# Characterization and Conversion of Sludge Palm Oil (SPO) into Biodiesel

Sim Siong Fong<sup>1\*</sup>, Lau Seng<sup>2</sup>, Benedict Samling<sup>1</sup>, Nur Syahidda Ani Binti Moksin<sup>1</sup>, Nursyafira Adzira Binti Halmi<sup>1</sup>, Amelia Laccy Anak Jeffrey Kimura<sup>1</sup>, Clarence Chin<sup>2</sup> and William Liang<sup>2</sup>

<sup>1</sup>Faculty of Resource Science & Technology, Universiti Malaysia Sarawak, 94300 Kota Samarahan <sup>2</sup>Hexa BRT Sdn Bhd, Lot 922, Block 7 MTLD, Jalan Demak Laut 3, Demak Laut Industrial Park, 93050, Kuching, Sarawak, Malaysia \*Corresponding author (e-mail: sfsim@unimas.my)

Sludge Palm Oil (SPO) is a potential low-cost feedstock for biodiesel production. The conversion efficiency and the quality of biodiesel produced is governed by the feedstock and the conversion reaction. To our knowledge, the potential of SPO produced by palm oil mills in Sarawak has yet to be explored. Hence, this paper aims to characterize the SPO obtained from Sarawak palm oil mills and to optimize the predominant conversion reaction. The SPO was converted into biodiesel *via* a sulphuric acid catalyzed esterification reaction, followed by transesterification using potassium hydroxide and methanol. This SPO contained 1.30 - 10.15% moisture, 2.91 - 7.63% sediment and 47.05 - 87.86% free fatty acids (FFA). The optimum esterification conditions identified were: SPO:methanol ratio (1:10), sulphuric acid concentration (1.84%) and reaction time (3.25 hr). Under these conditions, the biodiesel yield was 74% with an FFA conversion efficiency of 79.87%. The water produced during the esterification process was found to hinder the reaction, reducing both conversion efficiency and biodiesel yield. The findings of this study offer important insights about the challenges and feasibility of biodiesel conversion from SPO, where esterification is inevitable due to its high FFA content.

**Key words**: Esterification-transesterification; optimization; response surface methodology; fatty acid methyl esters; free fatty acids

Received: August 2021; Accepted: January 2022

Malaysia, the second largest producer of palm oil in the world, has a total of 457 fresh fruit bunch mills with a processing capacity of 116 million tonnes in 2020, of which Sarawak contributes 21% [1]. The processing of palm oil produces considerable amounts of palm oil mill effluent (POME); every tonne of crude palm oil (CPO) will result in 2.5-3.5 tonnes of POME comprising 93-95% water, 3-4% solid residue and 0.5-2% oil [2,3]. The term "POME" is often used interchangeably with palm oil sludge (POS), oil palm sludge (OPS), palm oil effluent (POE) and palm oil slurry (POS). A specific fraction at the upper layer of POME is regarded as Sludge Palm Oil (SPO) [4]; this fraction is rich (2.21 - 88.34%) in free fatty acids (FFA) and has an average moisture level of 0.99% [5]. The amount of FFA in SPO is appreciably higher than that in crude palm oil (CPO). As specified by the Palm Oil Refiners Association of Malaysia, the maximum FFA level for CPO is regulated at 5% while it should be < 0.1% for refined-bleached-deodorized oil [6]. SPO is widely used in soap manufacturing, as supplements in animal feed and as feedstock for biogas production [7,8,9]. It is also a viable and attractive feedstock for biodiesel production [4,10,11]. Conventionally, biodiesel is converted from vegetable oils or animal fats; however, this process is burdened by expensive raw materials which account for 60-70% of the total production cost. As a result, the price of biodiesel is higher than that of fossil fuels, rendering biodiesel production unsustainable [12,13,14]. In 2021, the price of CPO started at 990 USD/mt; it then soared above 1,000 USD/mt and continued to increase to the current price at 1,300 USD/mt. With the implementation of the biodiesel mandate in Malaysia, the demand for CPO is anticipated to rise. To ensure the sustainability of biodiesel production, there is a crucial need to identify a potential low-cost feedstock. With SPO quoted at about 50% of the price of CPO, it is an economically and environmentally attractive alternative feedstock for biodiesel.

The quality of biodiesel is governed by its raw materials, the conversion process and the separation

efficiency [15]. The ratio of oleic acid to linoleic acid (O/L) is used as an indicator of the feedstock quality for biodiesel production; feedstock with a higher O/L value is expected to produce biodiesel with better stability and a longer shelf life [16]. Other vegetable oils such as canola oil, coconut oil, soybean oil and corn oil have O/L values in the range of 0.42-3.58. In comparison, palm oil has a higher O/L ratio of 3.93 [15]. Although SPO has the advantage of cost over CPO, the quality of SPO may vary substantially as it is a by-product, thus contributing to inconsistencies in the biodiesel produced. Abdullah et al. [17] converted SPO into biodiesel via an esterificationtransesterification reaction using methanol (20:1) with 6% alum and 1.5% potassium hydroxide, KOH. The reaction yielded an average of 93% biodiesel, containing predominantly palmitic acid methyl ester, methyl oleate and methyl stearate. On the other hand, Aworanti et al. [18] optimized the transesterification reaction and attained a maximum biodiesel yield of 61.2% using a methanol-to-oil ratio of 12:1 and 1.5% KOH in 30 min. In general, SPO may be converted into biodiesel via various approaches with yields reported between 62.30% and 96.00% [4]. The conversion efficiency, though depending on the strategy used, is also profoundly governed by feedstock quality. This paper characterises the SPO obtained from palm oil mills in Sarawak and optimizes the conversion reaction of SPO into biodiesel. The findings of this study provide an insight into the potential of SPO from Sarawak mills as a low-cost feedstock for biodiesel production.

# MATERIALS AND METHODS

# 1. Samples

Samples of SPO were collected from palm oil mills in Sarawak as summarised in Table 1. A sample of POME was included for comparison. The sample code assigned represents the sample type, the date the sample was received (2020/2021) and the palm oil mill. To illustrate variability, photos of SPO from palm oil mills in Serian (SPO1512\_S01) and Kuching (SPO1512\_S02) are shown in Figure 1. The samples are different in colour.

# 2. SPO Characterisation

The samples were analysed (in triplicate) for their moisture, sediment and FFA content. Moisture content was determined based on the loss-on-drying approach [19]. The samples were dried in an oven at 105 °C overnight and the weight difference was obtained. Each oven-dried sample was then dissolved in 10 mL of toluene and centrifuged at 4000 rpm for 30 min. The sample was separated into two phases (residue and solvent phases), and dried in an oven at 105 °C for 2 hrs. The weight difference was recorded to represent the oil and sediment content.

The FFA content was determined by titration. 0.2 g of a fresh sample was dissolved in 100 mL solvent containing 95% ethanol and diethyl ether (1:1 v/v). The mixture was titrated with sodium hydroxide (0.05 M) in the presence of phenolphthalein as the indicator. The end point was reached when the solution turned pink and the FFA content was calculated according to the following equation.

$$FFA, \% = \frac{N \times V \times 25.6}{g}$$

N: Normality of NaOH V: Volume of NaOH (mL) used in titration g: sample weight

The functional group characteristics of SPO were determined using an ATR-FTIR (Thermo Scientific, Nicolet iS10). For functional group characterization, five replicates were scanned. The spectra in .csv format were analysed using the software's automated peak detection and matching algorithm [20]. The spectra were baseline-corrected and the peaks were detected based on the first derivative approach.

# 3. Biodiesel Conversion

The conversion of SPO into biodiesel was evaluated based on the model sample SPO1512\_S02. This sample was chosen as it contained a high FFA content, with almost all its oil fraction attributed to FFA. The sample was converted using the two-stage esterificationtransesterification process as shown below [21]. Typically, for feedstock with high FFA content (20-30%), the esterification process is required to transform the FFA into fatty acid alkyl esters before the conversion of triglycerides into biodiesel via the transesterification reaction [22].

Table 1. Samples of SPO and POM	E analysed
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Sample Code/Location of palm oil mill SPO1512\_S01 (Serian) SPO1512\_S02 (Kuching) SPO2912\_S03 (Sri Aman) SPO2912\_S04 (Betong) POME1803\_S05 (Mukah) SPO0504\_S06 (Sibu) SPO0804\_S07a (Serian) SPO1204\_S07b (Serian) SPO1204\_S08 (Mukah)



SPO1512\_S01 (Serian)



SPO1512\_S02 (Kuching)

Figure 1. Sludge palm oil samples from SPO1512\_S01 (Serian) and SPO1512\_S02 (Kuching)

Catalyst  $H_2O$ R-COOR R—COOH **+** R'—OH **◄** Fatty acid alkyl esters Fatty acids Transesterification reaction: -COO-R' -COO-R' H2C-OH  $H_2C$ —COO- $R_1$ Catalyst **+** H2Ċ—ОН  $H_2C - COO - R_2 + 3R' - OH$ H2Ċ—OH  $\dot{R}_3$ -COO-R'  $H_2\dot{C}$ —COO— $R_3$ Triacylglycerol Fatty acid alkyl esters Glycerol

Esterification reaction:

For the esterification process, the SPO sample was heated at 105 °C for an hour and the melted SPO was transferred into a round bottom flask. Methanol and 95% sulphuric acid were added to the flask and stirred at 400 rpm at 60 °C [22]. The esterification conditions such as methanol ratio, sulphuric acid concentration and reaction time were subjected to optimization according to the experimental design described in Section 2.5. The temperature of 60 °C was set in accordance with Ferdous et al. [23] who reported that a higher temperature could cause loss of methanol, thus reducing the conversion efficiency. The process was then continued with a transesterification reaction. 20 mL of 1% KOH in methanol was added and the mixture was stirred for 1 hr (400 rpm, 60 °C). The solution was dried using a rotary evaporator to remove excess methanol. The product was transferred into a separatory funnel and left for 24 hr to separate into fractions of biodiesel and glycerol. The separated solution was centrifuged at 4000 rpm for 15 mins. The transesterification parameters were set according to the conditions recommended by Hayyan et al. [11].

# 4. Fatty Acid Methyl Ester (FAME) Profile

The fatty acid methyl ester (FAME) profile was analysed using a Gas Chromatograph - Mass Spectrometer (GCMS) system (Shidmazu, model QP2020NX) installed with a SLB-5MS column (30  $m \times 0.25$  mm). One microlitre of the sample was injected using a split injector. The oven temperature programme was as follows: 50 °C for 1 min with 15 °C/min up to 180 °C, then 7 °C/min up to 230 °C and subsequently 15 °C/min up to 280 °C. The final temperature was held for 10 min. For MS detection, electron ionization with 70 eV was applied and mass fragments were detected between 40 and 400 m/z. The ion source temperature and transfer line temperature were 200 °C and 280 °C, respectively. The compound profiles and their corresponding areas were used to estimate the % FAME present in the biodiesel produced.

# 5. Optimization of Esterification Reaction

For feedstock with high FFA values, esterification prior to the transesterification reaction is crucial in determining the success of the conversion process [24]. Preliminary evaluation confirmed that the esterification conditions contribute prominently to the quality and yield of biodiesel. Hence, this study focused on optimization of the esterification parameters including the substrate:methanol ratio, concentration of sulphuric acid and reaction time [25]. The experiment was designed based on a circumscribed central composite design (CCD) which consisted of factorial points with centre and star/axial points. The factorial points were used to estimate the linear and two-factor interaction terms whilst the axial points,  $\alpha$ , contributed to estimations of the pure quadratic terms;  $\alpha$  was determined based on  $[2^k]^{1/4}$  where k is the number of factors (in this case, 3). The centre points served to estimate the error and precision of the model [26]. Table 2 shows the experimental design according to coded and uncoded factors; experiment no. 1-8 refers to the factorial points, 9-14 are the axial points and 15-24 are the centre points. The % FAME derived from the GCMS profile is a response to the function of the three aforementioned factors: methanol ratio, sulphuric acid concentration and reaction time.

# 6. **Response Surface Methodology (RSM)**

With the CCD design matrix, the predicted % FAME was modelled based on linear, interaction and quadratic functions where the variable coefficients,  $b_n$ , were determined using multiple linear regression,  $b_n = (D'.D)^{-1}.D'.y$  [D is the design matrix and y is the predicted response].

Linear	$y = b_o + b_1 x_1 + b_2 x_2 + b_3 x_3$
Interaction	$y = b_o + b_1 x_1 + b_2 x_2 + b_3 x_3 + b_{12} x_1 x_2 + b_{13} x_1 x_3 + b_{23} x_2 x_3$
Quadratic	$y = b_o + b_1 x_1 + b_2 x_2 + b_3 x_3 + b_{12} x_1 x_2 + b_{13} x_1 x_3 + b_{23} x_2 x_3 + b_{11} x_1^2 + b_{22} x_2^2 + b_{33} x_3^2$
Pure Quadratic	$y = b_o + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2$

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 Table 2. Coded /uncoded levels and the experimental design according to a circumscribed central composite design (CCD)

Coded level	-1	0	1
Reaction time (hr)	1	3	5
Methanol ratio	5	10	15
% H <sub>2</sub> SO <sub>4</sub>	0.95	1.84	2.74

Eve	Depation	Mathanal		Eve	Depation	Mathana1	
Exp	Keaction	Methanol		Exp	Keaction	wiethanol	
INO	time	ratio	% H <sub>2</sub> SU <sub>4</sub>	INO	time	ratio	% H <sub>2</sub> SU <sub>4</sub>
1	-1	-1	-1	1	1	5	0.95
2	-1	-1	1	2	1	5	2.74
3	-1	1	-1	3	1	15	0.95
4	-1	1	1	4	1	15	2.74
5	1	-1	-1	5	5	5	0.95
6	1	-1	1	6	5	5	2.74
7	1	1	-1	7	5	15	0.95
8	1	1	1	8	5	15	2.74
9	-1.68	0	0	9	0	10	1.84
10	1.68	0	0	10	6	10	1.84
11	0	-1.68	0	11	3	2	1.84
12	0	1.68	0	12	3	18	1.84
13	0	0	-1.68	13	3	10	0.36
14	0	0	1.68	14	3	10	3.3
15	0	0	0	15	3	10	1.84
16	0	0	0	16	3	10	1.84
17	0	0	0	17	3	10	1.84
18	0	0	0	18	3	10	1.84
19	0	0	0	19	3	10	1.84
20	0	0	0	20	3	10	1.84
21	0	0	0	21	3	10	1.84
22	0	0	0	22	3	10	1.84
23	0	0	0	23	3	10	1.84
24	0	0	0	24	3	10	1.84

The optimum conditions were modelled using the response surface modelling tool in MATLAB R2013a. The model fitness was evaluated based on the root mean squares error (RMSE); the smaller the RMSE, the better the model for % FAME prediction. The optimum conditions identified were then verified experimentally and the error of prediction was calculated.

$$RMSE = \sqrt{\frac{\sum_{i=1}^{l} (y - \hat{y})^2}{n - p}}$$

where

n = number of experiments; p = number of predictors.

y = Measured % FAME  $\hat{y} =$  Predicted % FAME

Error of prediction, 
$$\% = \frac{Experimental purity of biodiesel}{Predicted purity of biodiesel} \times 100\%$$

The resulting biodiesel was weighed to determine the yield. The purity of biodiesel was evaluated based on the % FAME area determined from the GCMS profile against the weight of SPO. The conversion efficiency was calculated from the % FFA in SPO before and after the conversion reactions.

Conversion efficiency, 
$$\% = \frac{\% FFA \text{ in } SPO - \% FFA \text{ in } residue after conversion}{\% FFA \text{ in } SPO} \times 100\%$$
  
Biodiesel yield,  $\% = \frac{Weight \text{ of } biodiesel}{Weight \text{ of } SPO} \times 100\%$   
FAME yield,  $\% = \frac{\% FAME \text{ area } from GCMS \times weight \text{ of } biodiesel}{\% FFAME } \times 100\%$ 

AME wield % -	$\%$ FAME area from GCMS $\times$ weight of biodiesel	× 100%
AML yield, 70 –	Weight of SPO	× 100 /0

Samples	% Moisture	% Sediment	% Oil Content	% Free Fatty Acids
SPO1512_S01	$10.15\pm0.72$	$7.63\pm0.71$	$82.09\pm0.90$	$77.12\pm0.48$
SPO1512_S02	$9.86\pm0.52$	$5.76\pm0.11$	$80.65\pm0.36$	$81.02\pm2.13$
SPO2912_S03	$5.20\pm0.27$	$2.77\pm0.29$	$92.03\pm0.56$	$81.02 \pm 1.88$
SPO2912_S04	$7.19\pm0.72$	$2.91\pm0.45$	$89.90\pm0.75$	$68.12\pm0.98$
SPO1803_S05	$1.30\pm0.10$	$3.29\pm0.72$	$95.41\pm0.79$	$47.05 \pm 1.10$
SPO0804_S07a	$6.57\pm0.56$	$3.55\pm0.31$	$89.87\pm0.69$	$86.94\pm0.54$
SPO1204_S07b	$3.45\pm0.59$	$3.39\pm0.35$	$93.16\pm0.93$	$85.96 \pm 0.21$
SPO1204_S08	$6.44\pm0.60$	$5.35\pm0.21$	$88.21 \pm 0.41$	$87.86 \pm 0.22$
POME0504_S06	$96.71\pm0.18$	$4.03\pm0.75$	$0.27\pm0.61$	$12.16\pm0.46$

Table 3. Characteristics of SPO/POME from palm oil mills in Sarawak (n=3)

Table 4. Comparison of the characteristics of SPO from palm oil mills in Sarawak with literature values

SPO	% Moisture	% Free Fatty Acids
This study	1.30 - 10.15	
Tennamaram Palm Oil Mill, Selangor <sup>1</sup> [29]	1.2	65
West Oil Mill, Carey Island, Selangor <sup>2</sup> [0]	1.2	25
Mixed Industrial Palm Oil (SPO+CPO), Selangor <sup>3</sup> [0]	1.1	8.5
Northern, Western, Central, Southern Peninsular Malaysia <sup>4</sup> [0]	0.05-2.08	2.2-88.3
West Oil Mill, Carey Island, Selangor <sup>5</sup> [0]	0.02	7.6
West Oil Mill, Carey Island, Selangor <sup>6</sup> [0]	1.2	21-25
Selangor <sup>7</sup> [0]		16
Thailand Palm Oil Mill <sup>8</sup> [10]		50
West Oil Mill, Sime Darby Plantation <sup>9</sup> [34]		51.6
West Oil Mill <sup>10</sup> [0]	0.9	23.2
Asian Indo Holdings Pte <sup>11</sup> [0]		36.7
Budi Oil Enterprise Sdn Bhd <sup>12</sup> [0]		30

#### **RESULTS AND DISCUSSION**

# 1. Characterization of SPO

Table 3 summarizes the characteristics of SPO/POME collected from palm oil mills in Sarawak. Generally, the SPO contained 1.30 -10.15% moisture, 2.77 - 7.63% insoluble particulates and 80.65 - 95.41% oil (organic soluble fraction). POME is distinctively different from SPO, demonstrating a much higher water content and < 0.5% of oil [3]. Compared to SPO from other locations (Table 4), those produced in Sarawak contain more moisture and a substantial FFA content ranging from 47.05 - 93.28%. In the palm oil refining process, SPO is discharged into cooling ponds and subjected to prevailing environmental conditions. The exposure time contributes to the variation in its FFA levels, as FFA content should increase with time as a result of triglyceride hydrolysis by the lipase enzyme [27, 28].

The IR spectra of SPO (Figure 2) exhibit major absorption bands at 2954-2848 cm<sup>-1</sup>

(aliphatic and methylene groups – fats and lipids), 1700-1732 cm<sup>-1</sup> (carbonyl group of esters and carboxylic acids), 1233-1463 cm<sup>-1</sup> (characteristics of parent materials) and 1009-1114 cm<sup>-1</sup> (C-O stretching of polysaccharides). The absorption signals of carboxylic acids (1699 cm<sup>-1</sup>) and aliphatic esters (1732 cm<sup>-1</sup>) evidently vary between SPO from different mills; samples with high FFA levels (SPO\_0804S07a & SPO12-4\_S07b, SPO1512\_S02 and SPO1204\_S08) demonstrate a pronounced absorption at 1699 cm<sup>-1</sup>. This characteristic can be used to evaluate the quality of SPO using the rapid FTIR technique.

#### 2. Optimization of Esterification Reaction

The % FAME in biodiesel derived from 24 CCD experiments ranged between 72.97 and 100% (Table 5). The major FAME compounds consistently identified were hexadecenoic acid (C16:1) methyl ester and 9-octadecenoic acid (C18:1) methyl ester. This is similar to the prevalent compounds present in biodiesel converted from freshwater algae [38], liquid waste of crude palm oil [22], mild melon seed oil [39] and palm oil [40].



Figure 2. Infrared spectra of sludge palm oils from various mills in Sarawak

Experiment		SPO:		% FAME in
No.	Reaction time, hr	methanol	% H <sub>2</sub> SO <sub>4</sub>	biodiesel
1	1	5	0.95	90.69
2	1	5	2.74	84.94
3	1	15	0.95	72.97
4	1	15	2.74	73.72
5	5	5	0.95	99.45
6	5	5	2.74	99.72
7	5	15	0.95	99.8
8	5	15	2.74	99.52
9	0	10	1.84	99
10	6	10	1.84	100.01
11	3	2	1.84	99.7
12	3	18	1.84	99.16
13	3	10	0.36	98.8
14	3	10	3.3	99.41
15	3	10	1.84	99.36
16	3	10	1.84	99.89
17	3	10	1.84	99.58
18	3	10	1.84	99.51
19	3	10	1.84	100
20	3	10	1.84	99.61
21	3	10	1.84	99.81
22	3	10	1.84	94.92
23	3	10	1.84	94.66
24	3	10	1.84	99.85

Table 5. FAME composition in biodiesel produced according to the experimental design

The design matrix was fitted into linear, interaction, full quadratic and pure quadratic models as tabulated in Table 6. The pure quadratic equation was the best fit model with the smallest RMSE. The response surface plots of the pure quadratic model (Figure 3) indicate that the optimum conditions for the three factors are a SPO:methanol ratio of 1:10, sulphuric acid concentration of 1.84% and a reaction time of 3.25 hr.

Table 6. Linear, interaction and quadratic models with their corresponding root mean squares of error (RMSE)

Model	RMSE	Equations
Linear	6.5565	$y = 91.86 + 3.11x_1 - 0.45x_2 - 0.36x_3$
Interaction	6.6259	$y = 107.57 - 1.19x_1 - 1.84x_2 - 3.08x_3 + 0.36x_1x_2 + 0.36x_2x_3 + 0.16x_1x_3$
Pure Quadratic	6.3065	$y = 74.86 + 6.65x_1 + 1.03x_2 + 7.83x_3 - 0.59x_1^2 - 0.07x_2^2 - 2.23x_3^2$
Full Quadratic	6.3398	$y = 90.78 + 2.35x_1 - 0.37 x_2 + 5.11x_3 + 0.36x_1x_2 + 0.36x_2x_3 + 0.16x_1x_3 - 0.59x_1^2 - 0.07x_2^2 - 2.23x_3^2$

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Figure 3. Response surface plots of methanol ratio, sulphuric acid concentration and reaction time for the esterification reaction

Under these conditions, the pure quadratic model predicts a biodiesel product with 100% FAME. As the catalyst concentration is increased, the biodiesel purity is expected to decrease, although not significantly. The model predicts 99% purity even with acid concentrations of up to 2.4% The improved conversion rate and FAME yield with increasing acid concentration were similarly reported by Akinfalabi et al., but this positive effect seemed to fade with higher catalyst concentrations [41]. Yujaroen et al. [42] likewise revealed that an optimum catalyst dosage was required to achieve the maximum yield, however a further increase in catalyst concentration was unnecessary as it did not improve the yield. The response surface plot further demonstrates that an oil-to-methanol molar ratio of 1:10 would produce biodiesel with an excellent FAME yield, but an increase in methanol would reduce the purity. The optimum molar ratio of 1:10 supports the findings of Akinfalabi et al. whose study involved the esterification of palm fatty acid distillate for biodiesel production [41]. An increase in methanol tended to encourage the forward reaction leading to a higher biodiesel yield [43,44]; however, the water generated during the esterification process could reverse the reaction, thus reducing the FAME content. The reverse reaction towards the reactants may be countered by increasing the amount of methanol [45] but this would increase production costs, rendering the conversion of SPO economically unfavourable. As water inhibition is critical in the production of biodiesel from low quality feedstock, Park et al. advocated the removal of water during esterification, or alternatively, the use of a water resistant catalyst [45]. The optimum reaction time was determined at 3.25 hr. In comparison, our preliminary study with esterification times of 1, 2 and 3 hr demonstrated marked improvements in FAME yield with increasing reaction time. The optimum methanol ratio, reaction time and sulphuric acid concentration reported in the literature were in the range of 9-24, 2-5 hr and 0.4 - 9%, respectively [46,47,48]. This variability is governed by the feedstock and its FFA level. The optimum levels established for the conversion of castor oil containing 13.6% FFA were a methanol ratio of 1:6, 2% sulphuric acid and 1 hr reaction time [23] whilst a palm fatty acid distillate with 90% FFA required a methanol ratio of 1:10 in 2% catalyst for 90 min [41]. Reșitoğlu et al. on the other hand converted cooking oil (65% FFA) using a methanol ratio of 1:9 with 9% sulphuric acid in 120 min [46].

# 3. Biodiesel Conversion

The optimum conditions determined based on a pure quadratic model were experimentally validated. The biodiesel produced contained 94.61% FAME in which hexadecanoic acid methyl ester (51.87%) and 9-octadecenoic acid methyl ester (34.48%) were predominant; this accounts for an error of 5.39% from the predicted FAME yield. Under the established optimum conditions, the biodiesel yield was 74% with a conversion efficiency of 79.87%. The estimated FAME yield in SPO was calculated at 70.00%. Table 7 shows the summary of yield and conversion efficiency of SPO to biodiesel. The conversion efficiency was below 80%, likely because the reaction was inhibited by the presence of water produced during the esterification process [45].

Amount of SPO subjected to conversion, g	5.00
Amount of biodiesel yielded, g	3.70
% FFA in SPO before conversion	81.02
% FFA in residue after conversion	16.31
% FAME area for GCMS	94.61
Biodiesel yield, %	$\frac{3.70}{5.00} \times 100 = 74.00$
FFA conversion, %	$\frac{\frac{81.02 - 16.31}{01.02}}{100} \times 100 = 79.87$
FAME yield in SPO, %	$\frac{\frac{0.9461 \times 3.70}{5.00} \times 100 = 70.00}{5.00}$

# Table 7. Summary of yield and conversion efficiency of SPO to biodiesel

#### CONCLUSION

The SPO collected from palm oil mills in Sarawak were found to contain considerable amounts of FFA (47.05 - 93.28%) and a high moisture content. The optimum esterification conditions established for conversion of SPO containing 81% FFA were 1:10 SPO:methanol, 1.84% sulphuric acid and 3.25 hr reaction time. Under these conditions, the FFA conversion efficiency was 79.87% with a biodiesel yield of 74%. However, the water produced during the esterification process could hinder the conversion efficiency, reducing the biodiesel yield. Due to the high FFA in SPO, esterification must be performed prior to transesterification. This could increase biodiesel production costs and outweigh the material cost of refined CPO. Additional treatment processes may also be required to deal with the water inhibition problem in the esterification process and the high moisture content of SPO.

# ACKNOWLEDGEMENTS

The authors thank Hexa BRT Sdn Bhd for funding this project (GL/F07/HEXA/2021). Thanks are also due to Assoc Prof Dr Tay Meng Guan for sampling and collection of SPO samples.

# CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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