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A Combination of Water-Steam Distillation and Solvent Extraction of *Cananga Odorata* Essential Oil

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Abstract: *Cananga odorata is an agricultural product with potential to produce essential oil because of its high volatile components content. In this study, the essential oil production of Cananga odorata from Indonesia was investigated using a combination of water-steam distillation and solvent extraction. Depending on extracted oil during distillation, the mass transfer modelling as a main extraction parameter to the design calculation was described. Meanwhile, in the solvent extraction, influence of difference solvent of oxygenated compound group of the produced oil was studied in order to increase in the oil quality. From the experiment results, the cananga oil yield of the water-steam distillation is about 0.936% v/w of fresh flower and the oil contains several components like trans-carryopyllene (39.03% w/w), alpha humulene (11.59% w/w), alpha bergamotene (11.29% w/w), and germacrene (10.94%). It is observed that the oil mass transfer occurs in two regimes i.e. constant mass transfer and decreasing mass transfer regimes. It is found that the oil mass transfer of water-steam distillation is about 2.52x10^{-6} mole/g.min.mmHg. In addition, the solvent extraction of the oil produced by the water-steam distillation can increase the oxygenated compounds, from 15.86% increase to 25.3 and 25.34% for chloroform and diethyl ether solvents, respectively.*

Keyword: *Cananga odorata, essential oil, water-steam distillation, solvent extraction, mass transfer coefficient*

I. INTRODUCTION

Cananga odorata, commonly known as Kenanga (Indonesia), ylang-ylang (Philippine), canang odorant (French); lanalana (Hawai‘i); makosoi, mokohoi, makasui, mokosoi (Fiji); moto‘i (Society Islands); moto‘oi, mata‘oi, mato‘oi (Tahiti); and cananga (English), is a medium-size tree at about 10–20 m in height that has been introduced for perfumes. This species can be found growing in forests, agro-forests, and garden. Its flowers blossom throughout the year in auxiliary, umbellate hanging clusters of 4–12 flowers, each with three and six petals up to 8 cm long. The young petals are twisted and are very fragrant with greenish yellow colour. Then they limp when reaches maturity which are less fragrant and deep yellow in colour. The fragrant flower can be extracted to produce the essential oil. The cananga essential oil can be used as an alternative source in the perfume industry and aromatherapy, flavouring agent in candles, icings, frozen dairy, pudding, baked goods, soft drinks and chewing gums, and food additive. As aromatherapy, the essential oil is useful for treatment of depression, distressed breathing, high blood pressure, anxiety, as an aphrodisiac, etc. Accordingly, cananga essential oil has been approved as food additives by US Food and Drugs Administration (FDA) and has been considered for food uses by Flavor and Extract Manufacturers Association (FEMA) and the International Organization of Flavor Industries (IOFIs). On other hand, in Java culture, the flower is used against malaria and to treat asthma.

In the literatures, cananga essential oil can be extracted using pressing/cold expressing [1], water distillation, water-steam distillation, steam distillation, effleurage, solvent extraction, supercritical fluid extraction, vacuum microwave hydro-distillation, and solvent-free microwave hydro-distillation methods [2]. Furthermore, there are many chemical compounds in the cananga essential oil, such as heavy oxygenated compound (benzyl benzoate); light oxygenated compound (eugenol, benzene acetate); oxygenated monoterpene (geranyl acetate, geraniol, linalool); sesquiterpen hydrocarbon (germacrene, trans-caryophyllene, p-methylanisole, camphene, anethol); nitrogenated compound (phenylacetonitrile, 4-methylbenzaldoxime, indole, methyl anthranilate) [2], [3]-[7]. Due to practicality, water-steam distillation method is opted more than other ones. In water-steam distillation, the mass transfer of the cananga essential oil is main parameters to determine the kinetics parameter of the extraction process in order to design the industrial scale equipment. In water-steam distillation, the mass balance equation can be expressed by.

$$\begin{pmatrix} the\ oil\ amount \\ of\ flower\ at\ time\ t \end{pmatrix} = \begin{pmatrix} the\ oil\ amount \\ of\ flower\ at\ t = 0 \end{pmatrix} - \begin{pmatrix} the\ oil\ amount \\ that\ has\ been\ produced \\ at\ time\ t \end{pmatrix} \quad (1)$$

$$x\left(W_{flower} - W_{oil}\right) = W_{flower}\, x_{oil\,0} - W_{oil} \quad (2)$$

$$x = \frac{W_{flower}\, x_{oil\,0} - W_{oil}}{W_{flower} - W_{oil}} \quad (3)$$

where: x is the essential oil content in flower during extraction.

In addition, the mass transfer of essential oil from flower surface occurs in two regimes i.e. constant mass transfer and decreasing mass transfer regimes. The mass transfer of essential oil is assumed constant when the essential oil yield does not yet reach a critical point. The essential oil yield in this step is higher than the critical yield and the mass transfer area in the flower surface is still constant. Whereas, when the essential oil content reached the critical yield, so the mass transfer of essential oil starts to decrease. A lot of researches that studied the mass transfer and kinetics of essential oil distillation as an important research component, such as a steam distillation for *Pimpinella anisum* essential oil [8], a steam distillation for *Cymbopogon winterianus* essential oil [9], a hydro/steam distillation for Ylang-ylang oil [10], and a steam distillation for *Ocimum Basillicum* [11]. In addition, the results of this model can be compared with the other models.

While, the extracted cananga essential oil by distillation method can produce not only volatile compound (essential oils) but also non-volatile compound (resin, wax, pigment, gums, fatty acids and other anhydrous oils) [2], [5]. Consequently, it is potential to purify of the cananga essential oil in order to increase the volatile compounds, especially the oxygenated compound group as the odoriferous components. The solvent extraction is one of chemically methods expect distillation and adsorption that can be applied to separate the impurities of cananga essential oil [2]. The distillation method to purify the essential oil produced by distillation must be operated at high pressure due to the boiling point of the essential oil components is high. However, the adsorption method must be conducted in very complex procedures in order to achieve the high efficiency of molecular sieve of the various oil components. Interestingly, in the present study, the essential oil purification that be conducted is solvent extraction method. In addition, the chosen solvent solutions are chloroform (a non-polar organic solvent) and diethyl ether (a non-polar organic solvent that slightly polar). This chosen has a prediction that the essential oils are complex mixtures of a great number of different components that can be divided in the functional groups like acid, alcohol, ketone, aldehyde, esther, ether, and alkane. These functional groups have different polarity that can be written from the most to less of the polar rank as follows: acid > alcohol > ketone > aldehyde > esther > ether > alkane. Thus, the influence of solvent polarity of solvent extraction can be studied. For that previously reasons, the aims of this research are to model and find the mass transfer coefficient of the *Cananga odorata* essential oil extraction using water-steam distillation and to study the influence of organic solvent solution of the solvent extraction in order to increase the volatile components of the essential oil.

II. Experiment And Method

A. The theoretically essential oil content analysis of *Cananga odorata* essential oil

The *Cananga odorata* flower was sliced and weighted at 5 g, and then it was extracted via soxchlet aparatus with n-hexane solvent at temperature of 70 - 80 ^{o}C until 20 of cycles. The attained cananga oil was distillated to evaporate the solvent at temperature of 70 ^{o}C until the oil solution volume of about 5 mL. After that, the essential oil was dried in oven to evaporate the residual solvent. The theoretically essential oil yield is a ratio number between essential oil with fresh flower weight.

B. The water-steam distillation of *Cananga odorata* essential oil

300 g *Cananga odorata* flower is extracted in 7 L pressure vessel operated at pressure of 1.5 atm (Fig. 1). The pressure vessel is equipped with a condenser, pressure gauge (in the range of 0-10 atm), ¼" pressure relieved valve, safety valve, basket, and glass level indicator. The condensed vapour flow through in 50 mL beuret in order to measure the volume of the essential oil and water every time interval of 30 min. During the process, the pressure distillation is kept constant at 1.5 bar. The pressure distillation was operated above the atmospheric condition in order to increase the extraction temperature, so that the essential oil yield can be increased. The extraction was stopped after the distillation time reach 8 hours. The essential oil density measurement was conducted using a liquid pycnometer. And then, the essential oil constituents were analysed by a Hewlett Packard HP5890 series II plus gas chromatograph equipped with a HPMS 5989b mass spectrometer using electron impact. The mass spectrometry (MS) conditions 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 other essential oils of known composition, with pure compounds and by matching the MS fragmentation patterns with WILEY229 mass spectra libraries and with those in the literature. The relative amounts of the individual components were obtained from GC analysis based on peak areas using the generalized equation of Van den Dool and Kratz [12].

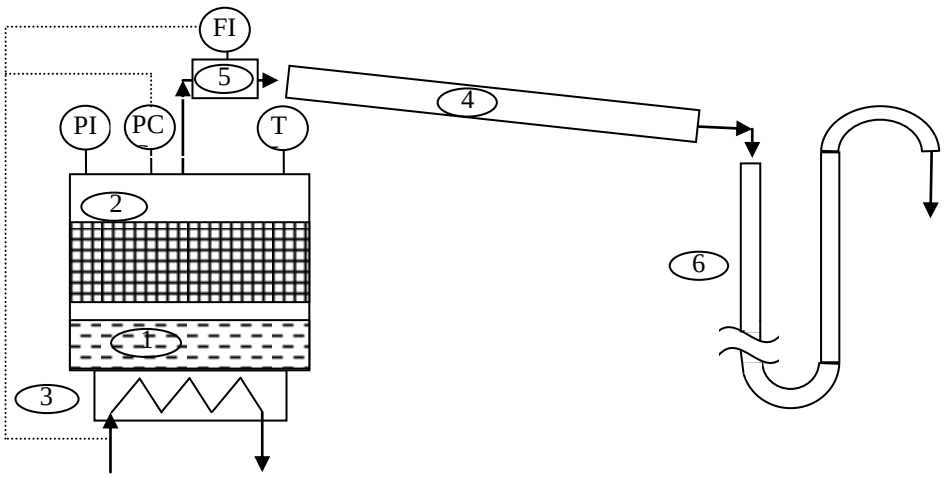

Fig. 1. Equipment schema of water-steam distillation (1: water area, 2: simplisia area, 3: heater, 4: condenser, 5: vapour valve, 6: beuret)

C. The solvent extraction of *Cananga Odorata* essential oil

Initially, the mixture of 50 mL of cananga essential oil and 50 mL of solvent and 1% w/v of sodium chloride was placed in the three neck extraction flask equipped with a magnetic stirrer and condenser to prevent the solvent to come out due to the solution high volatility. Sodium chloride was added to facilitate the emulsion breakdown. The extraction is stopped when the reaction time has reached 1 hour. The variable of the research is extraction solvent i.e. chloroform (SIGMA 3045) and diethyl ether (SIGMA 3098). Before the solvent was evaporated to its recovery from the mixture with essential oil, sodium sulphate was added to remove the organic phase followed by filtration. The evaporation was conducted under a vacuum in a rotary-evaporator (SIGM 1209) at temperature of 30 ^{o}C for 4 hours. The produced cananga essential oil was stored in dark bottles at 4 ^{o}C prior to composition analysis using GC-MS method.

III. RESULT AND DISCUSSION

From the experiment of essential oil content using a standard procedure [1], the theoretically essential oil yield is about 1.159 and 1.324% w/w of the fresh flower for experiments 1 and 2, respectively. From the water-steam distillation, the obtained essential oil yield was about 0.936% w/w of the fresh flower and the density of one was about 0.9026 g/mL. It is obvious that water-steam distillation at pressure of 1.5 bar is an efficient process that can extract the Cananga essential oil with the theoretically yield of about 71 to 81%. The oil and water volumes in distillate can be seen in Table 1 and the oil mole at various times is plotted in Fig. 2. The numbers of mole and mass were then calculated by the assumption of the water density = 1 g/mL (pure water), the water molecular weight = 18 g/mole, the oil density = 0.9026 g/mL (constant during the process), 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 1.

Table 1: Water and oil in the distillate

time	water			essential oil			time	water			Essential oil		
(min)	mL	g	mole	mL	G	mole	(min)	mL	g	mole	mL	g	Mole
0	0	0	0	0	0	0	165	1017	1017	165	1.80	1.6247	0.0099
15	42	42	15	0	0	0	180	1197	1197	180	1.90	1.7149	0.0105
30	72	72	30	0.30	0.2708	0.0017	195	1392	1392	195	1.95	1.7601	0.0107
45	117	117	45	0.40	0.3610	0.0022	210	1602	1602	210	2.00	1.8052	0.0110
60	177	177	60	0.50	0.4513	0.0028	225	1827	1827	225	2.05	1.8503	0.0113
75	252	252	75	0.70	0.6318	0.0039	240	2067	2067	240	2.10	1.8955	0.0116
90	342	342	90	0.90	0.8123	0.0050	255	2322	2322	255	2.15	1.9406	0.0118
105	447	447	105	1.10	0.9929	0.0061	270	2592	2592	270	2.20	1.9857	0.0121
120	567	567	120	1.30	1.1734	0.0072	285	2877	2877	285	2.25	2.0309	0.0124
135	702	702	135	1.50	1.3539	0.0083	300	3177	3177	300	2.30	2.0760	0.0127
150	852	852	150	1.70	1.5344	0.0094							
165	1017	1017	165	1.80	1.6247	0.0099							

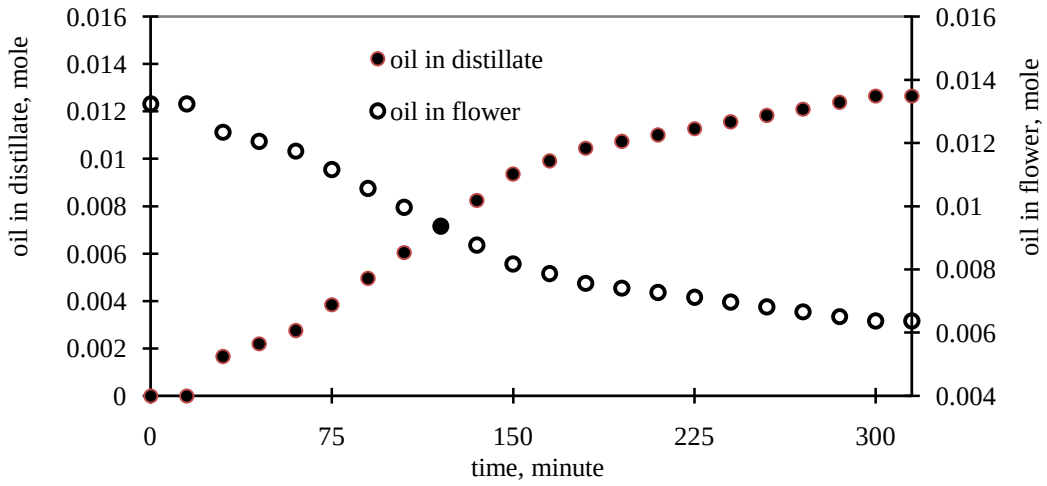

Fig. 2. Essential oil in distillate and flower

In Fig. 2, it is obvious that the correlation of the oil fraction mole between times has two phenomena. The first phenomenon is increasing of the oil mole during distillation from 0–105 minutes can be expressed by a linear curve. And the second is increasing of the oil mole starts to decrease (above 105 minutes). Accordingly, the point that increasing of oil mole starts to decrease is as a critical point of the extraction [13], [14]. The phenomena are caused by the mass transfer of essential oil from flower surface in water-steam distillation which occurs in two steps as follows.

The mass transfer of essential oil is constant

The process assumption can be applied until when the essential oil content reach a critical point. The essential oil yield in this step is higher than the critical yield and the mass transfer area in the flower surface is still constant. The essential oil yield in the flower can be calculated by Equation (3). The calculation results can be seen in Table 2 and Fig. 2.

Table 2: Essential oil fraction in flower

time (min)	oil fraction in flower (x)	Time (min)	oil fraction in flower (x)	time (min)	oil fraction in flower (x)
0	0.013240	120	0.009365	225	0.007116
15	0.013240	135	0.008767	240	0.006966
30	0.012349	150	0.008167	255	0.006815
45	0.012051	165	0.007867	270	0.006665
60	0.011753	180	0.007567	285	0.006515
75	0.011157	195	0.007417	300	0.006364
90	0.010561	210	0.007266	315	0.006364
105	0.009963				

From Table 2, the critical yield of the oil extraction attained at the time of 105 minutes and the essential oil yield in the distillate of 0.00939. This step occurs fast, consequently water and vapour are equilibrium and immiscible, so that the relation equations of pressure and water vapour fraction can be written as follows:

$$y_{water} = \frac{P^o_{water}}{P} \quad (4)$$

However, a relation equation of pressure and essential oil vapour fraction is as in Equation 5.

$$y_{oil} = \frac{P^o_{oil}}{P} \quad (5)$$

At the vapour phase, the water and essential oil fraction relation is

$$y_{water} + y_{oil} = 1 \quad (6)$$

Therefore the essential oil vapour fraction can be expressed into mole fraction in form

$$y_{oil} = \frac{m_{oil}}{m_{oil} + m_{water}} \quad (7)$$

Rearranging above equations gives Equation (8).

$$m_{oil} = \frac{y_{oil}}{1 - y_{oil}} m_{water} \quad (8)$$

Equation (8) show that the relation between essential oil mole with water mole in distillate in step 1 is a linear curve with slope of $y_{oil}/(1-y_{oil})$. From the experiment data we can make a plot of oil mole (m_{oil}) versus water mole (m_{water}), so that the value of essential oil fraction in vapour can be calculated. Consequently, with Equation (6) we can evaluate the essential oil pure vapour pressure. The results of the curve linear regression of oil mole versus water mole of the *Cananga odorata* essential oil distillation when the process time duration is 105 minutes can be seen in Fig 3. The slope obtained is about $2.6x10^{-6}$, so that the oil fraction and pure pressure in the vapour (y_{oil}) are about 0.00026 and 0.152 mmHg, respectively.

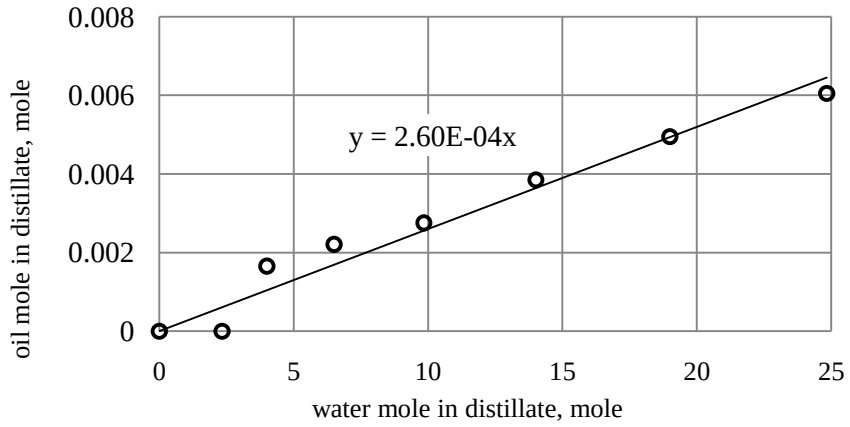

Fig. 3 Linear regression result between oil mole versus water mole in distillate

The mass transfer of essential oil decreases

The process can be applied if essential oil content reaches the critical yield and continue to decrease. In this step, the essential oil yield as well as the mass transfer area in the flower surface decrease. The mass transfer rate between phases from liquid phase to vapour phase can be arranged by Equation (9) and can be numerically solved by forming of Equation (10).

$$\beta . K_y a = \frac{\left(\frac{dm_{oil}}{dt}\right)}{m_{flower} . \left[P^o_{oil} - \left(\frac{dm_{oil}}{dm_{oil} + dm_{water}}\right) . P_T\right]} \quad (9)$$

$$\beta . K_y a = \frac{\left(\frac{\Delta m_{oil}}{\Delta t}\right)}{m_{flower} . \left[P^o_{m} - \left(\frac{\Delta m_{oil}}{\Delta m_{oil} + \Delta m_{water}}\right) . P_T\right]} \quad (10)$$

If $\frac{\left(\frac{\Delta m_{oil}}{\Delta t}\right)}{m_{flower} . \left[P^o_{oil} - \left(\frac{\Delta m_{oil}}{\Delta m_{oil} + \Delta m_{water}}\right) . P_T\right]}$ = Z, so equation (10) becomes:

$$Z = \beta . K_y a \quad (11)$$

The value of β can be assumed by Equation (12) and then substituted in Equation (11). The result is Equation (13).

$$\beta = e^{-\alpha (x - x_c)} \quad (12)$$

$$Z = e^{-\alpha (x - x_c)} . K_y a \quad (13)$$

Equation (13) can be solved by the logarithm procedure as follows:

$$\ln (Z) = -\alpha . (x - x_c) + \ln (K_y a) \quad (14)$$

Parameters α and $K_y a$ can be found by linear regression of $-(x - x_c)$ versus ln (Z) which the values of α and K_ya are values of slope and intercept of the line. Finally, from the data of oil and water moles in distillate, the mass transfer coefficient of *Cananga odorata* essential oil of water-steam distillation is about 2.52 x 10^{-6} mole/g.min.mmHg.

The GC-MS analysis results of the *Cananga odorata* essential oil

The GC-MS analysis results of the *Cananga odorata* essential oil extracted by water-steam distillation and solvent extraction can be seen in Fig. 4 and Tables 3. The composition can be accordingly divided by the oxygenated compound as in Table 4.

In Fig. 4 and Table 3, It can be seen that the main composition of *Cananga odorata* essential oil produced by water-steam distillation were trans-carryopyllene (39.03% w/w) followed alpha humulene (11.59% w/w), alpha bergamotene (11.29% w/w), and germacrene (10.94%). However, after the essential oil extracted by chloroform, the main composition were trans carryopyllene (26.10% w/w) followed by germacrene (12.04%),

and alpha humulene (10.79% w/w). After the essential oil extracted by diethyl ether, the main compositions were trans carryopyllene (25.01% w/w) followed germacrene (10.31%), and alpha humulene (10.86% w/w). At the crude oil, oil extracted by chloroform, and oil extracted by diethyl ether, the compound group of the essential oils were mainly the same. The most abundant group is sesquiterpen. Other compound groups with high amount are oxygenated monoterpen, high oxygenated, and heavy oxygenated compounds. The oxygenated compound is highly odoriferous, while the sesquiterpene hydrocarbon contributes only little to fragrance produced. The quality of cananga oil increases with the content of oxygenated compounds which is the result of this purification with the solvent extraction by chloroform. The oil extracted by chloroform and diethylether contain much more oxygenated compound than the crude oil (25.3 and 25.34% versus 15.86%). On the other hand, the sesquiterpen hydrocarbon is present in a higher quantity in the crude oil than the oil extracted by chloroform and diethylether (84.13% versus 76.09 and 75.33%). Therefore the purification of *Cananga odorata* oil by solvent extraction using chloroform yields better than the crude oil produced by water-steam distillation.

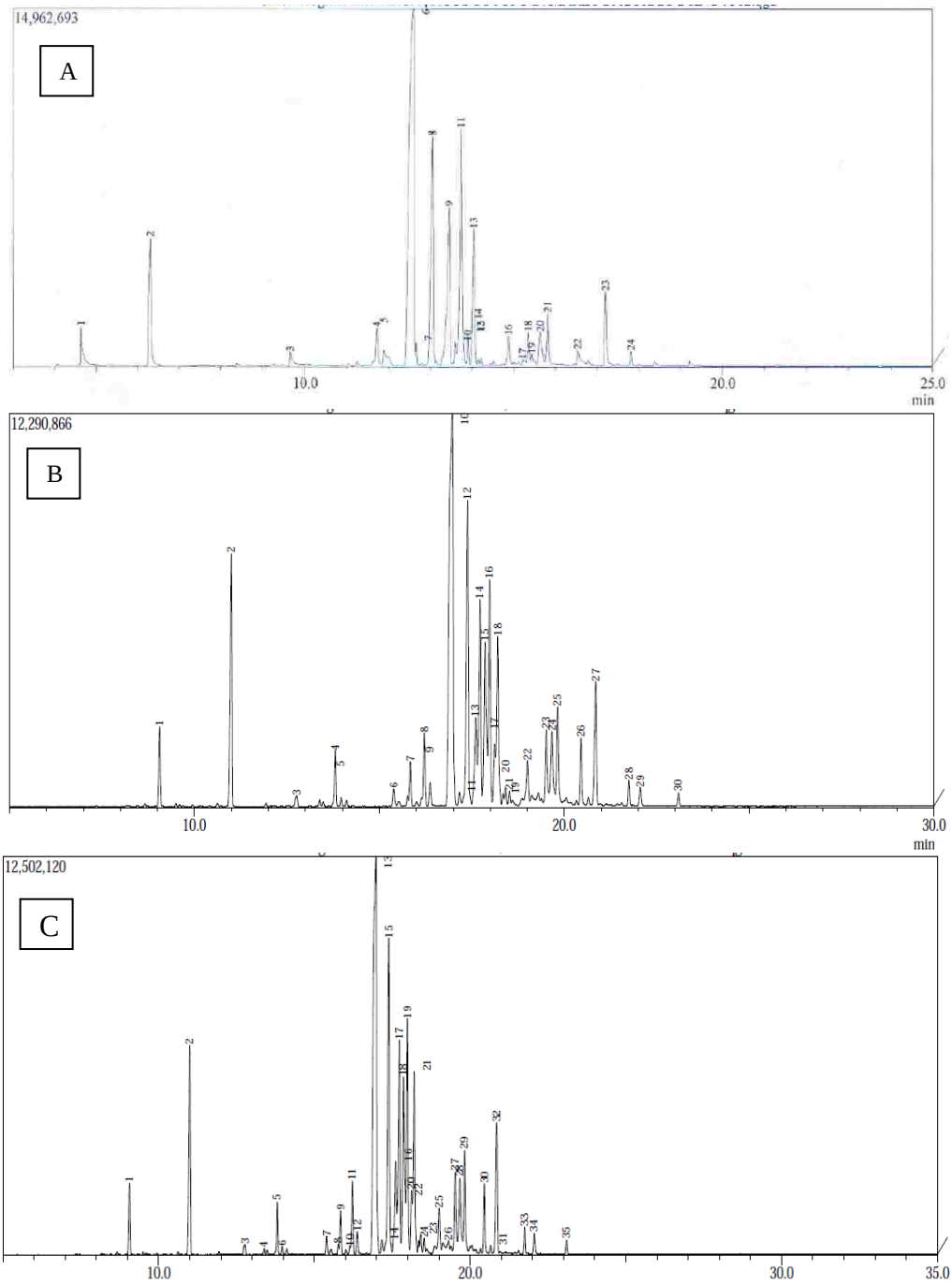

Fig. 4 Gas chromatography of A: *Cananga odorata* oil crude; B: *Cananga odorata* oil extracted by chloroform; C: *Cananga odorata* oil extracted by diethyl ether

Table 3. Chemical composition (mass %) of *Cananga odorata* crude oil; *Cananga odorata* oil extracted by chloroform; and *Cananga odorata* oil extracted by diethyl ether

No	Composition	Type	Crude oil	Oil extracted by chloroform	Oil extracted by diethyl ether
1	Anisole	S	1.42	2.38	2.1
2	Linalool	OM	6.37	7.36	6.76
3	Geraniol	OM	0.51	1.56	1.46
4	alpha Copaene	S	2.31	1.71	2.07
5	Geranyl acetate	OM	0.49	0.36	1.06
6	trans-Carryopyllene	S	39.03	26.1	25.01
7	Germacrene	S	10.94	12.04	10.31
8	alpha-Humulene	S	11.59	10.79	10.83
9	alpha Bergamotene	S	11.29	-	-
10	delta-Cadinene	S	5.44	5.36	7.64
11	Naphthalene	S	0.13	3.4	
12	Carryophylene oxide	S*	1.19	1.29	1.21
13	Humulene oxide	S*	0.2	-	0.65
14	Globulol	OM	0.93	2.29	2.42
15	alpha Longipinene	S	0.16	6.76	6.91
16	Torreyol	S	1.82	2.95	2.84
17	alpha Cadinol	OM	2.05	3.21	3.39
18	Farnesol	OM	0.69	1.78	1.98
19	Benzyl benzoate	HOC	2.98	1.75	4.14
20	Farnesyl acetate	OM	0.45	0.11	0.63
21	Eugenol	LOC	-	1.41	0.68
22	beta Elemene	S	-	0.65	0.63
23	alpha Farnesene	S	-	6.19	6.76
24	Cububene	S	-	0.26	0.23
25	Nerolidol	OM	-	0.28	0.28

S = sesquiterpen hydrocarbon
S* = oxygenated sesquiterpen
OM = oxygenated monoterpen
LOC = light oxygenated compound
HOC = heavy oxygenated compound

Table 4. Compound group of essential oil

Compound	Crude oil	Oil extracted by chloroform	Oil extracted by ether
S	84.13	76.09	75.33
S*	1.39	1.49	1.86
OM	11.49	16.95	17.98
LOC	0	2.82	1.36
HOC	2.98	4.04	4.14

IV. CONCLUSIONS

The *Cananga odorata* oil can be produced by a combination of water-steam distillation and solvent extraction using chloroform and diethylether. The mass transfer of the essential oil of water-steam distillation is about 2.52×10^{-6} mole/g.min.mmHg and the pure vapor pressure is about 0.152 mmHg. The main compound of the essential oil produced by water-steam are trans-carryopyllene (39.03% w/w), alpha humulene (11.59% w/w), alpha bergamotene (11.29% w/w), and germacrene (10.94%). The solvent extraction of the essential oil using chloroform and diethylether can be increase the oxygenated compounds, from 15.86% increase to 25.3 and 25.34% for solvent of chloroform and diethyl ether, respectively. On the other hand, the sesquiterpen compound can decrease due to purification using solvent extraction by chloroform and diethyl ether, from 84.13% decrease to 76.09 and 75.33%, respectively. Therefore the solvent extraction by chloroform and diethylether of the cananga essential oil produced by water-steam distillation can increase the oxygenated compound group.

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