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A COMPARATIVE STUDY OF BIODIESEL PRODUCTION FROM SCREW PINE FRUIT SEED: USING ULTRASOUND AND MICROWAVE ASSISTANCE IN IN-SITU TRANSESTERIFICATION

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Abstract

The main purpose of this research was the production of biodiesel from screw pine fruit seed (SPFS) oil as a new biodiesel feed-stock using in-situ transesterification with the assistance of microwave and ultrasound. The effect of independent variable such as the SPFS oil to methanol ratio, reaction time, catalyst concentration and temperature were investigated and compared the results of both methods. The physicochemical parameters of the biodiesel, such as kinematic viscosity, density and acid value were analysed using the EN 14214-2003 standard. The fatty acid methyl ester composition was identified using GC–MS. The experimental results with 25:1 w/w ratio of methanol to the SPFS oil, 1:1 w/w ratio of methanol to chloroform, 5% KOH, 50 rpm stirring and 65 °C reaction temperature showed that the maximum conversion efficiency was approximately 88.12% for the assistance of microwave with 4-min reaction time and 450W microwave power, and it was approximately 86.38% for the assistance of ultrasound with 80-min reaction time, 37 kHz ultrasound. The physicochemical properties met to the EN 14214-2003 standard, except for the acid value. Meanwhile, the GC–MS analysis identified five major fatty acid esters in the biodiesel.

Keywords: Biodiesel, Fatty acid, Screw pine fruit seed, Microwave, Ultrasound.

1. Introduction

The consumption of fossil fuel based oils has been dramatically increased from the past 30 years to answer the growth of world population and industries. The use of fossil fuel based oils, which is mostly for electricity generation and heating in industrial processes [1] has threatened continuously the global energy

sustainability. Almost all the contries in world have used their natural resource especially the sustainable and renewable ones to substitute diesel and to produce cleaner energy [2] including hydrogen power cells [3-6], solar power [7], and wind energy [8]. However, the technologies to produce cleaner energy are still costly to apply. Therefore, non-toxic biomass-based biodisel is promoting renewable energy because the low cost of technology to produce. It can be used for petroleum-derived gasoline, distillate fuel, diesel engine without the engine modification [9] with lower exhaust emissions, and it can reduce the level of pollutants and carcinogens [4-6, 10].

The main challenge in biodiesel production is how to solve the lack of feed-stock availability in large scale of biodiesel production [11-13]. In fact, types of oil which can use to produce biodiesel are edible oils (plant-based oils like palm oil, coconut oil, canola oil, corn oil, olive oil, etc., and animal-based oils), non-edible oils (jatropha curcas seed oil, rubber seed oil, Neem oil, etc.), and microorganism (algae, microalgae, bacteria and fungi) [12-20]. However, the edible oils is more globally needed for food security. Therefore, non-edible oils would be the best oils source for biodiesel production.

Screw pine (*Pandanus tectorius*) is one of the promising materials of non-edible oils whereas the fruit seed has not been investigated for biodiesel production. It is native to the Asia region and the Oceania region [21]. The plants can be also arisen from branch cuttings and start flowering after only 2 years to produce mature fruit [22]. It has been used for environmental services like controlling coastal erosion, for fibre, building material, shelter, shade and ornamental [21]. The fruit can be consumed, and it can supply up to 50% of all energy needed for a person. [23-24]. The fruit is generally ovoid-shaped with a diameter of 4–20 cm and a length of 8–30 cm, and it is made up of 38–200 wedge-like phalanges, which have an outer fibrous husk. Each phalange contains a minimum of two seeds and a maximum of eight seeds [21].

Several biodiesel production methods have been developed, and among others, in-situ transesterification in the presence of alkali catalyst gives a high level of conversion of triglycerides to their corresponding methyl esters. The process was affected by the reaction time, mole ratio of alcohol to oil, type of alcohol, type and amount of catalysts, reaction temperature and pressure, among other independent variables [25-27]. The application of ultrasonic stirring in biodiesel production improved transesterification yields, and reduced the reaction time [28-33]. Other method which was applied to increase the reaction rate and yields was microwave heating [34-37]. It can also reduce the production cost compared to the conventional heating [12, 13].

Based on the need of alternative non-edible oils whereas screw pine fruit seed (SPFS) has not been investigated for biodiesel production, and the advantages of ultrasound and microwave assistance on in-situ transesterification in biodiesel production including the effect of independent variables on the process, this paper aims to investigate the production of biodiesel from the SPFS using in-situ transesterification with the assistance of ultrasound and microwave. The effect of seed to methanol ratio, reaction time, catalyst concentration, temperature and microwave power were investigated, and physicochemical parameters of the biodiesel, such as kinematic viscosity, density and acid value were analysed and compared with the EN 14214-2003 standards.

2. Materials and Methods

The SPFS of Tonga type (entire fruits segements red-orange based on the literature [40]) were collected from Tsunami-affected coastal areas at Ujung Batee in Aceh Besar district, Aceh Province, Indonesia. The collected SPFS is shown in Fig. 1.

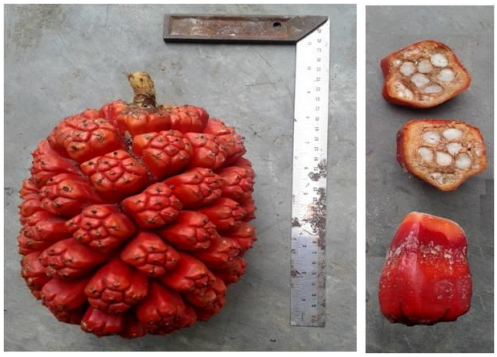

Fig. 1. Photograph of screw pine fruit seed (SPFS, Tonga type) collected.

The SPFS was cleaned and rinsed using tap water, and it was dried in an oven (Memmert, NN-ST342M, made in Germany) at 60 °C for about 48 h to remove the remaining moisture. The dried SPFS was crushed to particle with the average size of 2–3 mm in an electric blender (Sharp, SB-TI 172 G, made in China). The particle SPFS of 10 g was then used as the starting material. It was mixed with a mixture of solvent (methanol), co-solvent (chloroform) and catalyst (KOH) in a 500 ml Erlenmeyer flask (Pyrex).

Experiments of ultrasound-assisted in-situ transesterification (IST) were carried out in batch mode using ultrasonic reactor (Elmasonic, E300H, made in Germany). The IST system which was a mixture consiting of the particle SPFS, methanol, chloroform and KOH with the predetermined weight ratios, was stirred digitally using controlled mechanical stirrer (SciLOGEX OS20-S, made in USA) at 50 rpm in the Erlenmeyer flask. The mixture was sonicated at 37 kHz in the ultrasonic reactor for 20–90 min at the temperature of 55–70 °C. Following it, the mixture was filtered and placed in a rotary evaporator (Laborota 4003, made in Germany) for 2 h at 80 °C to separate the solvent and co-solvent. The mixture was allowed to stand for about 2 h while phase separation occurred by gravity settling with biodiesel on the top and glycerol at the bottom. The liquid glycerol phase was removed, and biodiesel was dried on a hot plate (IKA Hotplate Stirrer C-MAG HS 7) at 110 °C to remove the remaining water and impurities. The yield of biodiesel produced in this process was calculated using Eq. (1):

$$BY = \frac{BM}{mSPFS \times OC} \times 100\% \quad (1)$$

where BY (%) represents the biodiesel yield, BM (g) is the biodiesel mass, $mSPFS$ (g) denotes the SPFS mass, and OC (g/g) is the oil content from the in-situ transesterification.

The biodiesel products were analysed to determine the physicochemical properties, such as kinematic viscosity, density and acid value using the EN 14214-2003 standard. The same prosedure was taken into account to conduct the experiments of microwave-assisted in-situ transesterification, but the reaction time was set in the range of 2-6 min. The IST system was heated using a microwave (Samsung ME731K, 800W, 2450 MHz, made in China) at the power of 300-600W for 2–5 min. The experiments at an optimal condition resulting the highest biodiesel yield was repeated without the assistance of ultrasound and microwave to compare the results. The samples of biodiesel produced at the optimum conditions were used to identify the components and composition using gas chromatography–mass spectrometry (GC–MS, Shimadzu QP 2010 Plus, made in Japan). Each run of the experiments were conducted in multipled run, and the results were presented in an average value with standard deviation being 0.514, and each standard deviation is shown by the legend of figure presented in Result and Discussion part.

3. Results and Discussion

3.1. Effect of reaction time

Transesterification is one of the effective ways to produce biodiesel of methyl ester from the non-edible oils of triglycerides. Several method can be applied such as catalytic and non-catalytic transesterification depending on the properties of the crude [41]. KOH catalyst can be used for transesterification reaction to produce biodiesel from the SPFS using [17, 42]. The reaction time of transesterification is one of important independent variables which affect biodiesel conversion from the oil. Several methods have been applied in transesterification to reduce reaction time, energy and cost of biodiesel production [33, 43, 44]. Therefore, the effect of reaction time on in-situ transesterification of biodiesel production from the SPFS was investigated.

Experiments of ultrasound and microwave-assisted in-situ transesterification (IST) were carried out in batch mode with the experimental condition shown by the legend in Fig. 2. As can be seen in Fig. 2, reaction time had a significant effect on biodiesel yield up to 80 and 4-min reaction time for both the ultrasound and microwave assistance, respectively. The production rate of biodiesel by microwave assistance was obiously faster than the one by ultrasound assistance. For instance, it increased from 44.68 to 88.12% at 2 to 4 min, respectively reaching the highest conversion efficiency for microwave assistance, and it increased from 57.42 to 86.38% at 50 to 80 min, respectively.

The optimal reaction time of 4-min in the SPFS oil in-situ transesterification with microwave assistance was faster compared to the one using Chinese tallow tree seeds (20-min) [36] and Jatropha oil (6-min) [37]. It was longer than the ultrasound assistance-based in-situ transesterification using Salvia lerifolia seeds (9-min) [43], and it was the same as the one using Cynara cardunculus L. seed oil (20-min) [33].

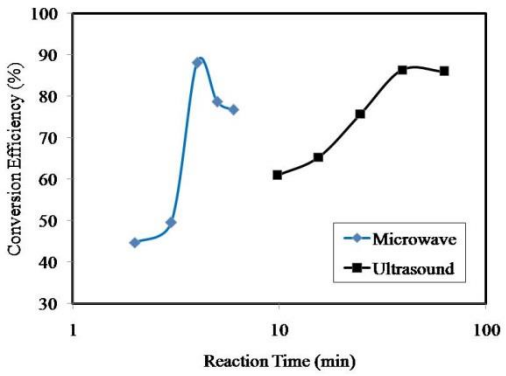

Fig. 2. Effects of reaction time on the biodiesel production from the SPFS using in-situ transesterification. Experimental conditions: 25:1 w/w ratio of methanol to the SPFS oil, 1:1 w/w ratio of methanol to chloroform, 5% KOH and 50 rpm stirring (1) Ultrasound assistance: 37 kHz ultrasound, 65 °C reaction temperature (2) Microwave assistance: 450W, hold at 65 °C. An average value of multipled run was taken, and the standard deviation was 0.635 on average.

3.2. Effect of methanol mass ratio

Based on the transesterification reaction [17, 42], methanol to the SPFS mass ratio on the biodiesel production should be higher than the stoichiometric molar (3:1) ratio in order to let the equilibrium reaction moving to the right hand side. In addition, larger ratio of methanol to the SPFS mass is also needed for the mass transfer and adsorption of triglycerides and methanol onto KOH catalyst. The effect of methanol to the SPFS mass ratio on the biodiesel production from the SPFS using in-situ transesterification are shown in Fig. 3.

As shown in Fig. 3, the highest biodiesel conversion efficiency was reached at 25:1 of methanol to the SPFS mass ratio, which was approximately 86.38 and 88.12% for the assistance of ultrasound and microwave, respectively. Decreasing the mass ratio to 20:1 and 15:1 decreased the biodiesel conversion efficiency to 76.81 and 65.32%, respectively for the microwave assistance, and decreased it to 79.36 and 64.68%, respectively for the ultrasound assistance. The same trend also occured when increasing the mass ratio from 20:1 to 30:1. This is reasonable because excess methanol improved the glycerols solubility in ester phase leading the lower product caused by more foam formed [44]. In addition, excess glycerin also drive the equilibrium back to the left hand side lowering the yield of biodiesel conversion when glycerin remain in solution, [45-46].

3.3. Effect of catalyst concentration

The amount of catalyst used in biodiesel production is an important independent variable among others. Catalyst concentration used to get optimal conversion efficiency by in-situ transesterification of the oil could be different for various condition and method. For examples, NaOH catalyst used was approximately 1% w/w oil for biodiesel production from Cynara cardunculus L. seed oil using with ultrasound assistance [33], and it was approximately 6% w/w oil for biodiesel

production from Jatropha oil [31]. The effect of catalyst concentration on the biodiesel production from the SPFS using in-situ transesterification is shown in Fig. 4.

As clearly shown in Fig. 4, the maximum conversion efficiency obtained at the KOH catalyst concentration being 5% w/w the SPFS oil was approximately 86.38 and 88.12% for the biodiesel production with the assistance of ultrasound and microwave, respectively. The catalyst concentration dramatically affect the biodiesel production with the assistance of microwave whereas it decreased drastically by 24.37% when the catalyst concentration decreased from 5 to 4%. It was caused by the less catalyst leading to less interaction between triglycerides and methanol. In contrast, more catalyst concentration applied caused more triglycerides supporting the saponification reaction to form more soap in the system. As the result, conversion efficiency drops by 26.32% when the catalyst concentration increases from 5 to 6%. The same trend but more moderate was shown by the one with the assistance of ultrasound.

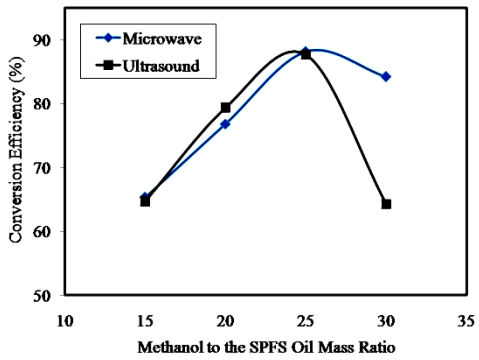

Fig. 3. Effect of methanol to the SPFS mass ratio on the biodiesel production from the SPFS using in-situ transesterification. Experimental conditions: 15:1 to 30:1 w/w ratio of methanol to the SPFS oil, 1:1 w/w ratio of methanol to chloroform, 5% KOH and 50 rpm stirring (1) Ultrasound assistance: 37 kHz ultrasound, 65 °C reaction temperature, 80-min reaction time (2) Microwave assistance: 450W, 4-min reaction time, hold at 65 °C. An average value of multipled run was taken, and the standard deviation was 0.380 on average.

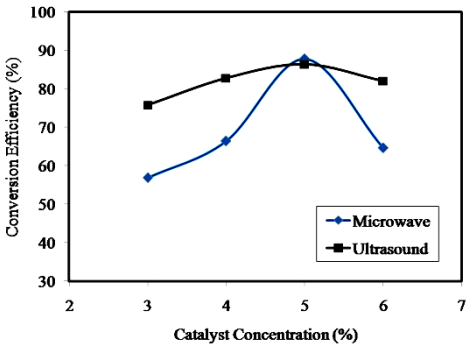

Fig. 4. Effect of catalyst concentration on the biodiesel production from the SPFS using in-situ transesterification. Experimental conditions: 25:1 w/w ratio of methanol to the SPFS oil, 1:1 w/w ratio of methanol to chloroform, 2-5% KOH and 50 rpm stirring (1) Ultrasound assistance: 37 kHz ultrasound, 65 °C reaction temperature, 80-min reaction time (2) Microwave assistance: 450W, 4-min reaction time, hold at 65 °C. An average value of multipled run was taken, and the standard deviation was 0.520 on average.

3.4. Effect of microwave power and reaction temperature

Microwave power really affects conversion efficiency in biodiesel production with the assistance of microwave. The highest conversion efficiency of 98% in biodiesel production from Palm oil was obtained at 70W with 1% HPAs, 1:85 ethanol and hold at 70 °C [47]. The maximum biodiesel yield of 96.2% from Yellow horn with 1% KOH, 1:12 methanol, was obtained at 500W and 60 °C [48]. The trend of conversion efficiency over microwave power was almost parabolic in the range of 100-500 W, and the maximum biodiesel yield from transesterification of Chlorella sp. obtained was 0.53% at 250W. A parabolic trend was also found for biodiesel production from the SPFS using in-situ transesterification, as shown in Fig. 5.

As clearly shown in Fig. 5, the maximum conversion efficiency in biodiesel production from the SPFS using in-situ transesterification for different reaction time and mass ratio of methanol to the SPFS oil was obtained at 450W. The conversion efficiency increased due to the increase in microwave power from 300 to 450W. It was caused by the increase in dipolar polarization of reactants leading to the decrease in activation energy [49]. The increase in dipolar polarization of reactants was indicated by the increase in reaction temperature from 60 to 65 °C for increase in microwave power from 300 to 450W, respectively. However, increasing microwave power to 600W resulted in decreasing conversion efficiency as can be seen in Fig.5. This is reasonable because the reaction temperature increased from 65 to 70 °C leading to methanol evaporation. As clearly shown in Fig. 5, the highest conversion efficiency is 88.12% with the optimal experimental condition being 4-min reaction time, 25:1 w/w ratio of methanol to the SPFS oil, 5% KOH, 1:1 w/w ratio of methanol to chloroform, 50 rpm stirring and 450 W microwave power.

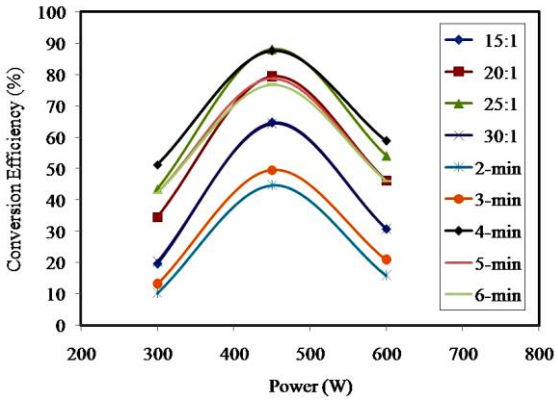

Fig. 5. Effect of microwave power on the biodiesel production from the SPFS using in-situ transesterification. Experimental conditions: 15:1 to 30:1 w/w ratio of methanol to the SPFS oil, 1:1 w/w ratio of methanol to chloroform, 5% KOH, 50 rpm stirring, 300-600W, 2-6-min reaction time. An average value of multipled run was taken, and the standard deviation was 0.481 on average.

The results just previously discussed is also in line with the effect of reaction temperature on conversion efficiency in biodiesel production from the SPFS with the assistance of ultrasound as shown in Fig. 6. Experiments were done with the

experimental condition shown by the legend in Fig. 6, and experiments at more than 65 °C reaction temperature were not conducted because the effect was known in the previous discussion. The graphs with KOH legend shown in Fig. 6 presents the experiments with 80-min reaction time, and the graphs with reaction time represent the experiments with 5% KOH.

As expected from the result of microwave assistance, reaction temperature influenced conversion efficiency in biodiesel production with the assistance of ultrasound, as can be seen in Fig. 6. The maximum biodiesel yield is 86.38% with the optimal experimental condition being 25:1 w/w ratio of methanol to the SPFS oil, 1:1 w/w ratio of methanol to chloroform, 5% KOH, 50 rpm stirring, 37 kHz ultrasound, 65 °C reaction temperature and 80-min reaction time. Meanwhile, the biodiesel yield without ultrasound assistance obtained was approximately 77.2%, which is a bit lower than the one with ultrasound assistance. This indicates that ultrasound increased mass transfer of reactants to form more ester. This increase could be due to the formation of cavitation bubbles stimulated by ultrasonic waves with sufficient activation energy, and the formation of microscale fine emulsions of oil and methanol to easily suspend each other [50].

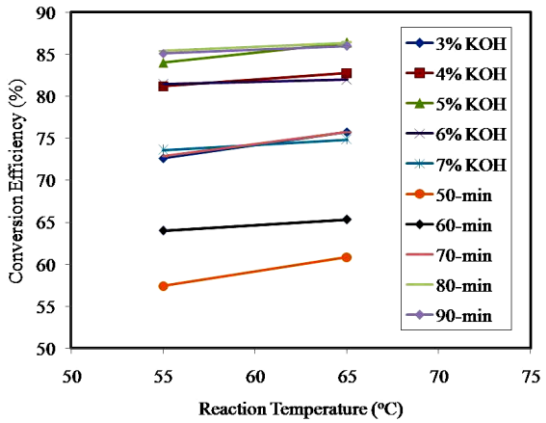

Fig. 6. Effect of reaction temperature on the biodiesel production from the SPFS using in-situ transesterification with the assistance of ultrasound. Experimental conditions: 25:1 w/w ratio of methanol to the SPFS oil, 1:1 w/w ratio of methanol to chloroform, 3-7% KOH and 50 rpm stirring, 37 kHz ultrasound, 55-65 °C reaction temperature, 50-90-min reaction time. An average value of multipled run was taken, and the standard deviation was 0.552 on average.

3.5. Quality assessment of produced biodiesel

The kinematic viscosity of biodiesel is one of the most important properties that affect the mechanism of atomization in the fuel injection equipment. Higher kinematic viscosity causes poor fuel atomization during spraying, increases carbon deposition on the fuel filter, needs more energy from the fuel pump, and wears the fuel pumps and injectors. In-situ transesterification was used to lower the viscosity of oil [51, 52]. Meanwhile, the density of the biodiesel was also a very important parameter, and the density values have been used to measure the amount of fuel in the fuel system by a volumetric method. The variation of the

density affected the power and the fuel spray characteristics during fuel injection and combustion in the cylinder [53, 54]. Acid value is a measure of free fatty acids contained in a fresh fuel sample and free fatty acids from degradation in aged samples. The acid value of biodiesel was influenced by the type of feed-stock used for fuel production and by its degree of refinement. Acidity can be generated by mineral acids introduced as catalysts, or by free fatty acids resulting from acid work-up of soaps. High fuel acidity caused corrosion and the formation of deposits within engines, particularly in fuel injectors [55].

In the current research, the quality assesment was performed using physical parameters such as kinematic viscosity, density and acid value. The obtained values were compared to Biodiesel Standards of European Norm (EN) 14214-2003 [56-58], and it is listed in Table 1. As presented in Table 1, the kinematic viscosity of the produced biodiesel from the SPFS oil was approximately 4.61 mm^2/s, and this value met the EN 14214-2003 standard. The biodiesel density was approximately 876 kg/m^3 which was in the range of the EN 14214 standard. Meanwhile, the acid value was approximately 0.8 mg-KOH/g, which was higher than the one based on the EN 14214-2003 standard.

Table 1. Quality test of biodiesel from screw pine seed.

Property	Test method	Limit	Biodiesel
Kinematic viscosity, mm^2/s	EN14105	3.5 – 5.0	4.61
Density, kg/m^3	EN12185	860 – 900	876
Acid value, mg-KOH/g	EN 14104	0.5	0.8

3.6. Component Analysis of Produced Biodiesel by Gas Chromatography–Mass Spectrometry (GC–MS)

Biodiesel samples at the optimum conditions were analysed using the GC–MS to identify the composition of fatty acid methyl ester in the biodiesel. The compositions of fatty acid methyl esters based on the GC–MS are shown in Fig. 7. Meanwhile, Table 2 shows esters found in the product with the percentage and retention time (RT).

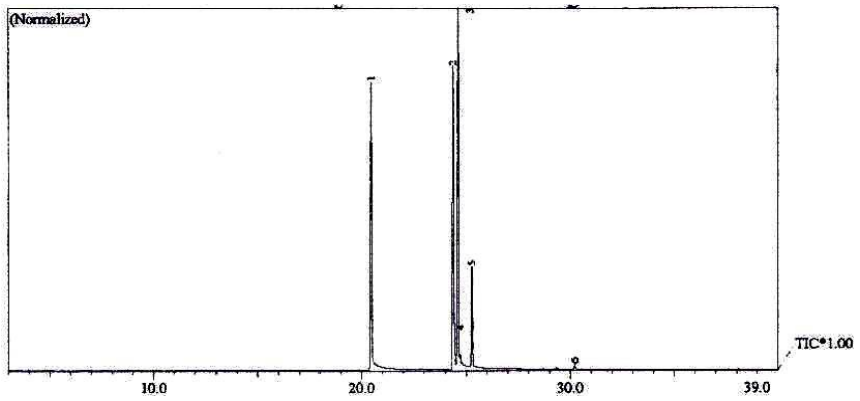

Fig. 7. GC–MS analysis of biodiesel from screw pine seed

Based on Figure 6 and Table 2, all the fatty acid content in the SPFS oil was converted completely into the respective fatty acid methyl/ethyl ester. The result of the GC–MS analysis shows that the major fatty acid esters in the biodiesel obtained are methyl palmitate (28.6%), methyl linoleate (26.18%), methyl oleate (31.1%), methyl oleate (3.19%), methyl stearate (10.42%) and methyl arachidic (0.51%).

Table 2. Physicochemical test of biodiesel from screw pine seed.

Peak	R. Time	Area%	Name of Component
1	20.473	28.6	Methyl palmitate
2	24.368	26.18	Methyl linoleate
3	24.610	31.1	Methyl oleate
4	24.698	3.19	Methyl oleate
5	25.279	10.42	Methyl stearate
6	30.216	0.51	Methyl arachidic

4. Conclusion

Screw pine fruit seed (SPFS) oil is promoting non-edible biodiesel production to develop in the future. Experiments of in-situ transesterification to produce biodiesel from the SPFS oil were carried out in batch mode with the assistance of microwave and ultrasound. The effect of independent variables such as the SPFS oil to methanol ratio, reaction time, catalyst concentration and reaction temperature were investigated with the comparisons along the independent variables. The fatty acid methyl ester composition was identified using GC–MS. The experimental results showed that the maximum conversion efficiency was approximately 88.12% for the assistance of microwave with 4-min reaction time, 450W microwave power, 25:1 w/w ratio of methanol to the SPFS oil, 1:1 w/w ratio of methanol to chloroform, 5% KOH, 50 rpm stirring and 65 °C reaction temperature. Meanwhile, it was approximately 86.38% for the assistance of ultrasound with 80-min reaction time, 37 kHz ultrasound. The physicochemical properties such as kinematic viscosity, density and acid value met to the EN 14214-2003 standard except for the acid value, and the GC–MS analysis identified five major fatty acid esters in the biodiesel.

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