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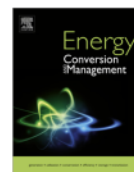
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Decanter cake as a feedstock for biodiesel production: A first report



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ABSTRACT

Decanter cake (DC), with an oil content of 11.5 ± 0.18 wt.%, was subjected to ultrasound-aided transesterification using boiler ash as a base catalyst, petroleum ether and hexane as co-solvents. Optimization work revealed that at MeOH:oil mass ratio of 6:1 and 2.3 wt.% catalyst (based on DC weight) with 1:2 co-solvents:DC mass ratio as the optimal reaction conditions. Both decanter cake and boiler ash, waste materials from oil palm mill, were successfully utilized to produce methyl ester (biodiesel) with highest conversion of 85.9 wt.% in a 1 h reaction period at 55 °C.

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1. Introduction

Processing of waste materials into value-added commodities is of great interest to researchers owing to its potential in solving simultaneously the issues related to the environment and depletion of natural resources. A large range of industrial wastes, both natural and synthetic, are disposed without extracting the useful components from them. Biomass is a promising source of renewable energy that contributes to energy needs and is the best alternative for guaranteeing energy for the future. In Malaysia alone, oil palm industry producing over 83 million dry tonnes of solid biomass annually and by year 2020 the figure is expected to be between 85 and 110 million dry tonnes [1]. Malaysia, being one of the largest producers and exporters of palm oil for the last forty years (total Malaysian palm oil plantation approximately 5 million hectares in 2011), has an estimated total amount of processed fresh fruit bunches (FFB) of 7.8 tonnes/ha (January–June 2012 production average), 70% of which is removed as waste, such as palm press fiber (30%), empty fruit bunches (EFB, 28.5%), palm kernel shell (6%), decanter cake (DC, 3%) and others (2.5%) [2,3]. As such, in the processing of 39 million tonnes of FFB annually (7.8 tonnes/ha \times 5 million ha), 1.17 million tonnes of waste DC (3% of 39 million tonnes FFB) is generated in Malaysia alone.

DC is a solid waste produced when the crude palm oil is centrifuged for purification where the supernatant is the purer palm oil and the sediment is the decanter cake. DC contains water (about 76%, on wet basis), residual oil (about 12%, on dry basis) and nutrients, cellulose, lignin and ash. There are previous reports on the use of DC in the area of bio-fertilizer, biofuel and cellulose [4,5]. Oil adsorbed on DC is a minor byproduct of palm oil purification process with appreciable magnitude that could be potential feedstock for production of biodiesel (methyl ester). The utilization of waste/used edible oils and animal fats as raw materials is a relevant idea, and there are many advantages for using waste feedstock for biodiesel production: (i) abundant supply, (ii) relatively inexpensive, (iii) environmental benefits [6–9]. Recently many researchers have focused on the exploitation of waste materials such as shells, ashes, rock, and bone as catalysts in transesterification. Due to their abundance and low cost, use of such waste materials has become very attractive. Indeed, the boiler ash (BA) used as a catalyst in this work is also a waste byproduct of palm oil mill. Generally, in palm oil mill, approximately 5% of BA is produced upon the burning of dry EFB, fiber and shell in boiler [10–12].

The current study focuses on *in situ* production of methyl ester (ME) using ultrasound as an eco-extraction process and the success of which had been reported previously [13,14]. Ultrasound promises simpler process with higher product purity and the process can be completed in shorter reaction time as well as reducing the amount of solvent as compared to conventional extraction methods. This study demonstrates the potential of DC as a low cost

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feedstock for *in situ* transesterification reaction aided by ultrasound and catalyzed by BA.

2. Methods

2.1. Material

Decanter cake (DC) and boiler ash (BA) were collected from local oil palm mill (Lepar Pahang, Malaysia). Methyl heptadecanoate of chromatographic grade, obtained from Sigma–Aldrich (Switzerland), was used as an internal standard whereas methanol, petroleum ether (PE) and hexane of analytical grades were purchased from Bendosen Laboratory Chemicals (Norway).

2.2. Catalyst preparation and characterization

The BA (catalyst) was prepared following the procedures published elsewhere [10,11]. BA was prepared by oven drying at $105 \pm 2^\circ\text{C}$ until constant weight, then calcined at 500°C for 5 h. The catalyst was characterized by X-ray diffraction (Rigaku) with Cu K α as a source, 2θ range from 25° to 125° with step sizes of 0.1° , at a scanning speed of 1°min^{-1} . Surface analysis of the catalyst was examined by using Micromeritics ASAP 2000. Prior to the analysis all the samples were degassed at 105°C and the adsorption of N_2 was measured at -196°C . The morphology of catalyst was observed by FE-SEM with electron dispersive X-ray (EDX) (JSM-7800F).

2.3. Preparation and determination of oil content in DC

The DC was dried in an oven at 105°C for 2.5 h. Subsequently, the dried DC was ground and sieved to obtain a smaller particle size. The oil content was determined by extracting 2 g dried DC with 20 ml of co-solvents (PE and hexane in equal ratio). The mixture was immersed in an ultrasound water bath (Bransonic at a working frequency of 42 kHz and the power supply of 235 W) at a temperature of $55 \pm 2^\circ\text{C}$ for 30 min. The sonicated mixture was then centrifuged at 1000 rpm for 5 min. The yellow-brown supernatant layer was transferred into a pre-weighed flask. The process was repeated two times and the accumulated supernatant solution

was evaporated by rotary evaporation. The oil content was calculated using the following formula:

$$\text{Oil content (\%)} = (M_1/M_0) \times 100$$

where M_1 and M_0 are the masses of the accumulated oil and DC in g, respectively. The acid value and acidity of the oil were determined following EN 14104 standard. Approximately 2 g of oil was weighed into an Erlenmeyer flask; 50 ml of neutralized isopropanol and a few drops of phenolphthalein indicator were added. It was then placed on a hot plate and the temperature was regulated to about 40°C . The sample was then shaken gently while titrating with a standard potassium hydroxide solution to the first permanent pink colour. The colour must persist for 30 s. The determination was repeated three times. The moisture content in DC was analyzed using the Karl Fischer titration method (784 KFT Titrimo, Metrohm). The morphology of DC was observed by FE-SEM with electron dispersive X-ray (EDX) (JSM-7800F) whereas the elemental analysis of extracted oil (O-DC) and DC (before and after extraction) was performed using ICP–MS (Agilent 7500 CX).

2.4. In situ transesterification reaction

Transesterification reaction was carried out in a test tube with the constituent of 2 g DC (0.23 g oil, at oil content 11.5 wt.%), 2.3 wt.% catalyst (0.046 g, based on DC weight), 1.35 g of methanol to give approximately 1:6 oil to methanol mass ratio and 1 g co-solvents (PE and hexane in equal ratio) to give 1:2 co-solvents:DC mass ratio. The test tube was immersed in the ultrasound water bath with the temperature set at 55°C for 1 h. Then the test tube was removed and centrifuged at 1000 rpm for 5 min. Three layers were formed; the top was co-solvents, followed by methanol and the DC layer at the bottom. The co-solvents and methanol layers were pipetted out separately, then about 3 ml of each PE and hexane were added into the test tube, homogenized and centrifuged, then the resultant top co-solvents layer was added to the portion of co-solvents that was pipetted out earlier. The step was repeated twice to obtain a total amount of about 14 ml of accumulated co-solvents. Upon solvent evaporation using rotary evaporator, pure ME was obtained. The resultant ME was subjected to chromatographic analysis for quantification using GC-FID (Agilent 7890A) following the European procedure EN 14214 with polar capillary

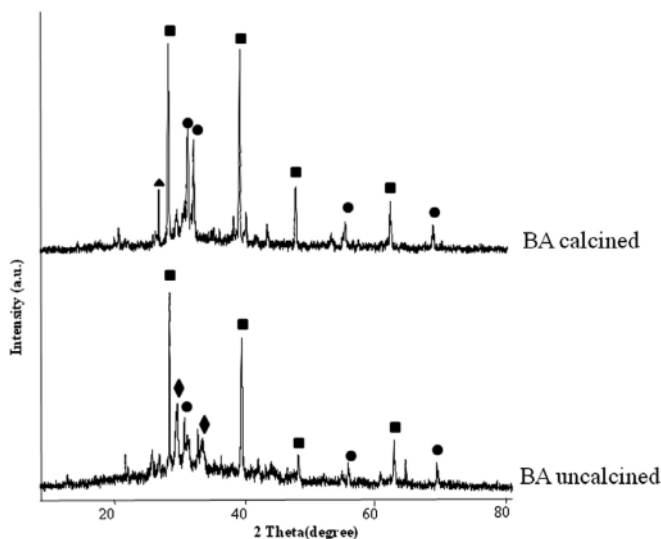


Fig. 1. XRD pattern of BA-calcined and BA-uncalcined; ▲: K_2MgSiO_4 , ■: $\text{K}_{9.6}\text{Ca}_{1.2}\text{Si}_{12}\text{O}_{30}$, ●: $\text{K}_4\text{CaSi}_3\text{O}_9$, and ◆: KAlO_2 .

column (DB-Wax, 30 m × 0.25 mm i.d. × 0.25 μm) using methyl heptadecanoate as an internal standard. Peaks of methyl esters were identified by comparing them with their respective standards.

3. Results and discussion

3.1. Catalyst characterization

X-ray diffraction (XRD) was used to determine the crystal structure of the BA, which is shown in Fig. 1. The diffractograms of BA-uncalcined (Fig. 1) showed a minor amorphous structure; with potassium silicate as a dominant structure and other silicates such as magnesium, aluminum and calcium. Calcination at 500 °C may have caused increasing bond formation between potassium, magnesium calcium and silicon oxide. Surface area and pore structure analyses were done using BET method. The uncalcined BA has total surface area is 106.6 (m²/g) and pore volume is 0.032 cm³/g. The calcination may have collapsed in the surface area and pore

volume of calcined BA-500 (55.86 m²/g and 0.024 cm³/g, respectively). The major pore distribution in BA-dried and BA-500 is between 20 and 40 nm, which can be classified as mesopores. The micrograph of BA-dried and BA-500 have shown similar spongy and porous structure (Fig. 2a and b) with particle size ranged in 25–30 nm and 15–20 nm, respectively for BA-dried and BA-500. The smaller size for BA-500 may result from decomposition of larger organics during calcination. In addition, from the EDX results, the BA-dried and BA-500 exhibited the mixture of the metals, namely potassium, calcium, phosphorus, silicon, aluminum, and magnesium, probably in the form of oxides due to the presence of high oxygen content. While the major element is potassium, the mixture of metal oxides could also contribute to basicity of the catalyst.

3.2. Oil content and characterization of DC

The oil recovered from DC (O-DC) was 11.5 ± 0.18 wt.% (on dry basis) and its acid value is 13.8 ± 0.02 mg/KOH g (equivalent to

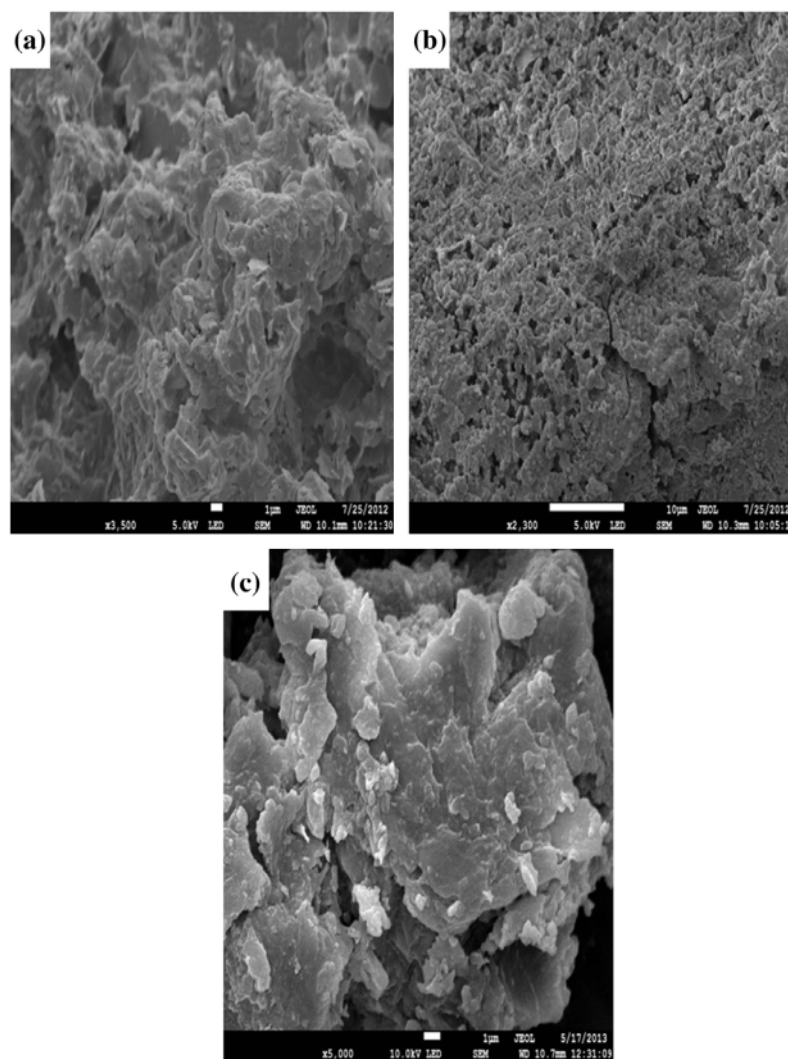


Fig. 2. SEM micrograph of (a) BA-calcined, (b) BA-uncalcined, and (c) DC.

Table 1
List of element detected in extracted oil and DC.

Element	O-DC (ppm) ^a	DC before extraction (ppm)	DC after extraction (ppm)
Be	<0.6 ppb	0.04	0.04
Na	<0.02	<0.02	<0.02
Mg	8.0	1275	1334
Al	<0.2 ppb	36,414	39,306
K	Not detected	5365	5830
Ca	359	4073	4145
V	0.2	3.1	3.2
Cr	1.1	9.2	9.7
Mn	<0.04 ppb	29.5	30.1
Fe	<0.01	1603.2	1649
Co	<0.01 ppb	0.1	0.1
Ni	<0.04 ppb	<0.04 ppb	<0.04 ppb
Cu	0.2	19.3	21.8
Zn	5.5	16.1	16.7
As	0.09	1.6	1.5
Se	0.3	0.07	0.05
Mo	<0.5 ppb	0.5	0.6
Ag	<0.6 ppb	0.1	0.1
Cd	<0.2 ppb	0.1	0.1
Ba	0.4	20.2	25
Pb	0.3	3.7	4.1
U	<0.6 ppb	0.3	0.3

^a Extracted using petroleum ether.

6.3% FFA as palmitic acid). The high acid content of O-DC could be due to the presence of substantial amount of free fatty acids in CPO, as reported previously [15]. The acid value of methyl esters prepared from oil adsorbed on decanter cake (ME-O-DC) revealed an average value of 1.5 ± 0.04 mg KOH/g (equivalent to 0.68% FFA as palmitic acid) and saponification number of 185 mg KOH/g.

Moisture could possibly influence transesterification reaction with base catalyst. The presence of moisture in the reaction could change the Lewis site of base catalyst into Brønsted base catalyst, and promote hydrolysis of triglyceride into FFA, which in turn trigger saponification and eventually decelerate ME conversion. In this study, the moisture in the extracted oil (O-DC) is found to be 0.30% and the water content in DC is 70.1%. The morphology of DC is depicted in Fig. 2c; the micrograph of DC exhibited bulk material with irregular particle shapes and small particles in the surface of the main material. EDX results of DC indicate the following composition; mostly carbon (60.30%) followed by oxygen (32.63%), and minor mixture metallic elements such as K (1.78%), Si (1.57%), Al (1.22%), Ca (1.05%), P (0.56%), Mg (0.45%) and S (0.44%). The average particle size of dried DC is 495 nm, and ranged from 43 to 2720 nm. Elemental composition of extracted O-DC and DC (before and after extraction) is summarized in Table 1.

The FTIR spectra of DC-before, DC-after and O-DC, ME-O-DC are shown in Fig. 3. As for spectra of DC before and after transesterification the characteristic absorption peaks at 546–776 cm^{-1} are attributed to the deformation vibration of M^+O^- and $\text{M}^+\text{O}-\text{M}^+$. The M^+OH stretching is shown at 1033 cm^{-1} and the adsorption peak around 1100–1641 cm^{-1} assigned to the presence of water vibration. The peak at 1753 cm^{-1} is characteristic band for oil ($\text{C}=\text{O}$) and band at 2855 and 2924 cm^{-1} is deformation characteristic of $-\text{CH}_2$. The typical broadband at about 3460 cm^{-1} is attributed to the O–H bending and stretching of the associated water molecules. The IR spectrum of the de-oiled DC (DC-after) shows the absence of vibrations corresponding to oil at 1753 cm^{-1} , 2855 and 2924 cm^{-1} ; indicating the oil extraction efficiency of the method.

In the transesterification of O-DC, the FTIR spectra of O-DC and ME-O-DC are very similar because of high chemical similarities that exist among O-DC and ME-O-DC. However, small differences in peak shape and peak split are observed in this three regions

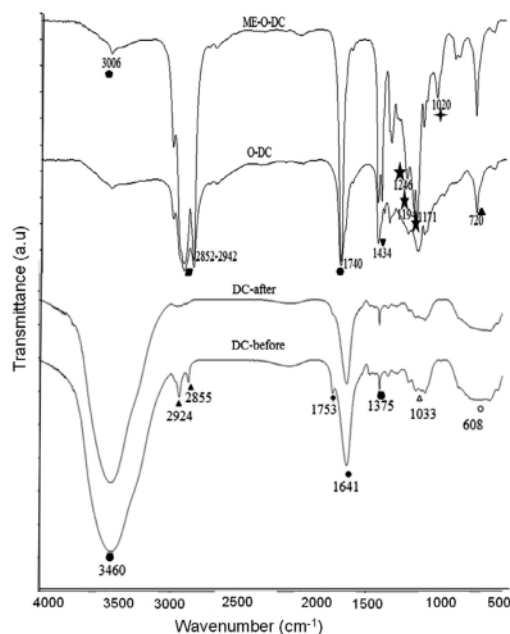


Fig. 3. FT-IR spectra of ME-O-DC, O-DC: \blacktriangle CH_2 ; \bullet $\text{C}=\text{O}$; \star $\text{C}-\text{O}$; \blacktriangledown CH_3 ; \blacksquare CH ; \bullet $=\text{CH}$; \blacklozenge CH deformation for alkyl group; DC-before and DC-after transesterification: \circ : M^+O^- and $\text{M}^+\text{O}-\text{M}^+$, \triangle : $\text{M}^+\text{O}-\text{H}$, \bullet : $-\text{OH}$, \blacklozenge : $\text{C}=\text{O}$, \blacktriangle deformation $-\text{CH}_2$.

($\text{C}=\text{O}$ ester, CH_3 and $\text{C}-\text{O}$ ester) because methyl ester (ME-O-DC) has a different compound bonded compared to oil (O-DC), such as the strong ester peaks at 1740 cm^{-1} ($\text{C}=\text{O}$ stretching band of ester) and at 1171, 1195, and 1246 cm^{-1} ($\text{C}-\text{O}$ stretching band of ester), furthermore, peaks at 1020 cm^{-1} in ME-O-DC is C–H deformation for alkyl group, are clearly present in the spectra. Outside these three regions, another characteristic peak that indicates the presence of CH_3 group in the mixtures of methyl ester can be observed at 1434 cm^{-1} . Strong and sharp signals between 2852 and 2924 cm^{-1} are due to C–H stretching. The absorbance at 3006 cm^{-1} indicates the $=\text{C}-\text{H}$ stretching and absorption peak at 720 cm^{-1} suggested the CH_2 rocking.

3.3. Effect of catalyst amount

The amount of catalyst (BA) was varied in the range of 0.5–3.5 wt.% (based on DC weight). As shown in Fig. 4a, transesterification was dependent on the amount of catalyst used. Increasing the catalyst from 0.3 to 3.5 wt.% increases methyl ester from 18.5 to 85.9 wt.%. The ME content reaches the highest value at the catalyst amount of 2.3 wt.%, due to the contact opportunity between catalyst and the reactants hence propels the reaction kinetics. Based on the oil weight, the amount of catalyst used in this work seems to be higher due to several reasons; part of the catalyst could be entrapped in the clay matrix; this portion of the catalyst may not have contributed to catalytic activity. In addition, a part of the catalyst (basic nature) is also used to neutralize the acids in CPO (acid value = 13.8 mg KOH g^{-1}). The ultrasound used in this work can affect the catalyst reactivity, positively, by enhancing the mass transfer between clay-catalyst-reactants as well as promising the presence of kinetic energy in the reaction media. Dispersion due to ultrasound increases the surface area available to the reactants. As such, the use of ultrasound promotes the efficiency of acyl conversion in a shorter reaction time.

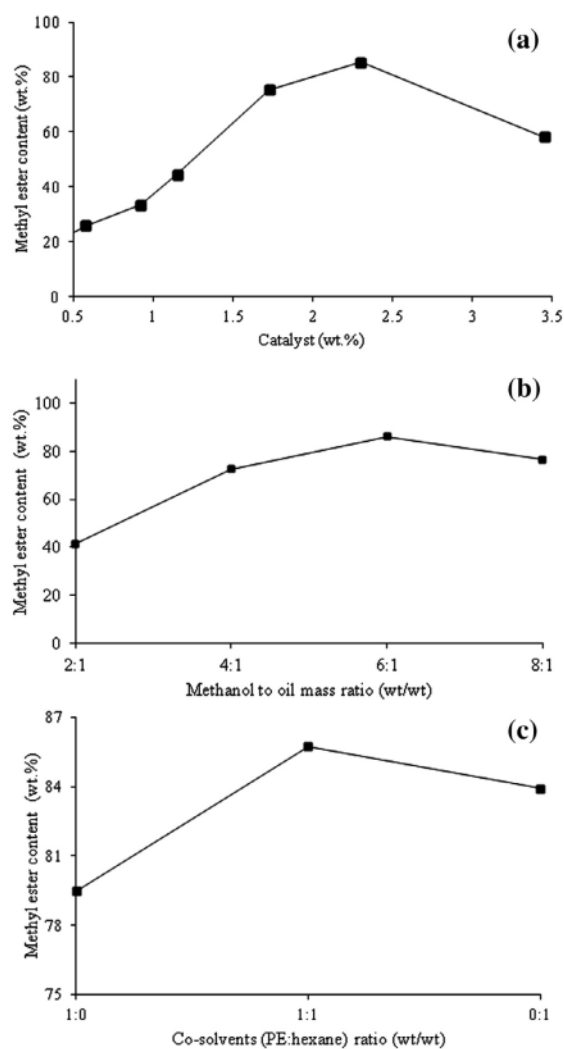


Fig. 4. Effect of (a) catalyst amount; (b) methanol to oil mass ratio; (c) co-solvents ratio on the methyl ester content (reaction conditions: temperature 55 °C; reaction time, 1 h).

3.4. Effect of methanol to oil mass ratio

The molar ratio of methanol to oil is one of the important variables which affects transesterification reaction. Stoichiometrically, the reaction requires three moles of methanol for each mole of oil (MeOH:oil = 3:1), and with excess methanol it would shift the equilibrium towards the direction of ME production. As observed in Fig. 4b, four different mass ratios of MeOH:oil were tested; 2:1, 4:1, 6:1 and 8:1. The ME content is increased as the MeOH:oil was raised from 2:1 to 6:1. A very sharp increase is observed between 2:1 and 4:1, which reached maximum performance at 6:1. However further increases to 8:1 has negative effects on ME conversion. Too much methanol could dilute the oil and as a result slows the reaction rate [16], which in turn, lowers conversions. In addition, a higher mass ratio of alcohol to oil increases the solubility of glycerol, and as a consequence, the separation of glycerol becomes more difficult and retards the forward reaction by promoting the backward equilibrium [17]. Relatively larger amount

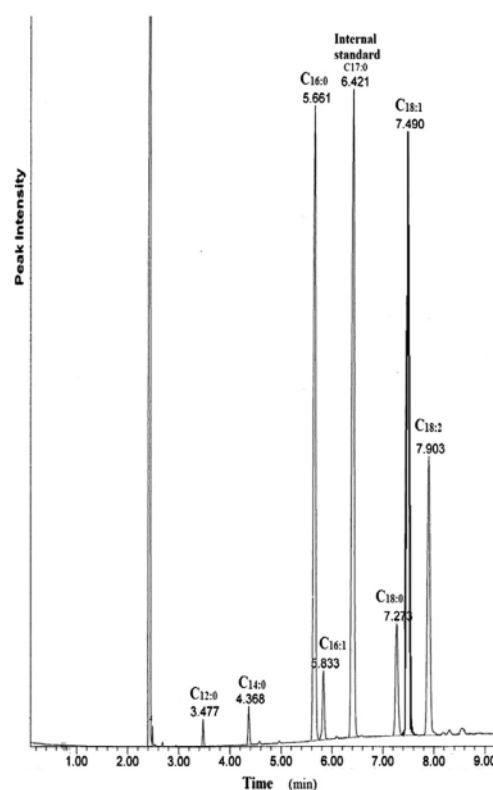


Fig. 5. Gas chromatogram of ME-O-DC.

of methanol was used in this work mainly to cater for the solvent-absorbing and solvent-retaining characteristics of the DC during reaction. Nevertheless, the excess of methanol can be readily recovered at the end of the reaction.

Ultrasound radiation causes methanol to disperse into the oil, thus increasing the contact surface between reactants, consequently accelerating the reaction. The effect of cavitations created by ultrasound supplies sufficient energy into the immiscible medium and the continuous formations and collapsing of micro bubbles accelerate the miscibility of reactants in addition to chemical and mechanical effects.

3.5. Effect of co-solvents

In this work, co-solvents (PE and hexane) were used to aid *in situ* oil extraction as well as oil solubility in the reaction media. Fig. 4c shows the influence of the co-solvents in the transesterification of the DC where ME content of approximately 80% and 84% were achieved by using PE or hexane alone, respectively. Higher conversion value for hexane is understandable as hexane (dielectric constant, 1.89) is less polar than PE (dielectric constant, 2.0–2.2), thus hexane is easily soluble in oil. On the other hand, the higher polar PE has better methanol solubility characteristic. This implies that by combining these two solvents, a better solubility between oil and methanol can be achieved as evident of highest ME content (86%) was achieved in the combination ratio of 1:1 PE:hexane. The reduction of energy consumption is one of the advantages of the ultrasound technique in methyl ester production. A study on the energy requirement for transesterification using ultrasound and hotplate (mechanical stirring) methods

revealed that transesterification using hotplate requires much more energy than ultrasonic process [18]. This shows that with appropriate reactor design, non-conventional techniques have potential to reduce the process energy requirement significantly.

3.6. Analysis of methyl esters

GC chromatogram proved that ME-O-DC mainly comprises of methyl esters of laurate ($C_{12:0}$) 1.5%, myristate ($C_{14:0}$) 2.2%, palmitate ($C_{16:0}$) 35.9%, palmitoleate ($C_{16:1}$) 3.9%, stearate ($C_{18:0}$) 6.4%, oleate ($C_{18:1}$) 34.3% and linoleate ($C_{18:2}$) 15.8%. Fig. 5 presents chromatogram of ME-O-DC and the internal standard (methyl heptadecanoate). The palmitic acid is the major fatty acid followed by oleic acid and linoleic acid. Stearic, palmitoleic, myristic, and lauric acids are present as minor constituents.

4. Conclusions

In this work, decanter cake (DC) was successfully utilized as a low-cost feedstock to produce methyl ester (biodiesel) via ultrasound aided *in situ* transesterification. Highest ME content of 85.9 wt.% was obtainable in an hour reaction time at 55 °C. Optimization of reaction parameters revealed that MeOH:oil, 6:1; catalyst, 2.3 wt.% (based on DC weight) and co-solvents to DC mass ratio of 1:2 as the optimal reaction conditions. The use of ultrasound undoubtedly assisted in achieving this remarkable result in 1 h reaction time. Materials derived from waste sources, as used in this work, should be given a priority for sustainable production of biodiesel.

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