Optimization and Kinetics of Essential Oil Extraction from Citronella Grass by Ohmic Heated Hydro Distillation

Muhammad Hazwan H., Hasfalina C. M., Hishamuddin J., and Zurina Z. A.

***Abstract*—An improved method for essential oil extraction was developed with the application of ohmic heated hydro distillation. In this study the parameters affecting citronella oil extraction by ohmic heated hydro distillation such as power input, extraction time, solvent to solid ratio and chopping frequency were investigated to achieve maximum oil yield. The kinetics of extraction was assumed and verified based on a second-order mechanism. The initial extraction rate, the saturated extraction capacity and the rate constant of extraction were calculated using the second-order model. The optimum parameters were found at voltage input of 77 V up to boiling point and 50 V until the end of extraction, 120 minutes extraction time, solvent to solid ratio of 3:1 and once chopping frequency. The kinetics and mechanism of ohmic heated hydro distillation of citronella oil has proved follow second-order model. The initial extraction rate (h) was 0.134 $gL^{-1}min^{-1}$; the extraction capacity (C_s) was 5.787 gL^{-1}; the second order extraction constant (k) was 0.004 $Lg^{-1}min^{-1}$ and the coefficient of determination (R^2) was 0.976.**

***Index Terms*—Ohmic heating; hydro distillation; citronella oil; optimization; kinetics**

I. Introduction

Citronella grass is native to India and Sri Lanka; whereby the plant can be found growing wild in most tropical Asian countries including Malaysia. Citronella belongs to the genus *Cymbopogon*, which very closely resembles and formerly was included in the genus *Andropogon* [1]. *Cymbopogon, which* is native to warm temperate and tropical regions of the Old World and Oceania, is a genus comprising about 180 species, subspecies, varieties and subvarieties [2]. The plant is also growing wild in most tropical Asian countries, America and Africa. There are two cultivated types of citronella grass, which are Ceylon (Mahapengiri) and Java (Lenabatu) citronella. Java citronella known as *Cymbopogon winterianus* Jowitt while Ceylon citronella known as *Cymbopogon nardus* (L) Rendle [3]. Normally, the plant is planted in the kitchen gardens in Malaysia. Recently, the plants are planted commercially by entrepreneurs for essential oils extraction [4]. The citronella oil is comparatively high demand due to its usage in traditional medication for antispasmodic, rubefacient, stimulant, insect repellant carminative, and diaphoretic. It also widely used in the perfume industry, soap manufacturing, cosmetics, flavoring industry and health purposes [5]-[7].

Despite of some successful practical technology used to extract the essential oil, there is still a need to consider procedure or method that able to produce essential oils at an optimum output in order to achieve acceptable production rate, energy consumption, process minimization and others. Ohmic heated hydro distillation was introduced to accelerate the extraction process. In ohmic heating, the electric current passes through the material which serves as an electrical resistance by converting the electrical energy into heat [8]-[9]. Thus, in ohmic heating the process is essentially heat generation within the material itself. Previous studies have shown the importance of ohmic heating in conjunction with several extraction processes such as lipid extraction from rice bran oil [10], extraction of sucrose from sugar beets [11], and orange peel oil extraction [12].

The application of ohmic heated hydro distillation for essential oil's plant from extraction has not been reported much. A study regarding hydro distillation of caraway (*Carum Carvi* L.) by direct induction heating assisted by a magnetic field process based on ohmic heating showed that the extraction time is much shorter than conventional hydro distillation [13]. Besides that, ohmic assisted hydro distillation of essential oils from *Zataria multifora* Boiss and *Thymus vulgaris* L. also proved that this method reduced extraction time and saved energy compared to hydro distillation technique [14]-[15]. Furthermore, preliminary study of a few plants oil extraction such as clove and lemon myrtle indicated that ohmic heated hydro distillation could be proposed as an improved method for essential oil's plant extraction as it had increased extracted oil [16]. The objectives of this study were to investigate the parameters affecting oil extraction from citronella grass by ohmic heated hydro distillation and to validate the kinetics and mechanism of ohmic heated hydro distillation of citronella grass based on a second-order model.

Second-order mechanism model means that the extraction occurs in two simultaneous processes. The amount of extracted oil increases rapidly with time at the beginning and then decreases slowly with the time until the end of extraction process [17]-[20].

The rate of dissolution for the oil contained in the solid to solution can be described by Equation (1)

$$dC_t/dt = k(C_s - C_t)^2 \tag{1}$$

where:

k = The second-order extraction rate constant ($Lg^{-1}min^{-1}$)
C_s = The concentration of oil at saturation (gL^{-1})

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C_t = The concentration of oil in the solution at any time (gL^{-1}), t (min)

The integrated rate for a second-order extraction was obtained by considering the boundary condition t = 0 to t and C_t = 0 to C_t:

$$C_t = Cs^2kt / (1+C_skt) \quad (2)$$

Linear form of the Equation (2) becomes:

$$t/C_t = (1/kC_s^2) + (t/C_S) \quad (3)$$

The extraction rate can be written as the following:

$$C_t /t = 1/[(1/kC_s^2) + (t/C_S)] \quad (4)$$

The initial extraction rate, h, when t approaches 0 can be written as:

$$h = kC_s^2 \quad (5)$$

Rearranging equation (4), the concentration of oil at any time can be obtained as:

$$C_t = t / [(1/h) + (t/C_S)] \quad (6)$$

By plotting t/Ct versus t, the initial extraction rate, h, the extraction capacity, C_s and the second order extraction constant, k, can be calculated experimentally.

II. Material and Methods

A. Raw Material Preparation

Citronella grass from Ceylon type (*Cymbopogon nardus*) was obtained from Malaysian Agricultural Research and Development Institute (MARDI) Kuala Linggi, Kuala Sungai Baru, Melaka. Four kg of citronella grass was chopped into smaller sizes by a EUMA™ Three-Phase Asynchronous Motor Y90L-2 chopper.

B. Set up of ohmic heated hydro distillation

The ohmic heated hydro distillation is as shown in Figure 1. The unit consists of a central stainless steel 316 electrode with 0.73 m long and 0.198 m diameters. An electrically insulated outer shell serves as the outer electrode. The electrodes were connected to a three-phase alternating current step down transformer 415/133/87 V power supply rated at 10 kVA. The distillation unit is then connected to a multi tube condenser made from stainless steel 304.

C. Optimization of Essential oil Extraction from Citronella Grass

The effect of four main parameters, which are power input, extraction time, solvent to solid ratio and chopping frequency were investigated to get the optimum parameters for achieving maximum yield.

The power input was investigated at two different power input namely (i) 77 V up to boiling point (±100°C) and 50 V until the end of extraction and (ii) 50 V throughout the extraction, extraction time from 15 minutes to 3 hours, solvent to solid ratio from 2:1 to 6:1 and the chopping frequency from once to three times. The optimization was varying one variable at a time method. The extracted oil was collected using the separator funnel. The oil was weighted after the extraction completed. The percentage of extracted oil was calculated by dividing the amount of extracted oil by the amount of citronella grass multiply by 100. All the experiments were repeated twice.

D. Kinetics of Extraction

After the optimized parameters were determined, the extraction was carried out in eight extraction interval time from 15 minutes to 3 hours. At the end of each interval time, the extracted oil was collected using the separator funnel. Each extraction interval time was repeated twice.

Fig. 1. An ohmic heated hydro distillation unit (1) Ohmic heated hydro distiller unit (2) Power supply (3) Multi tube condenser unit (4) Temperature sensor (5) Clamp meter

III. Results and Discussion

(i) Optimization of Essential Oil

A. Effect of Power Input

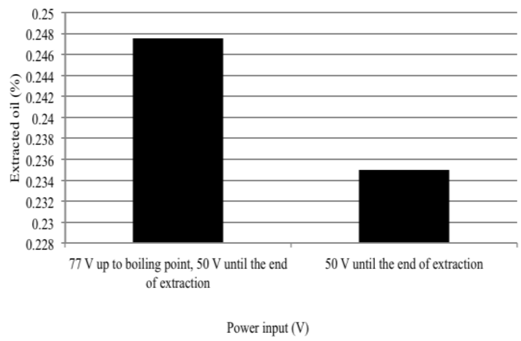

Fig. 2. Effect of power input on amount of extracted oil at 2 hours extraction time, solvent to solid ratio of 6:1 and once chopping frequency

Two different power input were used for optimization process. The extracted oil with different power input is shown in Fig. 2. Higher power input gave higher oil yield. The extracted oil of power input of 77 V up to boiling point, 50 V until the end of extraction was higher than the extracted oil of power input of 50 V throughout extraction with 0.248% and

0.235% respectively. Electrical conductivity is the most crucial factor in ohmic heating. There is a linear relationship between the heat generation rate in the center of the materials with the square of electric field strength and electrical conductivity of materials [21]. Adjusting the voltage applied can vary electrical field intensity. High voltage electrical discharges in water accelerate ‘shock waves’ during extraction [22]. In spite of this, ohmic heating speed up the breakage plant cell’s wall, hence reduce the mass transfer resistances and releases the cell’s contents into the extraction medium during hydro distillation process. Power input of 77 V up to boiling point and 50 V until the end of extraction is a better power input for citronella oil extraction.

B. Effect of Extraction Time

Fig. 3 shows the extracted oil from citronella grass at different extraction times. By increasing the extraction time, the amount of oil extracted also increase up to 0.348% whereby the maximum extracted oil is achieved at 90 minutes of extraction. Further increase in extraction process after 90 minutes did not significantly increase the oil yield. This experiment result confirmed the Fick’s second law of diffusion; whereby after a certain time, the solute concentrations in the plant matrix and in the solvent achieve final equilibrium [23]. Prolonged the extraction time was not significant to extract more oil yield. Moreover, the rate of diffusion strongly relate with the length of time required to achieve equilibrium between two phases [24]. The repetition of experiment was performed in kinetic study.

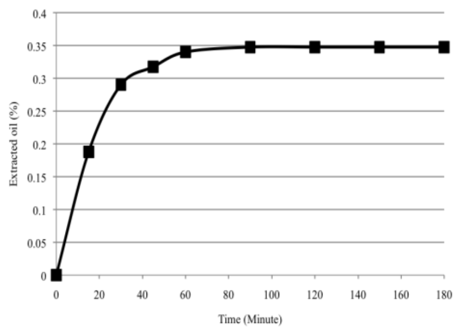

Fig. 3. Effect of extraction time on amount of extracted oil with power input of 77 V up to boiling point, 50 V until the end of extraction; solvent to solid ratio of 6:1 and once chopping frequency

C. Effect of Solvent to Solid Ratio

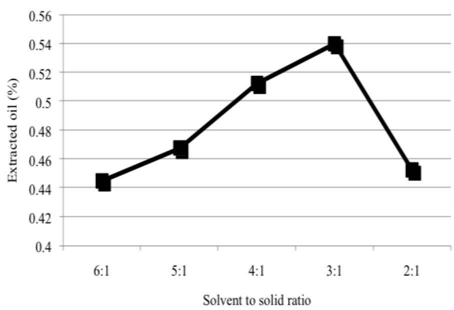

Fig. 4. Effect of solvent to solid ratio on amount of extracted oil at 90 minutes extraction time; power input of 77 V up to boiling point, 50 V until the end of extraction and once chopping frequency

Fig. 4 shows the extracted oil at five different solvents to solid ratio. By decreasing the solvent to solid ratio from 6:1 to 3:1, the extracted oil increased from 0.445% to 0.54%. However, the extracted oil decreased to 0.453% when 2:1 solvent to solid ratio was used. As the ratio 3:1 resulted the maximum yield, ratio 3:1 is the selected solvent to solid ratio. The effect of solvent to solid ratio is very essential consideration to exploit maximum extractability while scaling up or down the sample preparation method [25]. The procedures become more complex when larger solvent volumes were used. In addition, if smaller solvent volumes were used, it can make the target extraction incomplete [26].

D. Effect of Chopping Frequency

The primary purpose of chopping is to reduce the size of particle and increase the surface area that contributes to oil diffusion during extraction. The effect of chopping frequency on extracted oil is shown in Figure 5. The chopping frequency was varied from once to three times. The highest percentage of oil yield was obtained from once chopping frequency, which is 0.54%. As the chopping frequency increase, the extracted oil decrease. However, as the temperature rise when chopping or grinding process, sometimes it will affect the product in term of flavor and quality losses [27]. On the other hand, a study in food processing operation stated that during grinding, temperature is ranging from 42°C to 95°C, which leads to the loss of flavor, aroma and volatile oil of the ground powder [28]. This could probably due to the heat generated during chopping process that increases the vaporization of volatile oil. It can be concluded chopping once (using a similar chopper in this work) was sufficient to obtain a suitable particle size and hence maximum oil yield in citronella oil extraction.

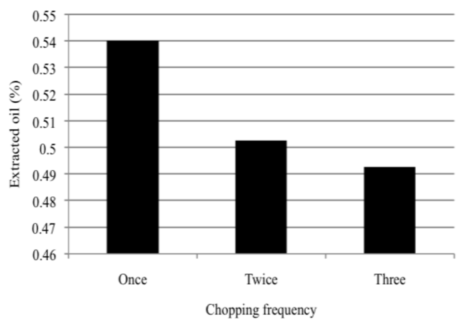

Fig. 5. Effect of chopping frequency on amount of extracted oil at 90 minutes extraction time; power input of 77 V up to boiling point, 50 V until the end of extraction and solvent to solid ratio of 3:1.

E. Kinetics of Oil Extraction

The kinetics of oil extraction from citronella grass was presented as in Fig. 6 and 7. The experiments were conducted at optimum parameters; power input of 77 V up to boiling point, 50 V until the end of extraction; solvent to solid ratio of 3:1 and once chopping frequency.

From Fig. 6, the extraction rate increased as the time of extraction increased until it reached plateau or constant after 120 min of extraction. An extractable oil of 3.65 gL^{-1} (0.37%) was obtained in the first hour of extraction until it became plateau 4.45 gL^{-1} (0.45%). Hence, 120 minutes extraction time is the optimal extraction time. The experimental result was plotted using a second-order model by plotting t/C_t versus time as shown in Fig. 7.

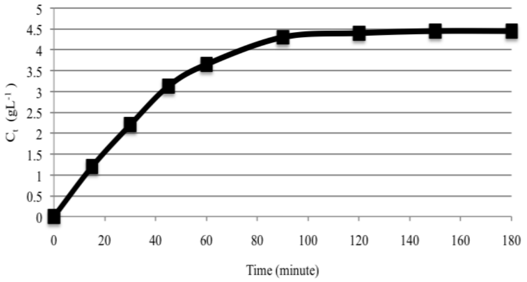

Fig. 6. Amount of extracted oil versus time of essential oil extraction from citronella grass by ohmic heated hydro distillation using power input of 77 V up to boiling point, 50 V until the end of extraction); solvent to solid ratio of 3:1 and once chopping frequency

The extraction rate fast at the beginning and slow until the end of the extraction process. Changes of solute concentration in the liquid phase affected the mass transfer of extraction process [29]. At the first stage, oil concentration was low. Thus, oil diffuses rapidly from solute to liquid phase. Besides, the free oil on the surface of citronella grass was solubilised when the solute was exposed to fresh solvent [30]. Diffusion rate decreased as the time of extraction increased due to the high solute concentration in liquid at the second stage. Although the extraction time increased after the maximum citronella oil was extracted, it did not show any changes or significant in amount of oil extracted. This finding confirms the second-order model kinetic study of citronella oil extraction [31]. According to M. Ahmed, steam distillation for citronella oil completed within three hours under normal pressure and the trend of oil recovery is as under distillation time of first hour (80%), second hour (19%) and third hour (1%) [32].

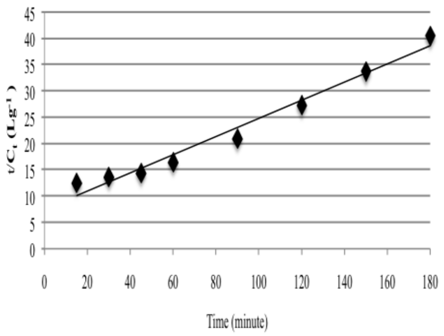

Fig. 7. Second-order extraction kinetics of citronella oil using power input of 77 V up to boiling point, 50 V until the end of extraction; solvent to solid ratio of 3:1 and once chopping frequency

TABLE I: LINEARIZATION OF SECOND-ORDER KINETIC MODEL OF OHMIC HEATED HYDRO DISTILLATION OF CITRONELLA

C_s (gL^{-1})	k ($Lg^{-1}min^{-1}$)	h (gL^{-1} min^{-1})	R^2
5.787	0.004	0.134	0.976

The straight line curve as shown in Fig. 7 proved and verified the assumptions that ohmic heated hydro distillation of citronella oil takes place in two subsequent stages. The initial extraction rate (h), the extraction capacity (C_s), the second order extraction constant (k) and coefficient of determination (R^2) were calculated experimentally by referring to the linear plot in Fig. 7. From graph t/C_t versus time, the slope is equal to $1/C_s$ and intercept is equal to $1/kC_s^2$. The data is showed in Table I.

Few studies have been conducted regarding the extraction of citronella from the same species (*Cymbopogon nardus*). 9.40% extracted oil obtained in the study of *Cymbopogon nardus* oil extraction by hydro distillation in terms of the effect of drying at different temperature of 50, 60 and 70 ℃ on the composition of oil extracted [33]. Reis et *al*. found that there were no significance difference on the composition of oil extracted even different temperature were used. Another study in the extraction of *Cymbopogon nardus* essential oil using supercritical CO_2, the operational parameters studied were from 313.15 to 353.15 K for the temperature and the applied pressures were 6.2, 10.0, 15.0 and 180.0 MPa. The best yield was 2.06% at 353.13K and 18.0 MPa of pressure [7]. Economic study should be conducted to further review this improved method which is ohmic heated hydro distillation in terms of costing such as labour, total operating cost and total cost of production for its application in small-scale industry.

IV. CONCLUSION

In this paper, an ohmic heated hydro distillation was optimized and modeled which ohmic heated hydro distillation could be proposed as an improved method for essential oil's plant extraction. Further, economic analysis and product quality analysis such as physical and chemistry oil analysis need to be analyzed. Also microscope study investigated for structural changes observation need to be conducted. Comparison with conventional method such as hydro distillation, steam distillation and solvent extraction could be suggested for further studies too.

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