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Optimization of Microwave-Assisted Hydrodistillation from Indonesian White Pepper (*Piper nigrum* L.) Essential Oils

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ABSTRACT

Pepper is one of the agricultural commodities that Indonesia widely exports. Pepper can be processed into various products, including essential oils. Pepper essential oil can be extracted using several methods, including the microwave. This method can speed up the essential oil extraction process. Optimization was carried out using the Box-Behnken design using the three independent parameters: solution ratio, time, and microwave power. The upper and lower limits used for the independent variables are solvent amount of 1,350 mL and 750 mL, time of 90 minutes and 60 minutes, and microwave power of 440 watts and 136 watts. The optimal data generated is treatment with a solvent amount of 1,050 mL, 90 minutes, and 440 watts of power, resulting in validation data of 99.046%. The optimal essential oil treatment gives a specific gravity of 0.8281, a refractive index of 1.4824, solubility in ethanol 1:2, acid number 0.739 mg KOH/g, ester number 19.599 mg KOH/g, and a hue value of 186,38 (green). Oil with optimum treatment also has an IC50 antioxidant activity of 32,919.9 ppm.

1. INTRODUCTION

Indonesia is among the top five largest producers of the spice pepper (Khew *et al*., 2022). Apart from being traded in whole form, pepper can be further processed into various products, including essential oil. The quality of essential oils is determined mainly by the properties and chemical compounds contained. Complex mixture of terpene hydrocarbon and oxygen compounds are known to be the main composition of pepper oil (Bastos *et al*., 2020). Several studies has identified the main compound that gives pungent aromas in *P. nigrum* essential oil or PEOs, such as β-pinene, α-pinene, phellandrene, linalool, D-limonene, limonene, myrcene, caryophyllene, α-copaene, and humulene (Al-Sayed *et al*., 2021; Katerina *et al*., 2023; Li *et al*., 2020; Menon *et al*., 2003). Variations in the composition of compounds found in PEO can be influenced by variety, land where it is grown, agro-climatic conditions, processing methods, and quality of raw materials.

Various techniques could be employed to yield PEOs, including the conventional (steam or hydro and solvent based extraction) and innovative/modern methods (supercritical fluid extraction/SFE, microwave-assisted extraction/MAE, ultrasonic-assisted extraction/UAE, and microwave assisted hydrodistillation/MAHD) (Wang *et al*., 2018). The hydrodistillation method is often used to obtain essential oils, however it can only produce low extraction yields with longer distillation time (Elyemni *et al*., 2019). Recently researchers has attracted in Microwave-assisted hydrodistillation or in short MAHD, because of its ability to reduce extraction time, volumetric heating, selectivity, and easiness to control the heating process (Asofiei *et al*., 2019; Bagade & Patil, 2019; Gumustepe *et al*., 2023). Furthermore, this method provides a higher yield and uses less water than hydrodistillation methods. This has made MAHD an attractive alternative in oil extraction than other extraction processes (Osorio-Tobón, 2020).

Black pepper extraction using the hydrodistillation method provides a lower yield for quite a long time than the MAE method (Shintawati *et al*., 2020). It has been proven by Rmili *et al*. (2014), the yield produced by the microwave extraction method is more significant than the hydrodistillation extraction method. In the microwave extraction hydrodistillation method, the yield was 1.5% for 35 minutes, while in the hydrodistillation extraction method, the yield was 1.24% for 1.5 hours. In the previous study that conducted by Tran *et al*. (2019), MAHD could run the extraction with shorter time without causing a negative impact to the composition of citronella oils. The yield resulting from this research also shows that extraction using the MAHD method is more significant than extraction using the conventional distillation method, with a yield value of 0.35% for extraction using MAHD and 0.15% for extraction using conventional distillation. So, it can be concluded that the MAHD method is relatively more efficient. However, there has not been much research on microwave assisted hydrodistillation for white pepper plants.

Research on antioxidant properties of MAHD of essential oils can be found widely. According to one research conducted by Moradi *et al*. (2018), extraction using the MAHD method produces a more excellent sesquiterpene b-caryophyllene content compared to conventional hydrodistillation extraction of rosemary essential oil. The composition of b-caryophyllene content is also found in white PEO. Percentage of sesquiterpenes and monoterpenes of white PEO are high include b-caryophyllene, sabinene, limonene, and torreyol. Research by Li *et al*. (2020) and Wang *et al*. (2021) shows that white PEO has potential as an antioxidant. However, the identification of antioxidants in this study was obtained from white PEO extracted using the usual hydrodistillation method, not by MAHD.

Microwave hydrodistillation has been a major technique for extracting PEO. However, new extraction technology utilization to enhance yield of PEO is still limited. In this study, microwave assisted hydrodistillation will be developed for the extraction of essential oil from pepper for improving the yield of PEO. An optimization test was carried out on extracting PEO using microwaves to increase the yield but also lower the time. The extraction process will affect the yield of the bioactive components in the essential oil. There needs to be a re-evaluation of the benefits of these chemical compounds. The extract was phytochemically characterized using GC–MS analysis and tested for ISO 3061:2008. The identified compounds were then studied for testing PEO's antioxidant properties.

2. MATERIALS AND METHODS

2.1. Materials and Reagent

In this research pepper seeds sample obtained from PT. Cinquer Agro Nusantara, Bandung, West Java, Indonesia. 1,1 diphenyl-2-picrylhydrazyl (DPPH) reagent was purchased from Sigma Chemicals, BaCl2 1%, H2SO4 1%, NaCl 0,85%, HCL 0,5 N, ethanol, 1,1-diphenyl-2-picrylhy-drazyl (DPPH), potassium hydroxide (KOH) 0.1 N and 0.5 N, and phenolphthalein indicator. All chemicals and solvents were of analytical grade.

2.2. Sample Preparation and Extraction Methods

Pepper seeds were ground into powder with a grinder (HC-800Y, Wuyi Haina Electric Appliance Co., Ltd., Zhejiang, China). The pepper powder was sieved with a 60-mesh sieve so that the sample used for extraction was uniform. The refined samples were then extracted using microwave-assisted hydrodistillation that can seen in Figure 1. In each run, the sample weighed 150 grams with a water. The sample is put into a flask which is then put into the microwave. The hot vapor will enter the hydrodistillation through the clavenger device. Hydro-distillation extraction was carried out using a clavenger device. The sample was put into a flask and distilled at a temperature of 100ºC. The essential oils are separated from the water in the reservoir flask and stored in an amber bottle to be further in the analysis stage. The amount of solution used following the experimental design provided from RSM on Table 1.

2.3. Experimental Design

This research method uses an experimental laboratory method using the RSM-type Box-Behnken design with the number of research treatments is predicted using Design Expert 13 software. This design combines three independent

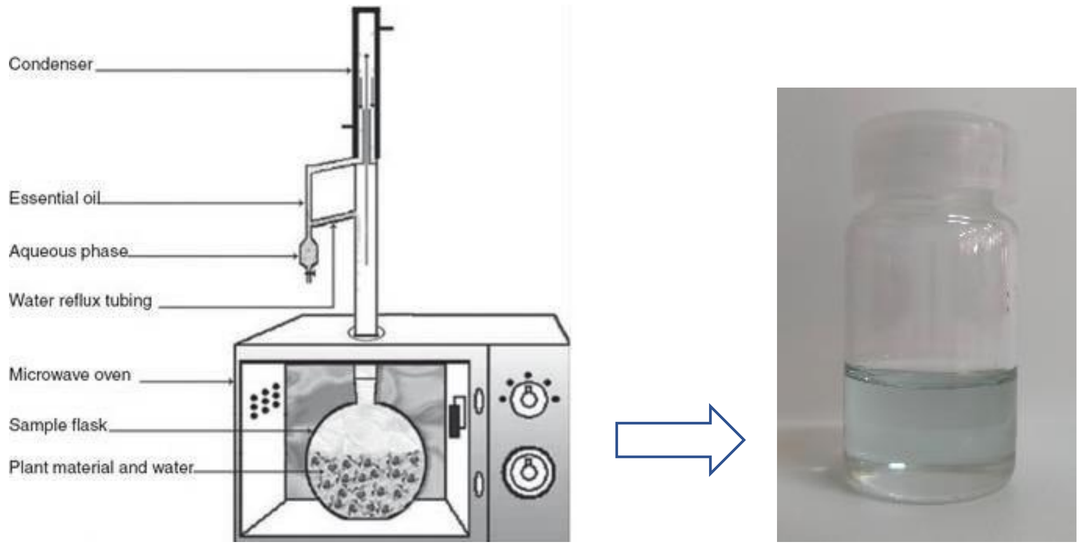


Figure 1. Schematic of MAHD and extracted oil with optimum color

variables: solvent amount, time, and microwave power. The dependent variable that will be the response is the yield of PEO. All variable has a minimum value (-) and a maximum limit (+), as seen in Table 1. Based on the running factors from Box-Behnken using Design Expert 13, there are 17 treatments (runs) with five middle points in the treatment. The results can be seen in Table 2. The experiment's results will then be evaluated using the analysis of variance (ANOVA). The results will then be compared with the yield produced by ordinary distillation extraction.

Table 1. Variable independent Box-Behnken

Variable Independent	Level		
	Low (-1)	Medium (0)	High (1)
A: Solvent amount (mL)	750	1,050	1,350
B: Time (min)	60	75	90
C: Power (Watt)	136	288	440

Table 2. Result from Box-Behnken

Run	Factor 1 A: Solvent Amount (mL)	Factor 2 B: Time (minute)	Factor 3 C: Power (Watt)	Response 1 Yield of PEO (%)
1	1,050	90	440	2.285
2	750	60	288	1.343
3	1,050	75	288	1.772
4	1,050	75	288	1.718
5	1,050	60	136	0
6	750	90	288	2.064
7	1,050	75	288	1.452
8	1,350	75	440	2.275
9	1,050	75	288	1.223
10	1,050	75	288	1.542
11	1,350	90	288	1.723
12	1,350	75	136	0
13	750	75	440	1.729
14	750	75	136	0
15	1,050	60	440	2.228
16	1,350	60	288	1.143
17	1,050	90	136	0

2.4. GC-MS Evaluation

The optimum oil will be analyzed for its supporting parameters as in Gas Chromatography-Mass Spectrometry (GCMS). GCMS were tested in Laboratorium Kesehatan Daerah (Jakarta, Indonesia). According to Hikmawanti *et al.* (2021), sample were analyzed using Gas Chromatography with AutoSampler (Agilent Innovations 7890), Mass Selective Detector 5975, and Chemstation data system. The separation was performed using a capillary column (HP Ultra 2) with a film thickness. Helium was use as the carrier gas with a consistent stream rate of 1.2 mL/min, injection port temperature set at 250°C. The starting temperature of the oven was set at 80°C and held for minutes, expanded at 3°C/minute to 150°C and held for 1 minute and lastly increased at 20°C/minute to 280°C and held for 26 minutes. The components were identified based on compatibility with true mass spectra within the Wiley electronic library. The basis for a major compound within the GC MS chromatogram was on the off chance that the compound had a percent range >5%. The similarity of the test mass range to the library was decided at a qualifying esteem of at slightest 80%.

2.5. Physical and Chemical Properties Analysis

The optimum oil will be analyzed for its supporting parameters as in ISO 3061:2008. The analyses that will be tested are specific weight, refractive index, solubility in ethanol, acid number, and ester number. The color was analyzed by Chromameter CR-400 Konica Minolta Spectrophotometer CM-5 (Bandung, Indonesia).

2.6. Antioxidant Activity

An antioxidant test will be carried out using the 1.1 Diphenyl-2-picrylhydrazyl (DPPH) method to evaluate the antioxidants produced by PEO. Make stock solutions (10 mg/ ml each) of PEO. All treatments are prepared in absolute ethanol. All samples were prepared in 6,250 – 100,000 µg/mL concentrations. All treatments were prepared in 2.0 mL mixed with 2.0 mL of 0.1 mM DPPH solution. Then, the solution was homogenized and left at room temperature for 30 minutes in a dark place. Absorbance will be measured at 517 nm using a Hitachi U-2900 UV/VIS Spectrophotometer (Bandung, Indonesia). The radical scavenger activity of DPPH will be calculated using the following formula:

$$Scavenging\ activity(\%) = \frac{A_{control} - A_{sample}}{A_{control}} \times 100\% \quad (1)$$

where $A_{control}$ is the absorbance of DPPH and ethanol, measurements will be carried out three times. The IC50 value is determined using the linear regression equation.

3. RESULTS AND DISCUSSION

3.1. PEOs Yield

Microwave hydrodistillation parameters were optimized by RSM using Box-Behnken design to achieve the maximum yield of PEO. According to the resulting, a small solvent amount with a long extraction time and a high power that we use will produce a high yield of PEO. The interaction effects between solution ratio and time, solution ratio and power, and time and power were highly significant. The power used in the microwave significantly affects the yield of PEO. The greater the power used, the higher the temperature and evaporation rate. The high microwave power will provide more energy to the material, which will then be converted into heat energy in the material (Hu *et al*., 2021; Yang *et al*., 2023). So using high microwave power will increase the rate of the extraction process and provide a high yield. However, using high microwave power may damage the essential oil cells, while low microwave power may lower the dielectric heating (Khruengsai *et al*., 2023; Radivojac *et al*., 2021). Although the initial irradiation in the MAHD system may improve essential oil solubilization, high microwave power use may diminish plant materials and enzymes and reduce essential oil yield (Abed, 2020; Lamberti *et al*., 2021).

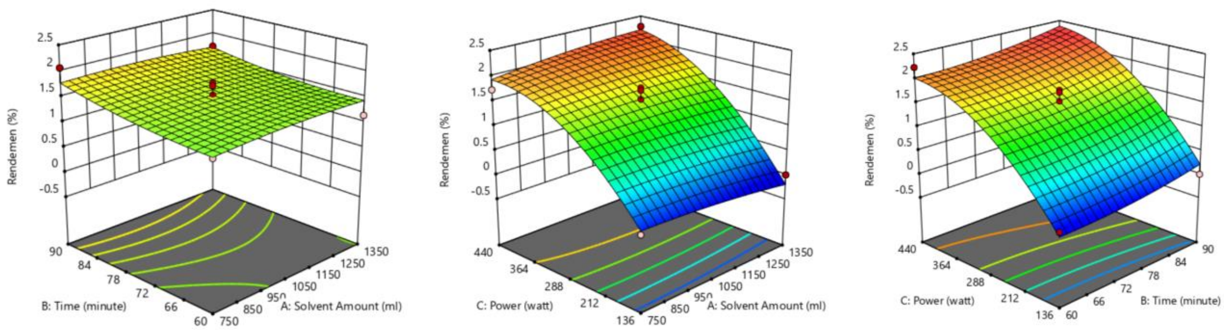
The response results obtained for the Box-Behnken design with the independent solution ratio (A), time (B), and microwave power (C) factors yielded PEO yield response values (%). The experimental values obtained from each treatment can be seen in Table 2. The significance of each variable and the coefficient (R^2) are demonstrated in Table 3. The model equation for the yield of PEO can be found as Y = –1.3535 + 0.1346A – 0.0335B + 0.0156C – 0.0011AB

+ 0.0004AC + 6.2134 BC – 0.0125A^2 + 0.0003B^2 – 0.000021C^2. ANOVA analysis for Box-Behnken design model concluded that the generated model was significant ($p < 0.0010$), and residual lack of fit was not significant ($p = 0.1914 > 0.05$). The adjusted coefficient and correlation coefficient were 0.8820 and 0.9484, respectively.

Table 3. ANOVA of response surface quadratic model for extraction yield of essential oil from pepper

Variables	Sum of Square	Df	Mean Square	F-value	P-value
Model	10.42	9	1.16	14.29	0.0010
A-solvent amount	3.125E-06	1	3.125E-06	0.0000	0.9952
B-time	0.2304	1	0.2304	2.84	0.1357
C-power	9.07	1	9.07	111.84	< 0.0001
AB	0.0050	1	0.0050	0.0613	0.8115
AC	0.0747	1	0.0747	0.9216	0.3690
BC	0.0008	1	0.0008	0.0099	0.9235
A^2	0.0106	1	0.0106	0.1309	0.7281
B^2	0.0249	1	0.0249	0.3076	0.5964
C^2	1.01	1	1.01	12.48	0.0096
Residual	0.5675	7	0.0811		
Lack of Fit	0.3739	3	0.1246	2.58	0.1914
Pure Error	0.1935	4	0.0484		
R^2	0.9484				
Adjusted R^2	0.8820				
C.V.%	21.52				
Adeq Precision	11.7975				
Cor Total	10.99	16			

RSM presents 3-dimensional graphic data containing the influence of 2 combinations of independent variables on the response to the yield of PEO. The values in the graph are obtained from the results of the ANOVA analysis, which are then plotted on the x, y, and z axes to form a 3-dimensional line. There are three treatment interactions, namely an interaction of the independent variables solvent and time ratio (AB), solvent and power (AC), and the last time and power (BC), which are presented in Figure 2. Figure 2(a) shows that a small solvent ratio with a long extraction time will produce a low yield of PEO. Figure 2(b) shows that the higher the power we use at both the low and the highest solvent ratios, the higher the yield of essential oils will be. However, the resulting yield will be low if you use low power. In Figure 2(c), the higher the power used at the lowest and highest times, the higher the yield produced. It can be seen in Table 2, run 1 with a power of 440 Watts produces a yield of 2.285%, while run 17 with a power of 136 Watts does not produce PEO yield. This is shown in Table 3 ANOVA where the resulting p-value of power is <0.0001 ($p<0.05$), so that the power variable provides a significant treatment of PEO yield.



(a) (b) (c)

Figure 2. (a) independent variables solvent ratio and time (AB) interactions; (b) independent variables solvent ratio and power (AC) interactions; (c) independent variables time and power (BC) interactions

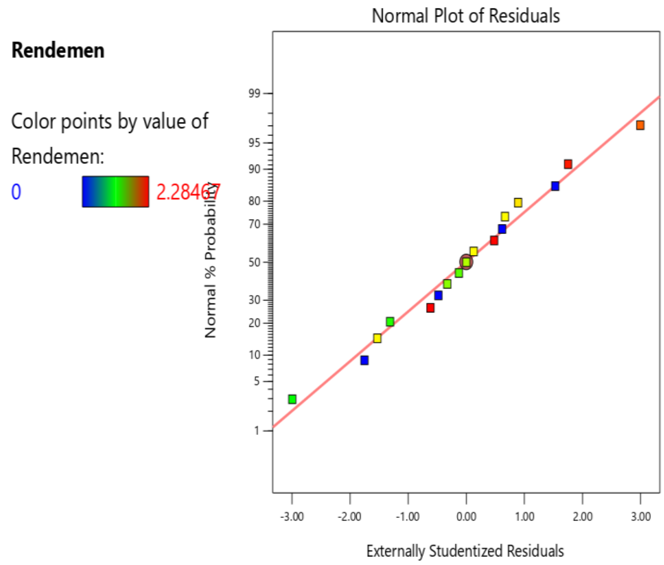


Figure 3. Normal plot of PEO yield

The relationship between the predicted yield data and the actual yield can be seen in Figure 3. The actual yield data is close to the predicted yield data, where the points are close to a straight line, so the actual data produced is excellent. If the existing data plot points are close to a straight line, the error that occurs in the system is very small. It was found in this study that the optimal microwave power was 440 W. The higher the power, the higher the energy received by the material, so that the yield of pepper essential oil is increased. High temperatures cause greater water movement because the kinetic energy between molecules increases and accelerates the diffusion process which causes all the oil contained in the material to be extracted in large quantities. The accuracy of the validation results obtained is very high, namely 97.64%, which indicates that the yield of PEO is almost comparable to the prediction from RSM. Actual data is also still in the 95% PI Low and 95% PI High range. If the data is between the prediction interval (PI), the model can predict the yield of PEO well.

The optimum treatment combination from the Box-Behnken design experiment will use a solution with a solvent amount of 1,079.1 ml, 86.815 minutes, and 428.559 watts of power. The selected process with high desirability can provide the highest estimated yield of 2.308%, where the recommended method will be rounded off to a solvent amount of 1,050 ml, 90 minutes, and 440 watts of power. The results showed that actual yield of PEO was 2.285%.

3.2. Characteristics and Chemical Composition

According to testing, the compounds produced in optimum PEO were found to be the highest compounds, namely Caryophyllene 33.32%, (+)-3-Carene 22.58%, D-Limonene 14.64%, β-Pinene 6.44%, dan α-Pinene 2.90%. The results presented on Figure 4 show that the compounds that shown in chromatogram. PEO primarily contains monoterpenes with various structures and sesquiterpenoids, with the major components being limonene, copaene, 3-carene, and caryophyllene (Kang *et al*., 2019)**.** This is in accordance with research conducted by previous researchers, although there are variations in chemical composition. Changes in essential oil content can be caused by geographic origin, season, and the extraction method (Khan *et al*., 2023).

The physical properties that evaluated in this study was compared to ISO 3061:2008 about Standard for Black Pepper Oil because there is no standard for white pepper. The material used in this research was pepper oil with optimum extraction. The results presented on Table 4 show that the characteristics produced in this research meet the ISO 3061:2008 standard. Color analysis results can be seen in Table 5 and the optimal PEO images is attached presented in Figure 1.

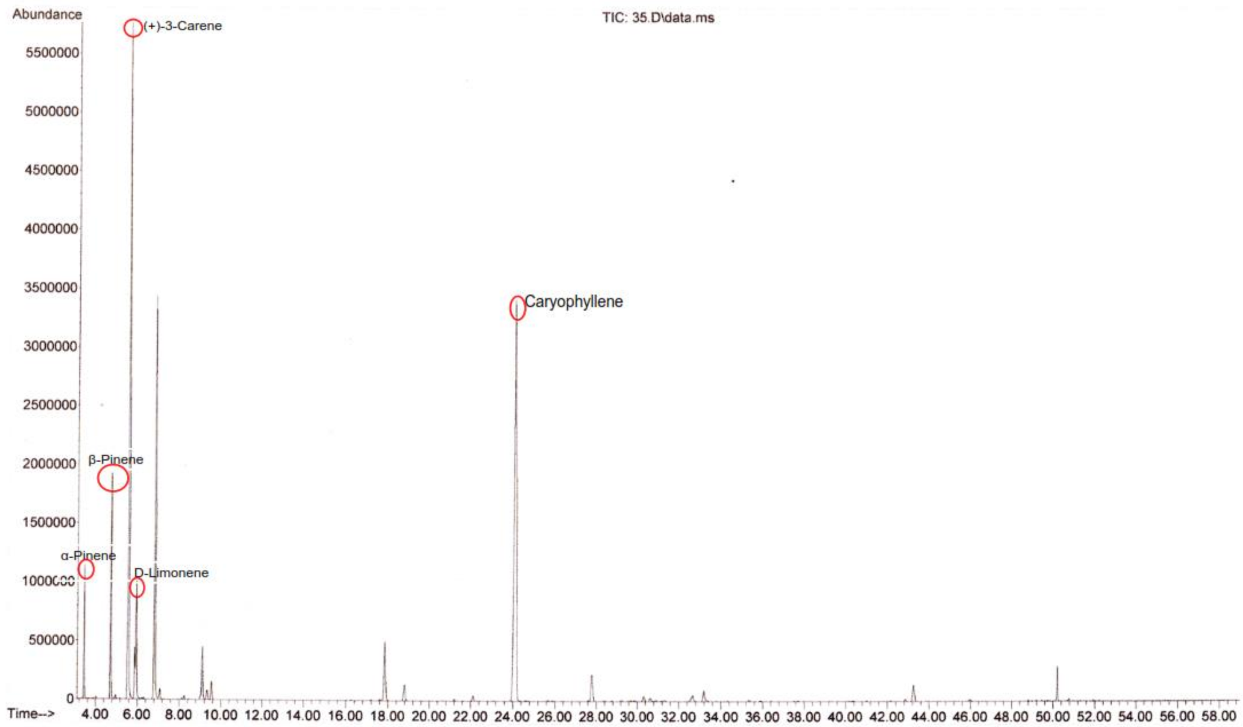


Figure 4. Chromatogram PEO Optimum

Table 4. Parameter Analysis

Parameter	ISO 3061:2008	Optimum
Specific weight	0.861 – 0.885	0.8281
Refractive index	1.480 – 1.493	1.4824
Solubility in ethanol	1 : 3	1 : 2
Acid number		0.739 mg KOH/g
Ester number		19.599 mg KOH/g

Table 5. Optimum Sample Color Analysis Results

Run	Repeat	L*	a*	b*	C*	H	Color
Optimum	1	87.35	-2.43	-0.28	2.44	186.5	Green
	2	87.35	-2.42	-0.27	2.44	186.38	Green

The optimum treatment PEO test results for specific gravity still far from ISO 3061:2008 standards. According to Yasin *et al*. (2013) and Yensusnimar (2021), the greater the heavy fraction in oil contained, the greater the specific gravity value. The greater the specific gravity of the material, the more components it has. The refractive index test results show that the values obtained meet the standards set by ISO 3061:2008. According to Muwaffaq & Supriyo (2021), essential oils with a high refractive index are of better quality than those with a low refractive index. Test results show that white PEO dissolves in 95% alcohol in a ratio of 1:2. This indicates that the ethanol solubility results meet the ISO 3061:2008 standard, which is based on Indonesian black PEO and requires 1 ml of essential oil in 3 ml of 95% alcohol. The easier the essential oil dissolves in alcohol, the easier it is to dilute the essential oil. According to Syahadat & Diningsih (2022), the alcohol solubility test reveals how easily the oil dissolves. The more efficiently the oil dissolves in alcohol, the more polar compounds it contains. Alcohol solubility is a crucial factor in testing essential oils, as it can indicate the quality of the oil. The experiment also revealed the acid number 0.739 mg KOH/g and the ester number 19.599 mg KOH/g. The L* value resulting from the optimum data is 86.22, where this figure is close to

100, indicating that the essential oil is bright or light in color. The a* value of -2.81 indicates a green chromatic color and a b* value of 0.16 indicates a yellow chromatic color. The optimum chroma value is 2.81, where both treatments produce chroma above 2. The Hue value in the optimum treatment is 176.79, which is included in green.

3.3. Antioxidant activity

Antioxidant activity was tested using the DPPH method. DPPH free radicals, which are stable and commonly used, help estimate antioxidant activity in neutralizing free radicals. Based on the research, the inhibition results of each concentration were obtained in Table 6. The inhibition value is obtained from the absorbance which is then calculated into the equation y = 0.0006x + 30.053 (R^2 = 0.9583). The results of IC_{50} for optimum PEO obtained from the difference of 50 minus 30.053 divide by 0.0006 is 32,919.9 ppm. Antioxidant properties are one of the common properties of food and medicinal plants, to which a variety of plant compounds, both secondary and primary metabolites, contribute. Antioxidants can neutralize the character of free radicals by transferring electrons or hydrogen atoms to DPPH (Baliyan *et al*., 2022). The antioxidant activity test indicated that PEO exhibited weak free radical scavenging capacity in the DPPH assay.

Table 6. Inhibition every concentration

Sample	Concentration (ppm)	Inhibition (%)
Optimum	6,250	29.678
	12,500	35.105
	25,000	49.058
	50,000	66.777
	100,000	87.043

4. CONCLUSIONS

The optimization model expressed in quadratic model that have a very significant effect of power (C) variable to the PEOs yields. The greater power of microwave provided the higher yield. It can be seen from the results that the highest microwave power is at 440 Watts. The optimal condition/ point was achieved by solvent amount of 1,050 mL, irradiation time for 90 minutes, and MW power of 440 W. The best PEOs have a specific gravity of 0.828 "units", refractive index of 1.482, ethanol solubility in 1:2, acid number 0.739 mg KOH/g, ester number 19.599 mg KOH/g, and the hue of 186.38 (identified as green). Antioxidant activities of PEOs have been evaluated that has the activity as IC_{50} of 32,919.9 ppm (weak).

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