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FULL PAPER

Optimization of palm oil extraction from decanter cake using Soxhlet extraction and effects of microwaves pre-treatment on extraction yield and physicochemical properties of palm oil

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Abstract

Response Surface Methodology (RSM) was applied to study the optimum condition of palm oil extraction from oil palm decanter cake (OPDC) using Soxhlet extraction and n-hexane as solvent. The main objective of this study was to achieve maximum oil extraction by determining the optimum of two parameters such as reaction time and solid to solvent ratio. The optimum parameters were found to be at 4.92 hrs of reaction time and solid to solvent ratio of 1:10. The proposed model shows R^2 value of 0.78 where the experimental parameters were significant to the result. The optimized data was employed for comparison of oil yield for OPDC without and with microwave pre-treatment. OPDC with microwave pre-treatment yielded 3.289±0.047 g of palm oil which was higher than OPDC without microwave pre-treatment which yielded only 3.107±0.085 g of palm oil. Fourier Transform Infrared Spectroscopy (FTIR) analysis also revealed the abundance of C-H alkene stretch and C=O stretch, two major functional groups indicated the presence of fatty acid within the palm oil derived from both samples. Scanning electron microscopy (SEM) of decanter cake provided evidence that the OPDC with pre-treatment has more shrinkage on the surface after Soxhlet extraction compared to OPDC without pre-treatment. Results of this study revealed that RSM helps to optimize parameters in agricultural processing.

1. Introduction

Oil palm sector is one of the most major industries in Malaysia. Malaysia is the world second largest palm oil exporter in 2016 after Indonesia where the main export market in India with an intake of 2.83 million tonnes or 17.6% of total palm oil export (Kushairi, 2017). Aside from being one of the largest palm oil producers, Malaysia also generated a profusion amount of industrial waste (Liew *et al.*, 2014). The estimated generated waste from every ton of fresh fruit bunch (FFB) is at the range from 0.6 to 0.8 m^3 of palm oil mill effluent (POME), 22 to 23% of empty fruit bunch (EFB), 13.5% of palm mesocarp fibre (PMF) and 4-5% of oil palm decanter cake (OPDC) from a typical palm oil mill (Sahad *et al.*, 2014).

Environmental issues are being concerned greatly in Malaysia as the palm oil mill contributed much on environmental pollution from its waste such as oil palm decanter cake (OPDC). The huge amount of OPDC production requires a large land area for composting and this will cause pollution hazards such as soil and water pollution (Farhana, 2010). Moreover, high production of biomass waste has negatively affected the total oil extraction rate (OER) of palm oil industry due to the losses of oil in the waste (Sahad *et al.*, 2014). The OER indicates the actual amount of oils extracted from FFB, as well as the overall efficiency of typical palm oil mills. Hence approaches should be done to convert these biomass waste into another form of energy or usage. The current biomass residues are being used as fuel in the boiler or be converted into fertilizer. The solid biomass wastes are being used as the main source of energy input for few palm oil mills to produce electricity and steam for palm oil production process such as sterilizer (Wu *et al.*, 2017). Few researchers have studied the suitability of OPDC as ruminant feed, plant fertilizer and composting material (Bakri, 2013; Sahad *et al.*, 2014). In addition,

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demand for biodiesel sector is increasing steadily (Szulczyk and Atiqur, 2018). Biodiesel is a renewable vehicle fuel based on biomass. Research showed that palm oil can be use as biodiesel for the vehicle with a diesel engine (Archer *et al.*, 2018).

In this preliminary study, Soxhlet extraction was a conventional method used in the process of recovering phenolic compounds from the plant matrix. Soxhlet extraction is the most commonly used method for extracting phenolic compounds as this extraction method has various advantages such as low processing cost; simple operation; high performance; favourable for the total recovery of extracts and less time and solvent consuming (Alara *et al.*, 2018). The soxhlet extraction method was used in this study to recover oil from OPDC. Hexane has high oil extraction efficiency, over 99% hence it is widely used in the vegetable oil industry. Vegetable oils are produced from oilseeds through solvent extraction such as hexane or a combination of mechanical processing and solvent extraction (Pirshahid *et al.*, 2018). A pre-treatment of microwave heating prior to Soxhlet extraction was also studied. A non-contact energy transfer process from electromagnetic energy into thermal energy occurred through microwave heating. If the electromagnetic energy is efficiently absorbed by the sample, the heating rate is increased. Based on the study by Sadeghi *et al.* (2017), changes in the cell structure of sample occurs which caused by electromagnetic waves, thus it affected the overall efficiency of the extraction process.

The objectives of this study were to study the optimization of palm oil extraction from decanter cake by Soxhlet extraction using Response Surface Methodology and to compare the performance of microwave pre-treatment prior to Soxhlet extraction in terms of the oil yield, chemical, physical and surface morphology of OPDC.

2. Materials and methods

2.1 Summary of Experimental Flow

The overall flow chart of the methodology was presented in Figure 1.

2.2 Sample Preparation

Oil palm decanter cake (OPDC) was collected from Jengka Pahang Palm Oil Mill and kept in the cold storage room at 5°C. n-Hexane (J.T.Baker™, United States) was used as the solvent. All chemicals used in this study were of analytical grade.

2.3 Experimental design

The optimized data of the following parameters were analysed using RSM: (A, reaction time, 4-8 hrs) and (B, solvent ratio, 10-13) also the affected response variable (Y, yield of oil extracted). The α value represented the distance of each star point from the centre, was set to 1.5 (α=1.5). Central composite design (CCD) was used for thirteen randomized experiments (8 non-centre points and 5 centre points) to develop a response surface that determines the optimal variables A and B that resulted in the highest response variable Y as shown in Table 1. The experiment was conducted in triplicate to obtain the average value of Y. The average oil yield data was keyed in the Design Expert Version 11 software, the optimization process was started. For optimized data, the software was set up to generate the minimum reaction

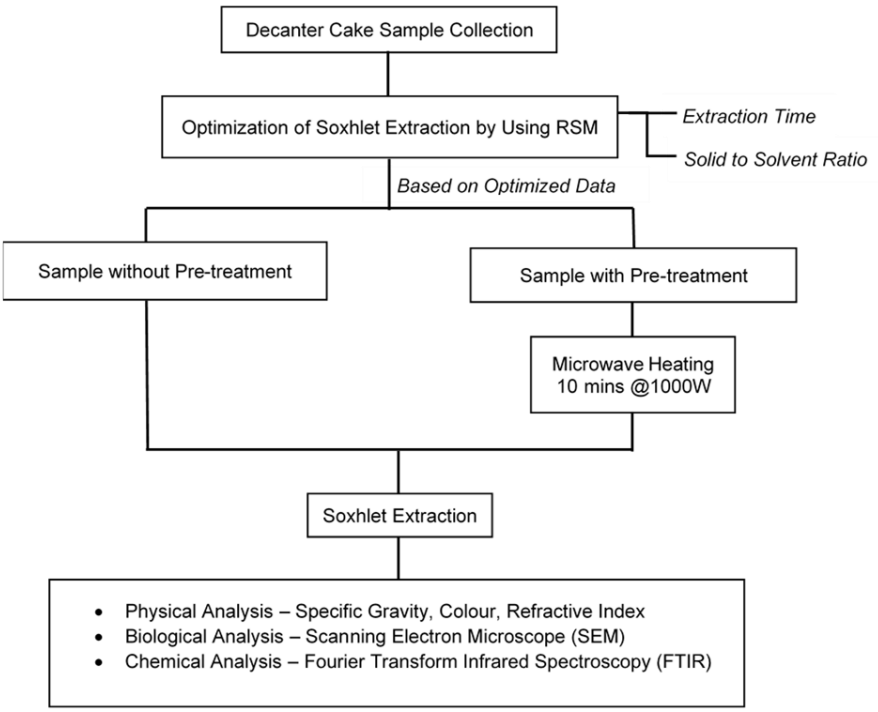

Figure 1. Flow chart of palm oil extraction from OPDC.

time, minimum solvent ratio and maximize yield of oil extract to achieve the 3 criteria as shown in Table 1. The chosen optimized data with the highest desirability was tested by carrying out another experiment to confirm the oil yield. Table 1 shows the criteria set up to obtain the optimized data.

Table 1. Experimental parameters for CCD of Soxhlet extraction.

Factors	Parameters	Limits		Criteria
		Lower	Upper	
A	Reaction time	4	8	Minimum
B	Solvent ratio	10	13	Minimum
Y	Yield of oil extracted	2.787	3.664	Maximum

2.4 Soxhlet Extraction

About 45 g of OPDC was measured by using an electronic balance (SHIMADZU Electronic Balance TX223, Japan) then moulded in a uniform rectangular cube. The sample was then dried in oven dryer (MEMMERT Universal Oven UN55, Germany) at 105°C for 24 hrs. A total of 20 g of dried OPDC was weighted and crushed into smaller pieces with a hammer and placed in a cellulose extraction thimble (CT30100, AquaLab Supplies, Spain) with internal diameter and length (30 x 100mm). The thimble was then filled with cotton as a stopper to prevent OPDC from leaking out during extraction. The amount of solvent used is based on the experimental data shown on Table 2 and the solvent was measured and filled into a bottom flask. The amount of solid used is fixed which is 20g and the solid to solvent ratio is (1: x) where x indicated the solvent ratio. The calculation of the amount of solvent used is calculated by using Equation 1:

$$Amount\ of\ solvent\ used\ (ml) = 20g \times solvent\ ratio \quad (1)$$

The temperature of Soxhlet extractor (JEX5/55 FAVORIT, Malaysia) was set to 3 for pre-heating and later changed to 4 (approximately 70°C). A total of six experiments were carried out following the experimental setup and shown in Table 2. Meanwhile, the solvent was separated from the oil using rotary vacuum evaporator (Heidolph Laborota 4000, Germany) and was collected in the receiving flask. All experiments were performed in triplicate. The amount of extracted oil was weighed and recorded.

Table 2. Experimental result

Run	Factor 1	Factor 2	Response
	A: Reaction Time	B: Solvent Ratio	Yield of extracted
1	6	11.25	3.334±0.25
2	3	11.25	2.787±0.21
3	6	11.25	3.082±0.05
4	6	11.25	3.037±0.12
5	4	12.5	2.991±0.17
6	6	11.25	3.025±0.11
7	4	10	3.006±0.14
8	8	11.25	3.286±0.26
9	6	10	3.298±0.19
10	6	11.25	3.064±0.07
11	8	12.5	3.328±0.06
12	8	10	3.314±0.23
13	6	13	3.644±0.06

2.5 Sample pre-treatment

Another experiment was carried out by using the optimized data of Soxhlet extraction for factor A and B. Microwave oven (Panasonic Microwave Oven NN-J993, Japan) was used for this experiment. Dried OPDC underwent microwave heating for 10 mins at 1000W following the method proposed by (Xern, 2017) as pre-treatment. After heating, 20 g of dried OPDC was used for Soxhlet extraction by using the optimized data from RSM (reaction time,4.9 hrs and solvent ratio, 10). The amount of extracted oil from the sample with and without pre-treatment was compared and discussed.

2.6 Physical analysis

Physical analysis on extracted oil for the sample with and without pre-treatment was determined. The specific gravity of the samples was calculated following Equation 2 by measuring the mass of oil sample using an electronic balance (SHIMADZU Electronic Balance TX223, Japan) and volume of oil sample using 100 mL measuring cylinder, colour measured by using colour reader (Konica Minolta Colour Reader Cr-10, Japan) while the refractive index was measured by using a refractometer (KRUSS Digital ABBE Refractometer AR2008, Germany). The temperature of oil samples was maintained at 50°C where the results were later compared with the references of standard values (Koushki and Nahidi, 2008; Chinedu and Ebere, 2017).

$$Specific\ gravity = \frac{(mass\ of\ oil\ sample)/(volume\ of\ oil\ sample)}{density\ of\ water} \quad (2)$$

2.7 Chemical analysis

Chemical analysis of extracted palm oil samples without special pre-treatment was carried out by using Fourier Transform Infrared Spectroscopy (FTIR-Spectrum 100 JASCO, Japan). The IR spectra were determined over a wavenumber range from 4000 to 650 cm^{-1}. The resolution was 4 cm^{-1} and 32 scans were averaged. FTIR is used to determine the functional group components from 2 extracted palm oil samples which were palm oil extracted from sample with and without pre-treatment. The pH value of oil samples was measured by using a pH meter.

2.8 Biological analysis

Biological analysis was carried out on OPDC

without pre-treatment and OPDC sample with pre-treatment by using Scanning Electron Microscope (SEM) (SEM S-3400N Hitachi, Japan) where the SEM images at 300x magnification of the samples was taken digitally. Each sample was coated with a layer of gold for 120s using a coater (Quorumtech EMscope SC500 Sputter Coater, United Kingdom). Images of 4 OPDC samples were captured: OPDC after oven dry; OPDC after Soxhlet extraction (without pre-treatment); OPDC after microwave pre-treatment; and OPDC after Soxhlet extraction (with pre-treatment).

3. Results and discussion

3.1 Sample without pre-treatment

The thirteen experiment runs were designed by RSM software where the response, yield of extracted oil was measured and recorded as shown on Table 2. Increasing solvent ratio causes the increment of the concentration gradient between the solid and the liquid phase where it favours good mass transfer thus higher yield of oil extracted (Sayyar *et al.*, 2009). A longer reaction time enhanced the diffusion process of oil in solvent which resulted in higher oil yield. As depicted in Table 2, the least yield of oil extracted (2.787 mL) was on experiment run 2 at 3 hrs of reaction time and the solvent ratio of 1:11.25, meanwhile the highest yield of oil extracted (3.644 mL) was on an experiment run 13 with 6 hrs of reaction time and solvent ratio of 1:13.

3.2 Data optimization by using RSM

Figure 2 shows the 3D surface plot for the yield of oil extract before optimization. The response surface plot showed the optimal condition between the variables such as reaction time and solvent ratio (Pirshahid *et al.*, 2018). The red dots on the 3D plot showed the data above the predicted value while the pink dots showed data below the predicted value. Figure 2(a) showed that the highest point on the 3D plot is below the expected value, hence an optimization was carried out and resulted in a more uniform 3D plot as shown in Figure 2(b). The optimized response surface plot as shown in Figure 2(b) revealed the increased oil yield with increasing variables A and B.

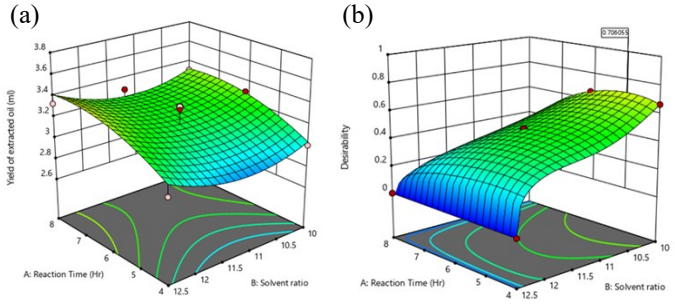

Figure 2. 3D response surface plot, (a) response surface plot before optimization, (b) response surface plot after optimization

There was a total of 10 solutions generated following the criteria set up where only solution with the highest desirability to achieve the criteria will be chosen to carry out a confirmation test on oil yield. The chosen solution with 0.706 desirability was at 4.923 hrs of reaction time and the solvent ratio of 10 which is 200mL. The expected oil yield should be 3.179 mL. To verify the yield, 3 confirmation tests were carried out based on the parameters in the chosen solution that achieved the criteria which were lowest reaction time and lowest ratio that gives the highest oil yield. Based on the confirmation test, the average oil yield based on the chosen solution was 3.107±0.085 mL where it is lower than the expected oil yield (3.179 mL). The difference between theoretical oil yield and experimental oil yield was 0.072 mL. The percent error is considered low which is 2.26% as the desirability to achieve the criteria was 0.706 (70.6%).

3.3 Statistical analysis

A statistical analysis of variance (ANOVA) is performed to see either the process parameters are statistically significant which is shown in Table 3. Finally, a confirmation test is conducted to verify the optimal process parameters obtained from the process parameter design.

Based on Table 3, the Model F-value of 4.84 implies the model is significant. There is only a 3.11% chance that an F-value this large could occur due to noise. Parameter A has the highest F value (7.77), hence it has the most influence in oil extraction process. P-values less than 0.05 indicate model terms are significant (Mushtaq *et al.*, 2015). In this case A, B^2 are significant model terms. Values greater than 0.05 indicates the model terms are not significant. Based on Table 2, B showed p-value greater than 0.05 which is not significant. The Lack of Fit F-value of 0.72 implies the Lack of Fit is not significant relative to the pure error. There is a 58.99% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good so that a fit model is obtained.

Table 3 shows the fit statistics analysis for the experimental run. R-squared is a statistical measure of how close the data are to the fitted regression line. R^2 approaching 1 showed the model is completely fit while R^2 approaching 0 showed that the model cannot be used due to large variance differences (Drennan, 1996). The predicted R^2 of 0.0561 is not as close to the adjusted R^2 of 0.6156 as one might normally expect; i.e. the difference is more than 0.2. This may indicate a large block effect or a possible problem with the model and/or data. Things to consider are model reduction, response transformation, outliers, etc. All empirical models should

Table 3. ANOVA Test for Response 1(Y): Yield of Extracted Oil

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.455	5	0.091	4.84	0.0311	significant
A-Reaction Time	0.1461	1	0.1461	7.77	0.027	
B-Solvent ratio	0.0094	1	0.0094	0.4981	0.5031	
AB	0.0002	1	0.0002	0.0112	0.9187	
A^2	0.0581	1	0.0581	3.09	0.1221	
B^2	0.1143	1	0.1143	6.08	0.043	
Residual	0.1315	7	0.0188			
Lack of Fit	0.0461	3	0.0154	0.7204	0.5899	not significant
Pure Error	0.0854	4	0.0213			
Cor Total	0.5865	12				
Std. Dev.	0.1371		R^2	0.7758		
Mean	3.19		Adjusted R^2	0.6156		
C.V. %	4.3		Predicted R^2	0.0561		
			Adeq. Precision	8.7604		

be tested by doing confirmation runs. Adeq. precision measures the signal to noise ratio. A ratio greater than 4 is desirable (Yang *et al.*, 2018). The ratio of 8.760 indicates an adequate signal.

Figure 3 shows that the levels of the oil yield predicted from the fitted empirical model are in line agreed with the observed values under the observed experimental conditions, with a sensibly high value of the coefficient of determination of 0.7758 (R^2) (Table 3) (Mushtaq *et al.*, 2015). The coloured dots showed the actual oil yield where the straight graph showed the predicted value. Based on Figure 3, there were few points fall below the expected value and a few points were above the predicted value. It shows the correlation between predicted and actual data are similar. The graph showed consistent data that proven this experiment data is acceptable.

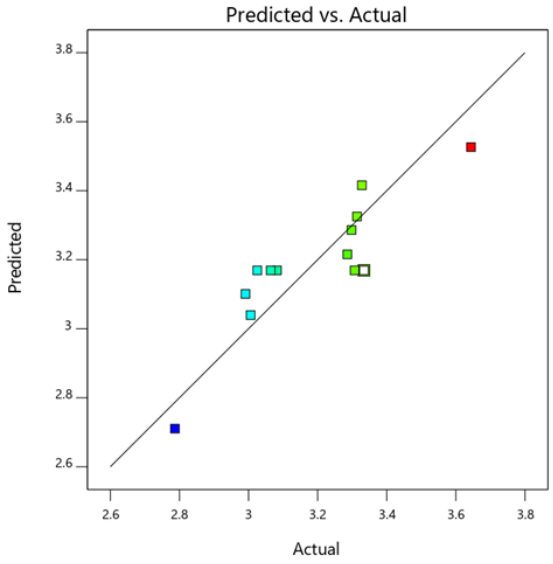

Figure 3. Graph of predicted vs actual value of oil yield

3.4 Regression model equation

The regression model equation generated by RSM to calculate the oil yield based on factors A and B with response Y. The equation in terms of actual factors can be used to make predictions about the response Y for given levels of factors A and B. Here, the levels should be specified in the original units for each factor. The regression model Equation 3 is as follow:

Yield of extracted oil	=
+14.19407	
+0.353781	Reaction Time (A)
-2.21912	Solvent ratio (B)
+0.002900	Reaction Time * Solvent ratio
-0.025944	Reaction Time²
+0.099206	Solvent ratio²

Yield of extracted oil (mL)

$$= 14.19407 + 0.353781(A) - 2.21912(B) + 0.0029(A * B) - 0.025944(A^2) + 0.099206(B^2)$$

3.5 Data comparison between sample without and with pre-treatment

The average oil yield for OPDC microwave pre-treatment was 3.289±0.047 g at 4.923 hrs of reaction time and the solvent ratio of 10 (200 mL) which was greater than OPDC sample without pre-treatment 3.107±0.085 g as shown in Figure 4. The difference between both yield value was 0.182 g or 5.533% more oil yield compare to sample without pre-treatment.

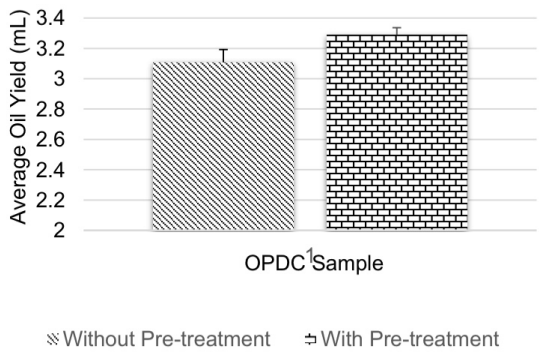

Figure 4. Comparison of average oil yield for both sample

Microwave pre-treatment is different compare to regular oven drying. Microwave heating used electromagnetic wave that heats throughout the OPDC sample while oven drying heats up OPDC sample from the outer surface only. Microwaves are a form of non-ionizing electromagnetic energy emitting at frequencies ranging from 300 MHz to 300 GHz (Sadeghi *et al*., 2017). This energy transmitted as wave and penetrated through biomaterials as well as interacted with polar molecules of materials, such as water to generate heat. Microwaves acted directly on molecules by ionic conduction and dipole rotation and thus only polar materials can be heated based on their dielectric constant (Sadeghi *et al.*, 2017).

3.6 Physical analysis of extracted oil

The physical properties (Specific gravity, colour and refractive index at 50°C) of extracted oil from OPDC with microwave pre-treatment and without microwave pre-treatment were carried out and the results were shown in Table 4. The result was compared to the standard at 50°C (Koushki and Nahidi, 2008; Chinedu and Ebere, 2017). Specific Gravity (SG) is a term used to define the density or weight of a liquid as compared to the density of an equal volume of water at a specified temperature (Chinedu and Ebere, 2017). Based on Table 4, the oil extracted from the sample without pre-treatment has a higher density (0.948 g/cm^3) and SG value (0.960) compared to the sample with pre-treatment. However, both oil samples had similar density and SG compared to standard.

Colour and appearance are important quality parameters for oil. Any colour within visible range was represented with the aid of three-dimensional coordinates L, a and b. Based on Table 4, both oil samples had similar L, a, b value. By referring to the CIELAB Colour Chart (Paravina, 2018), the colour of the oil sample was light yellowish colour at 50°C.

Table 4. Physical properties of palm oil based on standard at 50°C.

Physical Characteristics		Without Pre-treatment	With Pre-treatment	Standard value[#]
Density (g/cm^3)		0.948	0.886	0.889
Specific Gravity		0.96	0.897	0.906
Colour	L	58.8	58.7	nil*
	a	6	5.8	
	b	29.3	29	
Refractive Index (nd.)		1.462	1.462	1.455 – 1.462

[#]Adapted from Koushki and Nahidi (2008), Chinedu and Ebere (2017)

*the colour result was not reported

Refractive index (RI) showed how much light bends when it travelled through the soil sample (Chinedu and Ebere, 2017). The value of RI for oil sample without and with pre-treatment is the same and similar compared to reference standard value.

3.7 Chemical analysis of extracted oil

The FTIR spectra of oil extracted for the sample without and with pre-treatment by Soxhlet extraction are presented in Figure 5. Overall, the results showed a relatively similar FTIR spectra pattern. The major finding of extracted oil at different processing routes is that the oil showed very similar peaks where the first peak at 2854 to 2925 cm^{-1} indicated the presence of C-H alkene stretch and a representative of specific fingerprint around 1745 cm^{-1} attributed to C=O stretch which have similar reading compare to a study (Fauzi *et al.*, 2016). The result strongly supports that the extracted oil contains ester. Both oil samples were measured to be acidic where oil sample with pre-treatment showed

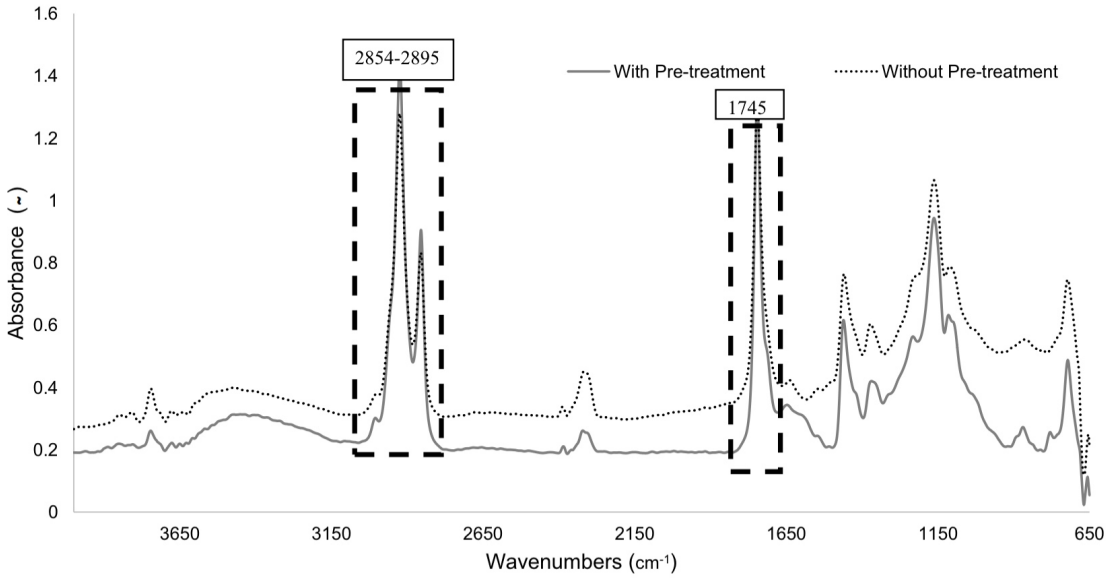

Figure 5. FTIR spectra of extracted oil samples.

higher acidic (pH 5.135) compared to oil extract without pre-treatment (pH 5.690). This explained the absorbance of 1st peak for the sample with pre-treatment was observed to be much higher than that of the sample without pre-treatment.

Ester is derived from carboxylic acid and alcohol ester is the main class of lipids which makes up the vegetable oil. Both functional groups indicated the presence of fatty acid in the oil extract (Fauzi *et al.*, 2016). Further analysis was suggested to be performed in future on oil extract by using Gas-chromatography to determine the component in oil extracts as palm oil contains oleic and palmitic acid the most which portray its oil quality.

3.8 Biological analysis of OPDC samples

The structure images of oil palm decanter (OPDC) samples were captured by using scanning electron microscope (SEM) as shown in Figure 6. Figure 6 (a-d) shows SEM images of OPDC without pre-treatment before Soxhlet extraction; OPDC without pre-treatment after Soxhlet extraction; OPDC with pre-treatment before Soxhlet extraction and OPDC with pre-treatment after Soxhlet extraction respectively.

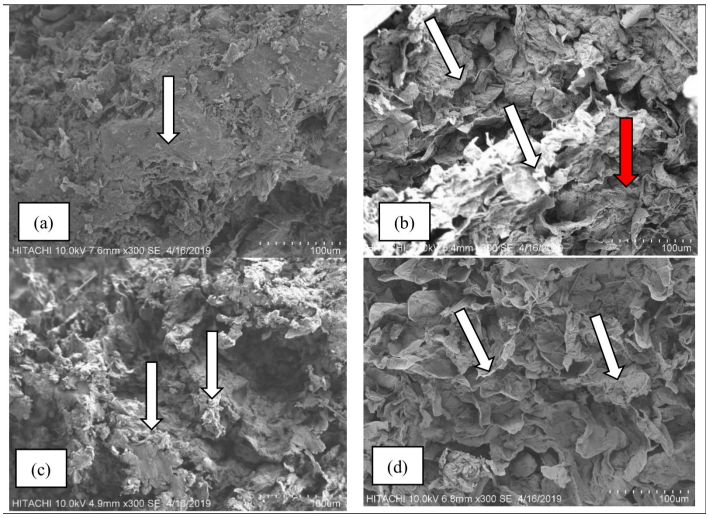

Figure 6. Scanning electron micrograph of OPDC; (a) SEM image of OPDC without pre-treatment before Soxhlet extraction; (b) SEM image of OPDC without pre-treatment after Soxhlet extraction; (c) SEM image of OPDC with pre-treatment before Soxhlet extraction; (d) SEM image of OPDC with pre-treatment after Soxhlet extraction.

SEM images showed the morphological changes on the surface of the OPDC samples. The surface of OPDC sample without pre-treatment before extraction appear to be flatter and smoother as shown in Figure 6(a) compare to OPDC with microwave pre-treatment before Soxhlet extraction as shown in Figure 6(c) where the surface has started to shrink. OPDC sample treated by microwave heating resulted in more breakage of sacs that contains oil and it is proved where OPDC sample after microwave showed darker in colour and appear to be wet. A study by (Sadeghi *et al.*, 2017) also stated electromagnetic waves changes the cell structure of the sample. For sample after Soxhlet extraction, Figure 6(b) image shows a red arrow where the labelled part is still smooth and flatten compare to Figure 6(d) as the overall surface shown to be more shrunk. This showed that more oil was extracted for OPDC with microwave pre-treatment compare to OPDC without pre-treatment as stated in the result of oil yield comparison in Figure 4. In contrast, OPDC sample shown shrunk surface as the oil was extracted from the sample.

4. Conclusion

Response Surface Methodology is a great tool for designing laboratory-scale experiment while providing statistical analysis to support the data. Optimization of palm oil extraction from decanter cake using Soxhlet extraction by RSM presented that at 4.923 hrs of reaction time and solid to solvent ratio of 1:10 has the highest oil yield which is 3.107±0.085 g. The proposed model design by RSM shows R^2 value of 0.776 where the experimental parameters are significant to the result. OPDC sample with and without microwave pre-treatment was compared and the results indicated microwave pre-treatment (1000W, 10 mins) yielded 3.289±0.085 g of palm oil which was higher than that of OPDC without microwave pre-treatment (3.107±0.085 g). Physical analysis of extracted oil from OPDC without and with microwave pre-treatment was carried out where both data were similar. Chemical analysis by using FTIR showed that both oil sample has two similar peaks on the FTIR Spectra where the 1st peak at the range of 2854 to 2925 cm^{-1} indicated the present of C-H alkene stretch and 2nd peak at 1754 which was C=O stretch ester. Both of these functional groups indicated the presence of fatty acid in the oil sample. Lastly, biological analysis with SEM images indicated that OPDC with pre-treatment had more shrinkage on the surface after Soxhlet extraction compared to OPDC without pre-treatment. The result proved that higher oil yield from OPDC with microwave pre-treatment.

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