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# Kinetics Study in Essential Oil Extraction from Basil Leaves by Microwave Assisted Hydrodistillation

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## ABSTRACT

Essential oil is compounds that contained in plants and have many benefits. Basil is an aromatic plant that can produce essential oils. Essential oils can be extracted from basil leaves using Microwave Assisted Hydrodistillation (MAHD) method. To determine the efficiency of the MAHD method, it's necessary to study the kinetics of the extraction method. It's because kinetic studies can be used to identify the optimal point in the process where the highest efficiency is obtained, so the extraction process can be optimized. The aim of this research is to evaluate the suitable kinetic model for the basil leaf extraction process using the MAHD method. This extraction using 100 grams of basil leaves and 200mL aquadest, heated at various microwave power 150W, 300W, and 450W. The extractions were carried out for 70 minutes with 10 min intervals. The results showed that the largest oil yield was obtained when using 450W power, which was 0.190 grams. Based on the experimental data obtained, the most suitable kinetic model is second-order model. This can be analyzed from the highest $R^2$ (0.9946 – 0.9999) and the lowest RMSE (0.0062 – 0.0349). Increasing the irradiation power will affect the extraction rate and parameter values of each kinetic model.

**Keywords:** essential oil, microwave assisted hydrodistillation, kinetic study.

## 1. INTRODUCTION

Essential oil is odourless compounds extracted from plants. Currently, the usage of essential oils is increasing not only as aromatherapy, antioxidant, anti-carcinogenic, and medicine. Extensively, essential oils can also be used in food and beverage flavourings and preservatives, cosmetics, and perfumes [1]. Essential oils in plants can be contained in specific structures such as glands, secretory hairs, and secretory cavities [2]. Essential oils can only be found in 10% of plants and a lot of them are found in Indonesia. There are a lot of types of essential oils that have been produced in Indonesia. This makes Indonesia a natural place for the essential oil industry to grow.

The types of plants that are widely extracted for their essential oil content such as citronella, cloves, eucalyptus, sandalwood, ylang ylang, basil, and many more [3]. Basil is one type of aromatic plant that has the potential to produce essential oils. The part of this plant that is usually used is the basil leaves. The essential oil content of the basil plant ranges from 0.3% to 3.6% of dry weight [4]. Considering its potential to extract essential oil, an appropriate method is needed

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to extract the essential oil from basil leaves. The most commonly used method is distillation. Distillation is a separation method using the principle of boiling point and volatility difference at a specified pressure and temperature [5]. Distillation methods that are still commonly used are the conventional methods such as hydrodistillation, steam-water distillation, and steam distillation. The use of conventional methods is not optimal because it takes a long time and has the potential to remove volatile compounds during the process [3]. An innovation to this conventional distillation method is the Microwave Assisted Hydrodistillation (MAHD) method, which combines rapid heating in the microwave field with conventional hydrodistillation [11]. Advantage of using microwave-assisted methods for essential oil extraction more effective heating, faster energy transfer, quicker response to heating control, earlier start-up, increased production, and elimination of some process steps [6]. Effectiveness of the MAHD method is proven using essential oil extraction kinetics. This method employs the help of microwaves as an additional technique so as to improve the performance of the extraction carried out [7]. Microwaves have the advantage of being easily absorbed by water [8]. This method offers greater potential for yield and is applicable on an industrial scale due to the economical configuration of the equipment [9].

Several organic solvents are volatile and harmful to humans and the environment so this research uses water as a solvent. This was carried out to reduce the increase in energy consumption and $CO_2$ emissions [10]. The use of water solvent is suitable for the MAHD method because it carries a high dielectric constant, which optimizes microwave absorption [11]. In addition, the application of water solvent is an application of the concept of "green solvent" because of its economical price, non-flammable, non-toxic, low contamination level and allows for clean procedures. Clean procedures meant that the solvent is reusable and easily biodegradable.

According to Kaderides et al. [12] the study of oil extraction kinetics and modelling is necessary because it can help in the design, optimisation and control of the extraction process. In addition, the study of kinetics can also provide a variety of information used to increase production capacity. Extraction kinetics and modelling are used to analyse the distillation process in terms of technology and process economics [13].

Previous studies on extraction kinetics have been conducted. Using the same method, research conducted by Haqqyana et al. [14] concerning the kinetics model of clove stem extraction, the most suitable kinetics model is weibull's exponential. Conversely, research carried out by Rostami [15] regarding the kinetics model for the extraction of red dates, the most suitable kinetics model is parabolic diffusion. Kusuma and Mahfud [16] also did the same research using sandalwood. Based on this, the most suitable model is second-order. Previously, the same research was conducted by Kusuma and Mahfud [16] about basil leaf extraction kinetics modelling. However, the modelling used is only limited to first-order and second-order.

The kinetics study using second-order modelling, power law model, hyperbolic model, Weibull's exponential equation, and Elovich's equation for the extraction of basil oil using Microwave assisted hydrodistillation (MAHD) method has never been done. Based on previous research, these kinetics models can be applied with flexibility to various types of reactions or extraction processes using natural materials. It is because these models take into a more detailed consideration of various factors such as temperature and concentration of materials to give a more accurate representation of the process. Looking at previous studies, different types of materials used will give different best kinetics modelling. Because the kinetics data on basil oil extraction is still limited, the main topic in this research is the rate of essential oil extraction kinetics using variable power in the microwave. The extraction kinetics assessment in this study used the second-order kinetics modelling approach, power law model, hyperbolic model, weibull's exponential equation, and Elovich's equation.

## 2. RESEARCH METHODS

### 2.1. TOOLS AND MATERIALS

The materials used are fresh basil leaves obtained from basil fields in Kebon Agung Village, Kaliwates Jember, Indonesia. Other materials are aquadest, and n-hexane. The tools used in this research are Electrolux microwave (type EMM 2308X, maximum power 800W, 23 litres, wave frequency 2450MHz, China), 1000mL round flask, and modified Clevenger distillation kit.

### 2.2. *MICROWAVE ASSISTED HYDRODISTILLATION* (MAHD)

In the application of the microwave-assisted hydrodistillation method we used a microwave (EMM2308X, Electrolux, 23 L, maximum power 800 Watt) the frequency of the waves contained in the microwave is 2450MHz. The dimensions of the PTFE-coated microwave are 46.1 x 28 x 37.3 cm. The microwave used has been modified by giving a hole at the top. A 1000 ml round bottom flask was placed inside the microwave and connected to the modified clavenger shown in Figure 1 [11].

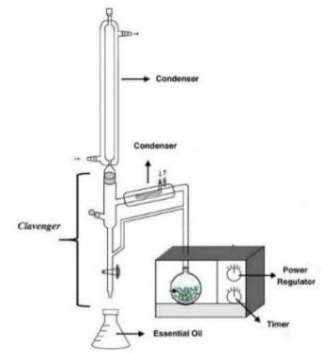


**Figure 1.** Modified clavenger.

The basil leaves to be used were first cut into two parts. After that, it is weighed until a mass of 100 grams is obtained. The leaves are cut into two parts in order to increase the surface area of contact with water or solvent. In other words, it can facilitate the evaporation of essential oils [11]. The cut and weighed leaves of O. Basilicum leaves that have been cut and weighed then added 200 ml of distilled water (leaf and water ratio 1: 2) are put into a round bottom flask and heated using microwave irradiation at 150 W, 300 W, and 450 W for 120 minutes.In the oil extraction process, the difference in density and the nature of the mixture causes water and essential oil to separate into two phases. Furthermore, both will be separated using a separating funnel. Basil oil is taken at 10-minute intervals and then placed in a vial bottle.

### 2.3. YIELD ANALYSIS

Yield is the ratio of the mass of oil produced to the raw materials used. Furthermore, the higher the amount of essential oil produced, the more result the yield will be [11]. Yield calculation calculated using the formula:

$$\bar{q} = \frac{q}{q_0} \quad (1)$$

$\bar{q}$ = the yield value of basil oil at each interval (gram)

q = amount of basil oil extracted in g/100g mass of basil extracted at time t

$q_0$= quantity of basil oil contained in basil in g/100g of basil mass measured.

### 2.4. EXTRACTION KINETICS MODEL

#### 2.4.1. Second Order Model

The second order reaction kinetics equation for the extraction rate can be seen in equation (2) below:

$$\frac{d[q]}{dt} = k_2[q - q_0]^2 \quad (2)$$

k = second order reaction rate constant ($L.g^{-1}.min^{-1}$)

The initial rate of extraction (h), reaction capacity ($q_0$), and second order reaction rate constant (k) can be determined experimentally from the slope and intercept by plotting the t/q values with t [6]. The linear equation of order 2 is given by the equation (3) and (4):

$$\frac{t}{q} = \frac{1}{q_0} \times t + \frac{1}{kq_0^2} \quad (3)$$

$$\frac{t}{q} = \frac{1}{q_0} \times t + \frac{1}{h} \qquad (4)$$

h = initial extraction rate (g/min)

### 2.4.2. Power's Law Model

The kinetic model that has previously been used in previous extraction processes is the power law model. This model shown in equation (5) is representative of the power law model.

$$\bar{q} = Bt^n \qquad (5)$$

B = power law model extraction rate constant (mL/g min)
N = exponent power law model(<1).

In simple terms, the power law model can be written in the equation (6).

$$\ln \bar{q} = \ln B + n \ln t \qquad (6)$$

By plotting $\ln \bar{q}$ versus $\ln(t)$, the values of B and n can be obtained from the intercept and linear line plot [17].

### 2.4.3. Hyperbolic Model

Hyperbolic model applied as Peleg's model:

$$\bar{q} = \frac{C_1 t}{1 + C_2 t} \qquad (7)$$

In this case,
$C_1$ = initial extraction rate ($min^{-1}$),
$C_2$ = Hyperbolic model constant ($min^{-1}$)
t = time (min).

From equation (7), it is important to state that the extraction that occurs at the beginning is first-order and drops to zero-order in the final phase of the extraction process. The linearized equation (7) will obtain equation (8) as follows:

$$\frac{1}{\bar{q}} = \frac{1}{C_1} \times \frac{1}{t} + \frac{C_2}{C_1} \qquad (8)$$

Result plot $1/\bar{q}$ versus $1/t$, will get $C_2/C_1$ as intercept and $1/C_1$ as a slope [18]

### 2.4.4. Weibull's Ekponential Equation

Application Weibull equation to the extraction of plant materials can be seen in equation (9) to determine the extraction yield:

$$\bar{q} = 1 - \exp^{\left(-\frac{t^m}{D}\right)} \qquad (9)$$

D = constant extraction rate
m = shape parameter

Different D values will produce different curves. In general, the higher the D value, hence the slower the extraction rate. Regarding the extraction process, a value of m < 1 with a parabolic curve that has a high initial slope and followed by an exponential shape [19].

Linear equations used in the Weibull equation as shown in equation (10) [15]:

$$\ln[-\ln(1 - \bar{q})] = -\ln D + m \ln t \qquad (10)$$

### 2.4.5. Elovich's Model

The reaction rate of the Elovich equation is obtained based on a decrease of compounds in reactants along with increasing extraction yield as in the following equation (11) [14]:

$$\bar{q} = E_0 + E_1 \times \ln t \qquad (11)$$

$E_0$ = initial yield
$E_1$ = Elovich's equation coefficient

From equation (11) to obtain $E_0$ and $E_1$ can be obtained by plotting the $q/q_0$ versus ln t.

## 2.5. STATISTICAL ANALYSIS

Statistical analysis used for evaluating the model in this study are the coefficient of determination or $R^2$ and RMSE (root-mean-square error) [20]. All statistical analyses and the effects of various models of extraction kinetics were assessed using Ms. Excel [21]. $R^2$ values nearest to 1 are effective and practical indicators of the model's prediction validity. While a lower RMSE value indicates that the model is suitable for use in experimental data (<0.1) [22].

## 3. RESULT AND DISCUSSION

Analysis of basil oil yield was conducted to determine the extraction effeciency using the Microwave Assisted Hydrodistillation (MAHD) [23]. The yield produced at 150W, 300W, and 450W power variations are 0.017, 0.138, and 0.190. The most optimal results can be developed for a larger scale or industrial scale. It is very possible considering the method used, MAHD makes the process more efficient compared to conventional methods due to the use of microwave energy. In terms of economics, the use of this method does require expensive initial costs. However, the process will be more efficient and provide more optimal yields by developing it according to the most suitable kinetics model.

In the 450W power variations it can be seen that the addition of basil oil yield is most significant in the first 40 minutes. However, after passing the first 40 minutes the addition of oil yield begat to decrease. But, this condition is opposite at 150W power where the most significant increase in basil oil yield occurs in the last 30 minutes. The decrease in basil oil yield after the first 40 minutes at 450W power is due to the essential oil content in the material has decreased [24]. While at 150W power, the yield of basil oil increased significantly in the last 30 minutes. This condition occurs because the power used is low, so it will take longer to reach the operating temperature and evaporation rate [24]. The low yield obtained at 150W microwave power is due to the low power so that the heat generated is less than the system temperature. This will cause a lower yield of basil oil in the same time as the other power [11].

Microwave power is an important factor that will be converted into heat energy, so it can be said that microwave power controls the amount of energy received by the material. The heat energy received by the material will help the process of releasing the oil contained in the material [25]. The higher the power used, the larger the yield produced [26]. The higher power is able to increase the operating temperature and evaporation rate. Based on this, it can be seen that the increase in power will affect the increase in temperature. This temperature increase occurs as a result of the absorption of energy from microwaves by the material. This means, the higher the microwave power used, the higher the energy received by the material to be converted into heat energy so that the yield produced is also larger [26]. The heat and mass transfer that occurs in materials with the MAHD method can be illustrated in Figure 2 [6]:

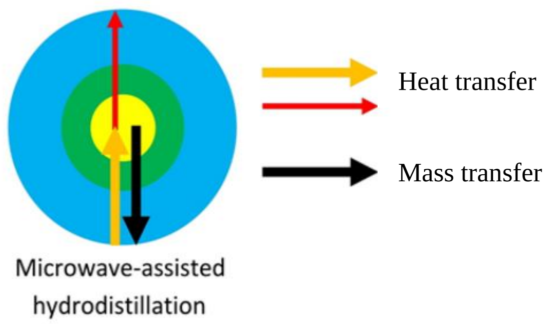


**Figure 2**. Heat and mass transfer mechanisms by MAHD method in basil oil extraction.

In Figure 2, the blue section indicates the water solvent, the green section indicates the structure of the basil leaves and the yellow section indicates the essential oil contained in the basil leaves. The red and yellow lines demonstrate the heat transfer that occurs in the extraction process from outside to inside and from inside to outside. Meanwhile, the black line represents the mass transfer of basil oil from inside to outside.

### 3.1. KINETICS MODEL

The reaction kinetics of basil oil extraction using the MAHD method can be evaluated through Figure 3.

In this research, the yield produced at each power variation will be evaluated through several kinetic models, namely, second order, weibull's exponential equation, Elovich's equation, power law, and hyperbolic model. For further analysis, the experimental results will be plotted into a graph as shown in Figure 3. The curve representing the change in basil leaf essential oil yield illustrates the occurrence of three stages in extraction. In the extraction process, there are three stages or phases of extraction. First, the equilibrium phase where solubility and partition intervention occurs. The substrate, which is the essential oil content, will be removed from the outer surface of the particles. The second phase is the diffusion phase, where mass transfer begins to be seen at the solid-liquid interface. In this phase, there is a process of mass transfer by convection and diffusion. The third phase occurs when the diffusion rate decreases as the extraction time increases. Although basil oil will still be produced, it will not make a significant difference [6].

In the extraction yield curve, for most power settings it can represent the three stages mentioned before. Where in the initial stage there is an almost linear increase of basil oil yield. Then, there is a rapid increase in basil oil yield because solutes will go to the surface of the solid material and diffuse back out of the solid material. This diffusion is caused by the solvent concentration containing the solute being greater than the solvent concentration outside the solid material that doesn't contain the solute. This is in accordance with the principle of diffusion, which is the transfer of mass from a high concentration area to a low concentration area due to molecular movement [27]. Then, there was a slow increase in basil oil yield until it reached a constant state after an extraction time of 70 minutes. The resulting yield will increase slowly at the end of the extraction stage due to the high concentration of solute, so this causes an insignificant or small increase in yield.

Based on the data in Table 1, the correlation between microwave power and parameters in each model is different depending on each model itself. The parameter values of $k_2$, $E_0$, B, $C_1$, and $C_2$ show an increase as the power increases up to 300W. However, the parameter values of $q_0$, m, D, $E_1$, n decrease. However, the opposite occurs when the power is increased at 450W, the values of $k_2$, h, $E_0$, B, $C_1$, and $C_2$ will decrease. This can be affected by several factors such as excessive heat due to increased power which will reduce the effectiveness of the solvent in the absorption of basil oil [11], thermal degradation of heat-sensitive substances [28], increased temperature which causes the interaction between solute and solvent to become weaker [29], and can cause undesired solvent extraction [28].

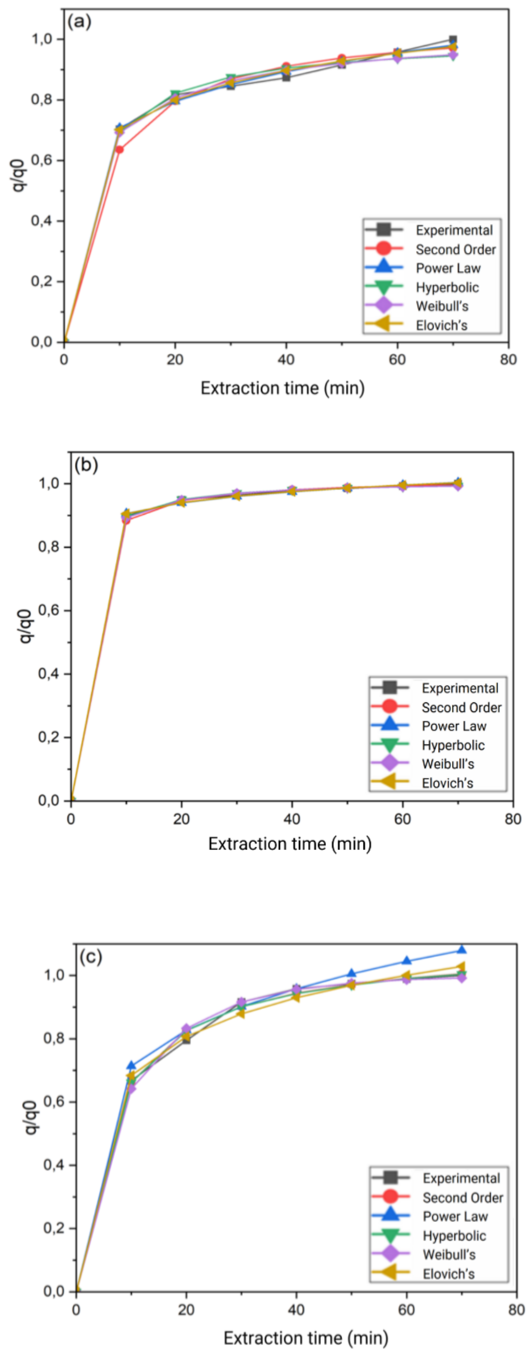


**Figure 3.** Comparison of basil oil yield at different microwave operating power (a) 150 W, (b) 300 W, (c) 450 W.

**Table 1**. Parameters of second-order, weibull's exponential equation, elovich equation, power law, and hyperbolic model for basil oil extraction by microwave-assisted hydrodistillation (MAHD).

| Kinetics Model | Parameter | *Microwave Power* (W) | | |
|---|---|---|---|---|
| | | 150 | 300 | 450 |
| Second-order | $K_2$ | 0.1389 | 0.6473 | 0.1379 |
| | $q_0$ | 1.0653 | 1.0183 | 1.0995 |
| | h | 0.1576 | 0.6712 | 0.1668 |
| | $R^2$ | 0.9946 | 0.9999 | 0.9992 |
| | RMSE | 0.0349 | 0.0062 | 0.0146 |
| Power's Law Model | n | 0.1674 | 0.0527 | 0.2125 |
| | B | 0.4816 | 0.8026 | 0.4376 |
| | $R^2$ | 0.9771 | 0.9723 | 0.9491 |
| | RMSE | 0.0142 | 0.0053 | 0.0441 |
| Hyperbolic Model | $C_1$ | 0.2255 | 0.7883 | 0.1669 |
| | $C_2$ | 0.2242 | 0.7793 | 0.1520 |
| | $R^2$ | 0.9339 | 0.9879 | 0.9844 |
| | RMSE | 0.0281 | 0.0038 | 0.0146 |
| Weibull's exponential equation | m | 0.4760 | 0.4063 | 0.8009 |
| | D | 2.5378 | 1.1401 | 6.1614 |
| | $R^2$ | 0.9345 | 0.9784 | 0.9838 |
| | RMSE | 0.0246 | 0.0597 | 0.0179 |
| Elovich's Model | $E_0$ | 0.3777 | 0.7905 | 0.2768 |
| | $E_1$ | 0.1408 | 0.0501 | 0.1770 |
| | $R^2$ | 0.9692 | 0.9770 | 0.9612 |
| | RMSE | 0.0159 | 0.0049 | 0.0225 |

**3.1.1. Second-order**

Based on Table 1, the parameters in the second-order kinetics model, $k_2$, are a constant for the second-order extraction rate and h is the initial second-order extraction rate. Based on the data obtained, there is an increase in $k_2$ with each additional microwave power up to 300W. The largest increase in extraction rate is at 300 W power where the $k_2$ and h values are 0.6473 and 0.6712, respectively. The high value of $k_2$ affects the length of time the extraction takes place, the higher the $k_2$ value, the shorter the extraction time [6]. This is in contrasts to $q_0$ which is second-order model parameter. The parameter $q_0$ which is the extraction capacity will decrease as the power increases. This is in the concept of second-order kinetics where the lower the extraction capacity, the shorter the time required, so that the value of the extraction rate will be higher. Extraction speed in MAHD is due to a combination of mass transfer and one-way heat transfer. Heat transfer with the microwave waves is from the outside to the inside of the basil leaf matrix and partly from the inside to the outside of the basil leaf matrix. Heat transfer from the inside of the matrix to the outside of the basil leaf assists mass transfer from the inside to the outside of the leaf. This is caused by microwave waves changing the matrix of basil leaves so that the oil gland ontained in basil leaves are dissolved and the oil diffused through the vapor more quickly [6].

**3.1.2. Power's Law Model**

Based on the data obtained in Table 1, the power law model parameters, B and n, are representing the extraction rate and diffusion rate. Parameter B shows an increase as the microwave power increases up to 300W. However, this is contrasted with the value of n which decreases as the power increases. The increase in B is indicated as the ability to transfer the concentration of basil oil [30]. An increase in B also indicates an increase in the ability of water to absorb the heat generated and transfer some heat to degrade the plant matrix. The degradation of the plant matrix will release the essential oil content into the liquid phase. The increasing temperature decreases the solvent viscosity and surface tension, leading to better interaction with the plant matrix and higher extraction rates. The transport process mechanism is indicated by the smaller value of the diffusion exponent n (n<1) [14].

### 3.1.3. Hyperbolic Model

Based on Table 1, the parameters of the hyperbolic model, $C_1$ and $C_2$, which are the initial extraction rate and the maximum extraction yield constant, increase as the microwave power increases, respectively. Table 1 shows that the parameters $C_1$ and $C_2$ increase as the microwave power increases. This can be related to the reason for the increase in basil oil yield which is affected by temperature. This is due to the thermodynamic effects of oil dissolution in the solid particles [18]. This is in accordance with the data obtained, that the parameters $C_1$ and $C_2$ are directly proportional to the increase in microwave power [18].

### 3.1.4. Weibull's Ekponential Equation

Other parameters observed in the kinetics model are m as a curve-shaping parameter and D as an extraction rate constant in the weibull's exponential equation model. Based on Table 1, it can be seen that D decreases along with the increase in power. The D parameter represents the time scale of the extraction process as the power increases. The decrease in D indicates that the extraction time used to produce the maximum yield is faster as the power increases. This is similar to the principles of the MAHD process when microwave heat in the electromagnetic area causes polar molecules' rapid rotation and ionic rapid motion in polar materials to produce friction. Volumetric heating determines the temperature distribution that affects extraction with MAHD. When the power is increased, the microwave irradiation raises the temperature and there is a buildup of pressure inside the basil leaf cells. This pressure causes the cell wall to explode and pushes the oil out [31]. The decrease in m is the same as the decrease in the parameter D.

### 3.1.5. Elovich's Model

The parameters used in the Elovich's equation model are $E_0$ and $E_1$. Table 1 shows the parameters in Elovich's model, namely $E_0$ as the phase transition coefficient of the extraction process, $E_1$ is the diffusion phase coefficient in the extraction process [14]. Based on the table, $E_0$ increases as the microwave power increases. This shows that the the higher the coefficient of the extraction process, $E_1$ is the diffusion phase coefficient in the extraction process [14]. Based on the table, $E_0$ increases as the microwave power increases. This shows that the higher the microwave power used will affect the mass transfer rate which becomes faster during the transition period. The $E_1$ value decreases as the power increases, which is related to the diffusion rate [32]. This parameter value is different from the research conducted by Haqqyana et al (2020) which states that the $E_0$ value does not change, this can occur because the power used is different and the type of material is different.

To evaluate the best kinetics model that can represent the experimental data, it can be analyzed using the coefficient of determination ($R^2$) and RMSE. The higher the $R^2$ value, the better the model is to be used to represent the experimental data. From all the kinetic models used in each power variation, the highest $R^2$ is obtained in the second-order kinetic model (0.9946 - 0.9999). The $R^2$ value in the second order represents that 99% of the variability in the dependent variable can be explained by the independent variables in the model. $R^2$ is used to evaluate the extent to which the kinetic model fits the experimental data obtained from the extraction process. The thing that needs to be considered to get a high $R^2$ value in the second order kinetic model is optimizing each parameter used. Another thing that needs to be considered is that the independent variable used will affect the $R^2$ value because of its influence on the dependent variable. Meanwhile, the other four kinetics models provide lower $R^2$ values than the second-order kinetics model (0.9339 - 0.9844). In relation to the best $R^2$ value in the second-order kinetics model, the RMSE value in the model is between 0.0062 - 0.0349 with the smallest RMSE value at 300W power usage. This shows that the experimental data and the calculated data are compatible.

## 4. CONCLUSION

The kinetics of basil oil extraction using MAHD with different power settings has been evaluated using five kinetics models, second order kinetics, power law model, hyperbolic model, weibull'sexponential equation, elovich's equation. The kinetic model studied proved to be suitable for modeling the kinetics of basil oil under the specified operating conditions. This is evidenced by the relatively high $R^2$ value (>0.93) and the relatively low RMSE value (<0.1) indicating a suitable fit between the experimental data and the kinetics model used. The second-order kinetics model proved to be the most suitable for the extraction of basil leaves using MAHD. This model has a high $R^2$ value of 0.9999 compared to other kinetics models. Meanwhile, the increased microwave power affects the parameter values in the second-order kinetics model. It can be concluded that the increased power will affect the extraction rate to produce a higher extraction yield in a shorter time.

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