microwave power of 600 W, F/S ratio of 0.0564 g/mL and extraction time for 50 minutes (with significant p-values of 0.0427 and insignificant lack of fit of 0.9645), indicating this model can represent this experimental. The comparisons have been done with several other techniques, such as SE, UAE and HRE, shows that microwave-assisted extraction (MAE) has been proven to save time than Soxhlet extraction and heat-reflux extraction methods.

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