Kinetics of Oil Extraction From Lemongrass (Cymbopogon winterianus Jowitt) using Hydrodistillation Method

by Sri Wahyu Murni

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Abstract

Volatile oil, or commonly known as essential oil, is a concentrated liquid that is insoluble in water and contains aromatic compounds. One source of essential oils is citronella. The extraction of lemongrass oil is carried out using the hydrodistillation method, with consideration of the solvent used in the form of aquadest so that it is easier to obtain and the time required for extraction is relatively faster. To increase the efficiency of the extraction process, kinetic modeling was carried out. This aims to predict the results before the extraction process is carried out, and considering that there are still few studies that discuss the kinetic model. So in this study, we modeled the process of extracting essential oils from lemongrass (*Cymbopogon winterianus* Jowitt) in 7 different kinetic models including pseudo-first-order and pseudo-second-order kinetics models in linear and nonlinear forms. By considering the value of the coefficient determination, from the 7 kinetic models, a non-linear form of pseudo-first-order (Non PFO-1) was chosen because it has a high R^2 value, the smallest percentage of q value and also small SSE, RMSE and MSE values.

1. Introduction

Lemongrass (*Cymbopogon winterianus* Jowitt) is a plant in the form of erect grasses, has very deep and strong roots, and the stems are erect and forming clumps. This plant can grow to a height of 1 to 1.5 meters. The leaves are single, complete and the leaf midrib is cylindrical, glabrous, and have red color on the inner surface, also have up to 70-80 cm long and 2-5 cm wide [1]. How to breed with roots that sprout. This plant can be harvested after the age of 4-8 months. Harvesting is usually done by cutting the clumps near the ground [2]. Lemongrass can produce essential oil. The main content of essential oils are citronella, citronellol, geraniol and citral. Geraniol is a compound consisting of two isopropene molecules, while citronellol is a condensation product of citronellal which belongs to the aldehyde group [3]. The extraction of citronella oil can be done through an extraction process with several methods such as hydrodistillation, steam hydrodistillation and steam distillation. Among these extraction methods, hydrodistillation is the most optimal method because it can extract oil from powdered materials (roots, bark, wood, etc.), the solvent used is aquadest which is easier to obtain, the time required for extraction is relatively faster. In addition, the hydrodistillation set are easy to move, more practical, simple and powerful [4]. Lemongrass can be utilized on the leaves, stems and pseudo-stems. According to Feriyanto et al. (2013) [5], the part of lemongrass that produces a high percentage of yield is in the leaves while the high quality of citronella oil is in the stem. Percentage of citronella in lemongrass on fresh leaves was 67.36%, withered leaves were 44.92 %, fresh stems were 75.16% and withered stems were 85.73%. In this case, the extraction will be carried out on the lemongrass stem, because according to research from Suradikusumah (1989) [6] the essential oil content in the lemongrass stem is 0.4% with the main component citronellal 66-85%. In addition, the selection of parts of citronella is also based on the utilization of lemongrass stems compared to the leaves and pseudo-stems. Therefore, extraction of lemongrass stems is carried out to increase the economic value of lemongrass stems. For the benefits that can be found in lemongrass oil, based on research that has been carried out using the spread plate method, it is known that lemongrass oil has antifungal and antibacterial activity. The active compounds in lemongrass oil that function as antibacterial are citronellal, geraniol, and citronellol which are able to inhibit bacterial activity [7]. Various industries have used lemongrass oil as a raw material for making shampoos, toothpastes, lotions, vegetable pesticides, antiseptics, and fragrance soaps [8]. Lemongrass essential oil is able to inhibit the development and even kill plant pests. Pests that can be controlled by lemongrass essential oil are citrus fruit borers, mealybugs, bed bugs, aphids, thrips, fruit flies, scale lice [9]. Other compounds in essential oils that are definitely effective for eliminating body odor are geraniol, linalool, and caryophyllene oxide. Other compounds are geranial, geranyl butyrate, eugenol and methyl eugenol [10]. In this case, to optimize the process of extracting lemongrass oil, ideally by doing a kinetic study. This is needed to predict the yield of essential oils that obtained before the extraction process, so as to increase the efficiency of the process [11]. The distillation process consists of two parts, rapid distillation (washing) of the component essential oil at or near the surface and distillation or slow diffusion through the particles followed by distillation from the outer surface of the raw material. The mathematical model is tested and established with dependencies of ln [(q$_0$-q)/q$_0$] vs t [12]. At present, kinetic modeling of extracts of various plant materials assumes: leaching and instantaneous diffusion as the basis for describing the extraction efficiency of essential oils with relate to extraction time. The applied model is simple, with relatively high precision and can be used in essential oil extraction techniques in production process practice. In particular, Markovic, et al. (2018) modeled the extraction kinetics of juniper berry material under microwave treatment showing that the wash-diffusion model is suitable for conventional and microwave-assisted extraction [13]. Based on the model created by Ho et al. (2005) [14],

Muhammad et al. (2012) [15], revealed that for essential oil extraction from materials such as grass, it was found that the distillation kinetics of lemongrass grass might follow the second model and suggested that ohmic heating could be an appropriate support to increase the yield of essential oils. Milojevic et al. (2013) also suggested that simultaneous washing and diffusion patterns might be suitable for processing plant materials [16]. An understanding of the desorption kinetics by which essential oils leave the feedstock plays an important role in practicing lab scale models. Furthermore, an understanding of the extraction kinetics of the process can also lead to improvements that can be made in existing utilization processes. For grapefruit peel material, the kinetics of essential oil extraction from this material is often modeled according to Largergren's pseudo-first-order kinetics and Ho et al's pseudo-second-order kinetics equations. This model is estimated by regression analysis to express the relation between variables and to evaluate the impact of one or more variables on the other. By observing the data obtained is not always linear, it is necessary to choose a non-linear model. It aims to select the model that best fits the experimental data obtained. Linear regression is also often used to determine the most suitable kinetic equation, especially because of its wide use in various adsorption data and some have a simple equation form [17,18]. With the development interest in the use of nonlinear optimization modeling in recent years, the transformation of non-linear equations to linear form is implicitly applied so that this will change the error structure and can also violate the error change and normality assumptions of standard least squares [19]. Therefore, different kinetic parameters will be obtained when using different forms of kinetic models. On the other hand, the non-linear method used to analyze experimental data has a more complex mathematical method in determining its kinetic parameters and is carried out on the same abscissa and ordinate, so can avoid the weakness of linearization. In this study, the comparison of the linear least squares method and the nonlinear regression method of the kinetic model was widely used to predict the best extraction kinetics and also to obtain kinetic parameters using lemongrass extraction experimental data.

To our knowledge, the kinetics of hydrodistillation of lemongrass essential oil has only been studied on a lab scale, so it may have little implications when used in large-scale instruments. In this study, we will compare linear and non-linear kinetic models of small-scale hydrodistillation processes to utilize essential oils from lemongrass as well as with various kinetic models discussed to predict the amount of essential oils obtained at a certain point in time during the process. The estimation of the kinetic model was evaluated through the coefficient of determination R^2, SSE, RMSE, MSE, and the percentage deviation between the amount of essential oil obtained at the time of saturation between the experiment and the prediction of the q model (%). The current results are expected to improve existing large-scale essential oil extraction processes and to give knowledge related to small-scale kinetic studies. Thus, in this study, we intend to carry out the Extraction of Lemongrass Oil using the Hydrodistillation Method to determine linear and non-linear kinetics modeling.

2. Materials and methods

2.1. Materials

The material used for this research is lemongrass which is taken from Jalan Godean Km. 7.5, Sidoarum, Godean District, Sleman Regency, Special Region of Yogyakarta which has an age of about 5-6 months. The part of the lemongrass to be extracted is the part of the stem where the lemongrass stems will be pretreated with uniform pieces of ± 0.5-1 cm. The sliced lemongrass is lined up to wither by allowing it to stand at room temperature for 1 day (24 h). The chemicals used are 96% ethanol, hexane and aquadest which can be obtained from CV. Progo Mulyo, Yogyakarta.

2.2. Extraction of Lemongrass Oil using Hydrodistillation Method

The raw materials used are lemongrass and aquadest. Both of these ingredients will be entered into a 1000 mL boiling flask with F/S ratio of 5:10 (w/v) (in this study, citronella of 200 g and 400 mL of aquadest were used). After the raw materials are added, the Liebig condenser is turned on and the materials are heated using heating mantle that has the following specifications: power of 350 W, voltage of 220 Volt/50 Hz and maximum temperature of 450ºC. This experiment was carried out within 120 min. The time when the distillate first drips is considered as t=0. The distillate that drips into the beaker glass every 5, 15, 30, 60, 90, 120 min will be separated from the mixture of aquadest and oil obtained using a separatory funnel. Then the essential oil obtained was weighed and the volume measured.
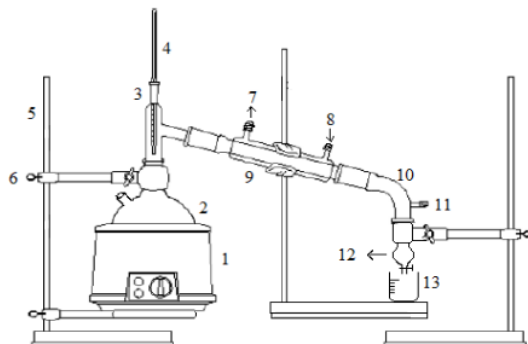
Figure 1 : Series of Lemongrass Oil Extraction Equipment using Hydrodistillation Method

The Description of Figure 1:
1. Heating Mantle   8. Cold Water Outflow
2. Two Neck Pumpkin   9. Liebig Condenser
3. Connection   10. Distillate Exit
4. Thermometer   11. Air Hole
5. Static   12. Separatory Funnel
6. Clamp   13. Beaker Glass
7. Cold Water Inflow

2.3. Kinetics of Extraction

Two kinetic models, are pseudo-first-order kinetic equations and pseudo-second-order kinetic equations are often used to describe the state of essential oil extraction from plant materials. The pseudo-first-order kinetic equation proposed by Lagergren (1898) [20] is denoted as follows:

$$\frac{dqt}{q_t} = k_1 \, (q_\infty - q_t) \qquad (1)$$

Where k_1 (min^{-1}) is the rate constant of the model, q_t (g) is the amount of essential oil obtained at time t and q_∞ (g) is the amount of essential oil at equilibrium. The pseudo-second-order kinetic equation developed by Ho et al. (2005) [14] is as follows:

$$\frac{dqt}{q_t} = k_2 (q_\infty - q_t)^2 \qquad (2)$$

Where k_2 (g.g^{-1}.min^{-1}) is the extraction rate constant. The linear and nonlinear forms of the above two equations were evaluated to determine the appropriate mechanism and estimate the kinetic parameters of citronella essential oil extraction.

3. Results and discussion

By integrating models (1) and (2) for boundary conditions ($t = 0$, $q_t = 0$, and $t = t$, $q_\infty = qt$), we can obtain linear and nonlinear forms of the kinetic model, as presented in Tables 1 and 2.

Table 1. Linear forms of Pseudo first order kinetic models

Eq.	Linear Form	Assumption	Plot	Parameter
	Linear Pseudo First Order			
(3)	$\log(q_\infty - q_t) = \log(q_\infty) - \frac{k_1 t}{2,303}$	Diffusion with no washing [13]	$\log(q_\infty, \exp - q_t)$ vs t	$q_\infty = 10^{intercept}$ $k_1 = $ -slope x 2.303
(4)	$\ln(q_\infty - q_t) = \ln q_\infty - k_1 t$	Diffusion with no washing	$\ln(q_\infty, \exp - q_t)$ vs t	$q_\infty = e^{intercept}$ $k_1 = $ -slope
	Non Linear Pseudo First Order			
(5)	$q_t = q_\infty(1 - e^{-k_1 t})$	Diffusion with no washing	q_t vs t	Solver

Tabel 2. Linear forms and solutions of Pseudo second order kinetic models

Eq.	Linear Form	Assumption	Plot	Parameter
	Linear Pseudo Second Order			
(6)	$\frac{t}{q_t} = \frac{1}{k_2 q_\infty^2} \frac{1}{t}$	Two simultaneous (a rapid and a slow extraction) [12,16,17]	t/q_t vs t	$q_\infty = slope^{-1}$ $k_2 = slope^2$ x $intercept^{-1}$
(7)	$\frac{1}{q_t} = \frac{1}{q_\infty} + \frac{1}{k_2 q_\infty^2} \frac{1}{t}$	Two simultaneous (a rapid and a slow extraction)	$1/q_t$ vs $1/t$	$q_\infty = 1/intercept$ $k_2 = intercept^2$ x $slope^{-1}$
(8)	$\frac{q_t}{t} = k_2 q_\infty^2 + k_2 q_\infty q_t$	Two simultaneous (a rapid and a slow extraction)	q_t/t vs q_t	$q_\infty = -intercept^{-1}$ x $slope^{-1}$ $k_2 = slope^2$ x $intercept^{-1}$
	Non Linear Pseudo Second Order			
(9)	$q_t = \frac{q_\infty^2 k_2 t}{q_\infty k_2 t + 1}$	Two simultaneous (a rapid and a slow extraction)	q_t vs t	Solver

Equation (5) was recently described by Milojevic et al. (2018) [13], which assumes leaching and diffusion of essential oils, found by Markovic et al. [13,21], hypothesized that diffusion is hindered and not hindered by

membranes or other obstructions present in the material. All equations in Table 1-2 were estimated with Microsoft Excel 2016. It should be noted that the value of q_∞ used to conform to Equations (3), (5) is the experimental value taken from the saturation point of the extraction. To evaluate the fit between the model and the experimental values, the coefficient of determination (R^2) and the percentage deviation q_∞ ($q(\%)$) were used to compare quantitatively the application of each model and were calculated as follows:

$$R^2 = 1 - \frac{\sum_{i=1}^{N}(q_{t,exp} - q_{t,cal})^2}{\sum_{i=1}^{N}(q_{t,exp} - q_{t,mean})^2} = \frac{\sum_{i=1}^{N}(q_{t,cal} - q_{t,mean})^2}{\sum_{i=1}^{N}(q_{t,cal} - q_{t,mean})^2 + \sum_{i=1}^{N}(q_{t,cal} - q_{t,exp})^2} \quad (10)$$

$$q\ (\%) = \frac{q_{t,exp} - q_{t,cal}}{q_{t,exp}} \times 100 \quad\quad\quad\quad (11)$$

$$SSE = \sum_{i=1}^{N}(q_{t,cal} - q_{t,exp})^2 \quad\quad\quad\quad (12)$$

$$MSE = \frac{1}{N}\sum_{i=1}^{N}(q_{t,cal} - q_{t,exp})^2 \quad\quad\quad\quad (13)$$

$$RMSE = \sqrt{\sum_{i=1}^{N}\frac{(q_{t,exp} - q_{t,cal})^2}{N}} \quad\quad\quad\quad (14)$$

Where,

q_t, exp $(g.g^{-1})$: Amount of essential oil obtained at equilibrium; namely the amount of essential oil distilled until it reaches saturation.

q_t, cal $(g.g^{-1})$: The amount of essential oil obtained from the model with the software;

q_t, mean $(g.g^{-1})$: The mean value of q;

$q\ (\%)$: Percentage difference between experimental q and calculated q;

N   : Number of data points

The kinetics of essential oil extraction from lemongrass used linear and nonlinear pseudo-first-order kinetics (Table 1) and pseudo-second-order kinetics (Table 2). The software is used to determine the kinetic parameters and predict the yield of essential oils at the saturation point, q_∞. Both can be calculated from the q_t versus t graph, as shown in Table 3. Similarly, the k_2 and q of the linear equations are obtained from the t vs q_t plot, as illustrated in Figure 2.
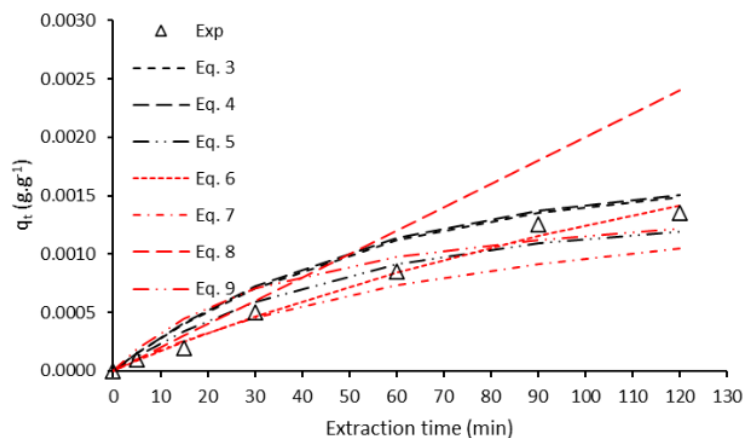
Figure 2. Experimental data and kinetic models of pseudo-first-order and pseudo-second-order in linear and non-linear forms from lemongrass oil extraction process

Figure 2 shows the experimental data of linear and nonlinear equations from kinetic models of pseudo-first-order and pseudo-second-order which describe the process of extracting lemongrass essential oil. Linear equations are often used to determine the most appropriate kinetic model and to find the least squares method used to find the parameters of the suitable kinetic model. So, in linear analysis, for the same kinetic model but different linear forms will significantly affect the calculation parameters. The difference represents a significant measure in the estimation of the kinetic parameters using the linearization technique. The different results for the linearized form of the pseudo-second-order model depend on the non-linear linearization equation so that the error factor will vary. Linearization will allow the distribution of errors better or worse. By using the non-linear method, it is not problem to transform non-linear pseudo-equations into linear form. Therefore, it is logical to use non-linear methods to represent the kinetic model efficiently and effectively. Moreover, when compared to linear equations, non-linear kinetic equations have the advantage that the error distribution remains unchanged, because all kinetic parameters are set on the same axis. From the figure, it can be seen that the experimental data points do not appear to have a straight distribution, while the distribution of data in equations 3 and 4 tends to follow a curved shape which is quite far from the distribution of experimental data, it means that the PFO 1 and PFO 2 model is not appropriate to describe the experimental data. Regarding the nonlinear form of pseudo-first-order (Non PFO-1), it was found that the experimental data points were consistent in the first and the second data with the predicted curve although they still tended to be visually visible for the next data, but this curve followed the trendline shape in the experimental data which tended to be constant at certain point. Meanwhile, for the pseudo-second-order kinetic model in linear form in equation 6 (PSO-1), the distribution of the data can be seen to be quite consistent, but the data points tend to increase continuously so that it can be said that it can not describe the process of extracting lemongrass oil. This is also for the case of equations 7 (PSO-2) and 9 (Non PSO-1) which follow a curved shape that is quite far from the experimental data and equation 8 (PSO 3) which appears to have a straight distribution. The kinetic parameters of the model include: amount of essential oil calculated in equilibrium, $q\infty$, fraction of essential oil extracted by washing, reaction rate constants on first order and second order (k_1, k_2). The parameter estimates for all models are listed in Table 3. R^2 has been a commonly used kinetics study indicator to determine the relation between experimental data and data from the model.

Tabel 3. Kinetic Parameters obtained from linear and nonlinear forms of two models

Kinetic Model	Eq.	qt calculation (g. g^{-1})	k_1 (min^{-1})	k_2 (g.g^{-1}min^{-1})	R^2	q (%)	RMSE	SSE	MSE
PFO-1	3	0.0017	0.0184	-	0.9169	22.8383	2E-04	2E-07	3E-08
PFO-2	4	0.0017	0.019	-	0.9061	24.8079	2E-04	2E-07	3E-08
Non PFO-1	5	0.0013	0.02	-	0.9419	0.0002	1E-04	8E-08	1E-08
PSO-1	6	0.0043	-	0.9280	0.9908	0.7242	5E-05	2E-08	2E-09
PSO-2	7	0.0018	-	6.1080	0.7617	17.2026	2E-04	2E-07	4E-08
PSO-3	8	20	-	5E-08	0.7654	50.5801	5E-04	2E-06	2E-07
Non-PSO-1	9	0.0016	-	16.3707	0.8908	9.5460	2E-04	4E-06	6E-07

Table 3 shows that the pseudo-first-order equation in linear form (Equation 3-4) is an equation that takes the form of a natural logarithm (Equation 3, PFO-1) with a higher R^2 coefficient, at 0.9169 than the general logarithm form (Equation 4, PFO-2). This can be caused by the transition from the exponential factor to the factor 10 of the logarithm, which causes different equations. The non-linear form of the pseudo-first-order (equation 5, Non PFO-1) shows a higher R^2 and a lower percentage of q than the linear form (equation 3-4), that means suitable for describing the experimental data. The highest percentage value of q was observed in the pseudo-first-order model (PFO-1 and PFO-2), and two of three linears models (PSO-2 and PSO-3) and pseudo-second-order non-linear model (Non PSO-1), showing that the model is not consistent with the experimental data. This is because each kinetic model has certain assumptions, example the linearization of kinetic curves to a straight line may have occurred in error, thereby inflating the oil yield calculated at the saturation point [22] and non-linear kinetics being able to adapt the data to suit different mechanisms more flexibly, i.e. the smaller difference between the calculated data and the experimental data. For the linear form of the pseudo-second-order model (PSO-1), it was observed that the value of R^2 was the highest compared to other kinetic models, but this method considered the error distribution only along the Y axis regardless of the Y axis , so resulting in differences parameters that determined for the three types of kinetic models of different pseudo-second-order for the same experimental data. Therefore, the linear method is imprecise in determining the most suitable kinetics and can not give a fundamental understanding of the extraction system kinetics, because it leads to inaccurate conclusions. This can be avoided by adopting a non-linear method to analyze experimental data, because non-linear analysis is carried out on the same abscissa and ordinate resulting in the same distribution and error structure [23]. In this condition, it will be more rational and reliable to predict the extraction data through a nonlinear regression process. This is reinforced by the results of previous studies which revealed that a non-linear pseudo-first-order kinetic model (Non PFO-1) has been found to be suitable for describing the extraction kinetics of essential oils from different materials including cinnamon, lavender and lemongrass [24]. With the data above, it is explained that the kinetic data on the pseudo-first-order kinetic model of the nonlinear form of equation 5 (Non PFO-1) has been found to be suitable for describing the mechanism of essential oil extraction from lemongrass. From the 7 models used, equations 3, 4, 5, and 6 achieve very high R^2, higher than 0.9 so it is suitable to describe the extraction kinetics. Therefore, the values of SSE, RMSE, MMSE and the percentage of q can be used as a further evaluation factor for selecting the appropriate model. From these values, it can be seen that the pseudo-first-order kinetic model of the non-linear form (Equation 5, Non PFO-1) is more suitable with the experimental data than the pseudo-first-order (PFO-1 and PFO-2) and pseudo-second-order (PSO-1) kinetic models of the linear form, because it has the lowest percentage value of q than the other models and also has the low value of SSE, RMSE, and MMSE. The selection of equation 5 (Non PFO-1) to describe the hydrodistillation process show that the transportation mechanism for the extraction of lemongrass uses diffusion without washing which is characterized by a large mass transfer at the beginning and in the end there is a slow diffusion of essential oils from the plant tissue to the outside, which is then washed away by steam and will tend to be constant over time.

4. Conclusion

Based on the 7 kinetic models discussed in our study, it was found that the non-linear pseudo-first-order kinetic modeling (Equation 5, Non PFO-1) was the most suitable to describe the extraction mechanism of lemongrass oil. These results indicate that this model fits the tested conditions, as evidenced by the high correlation coefficient ($R^2 \geq 0.9$) and the small values of SSE, RMSE, MSE and percentage of q. From the results obtained

in this model, it is hoped that it can contribute to simulation and optimization of the lemongrass oil extraction process, so that it can be used for the purposes of designing industrial plants with good operational conditions in the future.

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