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Extraction and Characterisation of *Musa balbiasana* cv. Saba Peel Oil

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In this study, *Musa balbiasana* cv. Saba peel oil and cooking oil were characterised for physicochemical characteristics and compared for potential biodiesel feedstocks. Chemical characterisation showed that *Musa balbiasana* cv. Saba peel oil was slightly more acidic compared to cooking oil, which were pH 5.23 and 6.06, respectively. The acid value of *Musa balbiasana* cv. Saba peel oil was lower than cooking oil, which were 0.64 mg/KOH and 0.74 mg/KOH, respectively. Free fatty acid analysis indicated that cooking oil has a higher value (0.37%) than *Musa balbiasana* cv. Saba peel oil (0.32%). In addition, the saponification value for *Musa balbiasana* cv. Saba peel oil was slightly lower compared to cooking oil, which were 74.09 (mg KOH/g oil) and 74.60 (mg KOH/g oil), respectively. The functional groups of both feedstocks were analysed using FT-IR to determine the -C=O stretching of methyl ester, -C-H stretch, -CH_2 bend, and -CH_3 bend asymmetric. The feedstocks were further analysed using GC-MS to detect the presence of the ester group. It was found that *Musa balbiasana* cv. Saba peel oil contained a higher ester group compared to cooking oil.

Key words: Extraction; *Musa balbiasana* cv. Saba; physicochemical

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The banana, or scientifically known as *Musa sapientum*, originates from the tropical region of Southern Asia. It is a herbaceous plant of the family *Musaceae*. It is one of the most important crops cultivated around the tropical region, including Malaysia and throughout the world. Bananas are highly nutritious and have been reported to prevent anaemia by stimulating the production of haemoglobin in red blood cell production [1]. The peel has been traditionally used to treat various ailments, such as burn, anaemia, diarrhoea, ulcers, inflammation, diabetes, cough, snakebite, and excess menstruation. In addition, bananas contain high levels of dietary fibre and phenolic compounds that are beneficial to our health.

Banana peel consists of higher phenolic compounds when compared to other fruit peels [2]. Phenolic compounds are secondary metabolites that may prevent cardiovascular diseases, cancer, diabetes, and obesity [3]. The recovery of these phenolic compounds and other major components such as lipid, proteins and carbohydrates from banana peel may add to the value of bananas. Furthermore, banana peel is used as a fertiliser by a simple decomposing process combined with cow dung or earthworms under aerobic or aerobic digestion. The resulting fertiliser contains high potassium and nitrogen levels, which is effective for application on various plants [4].

According to Emaga (2007) [5], banana peel contains high amounts of potassium, at approximately 55.23-63.52 mg/kg weight. In terms of nutrient content, it consists of protein (0.9%), lipid (1.7%), carbohydrate (59%), and crude fibre (31.90%). It was also reported that banana peel contains low levels of oxalate and phytate, which are also present in other feedstocks such as maize and sorghum [1].

In this work, *Musa balbiasana* cv. Saba peel was extracted by liquid-liquid extraction using hexane as a solvent. Palm oil and banana peel oil were characterised for physical and chemical properties as the feedstocks of biodiesel. Then, the feedstocks were analysed for functional groups, molecular weight, and molecular structures of fatty acids. To achieve this, Fourier Transform Infrared Spectroscopy (FT-IR) was used to identify the presence of functional groups, while Gas Chromatography Mass Spectroscopy (GC-MS) was used to measure the molecular weight of fatty acids in *Musa balbiasana* cv. Saba peel oil and palm cooking oil.

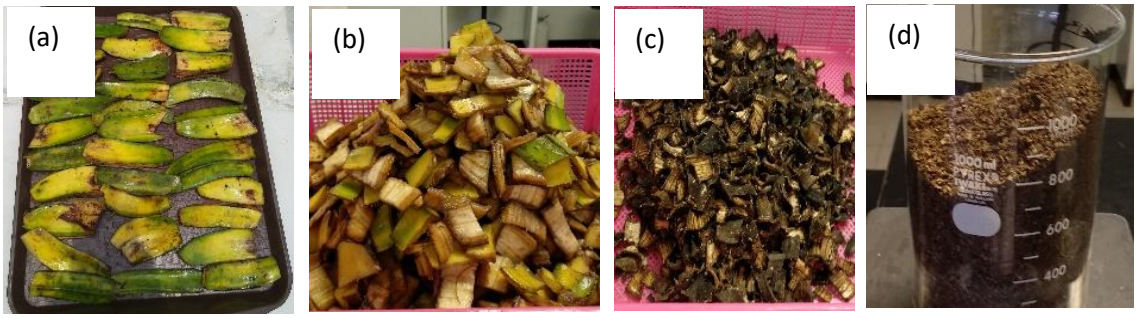

Figure 1. Sample preparation stages. (a) *Musa balbiasana* cv. Saba peels washed and stems removed. (b) Peels cut into smaller pieces to about 1cm x 1cm in size. (c) Peels dried in the oven for 24 h at a temperature of 40 - 50°C. (d) Dried peels ground for about 5-10 min to obtain a fine powder.

METHODOLOGY

1. Preparation of Samples

1.1. *Musa balbiasana* cv. Saba Peel

The sample of *Musa balbiasana* cv. Saba, also locally known as Pisang Saba, peels was collected at the residential college of Tun Fuad, Universiti Malaysia Sabah. The sample collected was in the ripe stage and yellow in colour. The sample collection was divided into two batches, with the same total weight of 10 kg each. The sample preparation stages are shown in **Figure 1.**

1.2. Palm Cooking Oil

Palm cooking oil (500 ml) was purchased from One Borneo Bataras Supermarket, located in Kota Kinabalu, at a low price of RM 5.30. The use of palm cooking oil as the standard sample for comparison is essential to characterise *Musa balbiasana* cv. Saba peel oil. Palm cooking oil can be used for up to 3 cycles due to its distinctive features in natural antioxidants. It is made from a high-grade pure palm olein which produces palm oils containing high contents of vitamin E without cholesterol.

1.3. Liquid-liquid Extraction of Musa balbiasana cv. Saba Peel

Generally, liquid-liquid extraction is also known as solvent or partitioning extraction. It involves the transfer of a solute from one solvent to another, the two solvents being immiscible or partially miscible with each other. The peel powder (200 g) was extracted in a 2 L beaker containing 1200 mL of hexane. The mixture was left shaken on an orbital shaker for 24 h. Then, the resulting oil and solvent mixture was filtered using a vacuum filtration to remove residual solids. The brownish solution was transferred into a round bottom flask and fitted to a rotary evaporator for 15 min at a temperature of 50°C until all hexane evaporated.

2. Characterisation of Feedstocks

Musa balbiasana cv. Saba peel oil and palm cooking oil were characterised for their physical and chemical characteristics. In physical characterisation, the oils were determined for pH, colour, and odour. Chemical characterisation involved the determination of acid value, free fatty acid, saponification value, FT-IR, and GC-MS.

2.1. Determination Colour and Odour

The colour of the oil samples was observed by visual comparison. The odour of the oil samples was determined by smell near the mouth of the centrifuge bottles.

2.2. Determination of pH

The pH of the oil samples was determined by using a pH meter. Each oil sample (30 mL) was measured in a beaker. The pH meter electrode was immersed in the oil samples, and the pH values were recorded.

2.3. Determination of Acid Value

2.0 g of oil sample was placed in a 250 mL conical flask. The sample was dissolved in 30 mL of neutral ethyl alcohol and the mixture was shaken thoroughly and allowed to boil in a water bath (78 - 80°C) until the substance completely dissolved. Two drops of phenolphthalein indicator were added into the solution. The solution was titrated against 1.0 M potassium hydroxide until a pink colour was obtained. The acid value was calculated by using the following equation:

$$Acid\ value\ (mg\ KOH/g) = \frac{(56.1 \times v \times N)}{w} \quad (2.1)$$

V *Titre value*
N *Normality of potassium hydroxide, KOH*
W *Weight of oil*
56.1 *Molecular weight of potassium hydroxide, KOH*

2.4. Determination of Free Fatty Acids

The value of free fatty acid of each sample was determined using the following equation:

$$Free\ fatty\ acid\ (\%) = \frac{(Acid\ value)}{2} \quad (2.2)$$

2.5. Determination of Saponification Value

1.5 g - 2.0 g of sample was poured into a 200 mL conical flask. Then, 25.0 mL of 0.5 M potassium hydroxide ethanol was added to the sample. The flask was heated (40-50°C) on a hotplate for 30 min with frequent shaking. It was cooled immediately after the heating process. 1 mL of 1% phenolphthalein indicator was added. Then, the sample was titrated with 0.5 M hydrochloric acid until it reached the endpoint, where it turned colourless. It was tested before the test liquid solidified. The same procedure was repeated three times by using a blank titration to obtain the mean value of titration volume of 0.5 M hydrochloric acid. Saponification value was calculated by using the following equation:

$$Saponification\ value\ (mg\ KOH/g\ oil) = \frac{[(s - B) \times M \times 56.1]}{w} \quad (2.3)$$

B *Titre value of blank*
S *Titre value of sample*
M *Molarity of hydrochloric acid*
W *Weight of oil*
56.1 *Molarity weight of potassium hydroxide*

2.6. Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

FT-IR was used to identify the functional groups that were present in a solution or a sample by interpreting their spectra. An FT-IR spectrometer was used to measure the spectra of carbonyl group (C=O), carboxyl group (COOH), and hydroxyl group (-OH) to confirm the presence of fatty acids in the samples. It was used to measure how well a sample absorbs light at each wavelength. The model of the FT-IR spectrometer was Perkin Elmer Spectrum 100, located at the Faculty of Science and Natural Resources, UMS. In this method, *Musa balbiasana* cv. Saba peel oil and palm cooking oil were used directly on the instrument.

2.7. Gas Chromatography Mass Spectrometry (GC-MS) Analysis

Chemical composition analysis of *Musa balbiasana* cv. Saba peel oil and palm cooking oil was determined by using GC-MS. 1 mL of feedstock was diluted into 50 mL until dissolved completely. Then, the solution was transferred into a 2 mL glass vial by using a 0.2 µm non-sterile membrane filter. The colour of the solution was transparent. The feedstock sample was analysed using GC-MS G1530N model with Agilent 6890 N network gas chromatography system, located at the Faculty of Engineering, UMS. The oven temperature was optimised for the separation of ester groups from 70℃ (1 min) to 220℃ (5 min) to 320 (10 min). The injection mode was split with a pressure of 120 kpa (75.0-150 kpa) and a split ratio of 2 mL/min. The ion source temperature and interface temperature for mass spectroscopy were 220℃ and 270℃, respectively.

RESULTS AND DISCUSSIONS

1. Determination of Musa balbiasana cv. Saba peel oil

Musa balbiasana cv. Saba peel was prepared as dried powder and extracted by liquid-liquid extraction using hexane as the solvent. During the preparation of the sample and the extraction, the weight and percentage weight of the sample were constantly reduced due to washing, cutting, heating, and grinding processes. Based on the proximate analysis, the highest percentage weight reduction was observed during the oven drying stage, in which the peel dried in the oven dropped from 2.98 kg to 0.42 kg after 24 h of drying (85.91% weight reduction). The significant weight reduction during the drying stage also reduced the moisture content, which affected the extraction yield of *Musa balbiasana* cv. Saba peel oil [6]. The volume of *Musa balbiasana* cv. Saba peel oil extracted from 200 g of dried *Musa balbiasana* cv. Saba peel was 18 mL.

2. Physicochemical Characterisation

2.1. Determination of Colour and Odour

The colours of *Musa balbiasana* cv. Saba peel oil feedstock and palm cooking oil feedstock are shown in **Figure 2**.

(a) (b)

Figure 2. Colours of (a) *Musa balbiasana* cv. Saba peel oil and (b) palm cooking oil.

Table 1: Properties of *Musa balbiasana* cv. Saba peel oil and palm cooking oil.

Sample	Colour	Odour	pH	Acid Value	Free Fatty Acid	Saponification Value
Musa balbiasana cv. Saba peel oil	Dark brown	Fruity fragrance	5.27 (0.04)	0.65 (0.16)	0.32 (0.08)	74.09 (4.80)
Palm cooking oil	Yellow	Odourless	6.06 (0.03)	0.74 (0.32)	0.37 (0.16)	74.60 (5.32)

Based on the results in **Table 1**, the colour of *Musa balbiasana* cv. Saba peel oil after liquid-liquid extraction was dark brown, while palm cooking oil was yellow. According to Bello *et al.* (2011), the colour of biodiesel may vary from ASTM standard of water clear to black, depending on the feedstocks [7]. The colour of biodiesel is also affected by the contamination of materials, method, and amount of purification performed. Oxidation causes the colour of biodiesel to change from yellow to dark brown, and results in a paint-like odour. However, the colour of biodiesel is not directly correlated with the quality of biodiesel [8-11]. The odour of *Musa balbiasana* cv. Saba peel oil was similar to a fruity fragrance. In contrast, palm cooking oil was odourless due to the presence of pure olein, which is known to be free from foreign and rancid odour.

2.2. Determination of pH

The pH values of *Musa balbiasana* cv. Saba peel oil feedstock and palm cooking oil feedstock were determined by using a pH meter. Based on **Table 1**, both feedstocks showed weak acidic properties due to low pH values. However, the pH of the banana peel oil was slightly higher compared to palm cooking oil. The low acidity value in both oils indicates that these feedstocks are suitable to be used as cooking oils.

2.3. Determination of Acid Value

The acid values for both feedstocks are shown in **Table 1.** The acid value of *Musa balbiasana* cv. Saba peel oil was lower (0.65 mg/KOH) than palm cooking oil (0.74 mg/KOH). According to ASTM standard D6751, the range of acid values for biodiesel is ≤ 0.8 mg/KOH [12-14]. Acid value determines the quality of free fatty acids in biodiesel. Biodiesel with a higher acid value requires a higher maintenance fuelling system due to corrosion. Acid value is also related to the purity of biodiesel. The results showed that the acid values of *Musa balbiasana* cv. Saba peel oil and palm cooking oil were relatively high. This may be due to a lack of purification treatment performed after the extraction process.

2.4. Determination of Free Fatty Acids

The results of free fatty acids for both feedstocks are shown in **Table 1.** The free fatty acid value (FFA) of *Musa balbiasana* cv. Saba peel oil (0.32%) was half the value of acid number. Palm cooking oil recorded a higher percentage of FFA (0.37%) compared to *Musa balbiasana* cv. Saba peel oil. High percentages of FFA (>1%) may affect biodiesel yield due to the formation of soap. Soap causes the formation of stable emulsions that prevent the separation of biodiesel from glycerine during processing. Therefore, both feedstocks are suitable as feedstocks for biofuel due to low acid values and low free fatty acid percentages.

2.5. Determination of Saponification Value

The saponification values for *Musa balbiasana* cv. Saba peel oil and palm cooking oil are shown in **Table 1.** Based on the results, the average saponification value for *Musa balbiasana* cv. Saba peel oil was 74.09 (mg KOH/g oil), while palm cooking oil was 74.60 (mg KOH/g oil). Saponification value is defined as the average molecular weight of oil and expressed in

milligrams of potassium hydroxide (mg KOH/g oil). It also indicates the presence of the acyl groups per unit of weight oil [11]. A higher saponification value leads to a higher percentage of FFA in the feedstock that will form soap during the separation of biodiesel [15].

Palm cooking oil has a higher saponification value than *Musa balbiasana* cv. Saba peel oil due to the high amount of triacylglycerol present. This requires more potassium hydroxide to react, which increases its saponification value. Both feedstocks showed low saponification values, which denote the presence of longer chains of unsaturated fatty acids, such as oleic acid, linoleic acid, and linolenic acid [13]. There is no specific saponification value from ASTM D6751 for reference.

2.6. Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

The functional groups and wavelengths of *Musa balbiasana* cv. Saba peel oil and palm cooking oil were analysed using FT-IR. The wavelength summary is recorded in **Table 2.** The FT-IR results were used to identify the presence of functional groups based on vibration and energy produced by various molecular bonding of the molecules.

Figure 3 shows the FT-IR spectra of *Musa balbiasana* cv. Saba peel oil and palm cooking oil. The primarily functional groups of the biodiesel feedstocks are carbonyl groups, based on the spectra. In addition, both feedstocks indicated the presence of -C=O triglycerides stretching for *Musa balbiasana* cv. Saba peel oil at 1737.21 cm^{-1} and palm cooking oil at 1744.13 cm^{-1}.

The transmittance bands between 2929 cm^{-1} and 2856 cm^{-1} indicated the presence of aliphatic hydrogen and a long carbon chain compound [13]. Both feedstocks showed -C-H stretching; *Musa balbiasana* cv. Saba peel oil between 2924.10 cm^{-1} to 2855.61 and palm cooking oil between 2921.30 cm^{-1} to 2852.95 cm^{-1}.

Table 2. Summary of FT-IR spectra of *Musa balbiasana* cv. Saba peel oil and palm cooking oil.

Functional groups	Wavelengths (cm^{-1})	
	***Musa balbiasana* cv. Saba peel oil**	**Palm cooking oil**
-C=O stretching of carbonyl group	1737.21	1744.13
-C-H stretch	2924.10 and 2855.61	2921.30 and 2852.95
$-CH_2$ bend	1453.21	1457.41
$-CH_3$ bend symmetric	1376.21	1374.02

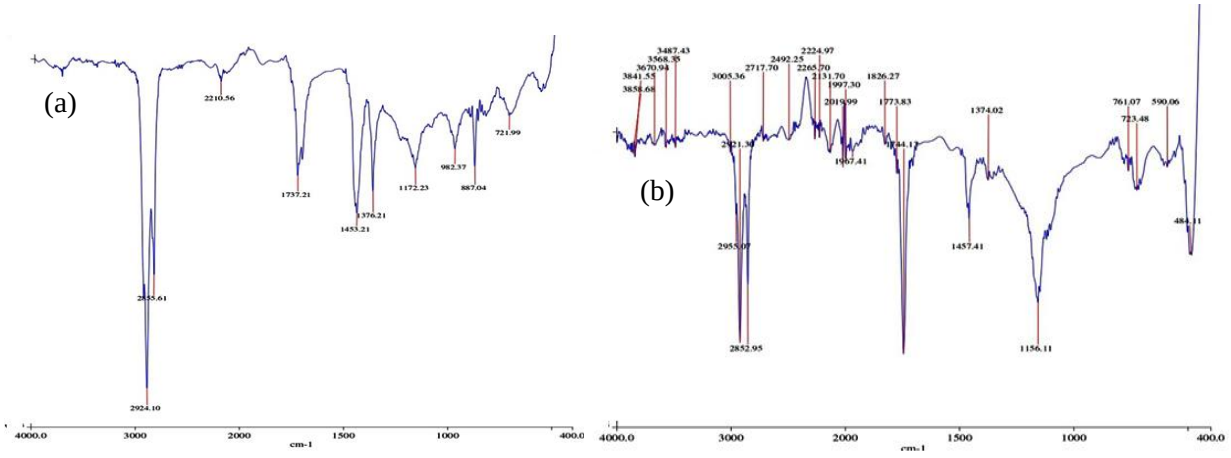

Figure 3. FT-IR spectrua of (a) *Musa balbiasana* cv. Saba peel oil; (b) palm cooking oil.

Both feedstocks showed -CH_2 bend, at 1453.21 cm^{-1} for *Musa balbiasana* cv. Saba peel oil and 1457.41 cm^{-1} for palm cooking oil. In addition, absorbances at 1376.21 cm^{-1} for *Musa balbiasana* cv. Saba peel oil and 1374.02 cm^{-1} for palm cooking oil denoted the presence of -CH_3 symmetric bend in the molecules of the feedstocks.

2.7. Gas Chromatography Mass Spectrometry (GC-MS) Analysis

The compounds present in both feedstocks are shown in **Table 3.** Four compounds were detected in palm cooking oil, which were l-(+)-Ascorbic acid 2,6-dihexadecanoate, Nonacosanal, Lanosta-8,24-dien-3-one, and 9,19-Cyclolanost-23-ene-3,25-diol,(3.beta.,23E)-.

Figure 4 shows GC-MS chromatograms of *Musa balbiasana* cv. Saba peel oil and palm cooking oil. The results indicated no presence of fatty acid methyl ester in palm cooking oil.

The GC-MS chromatogram of *Musa balbiasana* cv. Saba peel oil showed twelve major peaks, with four peaks confirmed as ester group. These included Glycidyl palmitate, 9-Octadecenoic acid (Z)-oxiranylmethyl ester, Hexadecanoic acid, 2-hydroxy-1-(hydroxy methyl)ethyl ester, and Dodecanoic acid, 1,2,3-propanetriyl ester.

Table 3. Compounds present in *Musa balbiasana* cv. Saba peel oil and palm cooking oil feedstocks.

Types of Feedstocks	Compounds	Molecular formulae	Retention time (min)	Ester group
Palm cooking oil	l-(+)-Ascorbic acid 2,6-dihexadecanoate	$C_{38}H_{68}0_8$	9.35	-
	Nonacosanal	$C_{29}H_{58}O$	16.69	-
	Lanosta-8,24-dien-3-one	$C_{30}H_{48}0$	19.76	-
	9,19-Cyclolanost-23-ene-3,25-diol,(3.beta.,23E)-	$C_{30}H_{50}O_2$	20.42	-
Musa balbiasana cv. Saba peel oil	2-Ethyl-oxetane	$C_5H_{10}O$	1.19	-
	2,4,-Decadienal,(E,Z)	$C_{10}H_{16}O$	5.25	-
	Glycidyl palmitate	$C_{19}H_{36}O_3$	13.51	/
	9-Octadecenoic acid (Z)-, oxiranylmethyl ester	$C_{21}H_{38}O_3$	14.87	/
	Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl)ethyl ester	$C_{19}H_{38}O_4$	15.10	/
	1-cis-Vaccenoylglycerol	$C_{21}H_{40}O_4$	16.05	-
	Supraene	$C_{30}H_{50}$	16.59	-
	beta.-Tocopherol, O-methyl-	$C_{28}H_{48}O_2$	18.26	-
	(R)-2,7,8-Trimethyl-2-((3E,7E)-4,8,12-trimethyltrideca-3,7,11-trien-1-yl)chroman-6-ol	$C_{28}H_{42}O_2$	18.50	-
	2H-1-Benzopyran-6-ol, 3,4-dihydro-2,5,7,8-tetramethyl-2-(4,8,12-trimethyl-3,7,11-tridecatryl)-	$C_{29}H_{44}O_2$	19.73	-
	gamma.-Sitosterol	$C_{29}H_{52}O_2$	19.03	-
	Dodecanoic acid, 1,2,3-propanetriyl ester	$C_{39}H_{74}O_6$	23.13	/

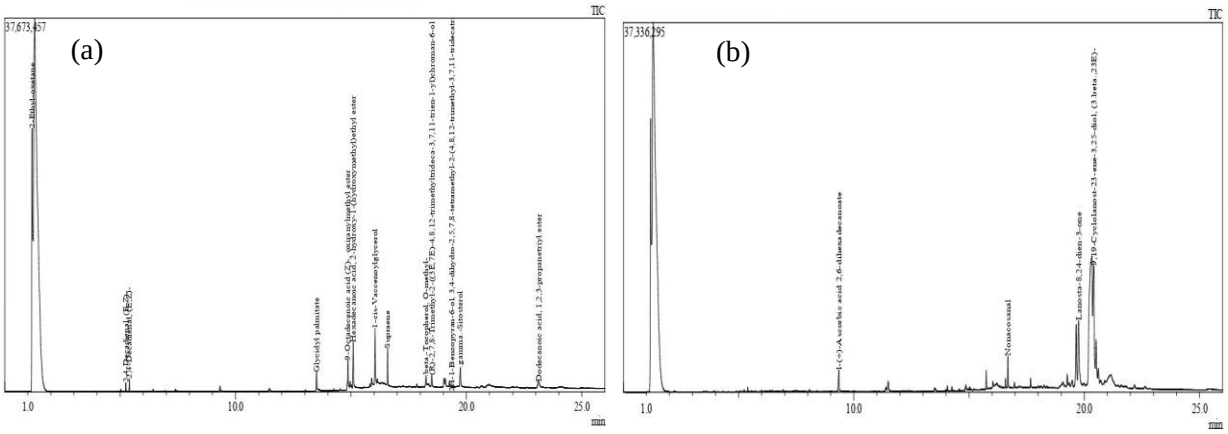

Figure 4. GC-MS chromatograms of (a) *Musa balbiasana* cv. Saba peel oil; (b) palm cooking oil.

CONCLUSION

This study demonstrates that *Musa balbiasana* cv. Saba peel oil and palm cooking oil possess the potential as sources of biodiesel feedstocks. The yield from the extraction of 200 g of powdered *Musa balbiasana* cv. Saba peel was 18 mL. The extracted oil of *Musa balbiasana* cv. Saba peel is more suited as a biodiesel feedstock than palm cooking oil as it has lower acid value, percentage of FFA, and saponification value. A low percentage of FFA in oils offers a higher yield of biodiesel due to the lower production of soap. Whereas, low saponification is preferable as it indicates low triacyl glycerides and FFA presence in oils. In the physical characterisation study, *Musa balbiasana* cv. Saba peel oil was found to be dark brown in colour, releasing a fruity fragrance. In comparison, palm cooking oil was yellow in colour, without any significant odour. The GC-MS analysis showed a higher presence of the ester group in *Musa balbiasana* cv. Saba peel oil compared to palm cooking oil.

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