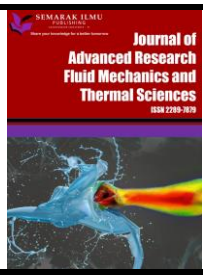
SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage:
https://semarakilmu.com.my/journals/index.php/fluid_mechanics_thermal_sciences/index
ISSN: 2289-7879



Simulation of Triglyceride Purification from Crude Palm Oil using Single Solvent Continuous Counter Current Extraction

Amani Salsabil Husodo[1], Juwari[1], Cheng-Hsiu Yu[2], Setiyo Gunawan[1,*]

[1] Department of Chemical Engineering, Faculty of Industrial Technology and System Engineering, Institut Teknologi Sepuluh Nopember, 60111 Surabaya, Indonesia
[2] Department of Chemical Engineering, National Taiwan University of Science and Technology, 10607 Taipei City, Taiwan

ARTICLE INFO

Article history:
Received 20 August 2024
Received in revised form 29 November 2024
Accepted 7 December 2024
Available online 20 December 2024

Keywords:
Continuous counter current extraction; cooking oil; crude palm oil; simulation; thermodynamic properties

ABSTRACT

As the world's largest crude palm oil (CPO) producer, Indonesia focuses on palm research, such as CPO-based cooking oil production. Generally, the widely applied production process in the industry is refining, which has several disadvantages, such as low energy efficiency and monochloropropane-1,2-diol (MCPD) formation. Under such conditions, CPO-based cooking oil production methods have been developed, with the continuous countercurrent extraction (CCE) method is considered the most effective for purifying triglyceride (TAG) as the main content of CPO-based cooking oil. The minimum content of TAG in cooking oil is 96% by mass according to SNI 7709:2019 (Indonesian cooking oil standard). Hence, to obtain the optimal production process that meets that standard, a simulation of TAG purification from CPO using the CCE method with a single solvent was conducted. This simulation obtained thermodynamic properties that correspond to the experimental extraction process, in which an industrial scale up can be acquired. The precise thermodynamic properties resulted from the precise selection of property package and Binary Interaction Parameter (BIP) estimation, and those are important because in this extraction process, liquid-liquid equilibrium works. This research used combinations of two property packages: Non-Random Two Liquid (NRTL) and Universal Quasi Chemical (UNIQUAC) and three BIP estimation methods: UNIQUAC functional activity coefficients (UNIFAC), UNIFAC-Lyngby, and UNIFAC-Dortmund. The simulation results were validated using existing experiments with statistical analysis such as relative error, R-Square, and p-value. The most valid simulation results were obtained by NRTL property package and UNIFAC-Lyngby BIP estimation and can be used to develop this TAG purification.

1. Introduction

With its Crude Palm Oil (CPO) production capacity of 50,07 million tons in 2023 and an increase of 7,15% comparing to 2022, Indonesia is one of the main CPO producers in the world [1]. Under such conditions, Indonesia could export 64.34% of its CPO, while the rest of it (that is not exported) can be used to produce several products, such as cooking oil [1]. The cooking oil production process

* *Corresponding author.*
E-mail address: setiyo.gunawan@its.ac.id

https://doi.org/10.37934/arfmts.125.2.5465

generally involves chemical and physical refining techniques. Unfortunately, these techniques have the following disadvantages: low energy efficiency and the formation of monochloropropane-1,2-diol (MCPD), which hydrolyzes in the body to produce 3-MCPD esters, which are carcinogenic [2,3]. Therefore, Triglycerides (TAG) purification from CPO has been developed. The purification of TAG by extraction has been carried out using batchwise solvent extraction (BSE) method and BSE combined with microwave-assisted extraction (MAE) [4,5]. In another study, the separation of TAG from crude nyamplung oil was performed by the Batchwise Solvent Extraction (BSE) and Continuous Counter-current Extraction (CCE) methods, the latter of which has advantages in terms of capacity and time efficiency [6,7].

To ensure that the cooking oil produced from TAG purification is in accordance with SNI 7709:2019, it is necessary to simulate it. These simulations are useful for obtaining precise thermodynamic modelling of experiments and for developing process designs to produce CPO-based cooking oils [8]. Simulations were performed using simulators such as Aspen Hysys, Aspen Plus, and CHEMCAD. Some simulations of TAG purification from CPO have been performed before using Aspen Plus. First, when the BSE method was used to purify TAG from CPO, it was found that Universal Quasi Chemical (UNIQUAC) was the best property package that suited the experiment [9]. Another simulation of CPO-based cooking oil using refining method was performed using Aspen Plus V.10 with UNIQUAC as its property package to analyze its exergy [10].

Based on previous experiments on TAG purification from crude oil (CPO and nyamplung) and those simulations, a research on simulating TAG purification for CPO-based cooking oil using the CCE method was conducted. The CCE method is well known for its material and time efficiency, so it is promising to develop TAG purification from CPO with the CCE method using simulation to obtain thermodynamic properties that match the experimental results [7]. Hence, more process designs for CCE-based TAG purification from CPO, in which the cooking oil produced meets the standard (SNI 7709:2019). While working with CCE, the essence of this process is liquid-liquid extraction. Liquid-liquid extraction calculation works with liquid-liquid equilibrium; thus, the selection of property package and Binary Interaction Parameter (BIP) estimation methods greatly determine the thermodynamic properties results.

2. Methodology

2.1 TAG Purification from CPO

This purification process uses the concept of liquid-liquid extraction carried out in an extractor with dimensions of 1.64 min height, 4 cm in diameter, and packed with Raschig rings [7]. The solvent used was 96% food-grade ethanol, whereas CPO had the following specifications (Table 1).

Table 1
CPO Product Specifications

Component	Mass (%)
Triglyceride (TAG)	87.30
Diglyceride (DAG)	3.90
Monoglyceride (MAG)	0.12
Free fatty acids (FFA)	5.60
Other	3.09

Purification in the extractor operates at a fixed pressure of 1 atm and a fixed temperature of 30°C. Ethanol is fed from the top, and CPO is fed from the bottom. The raffinate that comes out is a

non-polar fraction (NPLF), whereas the extract that comes out is a polar fraction (PLF). To obtain cooking oil, we used distillation to purify the NPLF.

2.2 Simulation of TAG Purification from CPO

The simulator used in this study was Aspen Plus V.11. Prior to simulations, the system should be analyzed to determine the appropriate thermodynamic properties [10]. In this system, thermodynamic equilibrium occurs in the form of liquid-liquid and liquid-vapor equilibrium. The extractor column works with liquid-liquid equilibrium, and the distillation column works with liquid-vapor equilibrium. Some compounds we used are non-polar compounds, such as: TAG and DAG. The rest are polar compounds such as: ethanol, water, and FFA. Hence, based on the compounds included in the process, we obtain polar and non-polar mixture, which is considered a non-ideal liquid-liquid system [11].

Therefore, we decided to use the UNIQUAC and NRTL property packages because both are highly recommended for liquid-liquid systems that are not ideal [11]. The BIP in this simulation was obtained from a databank equipped with the estimation results. Three BIP estimation methods are available: UNIFAC, UNIFAC-Lyngby, and UNIFAC-Dortmund [12]. The property package selection is shown in Figure 1, and the BIP estimation selection is shown in Figure 2:

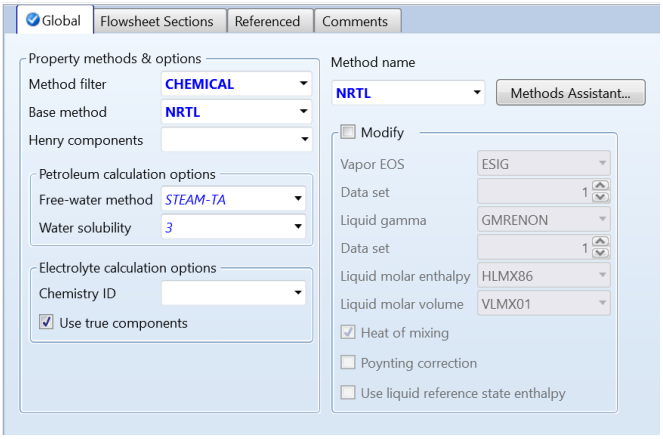


Fig. 1. Property package (method) selection scheme

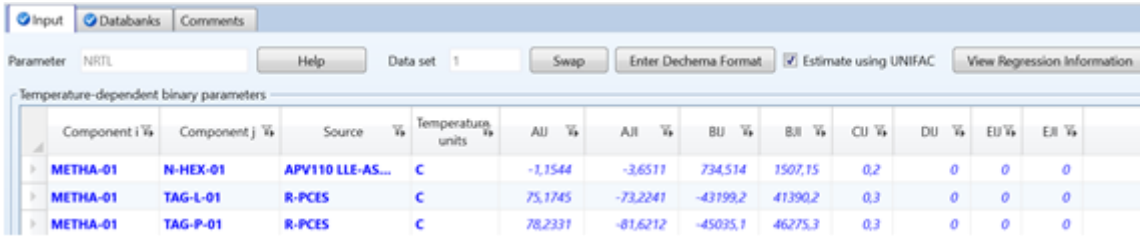


Fig. 2. Scheme of BIP values obtained from databanks and estimation

Aspen Plus V.11 has complete databanks for the constituent compounds of vegetable oils, so there is no need to hypothesize again, as in Aspen Hysys V.11 by using the "Hypo Manager" feature [13]. The existing simulation was validated with experiments conducted by Mahardika *et al.,* [4] in 2023. The validation was performed by analysing the R-squared values, p-values, and relative errors between the simulation and experiment, in terms of TAG purity, FFA purity, and NPLF yield to flow rate ratio. The result is valid if they meet these three criteria: 1) the R-Squared value is close to 1

(>0.99), 2) the greater the p-value (p-value > 0.05), and 3) the smaller the relative errors [14]. The unit operations used in the simulations were adjusted in the experiment as shown in Figure 3.

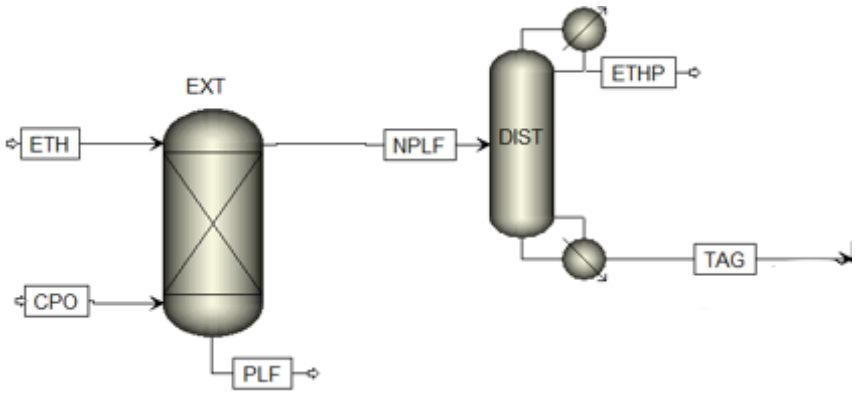


Fig. 3. Simulation of TAG purification from CPO using single solvent CCE

3. Results

3.1 TAG Purification from CPO

The experiment was conducted with 3 variables of CPO and ethanol flow rate ratios: 1: 2.5; 1: 5; and 1: 7.5. Regarding the FFA content, CPO is divided into two types: high FFA (HFFA) (mass % FFA > 5.00) and low FFA (LFFA) (mass % FFA ≤ 5.00) [15]. The CPO used in this experiment is considered a high FFA CPO, according to its FFA content (5.60 %) [4].

3.2 Simulation of TAG Purification from CPO

The simulation used 96% food-grade ethanol (96% ethanol and 4% water) and CPO. CPO is composed of TAG, DAG, MAG, FFA, and several other components. The polarity index of these components can be obtained from the LogP value. LogP is the partition coefficient between octanol and water, which is useful for measuring the hydrophilicity or hydrophobicity of molecules. Hydrophilic compounds tend to have low and even negative log P values, which indicate of polarity and are soluble in water and other polar solvents. If the LogP value is greater, the hydrophobicity is greater, and the compound is a non-polar compound [16]. Log P values are shown in Table 2.

Table 2
LogP-value [17]

Component	Log P Value
Triglyceride (TAG)	22.63
Diglyceride (DAG)	16
Monoglyceride (MAG)	6.3
Free fatty acids (FFA)	6.9
Other	8.9
Ethanol	-0.18

Based on the polarity index value, ethanol was chosen because it is expected to attract polar compounds such as FFA and MAG. Non-polar compounds such as TAG come out of the extract column and generate non-polar charge mixture (NPLF). The details of the compounds of each lipid class were adjusted based on research conducted by Japir *et al.,* [15] on 2017.

In the extraction column, a liquid-liquid equilibrium occurs. As shown in Figure 3, CPO flowing from the bottom inlet is a dispersed phase, whereas ethanol is a continuous phase. All simulations

performed using the NRTL and UNIQUAC property packages combined with three BIP estimation methods were convergent. However, after validation with the Mahardika *et al.,* [4] in 2023 experiment, the most accurate results were obtained with a combination of the NRTL property package and the UNIFAC-Lyngby BIP estimation method. Figure 4 shows the results of the simulation, Table 3 shows the best results of the simulation in terms of purity, and Table 4 shows the best results of the simulation in terms of yield.

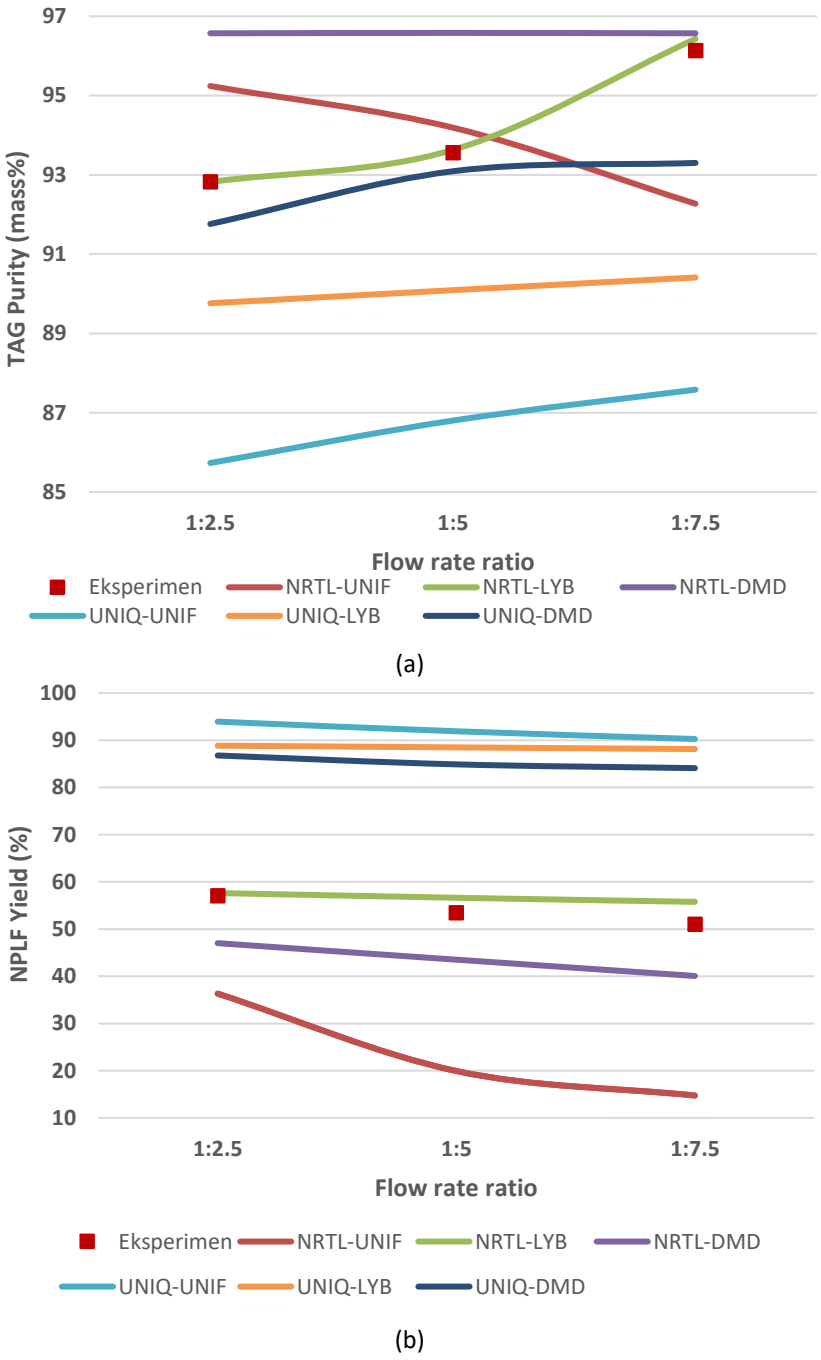


Fig. 4. Simulation results: (a) TAG content to flow rate ratio and (b) NPLF yield to flow rate ratio

Table 3
Simulation Results for Component Purity

Component	Flow Rate Ratio								
	1 : 2.5			1 : 5			1 : 7.5		
	Experiment	Simulation	%Error	Experiment	Simulation	%Error	Experiment	Simulation	%Error
Triglyceride (TAG)	92.83%	92.82%	0.01	93.56%	93.63%	0.07	96.14%	96.43%	0.30
R-Square (TAG)							1.0000		
p- value (TAG)							0.1658		
Free fatty acids (FFA)	2.20%	2.30%	4.35	3.45%	3.32%	2.48	2.17%	2.37%	2.80
R-Square (FFA)							0.9933		
p- value (FFA)							0.3097		

Table 4
Simulation Results for the NPLF Yield

NPLF Yield	Flow Rate Ratio								
	1 : 2.5			1 : 5			1 : 7.5		
	Experiment	Simulation	%Error	Experiment	Simulation	%Error	Experiment	Simulation	%Error
	57.03%	57.63%	1.03	53.46%	56.64%	5.62	51.01%	55.77%	108.54
R-Square							0.9947		
p-value							0.0719		

In this simulation, the NRTL equation combined with UNIFAC Lyngby's BIP estimation method gave the most accurate result, according to the statistical analysis between the simulation and experiment shown in Table 3 and Table 4. The statistical analysis used to analyze trends of purity and NPLF yield against flow rate ratios between simulation and experiment, in terms of relative errors, R-Square values, and p-values. As mentioned earlier, smaller relative error means that the simulation result is closer to experimental result [14]. The R-Square closer to 1 ($R > 0.99$) indicates that the simulation and experiment trends are similar [14]. Higher p-value (> 0.05) indicates that simulation and experiment are not significantly different, which means the simulation and experiment are more similar [14]. The trend of TAG purity against flow rate ratio of simulation and experiment are similar, as observed in Table 3 (relative errors < 1%, R-Square: 1.000, and p-value: 0.1658). FFA also expressed similar results with TAG with relative errors <5%, R-Square: 0.9933, and p-value: 0.3097). In Table 4, the experiment shows a decreasing NPLF yield when the flow rate ratio increases. The simulation result of the NPLF yield also manifested this trend (can be seen in small relative errors, R-Square: 0.9947, and p-value: 0.0719).

The accurate results observed in Table 3 and Table 4 are due to the accurate thermodynamic properties. To obtain such accurate properties, an accurate property package and BIP estimation method must be applied after understanding the equilibrium system in this process. It was mentioned before that this extraction system works with liquid-liquid equilibrium. The liquid-liquid equilibrium equation is a derivative form of liquid-liquid-vapor equilibrium equation. Because the

vapor phase is not involved, the gas phase factor can be ignored, and the form of the liquid-liquid equilibrium equation becomes:

$$x_i^{l1}\gamma_i^{l1} = x_i^{l2}\gamma_i^{l2} \tag{1}$$

where, x_i: component concentration, γ_i: activity coefficient, and l_n : nth fluid [18].

The property package was used to calculate the activity coefficient, BIP, and non-randomness parameter. In this experiment, NRTL performed better than UNIQUAC. The NRTL equation used to calculate activity coefficient can be expressed as follows:

$$\ln \gamma_i = \frac{\sum_{j=1}^{c}\tau_{ji}G_{ji}x_j}{\sum_{l=1}^{c}G_{li}X_l} + \sum_{l=1}^{c}\frac{x_jG_{ij}}{G_{lj}X_l}\left[\tau_{ij} - \frac{\sum_{r=1}^{c}X_r\tau_{rj}G_{rj}}{\sum_{l=1}^{c}G_{lj}X_l}\right] \tag{2}$$

where $\tau_{ji} = \frac{g_{ji}-g_{ii}}{RT}$, $G_{ij} = \exp\left(-\alpha_{ji}\tau_{ji}\right), \alpha_{ij} = \alpha_{ji}$.

G is an energy parameter describing the interaction between species *i* and *j*, x_i is a mole fraction of component i, τ_{ij} is a BIP, and α is a non-randomness parameter [19].

The NRTL equation above can be used at processes with one of the phase equilibriums: vapor-liquid, liquid-liquid, and vapor-liquid-liquid. This equation performs well with mixtures of non-polar and polar compounds such as alcohols and non-polar compounds, as well as with polar and polar compound mixtures such as alkenes with other non-polar fractions [20]. Eq. (2) requires the calculation of BIP (τ_{ij})and non-randomness parameter (α_{ij}). Both can be obtained from Eq. (3) and Eq. (4), respectively. The equations below are written in extended form as follows:

$$\tau_{ij} = a_{ij} + \frac{b_{ij}}{T} + e_{ij}\ln T + f_{ij}T \tag{3}$$

$$\alpha_{ij} = c_{ji} + d_{ij}(T - 273{,}15) \tag{4}$$

where a_{ij}: non-temperature dependent coefficient between components *i* and *j*, b_{ij}: temperature dependent coefficient between components *i* and *j* (K), c_{ij}: non-temperature dependent coefficient between components *i* and *j,* d_{ij}: temperature dependent coefficient between components *i* and *j* (K), e_{ij}: temperature dependent coefficient between components *i* and *j* (K), f_{ij}: temperature dependent coefficient between components *i* and *j* (K), and *T*: temperature (K). For Eq. (3), the system generally only requires the forms a_{ij} and b_{ij}. For Eq. (4), the system generally only requires the c_{ij} parameter; thus, α_{ij}= c_{ij} [20].

The coefficients used in Eq. (3) and Eq. (4) can be obtained from databanks equipped with BIP, estimated by the UNIFAC-Lyngby method. The method equation is written below:

$$a_{mn} = a_{mn,1} + a_{mn,2}(T - 298{,}15) + a_{mn,3}\left(Tln\frac{298{,}15}{T} + T - 298{,}15\right) \tag{5}$$

where $a_{mn,1}$ is a temperature-independent parameter, $a_{mn,2}$ and $a_{mn,3}$ are temperature-dependent parameters, and T is temperature (K) [21]. According to a study conducted by Liaw, *et al.,* [22] in 2011 that the prediction of miscible mixture coefficients by UNIFAC methods can be better than NRTL and UNIQUAC alone for certain types of mixtures. This is in line with what was obtained in this study, where the prediction of the BIP based on UNIFAC combined with NRTL as a property package produced accurate results.

Based on the simulation and experiment, the only flow rate ratio variable (between CPO and ethanol) for which the TAG content meets SNI 7709:2019 is 1:7.5 [23]. We can perform the column performance prediction using the best BIP estimation method and property package to develop this extraction process. The prediction of column performance was performed by analyzing the purity and yield to column height and, the purity and yield to the flow rate ratio. The column height scheme is shown in Figure 5. For the flow rate ratio predictions, we used an additional variable of CPO: ethanol ratios of 1:1 and 1:10. The prediction results are shown in Figure 6 and Figure 7.

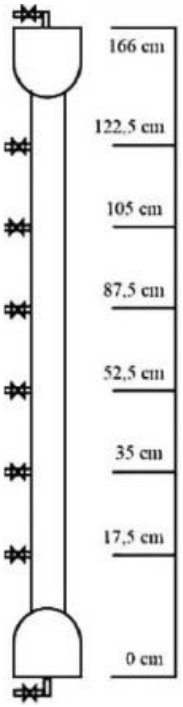
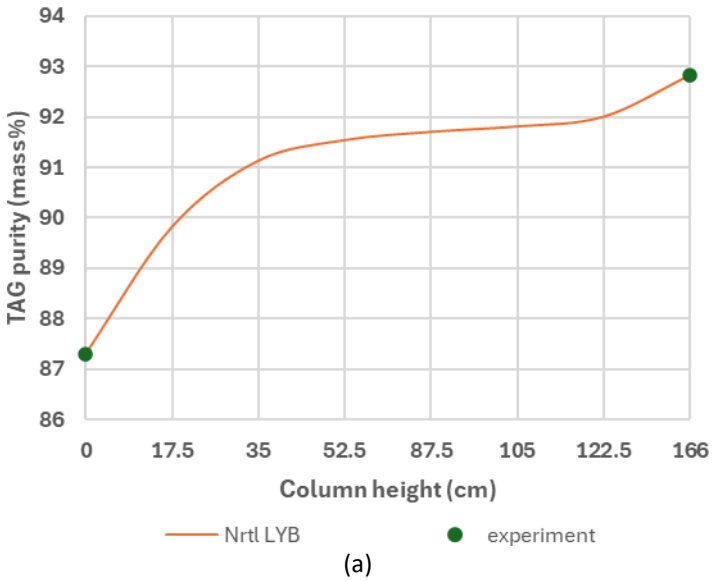
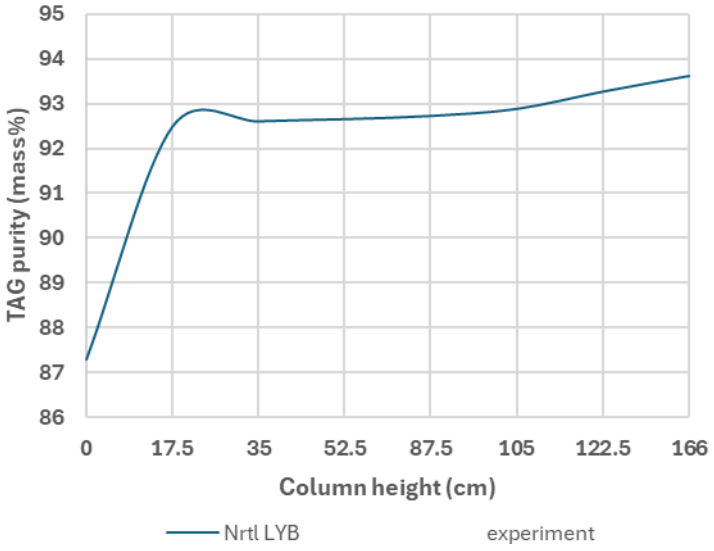


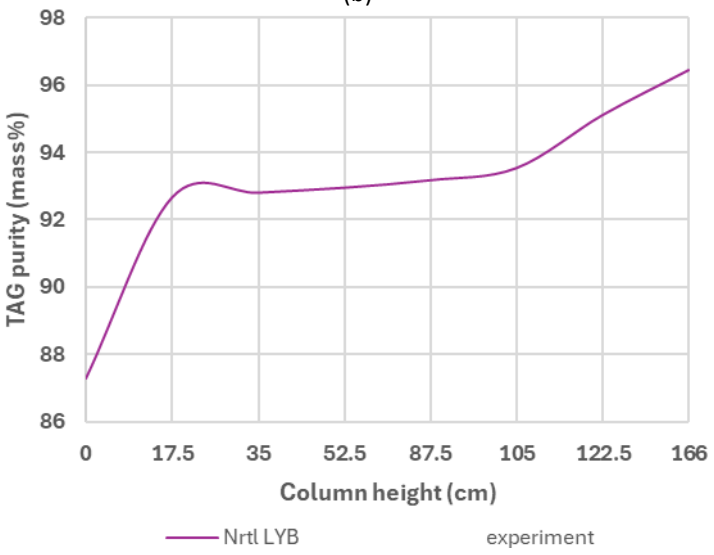
Fig. 5. Column elevation (height) point profile for prediction



(a)



(b)



(c)

Fig. 6. TAG purity prediction results against column height (a) Flow rate ratio 1:2.5, (b) Flow rate ratio 1:5, (c) Flow rate ratio of 1:7.5

Here are the results of TAG purity to the column height prediction, as shown in Figure 6: As for the prediction results of the flow rate ratio, it was used to predict the flow rate ratio that was not performed in the experiment. The results can be used to determine the optimum flow rate ratio for to be applied in process development. The results are shown in Figure 7.

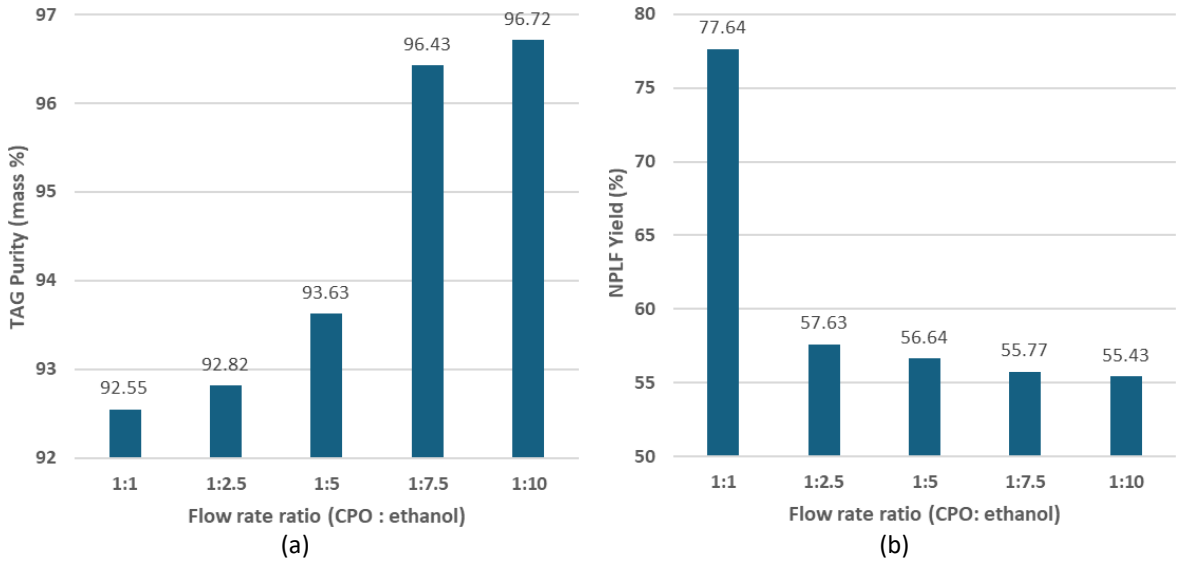


Fig. 7. Performance prediction results for the flow rate ratio: (a) Purity TAG, (b) NPLF Yield

The results of this prediction indicate that column specifications and flow rate ratio selection can be developed. From Figure 7, it can be observed that the higher the flow rate ratio, the higher the purity of TAG but the lower the NPLF yield. This means that the NPLF obtained for cooking oil is purer. However, under this condition, we should spend more solvent and it is not considered necessary. If the ratio is too low, the use of solvents can be minimized, but a large volume of cooking oil with low TAG purity can be obtained. We consider that flow rate ratios of 1:2.5 to 1:7.5 are necessary enough for process development because the NPLF yield, and TAG purity are not significantly different from those of the 1:10 flow rate ratio. From Figure 6, all flow rate ratios show the same trends in the prediction results of TAG purity against the column height. The higher the column, the higher the purity of TAG. Therefore, the results of this study need to be continued to obtain the best process flow for producing CPO-based cooking oil according to standards using this simulation because the thermodynamic properties of this simulation can model the experiment well.

4. Conclusions

CPO-based cooking oil from TAG purified from CPO using single solvent CCE method has a potential scale development. Prior to this, precise simulation of the process must be conducted. The simulation of TAG purification from CPO using CCE was performed using Aspen Plus V.11. To ensure that our simulation is accurate or has thermodynamic properties that best suit the experiment, it is necessary to understand the experiment's system process, the compounds included in the experiment, and the equilibrium system that works there. In this TAG purification process, a CCE method is applied. Hence, the system focus is on liquid-liquid extraction, in which liquid-liquid equilibrium occurs. Liquid-liquid equilibrium equations require activity coefficients, BIP, and non-randomness parameters. These parameters were calculated using the property package. The BIP and non-randomness parameters themselves require coefficients that obtained through the calculation of the BIP estimation method. Therefore, the precise selection of property package and BIP estimation yielded on precise thermodynamic properties of the simulation. This study performed simulation with two property packages, NRTL and UNIQUAC, combined with three BIP estimation methods: UNIFAC, UNIFAC-Lyngby, and UNIFAC-Dortmund. The most accurate thermodynamic properties of this simulation are obtained with combination of NRTL property package and UNIFAC Lyngby BIP estimation method. The determination is based on the validation using statistical analysis

such as relative errors, R-Squared values (>0.99), and p-values (>0.05) between simulation and experiment. This simulation is used to predict the column performance in terms of TAG purity and NPLF yield to flow rate ratios between CPO and ethanol, and to column height. The prediction results expressed that it is potential to develop this TAG purification process to generate an ideal CPO-based cooking oil process design.

Acknowledgement
The authors would like to acknowledge the financial support from Institut Teknologi Sepuluh Nopember for this work under the Funding project scheme (780/PKS/ITS/2024).

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