

Microwave Assisted Hydrotropic Distillation of Myrcene-Rich Essential Oil of *Cymbopogon Citratus*

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Abstract. Myrcene can be used as a versatile starting material for a myriad product for various applications. The drawbacks of the available myrcene separation techniques promote the investigation of application of environmentally clean, faster, more powerful, and cheaper methods. The combination of microwave heating system and application of urea in hydrotropic-based distillation of myrcene-rich essential oil of *Cymbopogon Citratus* was investigated in this work. A mathematical model of microwave assisted hydrotropic distillation (MAHtD) of essential oil based on the diffusion-control assumption is also proposed and validated. The MAHtD of *C. citratus* were performed by varying urea concentrations (20-30%) and temperature (105-110°C). The essential oil composition as well as its concentration were analyzed and determined by applying GC-MS. Research result shows that urea is proved as a good hydrotrop for MAHtD of myrcene-rich essential oil of *C. citratus*. MAHtD performed with 25% urea at a temperature of 105°C for 10 minutes gives a high myrcene content of up to 53.53%. The obtained myrcene-rich essential oil of *C. citratus* can be investigated for its utilization in various types of applications. Moreover, the proposed model was validated with the experimental data of MAHtD performed at a solid-liquid ratio of 1:10, 25% urea solution, and a temperature of 105°C. The experimental data show a good agreement with the proposed model with SSE of 0.033. The obtained effective diffusivity of the essential oil and the Henry constant in which essential for industrial scale-up process are $2.245 \times 10^{-7} \text{ cm}^2/\text{s}$ and 0.42, respectively. The effective diffusivity value was higher than those obtained from hydro distillation and microwave-assisted hydro distillation of other essential oil, which shows that urea, is a good solubilizing agent for *C. citratus* essential oil.

Keywords: *Cymbopogon citratus*; Hydrotropic; Microwave; Myrcene

1. Introduction

Myrcene (C₁₀H₁₆), an acyclic monoterpene in two isomeric forms (α and β - myrcene), is derived from the Myrcia species plant family. Bay leaves, verbena, pine, lemongrass, rosemary, juniper, rose, ginger, and celery are some of myrcene plant sources (Bai and Tang 2020). Beta-myrcene is often denoted as myrcene. The highly active diene structure of myrcene makes it a versatile starting material for menthol, geraniol, nerol, and linalool technical synthesis (Behr and Johnen 2009). Different types of myrcene

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derivatives are applied in various applications, such as fragrances, cosmetics, soaps, vitamins, pharmaceuticals, polymers, insect repellents, flavors, and biodegradable surfactants (Behr and Johnen 2009). Some literature also mention that myrcene exhibits biological activities such as anti-diabetic, antioxidant, anti-cancer, analgesic, anti-inflammatory, anti-biotic, sedative and anti-mutagenic properties (Bai and Tang 2020).

Industrial myrcene is produced from the pyrolysis of β -pinene, catalytic dimerization of isoprene, and the extraction from myriad myrcene plant sources (Behr and Johnen 2009). Hydro distillation, solvent extraction, and supercritical fluid extraction of myrcene are methods applied in the myrcene separation process. Hydro distillation and solvent extraction are commonly performed at high temperatures and require three-to-four hours of process. The possibility of losing the essential volatile compound and altering its odor characteristics are two examples of both techniques' drawbacks. Meanwhile, supercritical fluid extraction is effective, but the device is highly sophisticated, uneconomical, and operationally complex. The drawbacks of myrcene separation techniques promote the demand for investigating the potential application of environmentally clean, faster, more powerful, and cheaper methods.

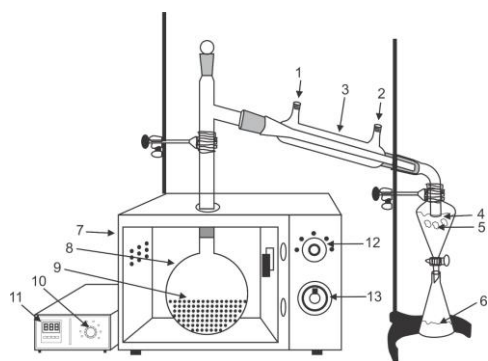
Hydrotropes are chemicals that increase the solubility of hydrophobic molecules, such as monoterpene in a water-based hydrotrope solution (Hartati *et al.* 2021). Hydrotropic solubilization has been applied in the separation of terpene, such as andrographolide from *Andrographis paniculata* Ness (Hartati, Anas, and Kurniasari 2015), abiatane diterpenoid (carnosic acid) from rosemary (Mazaud *et al.* 2020), and eugenol from clove buds (Ghule and Desai 2021). Sodium benzoate is applied in the hydrotropic separation of andrographolide (Hartati, Anas, and Kurniasari 2015). Short-chain alkyl polyethylene glycol ethers are used to separate carnosic acid from rosemary (Mazaud *et al.* 2020). Sodium salicylate and sodium cumene sulfonate are utilized in the extraction of eugenol and eugenol acetate (Ghule and Desai 2021). Urea, a cheap and environmentally friendly compound, is one of hydrotropes utilized in solubilization of several poorly-water soluble compounds as nifedipine (Cui 2013) and rice straw lignin (Hartati *et al.* 2021).

Considering the potential performance of hydrotropes in solubilizing monoterpene myrcene, urea in combination with microwave heating is applied in the separation of myrcene from *Cymbopogon citratus*. Microwave heating is prized in the chemical process as it gives benefits such as shorter time consumption, efficiency, low energy source, and considering an environmentally friendly method (Yustanti, Trenggono, and Manaf 2020; Maulida *et al.* 2020; Paramita, Yulianto, and Hartati 2019; Harahap *et al.* 2019; Kusuma and Mahfud 2015). Furthermore, mathematical modeling of MAHtD of essential oil, considered a fundamental step in establishing an efficient and scalable industrial process, is also proposed and validated in this research.

2. Methods

2.1. Materials

Cymbopogon citratus leaves were collected from Kalisidi, West Ungaran, Central Java. They were withered for 24 hours at room temperature and chopped into 1 cm length. Urea ($(\text{NH}_2)_2\text{CO}$) with a purity of 99% was produced by Pupuk Kujang Inc.



- | | |
|------------------|---------------------------------|
| 1. Water Outlet | 8. Distillation flask |
| 2. Water inlet | 9. Leaves of <i>C. citratus</i> |
| 3. Condenser | 10. Power button |
| 4. Water-EO | 11. Temperature panel |
| 5. Essential oil | 12. Time selection mode |
| 6. Water | 13. Power level mode |
| 7. Microwave | |

Figure 1 Microwave distillation unit

2.2. Procedure

Sixty g of dried and chopped *C. citratus* leaves, and 600 mL of urea solution were placed in the three-neck glass jar and inserted into the microwave distillation unit (EMM2308X model from Electrolux) with a condenser and temperature control (Figure 1). The microwave power level was set to a high level (800 W), and the microwave-assisted hydrotropic distillation (MAHtD) was performed at a temperature of 110°C. The microwave distillation unit is completed with the temperature control system (Figure 1). Samples were taken and separated using a pipette, and the oil volume was recorded.

2.3. GC-MS analysis

The essential oil was taken using a dropping pipette to measure its chemical compound using a Shimadzu single quadrupole GCMS-QP2010 SE type of GC-MS. The GC-MS non-polar fused silica capillary column has an inside diameter of 0.25 mm, a length of 30 m, and a film thickness of 0.25 µm. The column temperature was initially programmed at 60°C and increased at 3°C/min to 150°C and 10°C/min to 250°C. The final temperature was maintained for 15 minutes, while the injector and detector temperature was set to 250°C. Helium gas was used as the carrier gas with a flow rate of 0.52 mL/min, a total flow of 8.7 mL/min, a linear velocity of 26.3 cm/sec, a purge flow of 3.0 mL/min, and a split ratio of 1:10.

2.4. Modeling

The essential oil separation process can be considered solid-liquid extraction (Phat *et al.* 2021; Xiong, Chen, and Shen 2019; Cassel, Vargas, and Joseph 2006). In solid-liquid extraction, the solid is contacted with a solvent. Solubilized solute and the solute mass transfer proceed through two simultaneous steps, i.e., solute diffusion from the inner part of the solid particle to the solid surface followed by mass transfer of solute from the solid surface to the bulk of the liquid. The mathematical modeling of *C. citratus* essential oil was constructed by assuming that diffusion in the solid particle is the controlling step. The *C. citratus* leaves are thin slabs, and the cross-sectional area was relatively large. Solid particles' solute diffusion (essential oil) was processed in one direction (perpendicular to a large surface). Essential oil diffusion on solid particles follows Fick laws as presented in Equation 1.

$$N_A = -D_e \cdot \frac{\partial C_A}{\partial x} \quad (1)$$

Where C_A is the essential oil concentration (g of essential oil/cm³ of solid), x is the position, and D_e is the effective diffusivity of essential oil in solid particles (cm²/s). The mass balance equation of essential oil in slab volume element is written in Equation 2.

$$\frac{\partial^2 C_A}{\partial x^2} = \frac{1}{D_e} \frac{\partial C_A}{\partial t} \quad (2)$$

The initial and boundary conditions for Equation 2 are as follows:

$$C_A(x, 0) = C_{Ain} \quad (3)$$

$$C_A(0, t) = C_f/H \quad (4)$$

$$C_A(L, t) = C_f/H \quad (5)$$

where C_f is the essential oil concentration in the bulk of the liquid, and H is the Henry constant. The boundary condition (Equation 4 and Equation 5) are obtained by assuming that mass transfer of essential oil from solid surface to the bulk of the liquid is relatively fast so that the essential oil concentration in the solid surface is equal to the one in the liquid phase and thus is presented in an equation similar to Henry law.

To solve Equation 2 with the initial and boundary conditions as represented by Equation 3 to Equation 5, an equation representing C_f (Equation 6) is constructed based on the mass balance of essential oil in the solid and liquid phases.

$$C_f = C_{fo} + \frac{n \cdot S}{W} \left(L \cdot C_{Ain} - \int_0^L C_A \cdot dx \right) \quad (6)$$

Furthermore, the essential oil extracted can be calculated by applying Equation 7.

$$A_{extracted} = \frac{W \cdot C_f}{n \cdot S \cdot L \cdot C_{Ain}} \times 100\% \quad (7)$$

In this research, data on the essential oil concentration in the bulk of the liquid (C_f) and the percentage of essential oil extraction was acquired. The values of effective diffusivity of essential oil in solid particle (De) and Henry constant (H) were evaluated by trial and error using Microsoft Excel so that the minimum SSE (Equation 8) between the calculated and experimental values of the essential oil extracted was obtained.

$$SSE = (A_{extracted_data} - A_{extracted_calc})^2 \quad (8)$$

3. Results and Discussion

3.1. Microwave assisted hydrotropic distillation of *C. citratus*

3.1.1. The effect of hydrotrope concentration

The effect of urea concentration on the solubility of *C. citratus* essential oil was investigated by conducting MAHtD at 110°C, process duration of 10 and 15 minutes with a variation of urea concentration (20-30%) since the solubility of solute is known to increase with the increasing of hydrotrope agent applied. The yield of the essential oil obtained from MAHtD of *C. citratus* performed with 20-30% of urea solution at a temperature of 110°C for 10-15 minutes is illustrated in Figure 2a.

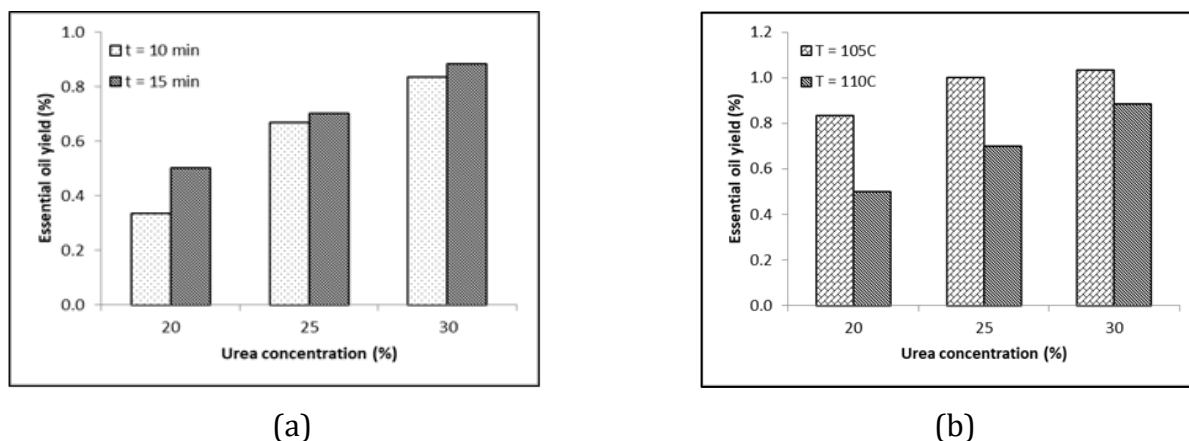


Figure 2 Essential oil yields of MAHtD of *C. citratus* performed with: (a) 20-30% of urea solution at a temperature of 110°C for 10-15 minutes, (b) 20-30% of urea solution at a temperature of 105-110°C for 15 minutes

The research showed that in two different process durations applied, yield of *C. citratus* essential oil increase with the increase of urea concentration used. *Cymbopogon citratus* essential oil yield of 0.5% was obtained from MAHtD with 20% urea solution while 0.88% was obtained from MAHtD with 30% urea solution. Other researchers have reported similar results using hydrotropic-assisted distillation processes for essential oils. The solubility of citral, eugenol, and eugenol acetate increased with the increase of hydrotropic solution (NaSal and NaCuS) applied. The ability of hydrotropes to solubilize solutes is attributed to the hydrotrope self-aggregation characteristic. Some hydrotropes are able to aggregate in a step-wise self-aggregation process and gradually increase aggregation size. It is also mentioned that the other possible mechanism for hydrotropic solubilization process is due to complexation (Choudhary and Nayal 2019).

Table 1 shows that yield of the MAHtD process applied here was higher than those obtained from microwave assisted hydro distillation (MAHD) of *C. citratus*. A yield of 0.67% was obtained from MAHD of 24 hours of withered leaves at a temperature of 105°C for 20 minutes. It showed that urea solution is able to increase the solubility of essential oil and has the ability to break the cell wall of leave where the essential oil is located. Essential oil of was detected to localized, and stored at the different parts of plants depending upon the producing species. Typically, essential oils are stored in secretory cells, glandular trichomes, secretory ducts, and secretory cavities (Saullea et al. 2018).

Table 1 *Cymbopogon Citratus* essential oil yield from various processes

No.	Process	Operation Condition	Yield (%)	References
1	MAHtD	24 h of withered leaves, 1 cm, T = 110°C, S/L = 1:10, t = 15 minutes, 30% urea	0.88	This research
2	MAHtD	24 h of withered leaves, 1 cm, T = 110°C, S/L = 1:10, t = 10 minutes, 30% urea	0.83	This research
3	MAHtD	48 h of withered leaves, 1 cm, T = 110°C, S/L = 1:10, t = 15 minutes, 30% urea	1.0	This research
4	MAHtD	24 h of withered leaves, 1 cm, T = 105°C, S/L = 1:10, t = 15 minutes, 30% urea	1.03	This research
5	MAHD	24 hours of withered leaves, 1 cm, T = 105°C, S/L = 1:10, t = 10 minutes	0.33	This research
6	MAHD	24 hours of withered leaves, 1 cm, T = 105°C, S/L = 1:10, t = 20 minutes	0.67	This research
7	Distillation	Dried leaves, 3 cm, t = 2 hours, bed volume 80%,	0.53	(Alam et al. 2018)
8	MAHD	Dried leaves of 1 week drying process, grinded, 250W, S/L = 1:6, t = 90 minutes	0.89	(Mathialagan, Nour, and Nour 2014)

3.1.2. The effect of Temperature

The MAHtD were conducted at two different temperatures, i.e. 105 and 110°C. Hydrotropic solubilization of solute is known to increase with increasing temperature. The effect of temperature on the aggregation behavior of aqueous solutions of sodium cumene sulfonate was investigated (Wagle, Kothari, and Gaikar 2007). It is found that the enthalpy of micellization is lowered with increasing temperature. Although the increasing temperature usually positively affects solute solubilization, in this research, the yield of MAHtD performed at a lower temperature (105°C) gives better yield than the one performed at 110°C (Figure 2b). The decreasing yield at high temperature process could be caused by the increase of thermal degradation of essential oil component. Essential oil is comprised of various aromatics compounds such as terpenes. In high temperature, terpenes could be converted into resinous products due to oxidation and poly condensation reaction (Abylaeva *et al.* 2020).

Table 2 Chemical composition of *Cymbopogon citratus* essential oil obtained from MAHtD performed 30% of urea solution, temperature of 105°C for 10 minutes

Peaks	Retention time (min)	Concentration (%)	Compounds
1	6.027	14.48	Beta.-Myrcene
2	10.837	21.46	6-Methyl-5-hepten-2-one
3	13.031	7.65	3,5-Heptadienal, 2-ethylidene-6-methyl-
4	18.614	11.45	Linalool
5	23.819	1.13	Z-Citral
6	25.744	1.27	E-Citral
7	26.699	12.63	Neryl Acetate
8	26.971	1.20	Beta.-Citronellol
9	29.920	28.73	Geraniol

3.1.3. Chemical composition of *C. citratus* essential oil

The essential oil of *Cymbopogon citratus* obtained by MAHtD performed with 25% and 30% of urea; the temperature of 105°C for 10 minutes was subjected to GC-MS analysis. The chromatogram of both samples is depicted at Figures 3 and 4. It was found that the *C. citratus* essential oil obtained from MAHtD performed with 30% of urea is dominated by geraniol (28.73%), 6-Methyl-5-hepten-2-one (21.46%), and myrcene (14.48%) (Table 2). A higher myrcene content was obtained from MAHtD performed with 25% of urea solution, where the *C. citratus* essential oil obtained is dominated by Beta-myrcene (53.53%), geraniol (14.23%) and 6-Methyl-5-hepten-2-one (12.57) (Table 3).

Table 3 Chemical composition of *Cymbopogon citratus* essential oil obtained from MAHtD performed 25% of urea solution, the temperature of 105°C for 10 minutes

Peaks	Retention time (min)	Concentration (%)	Compounds
1	6.071	53.53	Beta-Myrcene
2	7.751	0.77	cis-Ocimene
3	10.846	12.57	6-Methyl-5-hepten-2-one
4	13.033	0.82	Bicyclo[3.1.1]hept-3-en-2-one, 4,6,6-tri
5	18.608	3.66	Linalool
6	23.820	2.86	Z-Citral
7	25.747	6.19	E-Citral
8	26.689	3.02	Neryl Acetate
9	26.972	1.57	Beta.-Citronellol
10	28.157	0.78	Nerol
11	29.924	14.23	Geraniol

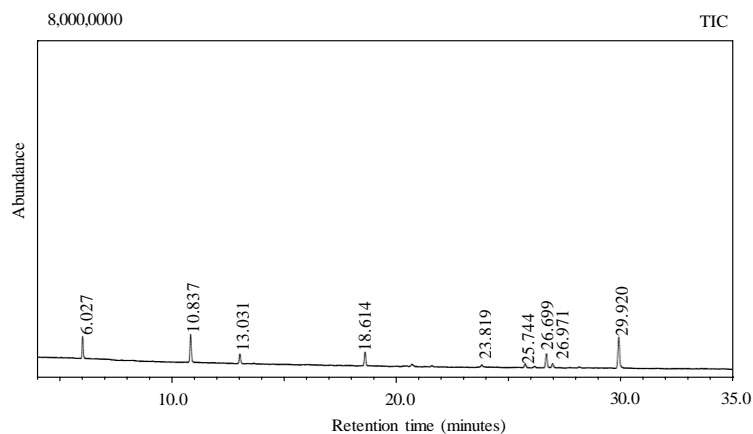


Figure 3 The chromatography of *Cymbopogon citratus* essential oil obtained from MAHtD performed 30% of urea solution, the temperature of 105°C for 10 minutes

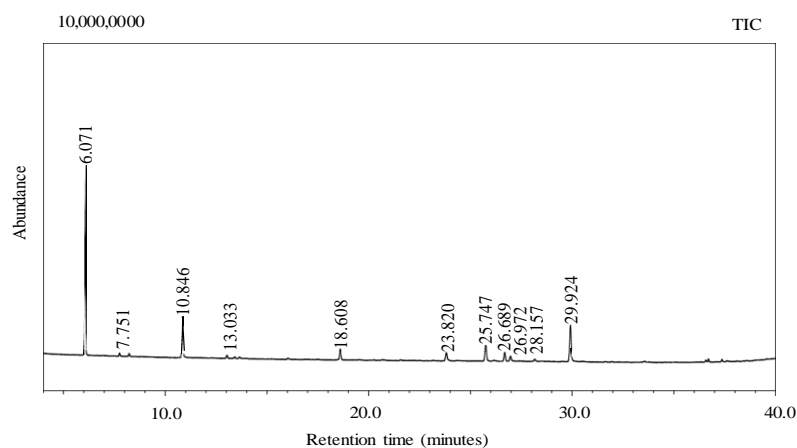


Figure 4 The chromatography of *Cymbopogon citratus* essential oil obtained from MAHtD performed 25% of urea solution, the temperature of 105°C for 10 minutes

The myrcene content of essential oil obtained from this research is relatively high. The myrcene content was in the range of 3.18-7.68% for three different maturity levels of the *C. citratus* plants in 6 hours of hydro distillation process at a temperature of 100°C (Tajidin 2016). Meanwhile, a relatively high myrcene content of *C. citratus* essential oil was mentioned by researcher investigating the effect of drying methods towards the chemical composition of *C. citratus* essential oil obtained from three hours of the hydro distillation process. Fresh, sun drying, shaded, and oven drying of *C. citratus* leaves gave essential oil having myrcene content up to 15.69, 16.16, 14.49 and 15.42%, respectively (Hanaa 2012). It was reported that the myrcene content of *C. citratus* is 27.83% (Gbenou *et al.* 2014) while hydro distillation of *C. citratus* from Cuba was reported to give 6.52% of myrcene in its essential oil (Pinto; *et al.* 2015). The high myrcene content of *C. citratus* obtained from this research indicates that geraniol undergoes dehydration and isomerization during MAHtD, resulting in the production of myrcene.

3.2. Mathematical model of microwave assisted hydrotropic distillation of *C. citratus*

The proposed model was validated by applying experimental data of extraction percentage of essential oil of obtained from MAHtD performed with solid-liquid ratio of 1:10; 25% of urea solution and temperature of 105°C. The validation shows that the calculated and the observed value is close (Figure 5a) with an SSE of 0.033. The obtained effective diffusivity of the essential oil is $2.245 \times 10^{-7} \text{ cm}^2/\text{s}$, and the Henry constant is 0.42.

The effective diffusivity value was higher than the ones obtained from hydro distillation and microwave-assisted hydro distillation of other essential oil. The effective diffusivity value of $4.98 \times 10^{-10} \text{cm}^2/\text{s}$ for hydro distillation of mace (*Myristicae arillus*) essential oil and of $1.65 \times 10^{-9} \text{cm}^2/\text{s}$ for microwave assisted hydro distillation of mace essential oil were reported (Megawati *et al.*, 2019).

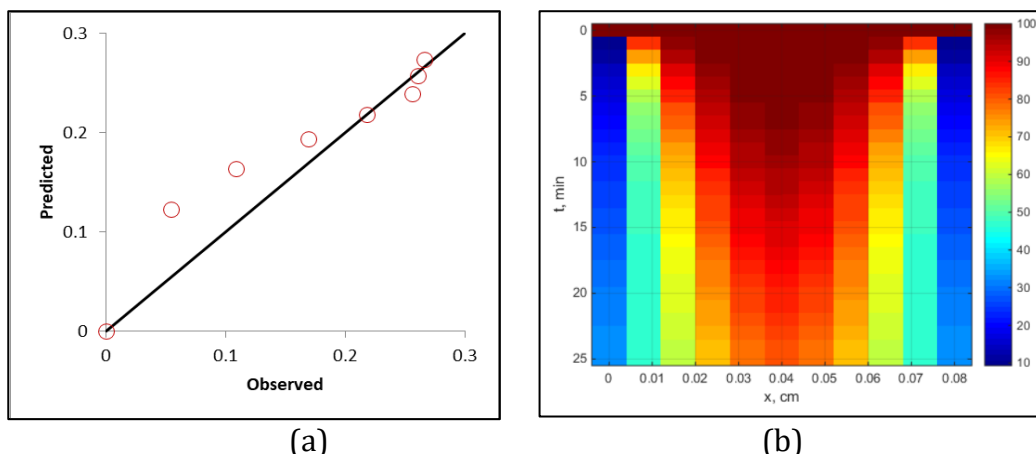


Figure 5 (a) Profile of the calculated and experimental data of extraction percentage of MAHtD process performed with solid-liquid ratio of 1:10; 25% of urea and temperature of 105°C; (b) Profile of essential oil concentration as axial position and time function

Validation of the mathematical model gives us a better understanding of the mechanism of MAHtD as it could provide us with the profile of essential oil concentration as axial position and time function (Figure 5b). It shows that initially, the essential oil in the center of the slab is high. It is logical since the hydrotrope solution is diffused from the outer part of the solid particle towards the center of the solid particle. During the diffusion, it solubilized the essential oil. As the separation process proceeded, the essential oil concentration started to decrease as it diffused into the surface of the solid particle's outer part.

4. Conclusions

Urea is proven as a good hydrotrope for MAHtD of myrcene-rich essential oil of *Cymbopogon citratus* since MAHtD of myrcene-rich essential oil of *Cymbopogon citratus* performed with 25% urea at a temperature of 105°C for 10 minutes resulted in a high myrcene content of up to 53.53%. The proposed model was validated with the experimental data of MAHtD performed at solid-liquid ratio of 1:10, 25% of urea solution, and a temperature of 105°C. The experimental data show a good agreement with the proposed model with an SSE of 0.033. The obtained effective diffusivity of the essential oil is $2.245 \times 10^{-7} \text{cm}^2/\text{s}$, and the Henry constant is 0.42. The effective diffusivity value was higher than those obtained from hydro distillation and microwave-assisted hydrodistillation of other essential oil, which shows that urea a good solubilizing agent for *C. citratus* essential oil. The development and validation of mathematical model of MAHtD gives us a better understanding of the mechanism of MAHtD and also provide basic data that are essential for design of process equipment of commercial scale. The scale up of the microwave assisted distillation unit in larger scale is remaining as the main challenge of the application of microwave-based processes. However, the availability and the relatively low cost of urea as well as the high content of myrcene content in essential oil of *C. citratus* obtained from MAHtD are the three main attractiveness of the application of this method.

Acknowledgments

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