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Extraction of P. cubeba Essential Oil by Microwave Assisted Hydrodistillation: Modeling and Process Optimization

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Abstract:

The industrial scale equipment design of P. cubeba essential oil extraction by microwave assisted hydro distillation (MAHD), as a relatively new extraction method, requires quantitative description of the mass transfer as well as explorative studies focusing on process optimization. The quantitative modeling was generated by several mathematical diffusion formulas and verified by the experimental mass transfer phenomena, while the optimization of particle-to-water ratio (0.033, 0.05, and 0.067 w/w) and times (0-52 min) was conducted by Response Surface Methodology (RSM). The results showed that the proposed mathematical model based on unsteady-state intra-particle diffusion theories well describes the rate of extraction of P. cubeba essential oil using MAHD method. The particle-to-water ratio as well as the extraction time significantly influences the oil yield. The optimum values obtained were 0.05 w/v and 32 min for particle-to-water ratio and time respectively, while the produced oil yield was theoretically 57.89%.

Keywords: essential oil; mass transfer modeling; microwave assisted hydrodistillation; Piper cubeba; response surface methodology

1. Introduction

P. cubeba essential oil is one of isolated essential oils from piperaceae family, members of traditional plant in many tropical and subtropical regions like Asia, African, Brazillian [1]-[4]. The chemical composition of *P. cubeba* oil is alkaloid, amides, terpenoid, benzoic acids, furanofuran, chormenes, chalcones, lignan (cubebin, hinokinin, yatein, isoyatein), and neolignan (kadsurin and piperenone) [1]-[2], [4]-[5]. The chemicals in *P. cubeba* oil can be used as spices, medicines to treat many ailments (gonorrhea, dysentery, abdominal pain, diarrhea, enteritis), antileishmanial, antimicrobial, antitumoral, fungicidal, and insecticidal [1]-[3], [5]-[5]. Extraction is one of the key technologies to isolate several essential oils. Existing methods to extract essential oil of *P. cubeba* seed is solvent extraction, water distillation, water-steam distillation, and steam distillation. These methods have many problems like low energy efficiency as well as high CO_2 emission and high solvent consumption [6]-[7]. Recently, microwave dielectric heating as power source is emerging as safer, more efficient, energy saving, and sustainable extraction process [7]. Reference [8] reported that to provide *Zygophyllum album L.* essential oil yield of 0.002% using microwave assisted hydrodistillation (MAHD) method required an extraction time of 30 min versus 3 h of hydrodistillation (HD) method. Likewise, reference [6] concluded that microwave steam diffusion (MSDf) for extraction of essential oil from orange peel is clearly advantageous in term of energy and time; the extraction time using MSDf method of 12 min versus 40 min of conventional steam

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diffusion (SDf) as well as the required energy to perform the two extraction methods are 300 kWh kg^{-1} essential oil and 120 kWh kg^{-1} essential oil for SDf and MSDf, respectively. On the other hand, no significant differences in chemical composition were found for the obtained essential oils by microwave steam distillation versus by steam distillation [7], and also by MAHD versus by HD [9]. Therefore, it is important to conduct further studies on the *P. cubeba* essential oil extraction using microwave as power source in order to improve the efficiency of the process. As the mentioned above, the essential oil extraction methods using microwave as power source usually conducted are water distillation, steam distillation, water-steam distillation, solvent free extraction, and solvent extraction. One of all, the most economically process is water distillation (hydrodistillation). The aim of the paper is to study the *P. cubeba* essential oil extraction by MAHD focusing on the chemical composition as well as phsyco-chemical properties of the essential oil obtained, the modeling of the mass transfer, and the process optimization.

The mass transfer theories are applied to develop the mass transfer modeling, one will contribute not only the technological analysis for fundamental understanding of the extraction but also the economic analysis for efficient process control. The mass transfer modeling of essential oil extraction can be developed in several fundamental understandings. Reference [10] has analyzed the mass transfer of ripe juniper berries oil during HD. They proposed that the oil mass transfer is basically the same as the diffusion of extractive substances through plant particles during solvent extraction (leaching). Referece [11] have studied the kinetic modeling of a continuous SD of aniseed essential oil and expressed as two regimes, (1) the first one is bounded by critical oil content, and (2) the second one corresponds to slower mass transfer from deeper parts to surface of the solid particles. In their study, the critical oil content was obtained as a point of the two linear lines with two different slopes on a curve of the oil content in seeds as function of time. The change in slopes is due to changes in the mechanisms, i.e. easily accessible oil (extra-cellular surface oil) as first regime and rather difficult oil to access (intra-cellular deeper inside the particle) as second regime. The same with [11], reference [6] modeled the lavender essential oil mass transfer of MSDf. In this study, the mass transfer modeling was also generated based on the fundamental understanding that can express the *P. cubeba* essential oil extraction mechanism as it rises during the conducted experiment.

Another aspect of this study is to find the correlation of process variable on *P. cubeba* oil yield. The important factor of the optimization is to find the optimum process is the selection of variables that influence the *P. cubeba* oil extraction using MAHD. It turned out that the process design variables of extraction using MAHD are microwave power, particle to water ratio, particle size, and time. In addition, it was observed that the essential oil extraction using MAHD method will be more efficient in the energy demand if the microwave power source is high. Meanwhile, for the particle size, the high essential oil extraction can be reached with smallest particle size. Thus, the process design variables studied were particle-to-water ratio and time. Methods that can be applied to optimize the process design variable are dynamics optimization, artificial neural network (ANN) [12]-[13], and response surface methodology (RSM) [12], [14]-[16]. Each technique has advantages and disadvantages. Recently, dynamics optimization is usually to optimize the complex process design like relationship of temperature steam, pipe diameter, and steam duty on pipe construction cost. Each variable process design has a unique formula of pipe construction cost. On other hand, RSM optimization is more advantage than ANN in showing a regression equation that can express the relationship of design variables. Thus, at the present study, we conducted the optimization of *P. cubeba* oil extraction with RSM. A two level two factor (2^2) full factorial central composite design (CCD) in RSM was developed to predict the relationship between the experimental variables (particle-to-water ratio and time) and the amount of the essential oil volume (response variable). Finally, the optimal solution offered by RSM was statistically verified by the coefficient of determination (R^2) based on the validation data set.

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2. Material and Methods

2.1. n-Hexane Extraction of P. cubeba Essential Oil in Order to Find the Oil Content

In this study, about 1 kg of dried *P. cubeba* seed were collected locally and it had the moisture of 2.163% w/w. The extraction procedure was referenced from the standard procedure by ASTM D5369-93(2008)e1. The grinded and screened *P. cubeba* seed of 5 g was extracted using 100 mL of n-Hexane in sochxlet apparatus for 20 of cycles. Then, the condensate essential oil and water has evaporated until the volume of 15 mL to solvent recovery and dried by oven in order to obtain the pure *P. cubeba* essential oil. The solvent extractions were performed at least three times.

2.2. The P. cubeba Essential Oil Extraction Using MAHD

Microwave assisted hydrodistillation (MAHD) has been conducted in a microwave oven (Samsung, ME731K, maximum output power of 800 W with 2450 MHz of microwave irradiation frequency). The reactor was a 1 L short-necked horizontal cylinder Pyrex glass. The powder *P. cubeba* seed of 40 g and aquadest of 600 mL (particle-to-water ratio = 0.0667 w/v) was put in the cylinder extractor and then was placed in the microwave oven that was equipped with Pyrex connecting tube to pass through the vapor to condenser. The condenser is equipped 50 mL of Pyrex burette in order to measure the essential oil and water condensate volumes every 5 minutes. The extraction was conducted until no distillate was obtained. After that, the collected condensate was separated using conical flash to remove the water from the essential oil, and then the essential oil was chemically dried under anhydrous sodium sulphate and stored at -4 ^{o}C. The experiment was repeated for various particle-to-water mass ratios (0.05 and 0.0333 w/v). The properties of the essential oil was analyzed: the composition by GC-MS (Perkin Elmer Clarus 680 coupled to a SQ 8T mass spectrometer), the density by picnometer, the acid number by KOH gravimetric, and the ethanol solubility of 1:1 v/v. The operation conditions of GC-MS analysis were as follows: ionization voltage, 70eV; emission current, 40 mA; scan rate, 1 scan/s; mass range, 35–300 Da; ion source temperature, 200 ^{o}C. The MS fragmentation pattern was checked with those of the other essential oils of known composition by matching the MS fragmentation patterns with NIST 56537 mass spectra libraries and with those in the literature.

3. Results and Discussion

The theoretically maximum yield of *P. cubeba* oil extraction as a result of n-hexane extraction was about 15.4% w/w. The result of the gas chromatogram analysis was shown in Fig. 1, while the produced essential phsyco-chemical properties such as chemical composition, density, acid number, and solubility in ethanol of 1:1 ratio can be seen in Table 1.

In Table 1, it can be seen that the main composition of the produced *P. cubeba* essential oil by MAHD were cububene (26.54% w/w) followed by naphthalene (25.85% w/w), azulene (10.39% w/w), carryopyllene (9.58% w/w), copaene (9.23% w/w), carene (1.45%). These compositions were similar to the ones of other extraction methods like n-Hexane solvent extraction which the main composition was in group of cubebin compound [5] and also extraction under sonification using MeOH [2].

The volumes of water and essential oil obtained every 5 minutes of interval time at various particle-to-water ratios were listed in Table 2. The assumptions that were taken to calculate the numbers of mole and mass were the essential oil and water phases are completely immiscible, the water density = 1 g/mL, the water molecular weight = 18 g/mole, the oil density = 0.9026 g/mL (only one component, i.e. cububene), and the oil molecular weight = 164 g/mole. The calculation results of mole and mass of the oil and water in distillate are listed in Table 2.

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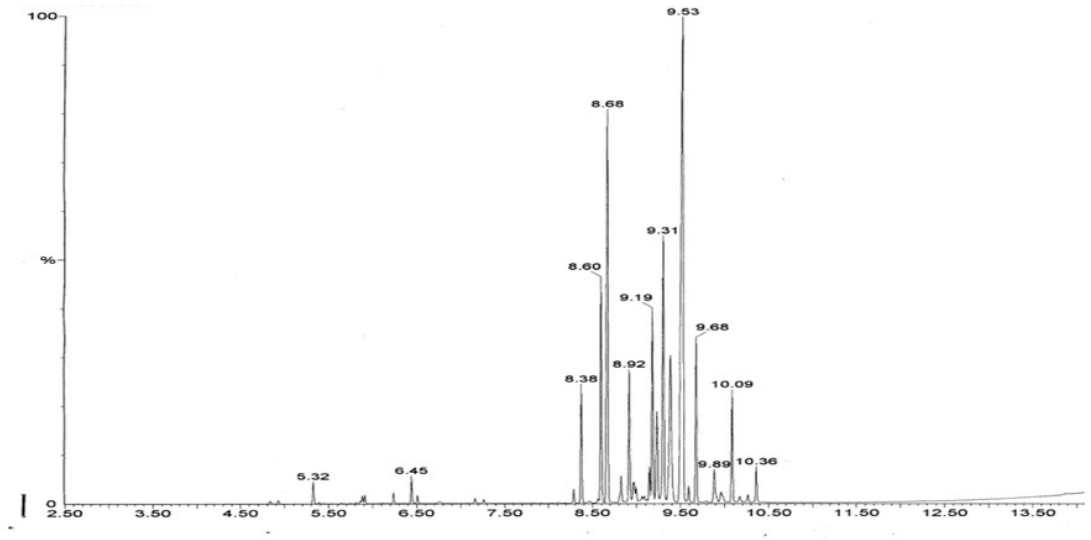

Figure 1. Gas chromatography of *P. cubeba* essential oil

Table 1. Chemical and physical properties of P. Cubeba essential oil

Retention time	Area	% Area	Chemical compound
5.325	838255680	0.70	*Carene*
6.445	905383744	0.75	*Carene*
8.376	4360846336	3.62	α-*Cubebene*
8.601	9091772416	7.56	α-*Cubebene*
8.671	18475495424	15.36	α-*Cubebene*
8.921	5222070272	4.34	*Caryophyllene*
9.186	6992571904	5.81	*Azulene*
9.306	11103812608	9.23	*Copaene*
9.521	29607204864	24.61	*Naphthalene*
9.681	6301551104	5.24	*Caryophyllene*
9.891	1492988928	1.24	*Naphthalene*
10.087	4207820800	3.50	*Azulene*
10.362	1294258176	1.08	*Azulene*
density		g/L	0.9108
acid number		mL KOH	0.799
solubility in ethanol of 1:1 ratio			clear soluble

In exraction, the mass balance equation can be expressed by :

$$x\left(W_{flower} - W_{oil}\right) = W_{flower}\, x_{oil_0} - W_{oil} \quad (1)$$

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$$x = \frac{W_{flower}\, x_{oil_0} - W_{oil}}{W_{flower} - W_{oil}} \qquad (2)$$

with x_{oil_0} is the initial essential oil content in flower (15.4% w/w = 0.154) and x is the essential oil content in flower during extraction. For the mass balace formula above, the essential oil content in the flower can be calculated and result as in Fig. 2.

From the experiment data as in Fig. 2, it can be logically concluded that the extraction was controlled by the diffusion of solute in the solid. By assuming that the particle is spherical, the radial diffusion can be described by Fiks's second law, the oil material balance in the particle would be as in Eq. (3).

$$\frac{1}{D_e}\frac{\partial C}{\partial t} = \frac{\partial^2 C}{\partial r^2} + \frac{2}{r}\frac{\delta C}{\delta r} \qquad (3)$$

with C is the concentration of essential oil in the particel at time t (m/v), r is the radius along the direction of diffusion, and D_e is the diffusion coefficient. The concentration of oil at the particle surface is assumed to be zero, so the boundary condition of the process is: $C(r,0) = C_o$ and $C(R,t) = 0$. It is well known that Eq. (3) can be solved with the method of separation of variables and the result is as given in equation (4) [17].

Table 2. Water and oil in the distillate

Particle to water ratio						0.033
Time	**Water**			**Essential oil**		
Min	Volume, mL	Mass, g	mole	Volume, mL	Mass, g	Mole
0	0	0	0	0	0	0
12	56	56	3.111	1.2	1.0930	0.0067
17	112	112	6	1.6	1.4573	0.0089
22	167	167	9	1.9	1.7305	0.0106
27	221	221	12	2.1	1.9127	0.0117
32	272	272	15	2.3	2.0948	0.0128
37	323	323	18	2.4	2.1859	0.0133
42	365	365	20	2.5	2.2770	0.0139
47	365	365	20	2.5	2.2770	0.0139
52	365	365	20	2.5	2.2770	0.0139
Particle to water ratio						0.05
0	0	0	0	0	0	0
12	55	55	3	1.5	1.3662	0.0083
17	108	108	6	1.9	1.7305	0.0106
22	162	162	9	2.3	2.0948	0.0128
27	215	215	12	2.6	2.3681	0.0144
32	267	267	15	2.9	2.6413	0.0161
37	317	317	18	3.2	2.9146	0.0178
42	357	357	20	3.4	3.0967	0.0189

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47	383	583	21	3.5	3.1878	0.0194
52	397	397	22	3.6	3.2789	0.0200
Particle to water ratio						0.067
0	0	0	0	0	0	0
12	50	50	3	1.8	1.6394	0.01
17	105	105	6	2.3	2.0948	0.01277
22	160	160	9	2.8	2.5502	0.01555
27	211	211	12	3.2	2.9146	0.01777
32	261	261	15	3.6	3.2789	0.01999
37	312	312	17	3.9	3.5521	0.02166
42	362	362	20	4.1	3.7343	0.02277
47	362	362	20	4.1	3.7343	0.02277
52	362	362	20	4.1	3.7343	0.02277

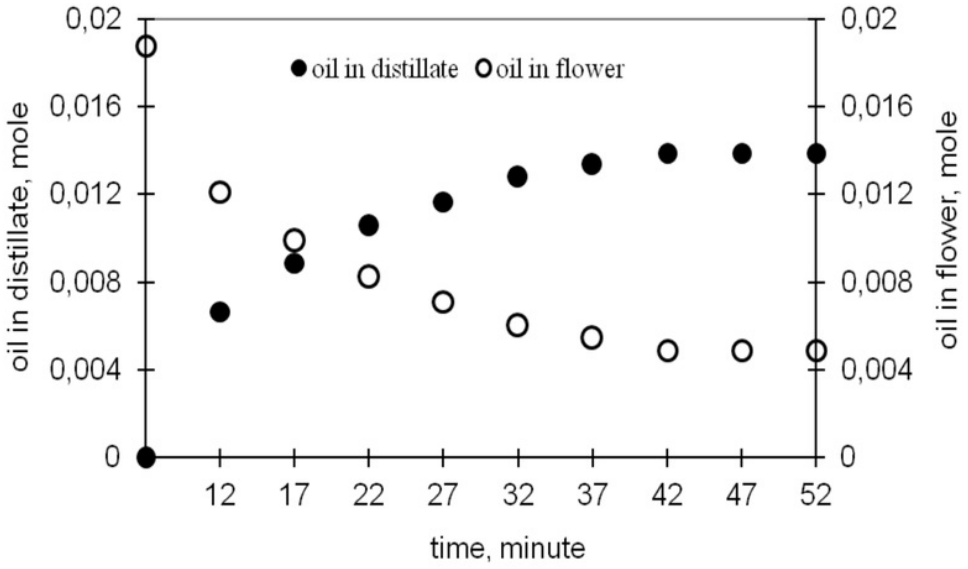

Figure 2. Essential oil in distillate and flower of *P. cubeba* extraction at particle-to-water ratio of 0.033 w/v

$$C(r,t) = \frac{2C_0R}{\pi}\sum_{t=0}^{t=t}\frac{(-1)^n \sin\left(\frac{n\pi r}{R}\right)}{n}\frac{}{r}e^{-\left(\frac{n\pi}{R}\right)^2 D_e t} \quad (4)$$

The extracted oil mass from single particle can be expressed by:

$$m = \int_{t=0}^{t=t} N_A dt \quad (5)$$

with N_A is the extraction rate that can be calculated by:

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$$N_A = -D_e 4\pi R^2 \left(\frac{\delta C}{\delta r}\right)_{r=R} \tag{6}$$

$$N_A = 8\pi C_0 D_e R \sum_{n=1}^{n=\sim} e^{-\left(\frac{\pi^2 D_e}{R^2}\right)n^2 t} \tag{7}$$

The extracted mass fraction (essential oil yield) can be calculated with the formulation as follows:

$$x = \frac{m}{\frac{4}{3}\pi R^3 C_0} = \frac{6D_e}{R^2} \sum_{n=1}^{n=\sim} \int_{t=0}^{t=t} e^{-\left(\frac{\pi^2 D_e}{R^2}\right)n^2 t} \tag{8}$$

$$x = 1 - \frac{6}{n^2} \sum_{n=1}^{n=\sim} \frac{1}{n^2} e^{(-\beta n^2 t)} \tag{9}$$

$$\beta = \frac{\pi^2 D_e}{R^2} \tag{10}$$

The parameter β was adjusted and estimated by minimization of the sum of square of errors between the essential oil data from Table 1 and the generated mass transfer calculated. Then for n = 20, the best β values obtained were $5.2.10^{-4}$, $3.6.10^{-4}$, and $2.85.10^{-4}$ min^{-1} for particle-to-water ratios of 0.033, 0.05, and 0.067, respectively. The comparison between the essential oil yield from experiment data and calculated result can be seen in Fig 3. It can be seen that the model proposed can well described the extraction process. The value of diffusional coefficient can be calculated by Eq. (10) with the particle size of *P. cubeba* was about 0.105 mm (throught 150 mesh) and result is $(2.12 \pm 0.619).10^{-11}$ m^2/s.

In the explorative process optimization, a total of 26 experiments were conducted to generate the CCD matrix in Table 3 and the average values were used to analysis the data by RSM software of Design Expert Version 6.0.8. From the ANOVA analysis, the suggested correlation model between the independent variables and response was a quadratic model due to the associated Prob. > F value for the model is lower than 0.0001. The quadratic model can be written as in:

$$Oil\ yield = 0.08915 + 0.0016A + 0.00849B + 5.85.10^{-6}AB + 5.37.10^{-5}A^2 + 4.22.10^{-4}B^2 \tag{10}$$

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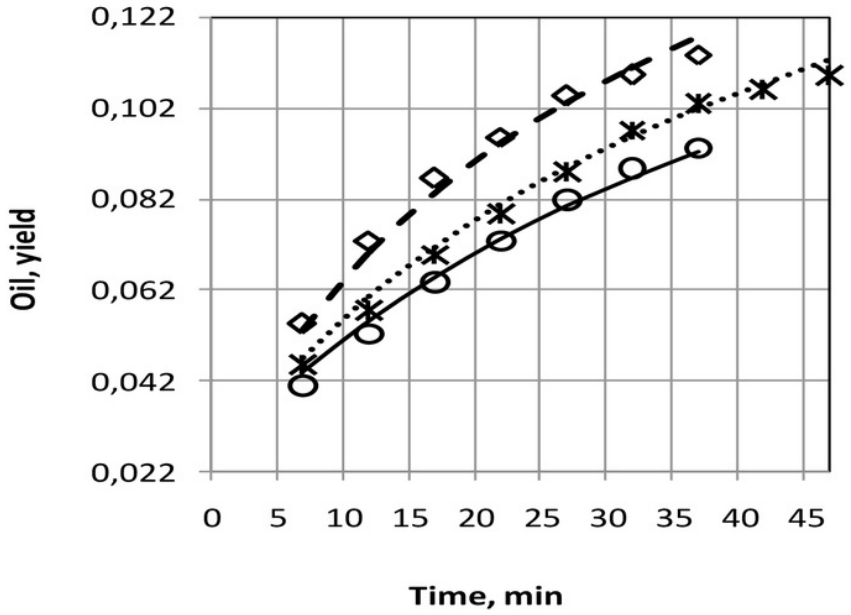

Figure 3. Essential oil yield curve of the *P. cubeba* microwave assisted hydrodistillation, mass transfer model (line) and experimental data (marker) at various particle to water ratios of 0.033 (circle), 0.05 (triangular), and 0.067 w/v (square).

Table 3. CCD for two independent variables

Run	Coded value		Real value		Response
	A	B	A	B	Oil yield
	w/w	min	w/w	Min	w/w
1	-1	-1.00	0.033	12	0.05465
2	-1	-0.75	0.033	17	0.07286
3	-1	-0.50	0.033	22	0.08653
4	-1	-0.25	0.033	27	0.09563
5	-1	0.00	0.033	32	0.10474
6	-1	0.25	0.033	37	0.10930
7	-1	0.50	0.033	42	0.11385
8	0	-1.00	0.050	12	0.04554
9	0	-0.75	0.050	17	0.05768
10	0	-0.50	0.050	22	0.06983
11	0	-0.25	0.050	27	0.07894
12	0	0.00	0.050	32	0.08804
13	0	0.25	0.050	37	0.09715
14	0	0.50	0.050	42	0.10322
15	0	0.75	0.050	47	0.10626
16	0	1.00	0.050	52	0.10930

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17	1	-1.00	0.067	12	0.04099
18	1	-0.75	0.067	17	0.05237
19	1	-0.50	0.067	22	0.06376
20	1	-0.25	0.067	27	0.07286
21	1	0.00	0.067	32	0.08197
22	1	0.25	0.067	37	0.08880
23	1	0.50	0.067	42	0.09336
24	1	0.50	0.067	42	0.09336
25	0	1.00	0.050	52	0.10930
26	-1	0.50	0.033	42	0.11385

The RSM results were shown in the form of diagnostic plot and contour in Figures 4 and 5, respectively. Furthermore, in the diagnostic plot, the goodness of the model was checked by the correlation coefficient (R^2 value) between the experimental and model of the response variable. The R^2 values was about 0.9957, in which the value implies that the quadratic model was significant. In the contour, the particle to water ratio and extraction time significantly influenced the oil yield. More clearly, a regression analysis of the quadratic model (Table 4) shown that the main (A, B) and square (A^2, B^2) term of the independent variables were significant ($P<0.05$). On the other hand, the interaction term (AB) was not significant ($P>0.05$).

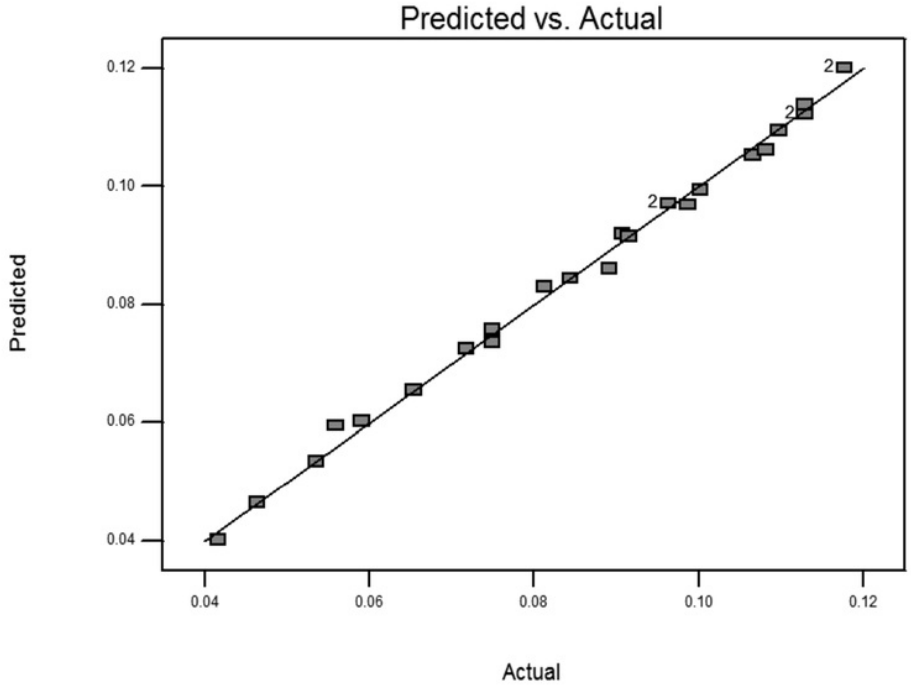

Figure 4. Comparison of oil yield predicted by the RSM model with experimental data

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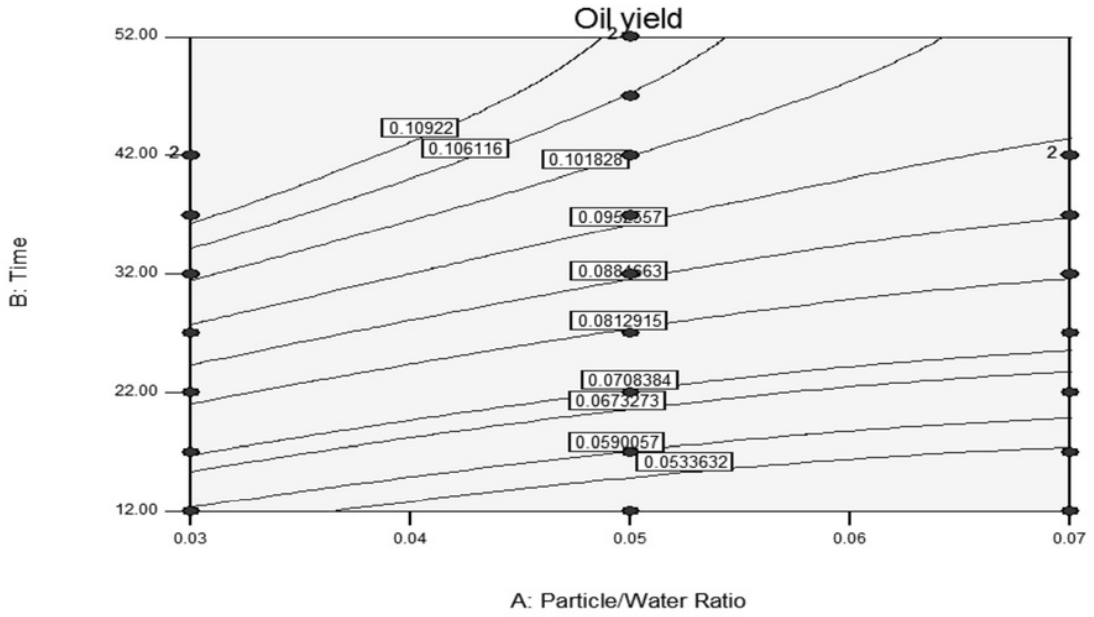

Figure 5. Contour plot showing the effects of combined particle-to-water ratio and time on the oil yield

Table 4. Regression analysis of the quadratic model using 2^2 factorial CCD

Factor	Coefficient estimate	Standard error	F-value	P-value
Intercept	0.089150	0.000702	0.087686	0.09061
A	0.001600	0.0016	600.2538	< 0.0001
B	0.008490	0.00849	3185.903	< 0.0001
A^2	0.000054	5.37E-05	20.1398	0.0002
B^2	0.000422	0.000422	158.4397	< 0.0001
AB	5.85E-06	5.85E-06	2.196463	0.1539

4. Conclusion

The mass transfer modeling of *P. cubeba* essential oil extraction using MAHD can be formulated by Fiks's second law of one-diffusional unsteady state diffusion in spherical particle with diffusion coefficient of $(2.12\pm0.619).10^{-11}$ m^2/s. In addition, the correlation of particle to water ratio and extraction time on oil yield can be expressed by quadratic regression model in which the main (A, B) and square (A^2, B^2) term were more significant than the interaction term (AB). By the point prediction of independent variable optimization it is obtained that the optimum values of particle to water ratio and time were 0.05 w/v and 32 min, respectively, while the theoretical oil yield was 57.89%.

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