Essential Oil from Extraction and Steam Distillation of Ocimum Basillicum

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Abstract— ***Indonesia is one of the essential oil producers in the world. Ocimum Basillicum can be easily found in Indonesia, nevertheless the production of essential oil from this plant has not been explored widely, and therefore the aim of the research is to find the optimum condition for extraction of Ocimum Basillicum with two solvents, i.e. ethanol and n Hexane. In this study, the steam distillation was also applied to Ocimum Basillicum.***

Two grams of air dried Ocimum Basillicum leaves and stems and 400 mL of solvent were placed in the extraction flask, equipped with stirrer and waterbath to control the temperature. Samples (1 mL) were taken every 5 minutes, until the equilibrium was reached. Concentration of essential oil in the solvent was determined by using UV-visible spectrophotometer. The same procedure was run for three different temperatures: 33 ^oC, 45 ^oC and 55 ^oC. Two different solvents: n Hexane and ethanol were used in the experiment.

From the GCMS analysis showed that leaves and mixture of leaves and stems gave the same compounds in the essential oil produced, but the mixture of leaves and stems gave a better product in terms of appearance although leaves gave higher yield. The higher the extraction temperature, the better the extraction process was. N Hexane was a better solvent for extraction of Ocimum Basillicum than ethanol, not only in terms of equilibrium but also in term of kinetics. The mass transfer coefficient was evaluated by fitting the experimental concentration profile to the unsteady state mass balance equation, followed the dimensionless equation:

$$Sh = 0.225\ Sc^{0.3}\ Re^{0.6}$$

The average relative errors were 2%.

From a 300 g of Ocimum Basillicum leaves and stems, 1.9 g of essential oil was produced using steam distillation compared to 4 g of oil produced from solvent extraction.

***Index Terms*— Distillation, essential oil, mass transfer, Ocimum Basillicum , solvent extraction.**

I. Introduction

Ocimum Basillicum means aromatic plant [2]. The oil of *Ocimum Basillicum* is categorized as high essential oil, which means the aroma will evaporate within 24 hours after it is applied to the body. *Ocimum Basillicum* oil can be used for aroma therapeutic massage by lightening and refreshing the body. It can also reduce the intensity of digestion problem, head aches, strained muscles and nervous breakdowns. (*Center for New Crops and Plant Products, Purdue University, US).*

Indonesia currently produces some essential oil, namely Ylang – ylang, vetiver and sandalwood oil [4], but the quality of the oil can not be maintained consistently to satisfy the world requirement. *Ocimum Basillicum* essential oil yet to be produced in Indonesia, eventhough *Ocimum Basillicum* is thriving in Indonesia. The manufacturing of *Ocimum Basillicum* as essential oil, both in small and middle scale industry has not been developed yet, due to the unavailable of optimum condition for the extraction process, whereas in fact there is a quite high demand from overseas.

The aims of this research are to find the optimum condition of essential oil extraction from the *Ocimum Basillicum*, to determine the equilibrium and kinetics parameter of the extraction process and to compare it to the steam distillation method. From the following result, the small and medium scale industry may wish to apply the technology.

II. THEORY

Essential oil is an easily evaporating compound and insoluble in water. Essential oil can be isolated from the plant's tissue by using either the distillation or extraction processes [3]. However, many industries prefer to use the distillation process. Essential oil comes from every part of the plant including ; leaf, stems, flower, seed, branch, root, additionally essential oil is commonly used in cosmetics, drugs, and perfume [2]. There are 80 kinds of essential oils in the international market, yet Indonesia only exports twelve oils, Patchouli Oil, Vertiver Oil, Cintronella Oil, and Cloves Oil are a few examples [1].

Ocimum Basillicum belongs to *lamiaceae* family, as shown in Fig 1, is 50 cm tall, contains oval-shape leaves, white or purple flower, and has a distinctive-aroma [10].

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Fig 1. *Ocimum Basillicum* source [10]

There are many chemical compounds in *Ocimum sp*., 0.20-1% of essential oil is contained in the dried leaf. The most common compounds in *Ocimum Basillicum* are *linalool and methylchaviol,* with cineol represented to a lesser extent [10].

There are several standards which must be fulfilled to mantain the quality of *Ocimum Basillicum* essential oil [9], namely :

- Appearance, color, fragrance : transparent fluid or pale yellow, *Ocimum Basillicum's* distinctive flavour and fragrance.
- Specific Gravity on 25°C **:** 0.95200 **–** 0.97300
- Refractive Index on 20°C : 1.51200 - 1.51900
- Optical gyration on 25°C : [-] 8.85º to [-] 11.85º
- Well mixed with Hydroxycitronellal
- Dissolved in paraffin oil
- Insoluble in water

References [5] and [6] studied the extraction of Citronella oil from lemon grass using the n Hexane and ethanol as solvent, from the data obtained, n Hexane was better than ethanol, in terms of equilibrium and kinetics.

The solid-liquid extraction process was used for this research. Both equilibrium and kinetics data are needed to design the industrial scale equipment.

In solid-liquid extraction, the solute mass transfer from solid to liquid occurs in two stages of process [8], namely:
1. Diffusion from the inside of the solid material to its surface.
2. Mass transfer from the surface of the solid material to liquid.

Because the size of the solid material is small enough, it is assumed that the solute concentration within the solid material is always homogeneous. Thus there is no gradient concentration within the solid material. In other words, the effective diffusivity within the solid material can be neglected. Therefore mass transfer between phases controls the overall mass transfer, in this case kca is the determining factor
From the mass balance of the essential oil in the liquid phase:

$$k_c \frac{A}{V}(C_A^* - C_A) = \frac{dC_A}{dt} \quad \text{.....(1)}$$

with ; $\frac{A}{V} = a$

$$k_c a\,(C_A^* - C_A) = \frac{dC_A}{dt} \quad \text{.....(2)}$$

C_A	=	oil concentration in the liquid phase	,g oil/ cm^3
A	=	Interfacial area of solid	,cm^2
C_A^*	=	oil concentration in the liquid phase which in equilibrium with the oil concentration in the solid material,	,g oil/ cm^3
$k_c a$	=	Volumetric mass transfer coefficient	,1/s
V	=	Solvent volume	,cm^3
t	=	Time	,s

Overall mass balance of the essential oil at a certain time:
The initial mass of the essential oil = mass of oil within the soluble + mass of oil within the solid material

$$W \,.\, X_o = C_A \,.\, V + W \,.\, X_A$$

$$X_A = \frac{WX_0 - C_A V}{W} \quad \text{.....(3)}$$

W	=	Solid weight	,g
X_A	=	Oil concentration in the solid material	,g oil/g solid
X_0	=	initial oil concentration in the solid material	,g oil/g solid

From the above differential equations with the result data of C_A as a function of time, the mass transfer coefficient was evaluated by fitting the experimental concentration profile to the unsteady state mass balance equation. The value of $k_c a$ can be determined by using the Matlab program.

The mass transfer coefficient was related to the affecting variables by dimensionless equation:

$$\frac{kca.dp^2}{D_L} = K\left[\frac{N.dp^2.\rho}{\mu}\right]^a \left[\frac{\mu}{\rho.D_L}\right]^b \quad \text{.....(4)}$$

dp	=	solid size	,cm
D_L	=	diffusivity of the essential oil in solvent	,cm^2/s
N	=	stirrer speed	,rps
ρ	=	solvent density	,g/ cm^3
μ	=	solvent viscosity	,g/cm.s

$$Sh = K \,.Re^{\,a}\, .Sc^{\,b} \quad \text{.....(5)}$$

Re = Reynolds number , $\left[\frac{N.dp^2.\rho}{\mu}\right]$

Sh = Sherwood number , $\frac{kca.dp^2}{D_L}$

Sc = Schmidt number , $\left[\frac{\mu}{\rho.D_L}\right]$

III. EXPERIMENTAL

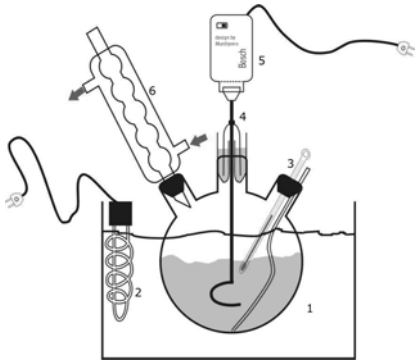
The materials used are: *Ocimum Basillicum,* water, aquadest, solvents : n-Hexane and Ethanol

The schematic diagram of the equipment used in this experiment is seen in Fig 2.

Four hundreds cm^3 of n Hexane, were placed into the three neck extraction flask, equipped with a stirrer and water bath, to control the temperature of the process. The temperature was set at 55 ^oC. Two grams *Ocimum Basillicum* were put into the extraction flask, and the stirrer speed was set to 5 rps. Samples were taken every 5 minutes and repeated until the 7-hours-stirring process was completed. The experiment concluded when the equilibrium was reached (concentration within the soluble was relatively constant). The same procedure was carried out for three different temperatures: 35^oC , 45^oC and 55^oC , and the same experiment was carried out using ethanol as a solvent.

Equilibrium data for n Hexane and ethanol was collected by conducting a series of experiment using different weights of *Ocimum Basillicum* leaves and stems at three temperature 35^oC , 45^oC and 55^oC respectively,

The analysis of *Ocimum Basillicum* essential oil in the samples were determined by using a UV-visible spectrophotometer. Steam distillation was applied to *Ocimum Basillicum* leaves and the mixture of leaves and stems.



1. Waterbath
2. Heating coil
3. Thermometer
4. Stirrer
5. Stirrer motor
6. Reverse cooler

Fig 2. Diagram of Extraction Equipment

IV. RESULT AND DISCUSSION

Steam distillation was applied to *Ocimum Basillicum* leaves and a mixture of leaves and stems. The result of the experiment is shown in Table I:

Table I Steam distillation of Ocimum Basillicum plant

Raw material	Leaves	Mixture	Unit
Weight of material	300	300	g
Distillation time	135	135	min
Distillate rate	0.5083	0.526	cm^3/s
Distillate Volume	3600	3800	cm^3
Essential oil	1.9790	1.7208	g
Yield, %	0.6597	0.5736	%
Appearance of oil	Turbid pale yellow	Clear pale yellow	
Water content	11.4	10.75	%

The result shows the yield of essential oil from only leaves was slightly higher than the essential oil from mixture of leaves and stems. The appearance of the later was better, hence the next experiment was conducted by using the mixture of leaves and stems as raw material. Essential oil from distillation process was analyzed using GCMS, and the result is shown in Table II.

Table II. GCMS Analysis of Ocimum Basillicum essential oil

Name	Formula	Leaves,%	Mixture,%
3,7-dimethyl 2,6-Octadienal	$C_{10}H_{16}O$	84,8	85,46
Linalool	$C_{10}H_8O$	3,88	2,62
Linalool Oxide	$C_{10}H_8O_2$	2,67	2,24
Carryophyllene Oxide	$C_{15}H_{24}O$	1,96	1,9
Others organics material	-	6,69	7,78

International quality standards of *Ocimum Basillicum* essential oil, requires 85 % of 3,7-dimethyl 2,6-Octadienal (Methylchaviol) and 3 % Linalool. It is shown in Table II, both oil from only leaves and a mixture of leaves and stems satisfy the specification.

The equilibrium experiment data were oil concentration in solvent at different weight of Ocimum Basillicum leaves and stems. The oil content in the solid (X_A) was calculated by Equation (3), and the result of calculation n Hexane is shown in Fig 3.

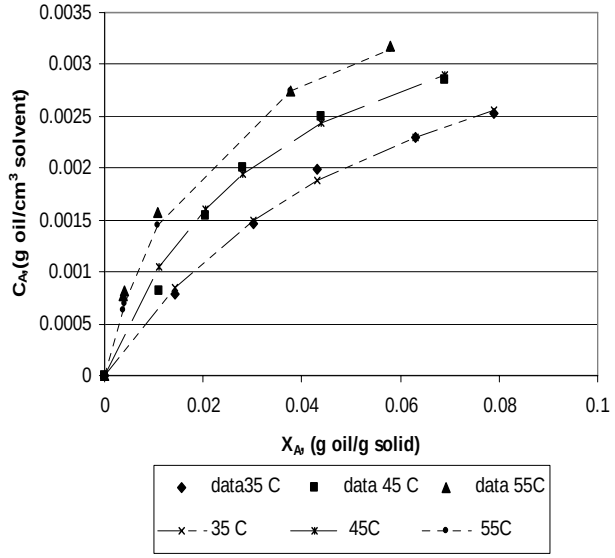


Fig 3. Equilibrium curves of the Ocimum Basillicum essential oil in n Hexane solvent

The equilibrium data of *Ocimum Basillicum* essential oil in Ethanol can be seen in Fig 4.

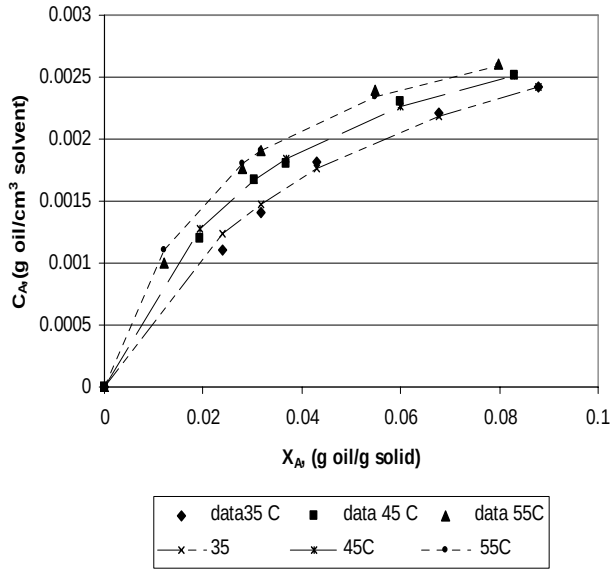


Fig 4. Equilibrium curves of the *Ocimum Basillicum* essential oil in Ethanol solvent

Three equations were applied to the equilibrium data , i.e., Henry, Langmuir and Freundlich. Based on the relative error, the fit equation was chosen. Both for n Hexane and Ethanol, the Langmuir equation fit the equilibrium data best.

$$C_A^* = \frac{k.C_{As}.X_A}{1 + k.X_A} \quad \text{.....(6)}$$

k = Langmuir constant, g solid/g oil
C_{As} = maximum oil concentration in the liquid phase, g oil/ cm^3

The Langmuir constant for both solvents is shown in Table III.

Table III. Langmuir Constant for equilibrium using the n Hexane and Ethanol solvents

		35C	45C	55C
N Hexane	C_{As}	0.004556	0.00442	0.0043
	k	16.2	27.85	47
Ethanol	C_{As}	0.003776	0.003563	0.003408
	k	20.2	28.9	40.17

From the equilibrium curves, both for n Hexane and Ethanol, the higher the extraction temperature, the better the extraction process was. N Hexane was a better solvent for extraction of *Ocimum Basillicum* than Ethanol, so that the required solvent was less.

The kinetics experiment data were oil concentration in solvent as a function of time. The experiment was carried out at three different temperatures, 35^{o}C , 45^{o}C and 55^{o}C. Fig 5 shows the data obtained for n Hexane. The higher the temperature, the faster equilibrium reached, means the faster the extraction process.

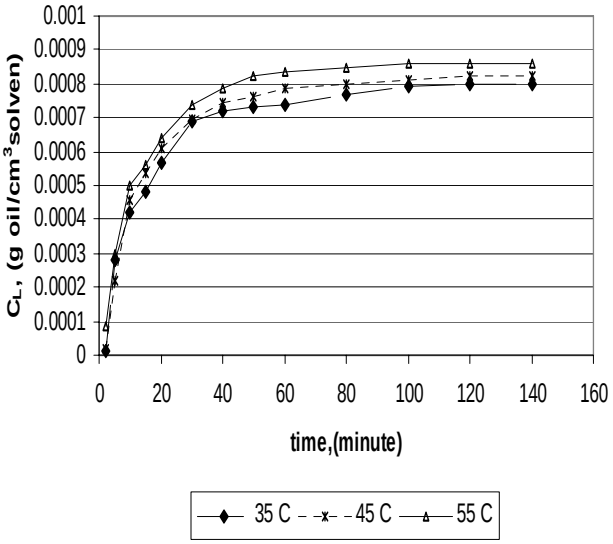


Fig 5. Kinetics experiment data of the Ocimum Basillicum essential oil in n Hexane solvent

Kinetics experiment data for Ethanol can be seen in Fig 6.

Using the Matlab program, the value of volumetric mass transfer coefficient between phases k_{ca} can be determined. The calculation results for both solvents can be seen in Table IV.

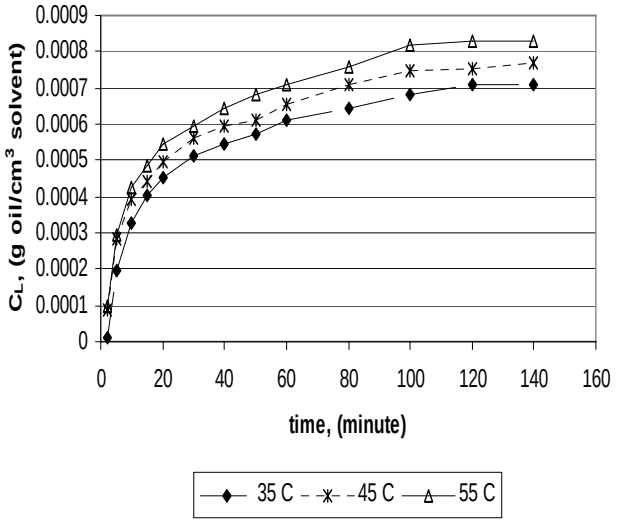


Fig 6. Kinetics experiment data of the Ocimum Basillicum essential oil in Ethanol solvent

Table IV Kinetics parameters

Temp, K	$D_L,10^5$ cm^2/s	kca, 1/s	Sc	Re	Sh
Hexane					
308	2.02	0.00145	219.74	1126.8	71.78
318	2.08	0.00152	196.16	1222.6	73.07
328	2.15	0.0016	173.85	1337.5	74.4
Ethanol					
308	0.638	0.00046	2113.2	370.36	72.1
318	0.659	0.000496	1705.6	444.44	75.2
328	0.68	0.000535	1404.3	523.33	78.67

The mass-transfer coefficient of the *Ocimum Basillicum* essential oil in Ethanol is less than the mass-transfer coefficient of the *Ocimum Basillicum* essential oil in N Hexane. Thus, it took less time to extract the *Ocimum Basillicum* oil by using the n Hexane compared to using ethanol. N Hexane was a better solvent for extraction of *Ocimum Basillicum* than ethanol, not only in terms of equilibrium but also in terms of kinetics.

The resulting equation is :

$Sh = 0.225\ Sc^{0.3}\ Re^{0.6}$

The average relative errors were = 2 %

Using steam distillation, 1.9 g of essential oil would be produced compared to 4 g of oil when the extraction process was applied, from a 300 g of *Ocimum Basillicum* leaves and stems. However, using the extraction method, the oil should be separated from its solvent by using the distillation method. Both methods, solvent extraction and steam distillation should be considered based on economic evaluation.

Microwave-assisted extraction of tea polyphenols and tea caffeine from green tea leaves was investigated by [7], and found that yield of the microwave assisted extraction was higher compare with the ordinary solvent extraction. Therefore applying this method might be a better idea to get the higher yield.

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