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The effect of NaOH catalyst concentration and extraction time on the yield and properties of *Citrullus vulgaris* seed oil as a potential biodiesel feed stock

J.K. Efavi [a], Dindomah Kanbogtah [a], V. Apalangya [b], E. Nyankson [a], E.K. Tiburu [c], D. Dodoo-Arhin [a], B. Onwona-Agyeman [a], A. Yaya [a, d, *]

[a] Department of Materials Science & Engineering, School of Engineering Sciences, CBAS, University of Ghana, Legon, Ghana
[b] Department of Food Process Engineering, School of Engineering Sciences, CBAS, University of Ghana, Legon, Ghana
[c] Department of Biomedical Engineering, School of Engineering Sciences, CBAS, University of Ghana, Legon, Ghana
[d] Institut des Matériaux Jean Rouxel (IMN), UMR 6502, CNRS, Université de Nantes, 2 rue de la Houssinière, 44322 Nantes Cedex 3, France

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ABSTRACT

In this work, oil was extracted from *Citrullus vulgaris (watermelon)* seeds for potential feedstock in biodiesel production. The results showed that, the oil content from *Citrullus vulgaris* seeds oil during extraction reached an average yield of 50%. Biodiesel was produced via transesterification using NaOH as catalyst. The effect of NaOH on the yield of the biodiesel was investigated at three different concentrations; 0.13, 0.15 and 0.18 g and oil to methanol ratio of 5:1 under different reaction times; 90, 120 and 150 min at 60 °C. The yield of biodiesel from NaOH concentration of 0.13 g was found to be 70% as compared to those of concentrations, 0.15 g and 0.18 g which were 53% and 49% respectively.

Gas chromatography was used to identify the methyl ester groups present in the biodiesel and the results revealed both concentration and time-dependent increase in oil yield. The physicochemical properties of the watermelon seed oil such as flash point (141.3 ± 0.4–143.4 ± 0.2), density (0.86 ± 0.04 –0.91 ± 0.01 g/cm^3), kinematic viscosity (30.50 ± 0.1–31.20 ± 0.04 mm^2/s) and acid value (mg KOH/g) are similar to conventional vegetable oils. This work therefore, highlights the potential utility of water melon seeds for biodiesel production.

1. Introduction

The elevated interest in biodiesel fuels as alternative source of energy is due to its beneficial impacts in the reduction of greenhouse gasses, improvement in energy security, and its potential source of income (De Fraiture et al., 2008; Rahman et al., 2015; Tshizanga et al., 2017). Furthermore, rising energy prices and release of climate warming greenhouse gases from fossil based fuels will continue to spur research interest in biodiesel as cleaner and potentially cheaper alternative (Demirbas, 2008; Elum and Momodu, 2017; Ritchie and Dowlatabadi, 2017). Currently most biodiesels are produced using various edible vegetables as feedstock, however, production of biodiesel from these edible vegetable oils competes with food consumption and consequently resulting in high cost of food (Banerjee et al., 2014; Barnwal and Sharma, 2005; Demirbas, 2005; Haas, 2005; Sundus et al., 2017; Mitchell, 2008; Mohamad et al., 2017). In addition, the negative impact of the utilisation of food derived biodiesel production may compromise the environment in terms of deforestation and destruction of the ecosystem (Agarwal et al., 2017; Fargione et al., 2008; Naylor et al., 2007; Tilman et al., 2009).

Despite the environmental consequences of the utilisation food sources for biodiesel production, the world energy burden cannot be supported solely from these sources (Hill et al., 2006; Yang et al., 2014). Furthermore, the cost associated with the production of vegetable oil as a source for biodiesel, is not commercially scalable. It is estimated that, the price of biodiesel per litre is higher as compare to petroleum based diesel. Also, it has been reported that, the high cost of biodiesel is mainly as a result of the cost of virgin vegetable oil (Pleanjai and Gheewala, 2009; Takase et al., 2015). In view of this, there has been the need to use non-edible oil as

* Corresponding author. Department of Materials Science & Engineering, School of Engineering Sciences, CBAS, University of Ghana, Legon, Ghana.
E-mail address: ayaya@ug.edu.gh (A. Yaya).

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feedstock to produce biodiesel. The potential replacement of edible vegetable oil with non-edible vegetable from jatropha, neem, moringa, casper, melon seeds as biodiesels have been explored with considerable success (Anya et al., 2012; Mabaleha et al., 2007; Martín et al., 2010; Mohammed & Nemit-allah, 2013; Nakpong and Wootthikanokkhan, 2010).

Watermelon is a drought tolerant fruit which belongs to the cucurbitacea family of flowering plants (Górnaś and Rudzińska, 2016; Razavi and Milani, 2006). It is botanically named as *citrullus vulgaris* which originated from southern part of Africa (Council, 2006). Watermelon seed oil is also known as Ootanga oil or Kalahari oil (Jones, 2011). *Citrullus vulgaris* seed oil contains high amounts of unsaturated fatty acids and oleic acids which is similar to vegetable oil hence making it suitable for biodiesel production (Albishri et al., 2013). Watermelon is cultivated on large scale and around 20% of total production goes waste due to surface blemishes and the juice also serves as feedstock for ethanol production (Rao et al., 2012). However, there is limited work on the use of watermelon seeds as source of oil for biodiesel production. Another variety of watermelon, *Citrullus colocynthis* L., has been thoroughly exploited for biodiesel production as compared to watermelon seeds (*citrullus vulgaris)*, even though the latter is widely cultivated in sub-Saharan Africa (Giwa et al., 2010; Jarret and Levy, 2012; Panneerselvam et al., 2017; Rao et al., 2012).

This work seeks to investigate the production of biodiesel from watermelon waste (seeds) using analytical techniques such as soxhlet extraction methods by using sodium hydroxide as a catalyst via transesterification reaction. The end product was then quantified and estimated based on known amount of the starting material as well as a function of the catalytic concentration and the time of reaction.

2. Methodology

2.1. Citrullus vulgaris seed oil extraction

The *Citrullus vulgaris* (watermelon) seeds were obtained from a nearby farm in Accra. The seeds were milled using attrition mill for 10 min. Methanol (99.5%), anhydrous sodium sulphate and sodium hydroxide (99.5%) were obtained from Sigma Aldrich, Germany.

Currently there is no commercial production of *Citrullus vulgaris* seed oil; hence it is not available in the market. The oil was extracted using a soxhlet extractor as follows. Prior to the extraction, the seeds were milled and dried in an oven at 60 °C for 120 h. The milled seeds (10 g) were placed in the extraction thimble and inserted into the soxhlet assembly fitted with a 200 ml flash. A 120 ml portion of petroleum ether was added and the assembly was reflux for a period of 15 h. The extract was concentrated to a total volume of 50 ml using rotary evaporator at 80 °C to remove the solvent and subsequently dried over anhydrous sodium sulphate to remove residual water. This extracted oil and the yield were calculated based on thier dry weight.

The relation below was used to calculate the % oil yield;

$$\%\ \text{Oil yeild} = \frac{\textit{weight}\ \text{of oil}}{\textit{weight}\ \text{of seed}} \times 100 \quad (1)$$

2.2. Transesterification

The transesterification reaction of the oil was carried out in a 100 ml conical flask with methanol in a molar ratio of 1:5 (methanol:oil), using anhydrous sodium hydroxide as catalyst (0.13, 0.15, and 0.18 g). The reaction was carried out at 60 °C for 90 min, with the reaction mixture funnel filtered to separate the glycerol from the ester within a 24 h period. Separation of the two phases and removal of excess solvent in each phase gave biodiesel and glycerol with good yield. During the purification process, methyl ester was washed with water to remove the catalyst, residual amount of glycerol and methanol. Furthermore, traces of solid waste were also removed to recover an appreciable amount of the biodiesel. This was essential because, the purity level of the biodiesel has strong effects on its fuel functionalities and properties (Razavi and Milani, 2006).

2.3. Gas chromatography

Analysis was performed using a varian 240 -MS IT Mass Spectrometer fitted with a factor four (4) TM VF-5MS capillary column (length 30 mm, internal diameter 0.25 mm, film thickness 0.25 μm). Temperature of the front injector was set at 280 °C with a column oven temperature of 270 °C for a total time of 35 min. The detector middle temperature was also at 280 °C.

2.4. Determination of fuel properties of water melon oil

The biodiesel properties of watermelon seed oil, such as density, kinematic viscosity, flash point and acid value were determined according to biodiesel test standards: density (ASTM D4052), kinematic viscosity (ASTM D445), flash point (ASTM D93) and acid value (EN 14104). All tests were run in triplicate and mean values were reported.

3. Results and discussion

3.1. Percent oil yield per gram of seed

The extraction for each batch was subjected to the same condition of temperature and pressure. The oil yield was calculated based on the dry weight using equation (1). From the results shown in Table 1, the oil content of the extracted water melon seeds before the conversion to biodiesel (crude oil) is 50% (obtained as the average of the 4 batches, see Table 1 which is higher *than other sources such as Mahua (35–40%), Sal (10–12%), Linseed (35–45%), Neem (20–30%), Pongamia (30–40%)* and 45.5% crude vegetable oil extracted from coconut oil (Musa et al. 2016; Singh and Singh, 2010).

The oil was used to estimate biodiesel production using anhydrous NaOH as catalyst with concentrations (0.13 g, 0.15 g, & 0.18 g).

As shown in Fig. 1, the production of biodiesel decreases as a function of catalyst concentration with the highest yield observed at NaOH concentration of 0.13 g and the lowest at 0.18 g. The concentration of catalyst can affect the yield of biodiesel. The most commonly used catalyst for biodiesel productions can be a base catalyst (KOH, NaOH) or Acidic catalyst (H_2SO_4). The base catalysts are mostly preferred due to their non-corrosive nature. In the present study, we used NaOH for the simple fact that, it is readily available and cheap. The NaOH catalyst first reacts with the methanol, reducing it to a methoxide which then attacks the triglycerides in the oil breaking it into fatty acid methyl esters (FAME)

Table 1
The percent oil yield from water melon seeds after the extraction (crude oil).

Batch	Weight of seed (g)	Weight of oil (g)	% Yield
1	48.530	25.37	52
2	82.226	40.226	49
3	69.367	33.967	48
4	56.040	26.339	47

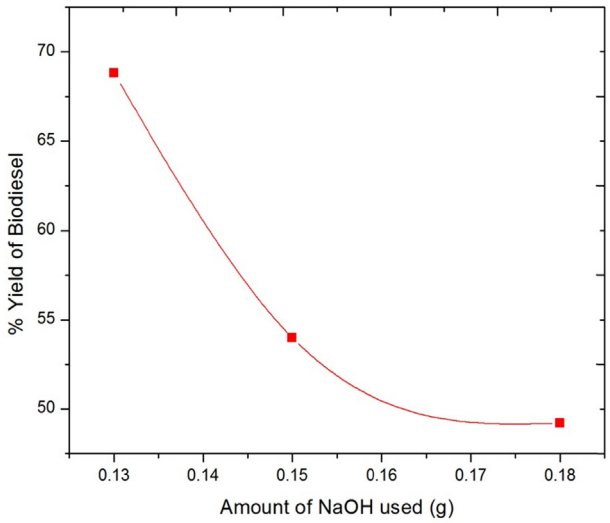

Fig. 1. Graph of biodiesel yield against amount of NaOH used ("the line is a guide to the eye").

and glycerol.

In Fig. 1, it is observed that the maximum yield of the biodiesel (FAME) is about 70%, obtained with the lowest NaOH concentration of 0.13 g. However, increasing the concentration of the catalyst leads to a gradual decline in the yield (about 49%) for 0.18 g NaOH concentration. The reduction in the yield is due to the addition of excess NaOH catalyst which leads to more triglycerides which reacts with NaOH to form soap (Leung and Guo, 2006 & Freedman et al., 1984).

To estimate the duration of the reaction that generated the percentage yields in Fig. 1, a biodiesel reaction profile was generated in Fig. 2. It was found that, there is an increase in biodiesel production as a function of time and almost plateaus after 3 h.

3.2. Effect of reaction time on biodiesel yield

The effects of reaction time on the yield of biodiesel at different time intervals from 90, 120, 150 min at constant temperature was investigated with catalyst content of 0.13 g and at constant oil to methanol ratio of 5:1, see Fig. 2. It can be argued that, the conversion rate of fatty acid esters increases with reaction time. At the start, the reaction is slow due to the mixing and dispersion of alcohol into the oil. After some time, the reaction proceeds steadily fast. Ideally, the yield reaches a maximum at a reaction time of 120 min, and then remains relatively constant with a further increase in the reaction time. Thus, it suggests that, the suitable reaction time is somewhere between 90 and 120 min. However, excess reaction time at about 150 min, will lead to a reduction in the product yield due to the backward reaction of transesterification, resulting in a loss of esters as well as causing more fatty acids to form soaps (Albishri et al., 2013, Eevera et al. (2009) & Ma et al. (1998)).

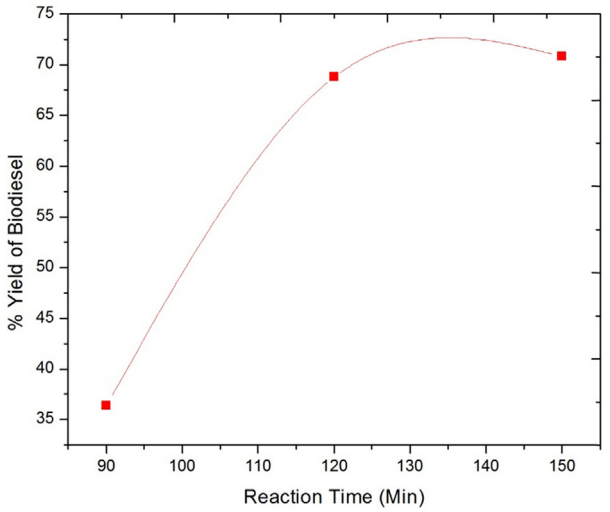

Fig. 2. The percent yield of biodiesel against reaction time.

3.3. Effect of catalyst concentration on the physicochemical properties of watermelon seed oil

The physicochemical properties such as density, kinematic viscosity, acid value and flash point of the extracted watermelon seed oil were determined using freshly extracted crude watermelon seed oil. The results of these measurements are recorded in Table 2. Density is an important parameter for biodiesel fuel injection systems and therefore the current study seeks to also determine the density among other parameters of the generated biodiesel. The density is directly proportional to the kinematic viscosity which determines how well the biodiesel will flow in a fuel system. According to the European standard EN ISO 12185, the value of the density at 15 °C should be between 0.86 and 0.90 g/cm^3. The obtained value for the biodiesel produced in the current study was within the specification limits as shown in Table 2. The kinematic viscosity of the extracted watermelon seed oil ranges between 30.5 ± 0.1 and 31.2 ± 0.04 mm^2/s as shown in Table 2, which was similar to that reported for Egusi melon, which are within the range reported for vegetable oil (Giwa et al., 2010; Kim et al., 2004). Also, the acid value of the oil ranges from 0.96 ± 0.21 to 0.97 ± 0.12 mg KOH/g. This range was similar to the acid value of 0.98 reported by Giwa et al. and matches with the fatty acid value of 0.49% needed for transesterification without the formation of soap (Demirbas, 2009).

The flash point is the temperature at which the fuel will ignite when it comes into contact with flame. As shown in Table 2, the flash point ranges from 141.2 ± 0.1 for sample 1 to a maximum of 143.4 ± 0.2 (for sample 2). However these flash point values for watermelon seed oil are demonstrably higher than that for most reported diesel fuels. As a result watermelon biodiesel is potentially safer fuel for transportation purposes as compared to most diesel oils with lower flash points. These flash point are also consistent with other reported flash point of palm and pongamia biodiesels (Sarin et al., 2007). Additional all the properties recorded with the exception of the yield percent of the biodiesel were similar irrespective of the NaOH concentration. This was expected because a single source for the biodiesel was used for the transesterification reaction.

Table 2
Physicochemical properties of biodiesel obtained from watermelon seed oil prepare with 0.13 g (Sample 1), 0.15 g (Sample 2) and 0.18 g (sample 3) NaOH catalyst concentrations at a reaction time of 120 min.

Properties	Sample 1	Sample 2	Sample 3
Density (g/cm3)	0.88 ± 0.02	0.91 ± 0.01	0.86 ± 0.04
Kinematic viscosity (mm^2/s)	30.80 ± 0.0	30.50 ± 0.10	31.20 ± 0.04
Acid value (mg KOH/g)	0.96 ± 0.21	0.97 ± 0.12	0.97 ± 0.10
Flash point (°C)	141.2 ± 0.1	143.4 ± 0.2	141.3 ± 0.4
Yield (%)	70	55	49

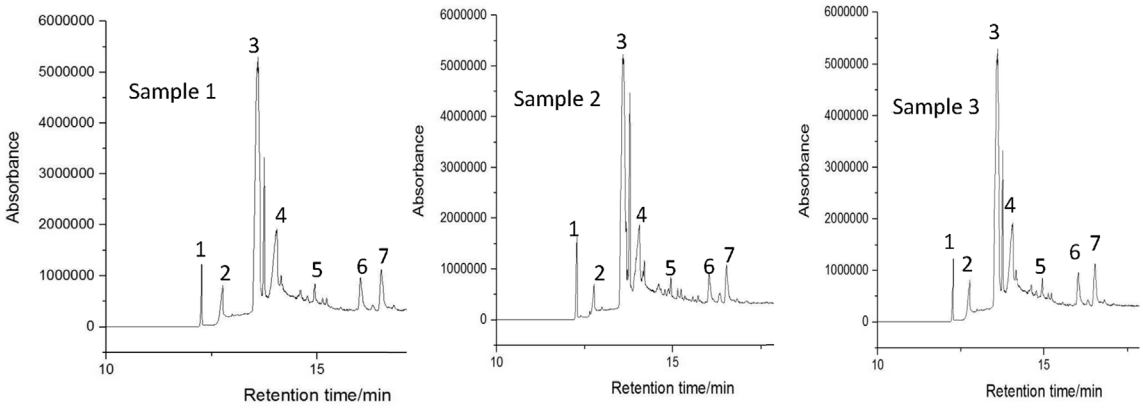

Fig. 3. Gas chromatographs of biodiesel oil samples with different NaOH concentrations and their fingerprints fatty acid methyl esters peak identification; where each of the corresponding peak numbers are given in Table 3.

Table 3
Identifications of peaks corresponding to fatty acids chain with peaks numbering (1–7) from Fig. 3.

Peak No	Retention time (mins)	Fatty Acids	Corresponding acid	Chemical formulae
1	12.77	Hexadecanoic acid	C16:0	$CH_3(CH_2)_{14}COOH$
2	13.61	*cis*-9-Octadecenoic acid	C18:1	$C_{18}H_{34}O_2$
3	14.05	*cis*-9,12-Octadecadienoic acid	C18:2	$C_{18}H_{32}O_2$
4	14.55	Octadecanoic acid	C18:0	$C_{18}H_{36}O_2$
5	15.00	9-Hexadecenoic acid	C16:1	$C_{16}H_{30}O_2$
6	15.50	(9Z,12Z,15Z)-octadeca-9,12,15-trienoic acid	C18:3	$C_{18}H_{30}O_2$
7	15.75	Heptadecanoic acid	C17:0	$C_{17}H_{34}O_2$

3.4. Gas chromatography analysis

GC/MS analysis was used to study the chemical composition of the synthesized biodiesel. The gas chromatogram of samples 1, 2 & 3, which were respectively treated with 0.13, 0.15 and 0.18 g NaOH are shown in Fig. 3.

The nature and percentage of fatty acids present in vegetable oils is dependent on the plant species. The profile of fatty acids in vegetable oils is a primary factor that influences oxidation because; the rate of oxidation depends on the number and positions of double bonds. In order to confirm this, a report on the oxidation stability, found that, oxidation decreases with increase in poly-unsaturated methyl esters such as linoleic and linolenic esters (Giwa et al., 2010).

In Fig. 3, the chromatographs of the three samples are shown. The graph shows seven identical vibrational peaks (numbered 1–7, Fig. 3) are present in each of the samples which indicate that, there are seven different compounds present in the biodiesel. The higher intensity peaks signify compounds with the highest abundance. In order to identify the corresponding compounds of the peaks in the samples, matching patterns from GC/MS data library was used and their methyl ester compounds are given in Table 3.

4. Conclusions

Biodiesel of high quality can be produced from watermelon seed oil by transesterification reaction using NaOH as catalyst. The biodiesel yield is affected by the quantity of catalyst used and the reaction time. Using a concentration of 0.13 g of the NaOH catalyst, the yield of biodiesel was 70%. But, increasing the concentration on the other hand, resulted in a gradually decreased in yield of 49% for 0.18 g catalyst. This further confirms soap formation as a result of excess catalyst with a slower conversion of the triglycerides to FAME which invariably affected the biodiesel yield. The highest biodiesel yield was recorded at a reaction time of 120 min with a catalyst content of 0.13 g. The density of biodiesel samples matches with European standard EN ISO 12185. The gas chromatography analysis showed similar organic molecules but with different atomic weights for each of the biodiesels.

Conflicts of interest

None.

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