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Biodiesel Generation from Palm Kernel Biomass *via* an *In-situ* Transesterification Approach

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Abstract

This study investigated an in-situ transesterification of palm kernel biomass at variable reaction times (4 h, 5 h and 6 h) and catalyst loading (2 wt%, 3 wt.% and 5 wt.%). The biodiesel yields were observed to vary with these parameters. GC-MS analysis showed high fatty acid methyl esters (FAMEs) contents for all the prepared biodiesel samples (C-18:1, C-14:0, C-16:0, and C-18:0). 2 wt.% catalyst loading and 4 h reaction time were observed to be the best reaction conditions from the experimented parameters that gave the best biodiesel yield at 300 rpm mixing intensity. The in-situ method recorded a higher degree of oil extraction and efficient conversion of oil to biodiesel (93.93%) compared to the conventional transesterification method which gave 89.45%. The kinematic viscosity, acid value and iodine value of 3.96 mm^2/s, 0.56 mg KOH g^{-1} and 120 g 100 g^{-1} were found to be within the limits of ASTM D6751-09 and EN14214 standards. These results confirm that efficient production of biodiesel from palm kernel biomass could be possible through an in-situ acidic transesterification process.

Keywords: Biodiesel, fatty acid methyl ester, palm kernel biomass, transesterification.

Introduction

The immense demands for energy consumption in the industrialized world and human existence have increased due to the increase in population (Onaifo et al. 2016, Olugbemide et al. 2020). At the moment, there is depletion in the major sources of non-renewable energy causing depletion in the ozone layer and climate change (Atadashi et al. 2011). This has resulted in the development of renewable energy sources with little or no environmental pollution when compared to conventional sources. Examples of renewable energies that have been used include geothermal energy, biofuels, solar energy, wind energy, hydrothermal energy, biomass, biofuels and others (Marchetti et al. 2007). Nevertheless, biofuels such as biodiesel are the most widely used fuels in diesel engines for replacing the dwindling conventional petroleum diesel because they have similar properties (Simasatitkul et al. 2011, Giwa et al. 2014, Giwa et al. 2015). The high flash point of biodiesel (150 °C) makes it less volatile and safer to transport and handle (Hoekman et al. 2012).

Biodiesel is a viable alternative transportation fuel that constitutes mono-alkyl esters of long-chain fatty acids from vegetable oils or animal fats (Kukana and Jakhar 2021). It is also known as a carbon-neutral fuel and a national product that reduces the dependency on foreign energy resources and creates jobs for the economy (Fukuda et al. 2001). It enjoys a positive social impact, by promoting rural revitalization (Kiss et al. 2006) and converting waste materials of no economic value to products with a more improved worth. Biodiesel is composed of long-chain hydrocarbon groups with essentially no branching or aromatic structures. The presence of a significant amount of oxygen (9%) in its structure makes its combustion efficiency higher than that of petroleum diesel (Hoekman et al. 2012). Previous studies have produced biodiesel from high-oil content agricultural products such as coconut, palm oil, soybean oil, Jojoba oil, rice bran, sunflower, canola, rapeseed, soybean, sunflower oil, neem oil, peanut oil and cottonseed oil via the transesterification process (Hailegiorgis et al. 2011, Sinha et al. 2008, Issariyakul and Dalai 2010, Maliki and Ifijen 2020, Ifijen and Nkwor 2020, Maliki et al. 2020). This process involves the reaction of an alkyl-alcohol with a long chain ester linkage to yield mono-alkyl esters (biodiesel) and glycerol in the presence or absence of a catalyst to chemically break the molecule of triglyceride (Ifijen et al. 2018, Kafuku et al. 2010).

Several alternative fuels such as biogas, producer gas, ethanol, methanol and vegetable oils have been utilized. However, biodiesel offers an advantage because of its comparable fuel properties with that of petroleum diesel and its renewable and environmentally friendly nature (Zaher and Gad 2016). Biodiesel releases less particulate emissions, soot and carbon monoxide than petroleum-based fuels (Aalam and Saravanan 2015, Selvakumar et al. 2015, Ahmed et al. 2016). This eco-friendly property has led to its use in many countries, especially in environmentally sensitive areas like Malaysia, Thailand, South Korea, USA, Philippines, Indonesia, China, Australia, Argentina, Malaysia, Brazil, Czech Republic, Germany, France, Italy, Slovakia, Spain, etc. A major challenge to biodiesel commercialization as an alternate fuel to petroleum diesel is the cost of production. However, the cost can be reduced by the increase in feedstock supply through the expansion of current agricultural lands or the development of new sources of vegetable oils (Demirbas 2008). Several kinds of bio-based fuels such as vegetable oils (raw, refined, or used), oil methyl esters, and liquid biomass fuels as options to fossil fuels have been studied (Karabas et al. 2014). To be as efficient substitutes for petroleum diesel, vegetable oils have to be modified. The four primary techniques that can be used in the modification are micro-emulsion, dilution, transesterification and pyrolysis (thermal cracking) (Kumar and Sharma 2011). The leading technique for the advancement of an eco-friendly sustainable and reliable fuel from raw vegetable oils is transesterification (Aalam and Saravanan 2015). The transesterification process has been proposed by several studies as one of the encouraging techniques for converting vegetable oils to fatty acid alkyl esters that can be utilized in diesel fuel-based engines. The factors that affect transesterification are catalytic concentration, methanol-to-oil molar ratio, reaction temperature, reaction time and free fatty acid content (Thoai et al. 2019). Biodiesel is a fuel that is sustainable, biodegradable, environmentally safe, and nontoxic and can be applied without any engine remodeling in conventional diesel engines with consistent performance. This study made use of an in-situ transesterification process to increase the conversion of biodiesel. In-situ transesterification, therefore, facilitates the conversion of the biomass oil to biodiesel directly from the oil-bearing biomass through the simultaneous extraction and transesterification process of the biomass oil (Ehimen et al. 2010). The major advantage of this process is the capability of generating biodiesel from oilseeds without first carrying out oil extraction and the low amount of solvent/catalyst required during the extraction/synthetic steps. This helps to overcome the challenges of the large amounts of hazardous solvent wastes produced from the oil extraction/transesterification processes (Li et al. 2011). As a consequence, this method has proven to be an efficient and economical method to produce biodiesel compared to the conventional route (Johnson and Wen 2009).

This study generated biodiesel from palm kernel biomass via an in-situ transesterification process and investigated the certain reaction conditions such as the effects of catalyst loading and reaction time on the biodiesel yields.

Materials and Methods: Experimental Chemicals

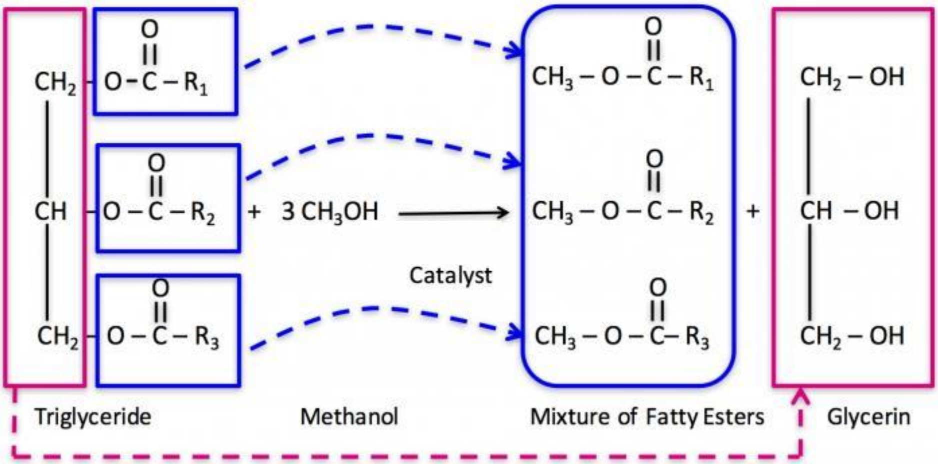
Methanol, hexane, tetraoxosulphate(VI) acid, calcium chloride, sodium thiosulphate, potassium iodide, potassium hydroxide, calcium oxide, phenolphthalein solution, starch solution, Wijis solution and distilled water were used. All reagents were of analytical grade.

Biomass and sample preparation

The palm kernel seed samples used in this work were obtained from Oluku in Benin City, Edo State, Nigeria. The obtained seeds were screened to remove the defected seeds, sundried and cracked to obtain the kernel seeds. Thereafter, they were washed, sundried and ground into powder form. The seeds samples were repeatedly weighed and dried in the oven at 105 °C until a constant weight was achieved.

In-situ transesterification palm kernel biomass

To reduce moisture content, pre-treatments of the milled palm kernel were carried out using methanol as described by Qian et al. (2013). The in-situ transesterification process was carried out as described by Johnson and Wen (2009). In a typical experiment, 120 g of milled palm kernel biomass was placed in a 1 L three-neck round bottom flask with a reflux condenser and mixed with methanolic solution containing sulfuric acid, which was freshly prepared to maintain its catalytic activity. n-Hexane (95% purity) and a calcium oxide catalyst concentration of 2 wt.% were then added to the mixture of methanol and sulfuric acid. The reaction temperature was maintained at 50 °C for 4 h. The reaction mixture was filtered, centrifuged and separated to obtain the biodiesel (fatty acid methyl esters (FAMEs)) which was washed with warm distilled water and also dried with calcium chloride. Other biodiesel samples were similarly prepared by varying the reaction parameters at a constant stirring speed of 300 rpm. The chemistry of the biodiesel synthesis is depicted in Scheme 1.



Scheme 1: Chemistry of biodiesel production.

Characterization technique

The fatty acid methyl esters (FAMEs) present in the generated biodiesel were characterized using GC-MS with a variable split-flow injector (Agilent 7890, Agilent Technologies) coupled with a mass selective detector (mass spectrometer) of 5975 Agilent technologies equipped with a HP5MS Agilent technologies capillary GC column (length of 30 m, internal diameter of 0.320 mm, 0.25 mm Agilent HP-5MS column (film thickness of 0.25 µm). The injector and detector temperatures are 250 °C and 280 °C, respectively. Helium was used as the carrier gas.

Results and Discussion

Table 1 reveals the fuel features of biodiesel synthesized *via* an *in-situ* transesterification process. The results revealed that the acid values of the developed biodiesel samples were within the detailed limits of ASTM (0.8 max mg KOH g^{-1}) standards, confirming that the free fatty acids have been totally transformed to biodiesel (Kiss et al. 2006). The determination of a fluid's internal resistance to the flow of biodiesel under gravitational forces is known as *kinematic viscosity*. It is an indicator of fuel atomization and complete combustion in the diesel engine. The generated biodiesel samples were within the range of kinematic viscosity specifications (determinations at 40 °C) of 1.9–6.0 mm^2/s and 3.5–5.0 mm^2/s specified by the American standard ASTM D6751 and European standard EN 14214, respectively (Knothe and Steidley 2005). The observed kinematic viscosity suggests that the prepared biodiesel will provide sufficient lubrication for fuel injection pumps, resulting in decreased wear (Noureddini et al. 1992). Iodine value is an essential quality parameter for estimating biodiesel oxidative stability. It can be used to determine the degree of unsaturation of the biodiesel (Knothe 2002). The observed iodine values were within the ASTM standards (ASTM 1998). Notwithstanding, some of the iodine values of the generated biodiesel samples were slightly higher than the ASTM standard.

Table 1: Effects of the reaction process variables on the physiochemical properties of biodiesel produced

	Catalyst loading (%)			Reaction time (h)		
Biodiesel characteristics	2	3	5	4	5	6
Appearance	Light yellow	Light yellow	Light yellow	Light yellow	Light yellow	Light yellow
Acid value mg KOH g^{-1}	0.56	0.56	0.51	0.56	0.59	0.73
Iodine value g 100 g^{-1}	120	119	116	120	122	120
Kinematic viscosity (at 26 °C) mm^2/s	3.96	3.51	2.80	3.96	2.48	2.42

Effects of variable weight of catalyst loading

The extraction rate of 9.44% makes methanol a poor solvent whenever it is used in lipid extraction (Qian et al. 2008). Hence, catalysts will assist in breaking the cell walls of the oilseed to enable the methanol to react with the oil. The consequence of the weight of catalyst loading on the in-situ transesterification reaction was investigated by adopting three different weights of catalyst loading**,** 2 wt.%, 3 wt.% and 5 wt.% of CaO that have been activated with methanol at room temperature. Figures 1-3 and Tables 2-4 revealed the detected fatty acid methyl esters (FAMEs) of the biodiesel samples at variable catalysts loading obtained from the GC-MS analysis. The generated yields of fatty acid methyl esters from the catalytic performance of hydrogen tetraoxosulphate (vi) acid (H_2SO_4) at varying catalytic loading of 2 wt.%, 3 wt.% and 5 wt.% were very good. An increase in the catalytic loading led to a remarkable improvement in the FAMEs yields from 96.24 to 97.87% and then to 99.99%. This may be ascribed to the unusual surface area of the catalyst and its comparatively simple diffusion rate in the mesopores. The impact of the catalyst on the FAME yields was relatively predominant and the utilized acid catalyst (H_2SO_4) also exhibited tremendous potential as a catalyst for biodiesel generation. Consequently, this proves that tremendous FAMEs can be accomplished by employing the acid-catalyzed *in-situ* transesterification technique. Furthermore, high content of methyl tetradecanoate (C14:0), octadecenoic acid methyl ester (C18:1), methyl stearate (C18:0) and hexadecanoic acid methyl ester (C16:0) at multiple catalyst loading were obtained by the biodiesel fatty acid profiling. Octadecanoic acid methyl ester had the highest amount of fatty acid methyl ester content (37-40%), while methyl stearate (6-8%) showed the lowest fatty acid methyl ester content. The amounts of octadecenoic acid methyl ester (C-18:1) and methyl stearate (C-18:0) were seen to be the highest at 5 wt.% and lowest at 2 wt.% apiece. Notwithstanding, greater amounts of hexadecanoic acid methyl ester (C-16:0) and methyl tetradecanoate (C-14:0) were recorded by 2wt. % catalyst loading, whereas the lowest amounts of C-14:0 and C-16:0 were observed at 3 wt.% and 5 wt.% catalyst loading, respectively. Total saturated fatty acid methyl esters (SAFA) of 59.09%, 57.34% and 55.97% were demonstrated at catalytic loading of 2 wt.%, 3 wt.%, and 5 wt.%, respectively. Also, total monounsaturated fatty acid contents (MUFA) of 37.15%, 39.53% and 40.03% were revealed at the aforementioned catalyst loading.

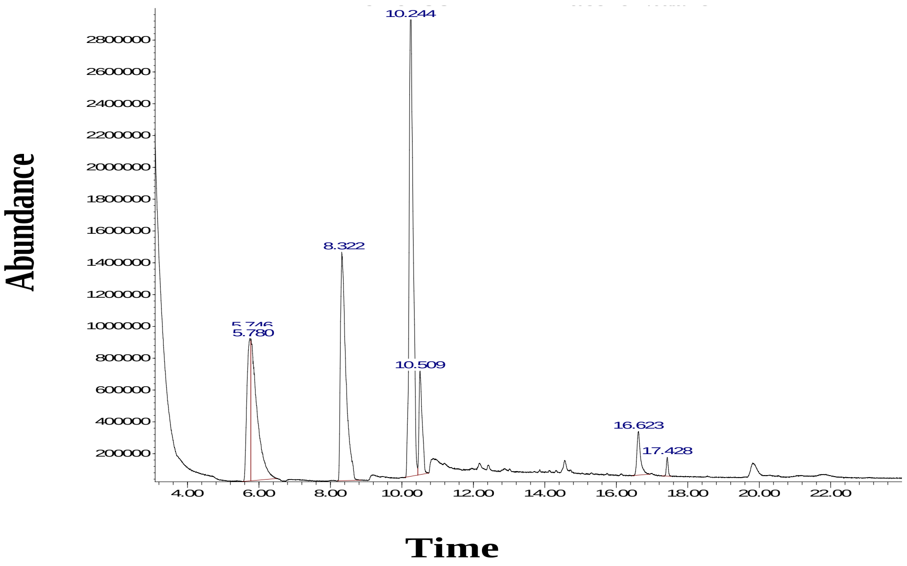


Figure 1: Chromatogram of the observed fatty acid methyl esters at 2wt. % catalyst loading.

Table 2: Fatty acid methyl esters detected at 2 wt.% catalyst loading

Names of fatty acid methyl esters (FAMEs)	Retention time (mins)	FAMEs yield (%)	Molecular formula	Molecular weight
Methyl tetradecanoate	5.763	28.086	$C_{15}H_{30}O_2$	242
Hexadecanoic acid, methyl ester	8.322	24.534	$C_{17}H_{34}O_2$	270
9-Octadecenoic acid, methyl ester *(E)*-11-Octadecenoic acid, methyl ester *Cis*-13-Octadecenoic acid, methyl ester	10.244	37.152	$C_{19}H_{36}O_2$	296
Methyl stearate	10.509	6.472	$C_{19}H_{38}O_2$	298
% Total		96.244		

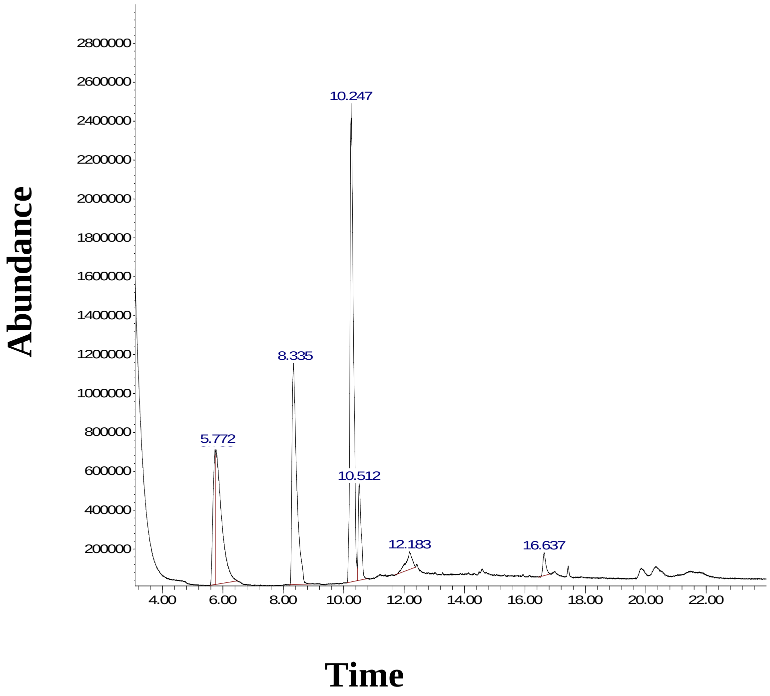


Figure 2: Chromatogram of the observed fatty acid methyl esters at 3wt. % catalyst loading.

Table 3: Detected fatty acid methyl esters at 3 wt.% catalyst loading

Names of fatty acid methyl esters (FAMEs)	Retention time (mins)	FAMEs yield (%)	Molecular mass	Molecular formula
Methyl tetradecanoate	5.738	26.386	242	$C_{15}H_{30}O_2$
Hexadecanoic acid, methyl ester	8.335	24.360	270	$C_{17}H_{34}O_2$
9-Octadecenoic acid, methyl ester *(E)*-11-Octadecenoic acid, methyl ester *Cis*-13-Octadecenoic acid, methyl ester	10.247	39.530	296	$C_{19}H_{36}O_2$
Methyl stearate	10.512	6.596	298	$C_{19}H_{38}O_2$
% Total		97.872		

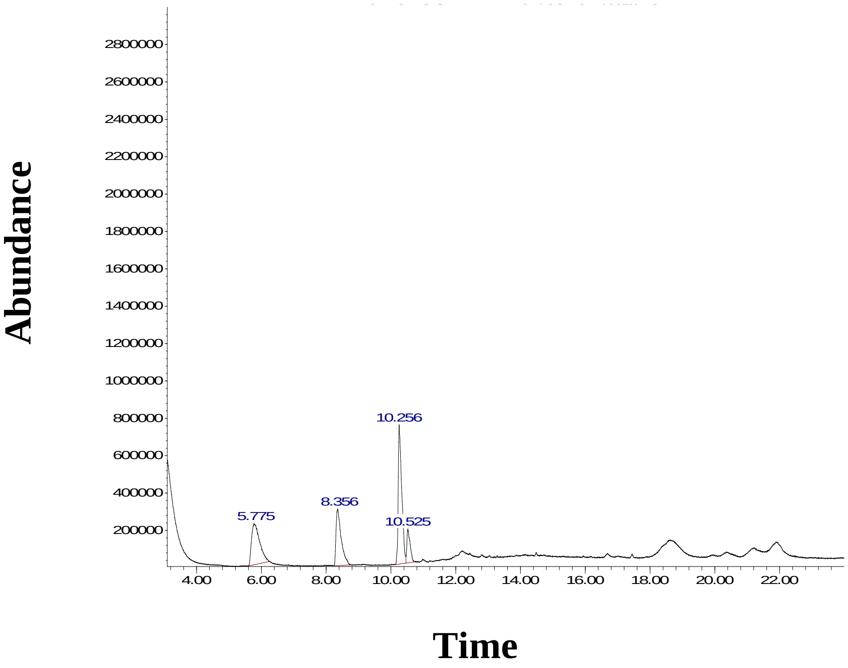


Figure 3: Fatty acid methyl esters revealed at 5 wt.% catalyst loading.

Table 4: Detected fatty acid methyl esters at 5 wt.% catalyst loading

Names of fatty acid methyl esters (FAMEs)	Retention time (mins)	FAMEs yield (%)	Molecular weight	Molecular formula
Methyl tetradecanoate	5.775	27.368	242	$C_{15}H_{30}O_2$
Hexadecanoic acid, methyl ester Pentadecanoic acid, 14-methyl ester	8.356	23.786	270	$C_{17}H_{34}O_2$
(Z)-9-Octadecenoic acid, methyl ester 8-Octadecenoic acid, methyl ester *(Z)*-6-Octadecenoic acid, methyl ester	10.256	40.034	296	$C_{19}H_{36}O_2$ $C_{19}H_{36}O_2$ $C_{19}H_{36}O_2$
Methyl stearate	10.525	8.811	298	$C_{19}H_{38}O_2$
%Total		99.999		

Effects of variation in reaction time

The most significant factor which influences in-situ transesterification is the reaction time (Qian et al. 2013). The effectiveness of biodiesel production and the characteristic of biodiesel outcomes can be influenced by reaction time. The reaction time was varied from 4 h to 6 h to study its effects in the in-situ transesterification of palm kernel using CaO catalyst. Figures 4-6 and Tables 5-7 depict the detected fatty acid methyl esters (FAMEs) of the biodiesel samples obtained from the GC-MS analysis at variable reaction times of 4 h, 5 h, and 6 h. There was an increase in the yields of the FAMEs from 96.88% to 97.85% and finally to 99.94% as the reaction period was extended. This suggests that the FAME yield improves as the reaction period is prolonged. The highest yields of FAMEs obtained at the variable times of 4 h, 5 h, and 6 h were observed to be 37.792%, 34.406% and 37.870% for 9-octadecenoic acid methyl ester, *(E)*-9-octadecenoic acid methyl ester and 11-octadecenoic acid methyl ester, respectively. The lowest contents were seen at 6.472%, 0.310% and 7.797% for methyl stearate, 9,12-octadecadienoic acid (*Z*, *Z*)-methyl ester, and methyl stearate, respectively. The total saturated fatty acid methyl ester contents (SAFA) at various reaction times of 4 h, 5 h and 6 h were observed to be 59.09%, 62.27%, and 62.13%, and the total monounsaturated fatty acid contents (MUFA) were found to be 37.79%, 34.41% and 37.87%, respectively. However, polyunsaturated fatty acid methyl acid (PUFA) content was only observed in the sample fabricated adopting the 5 h reaction time to be 1.17%.

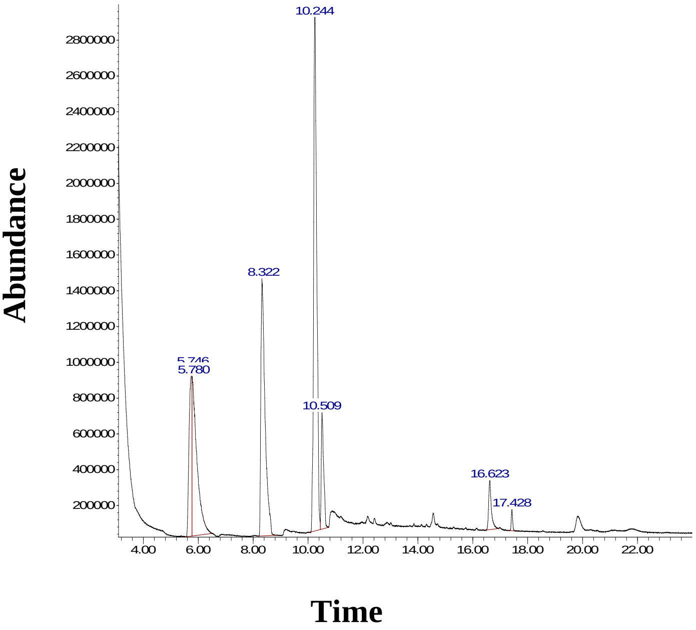


Figure 4: Chromatogram of the observed fatty acid methyl esters at 4 h reaction time.

Table 5: Detected fatty acid methyl esters at 4 h reaction time

Names of fatty acid methyl esters (FAMEs)	Retention time (mins)	FAMEs yield (%)	Molecular weight	Molecular formula
Methyl tetradecanoate	5.746	10.214	242	$C_{15}H_{30}O_2$
Methyl tetradecanoate	5.780	17.872	242	$C_{15}H_{30}O_2$
Hexadecanoic acid, methyl ester	8.322	24.534	270	$C_{17}H_{34}O_2$
9-Octadecenoic acid, methyl ester *(E)*-11-Octadecenoic acid, methyl ester *Cis*-13-Octadecenoic acid, methyl ester	10.244	37.792	296	$C_{19}H_{36}O_2$
Methyl stearate	10.509	6.472	298	$C_{19}H_{38}O_2$
% Total		96.884		

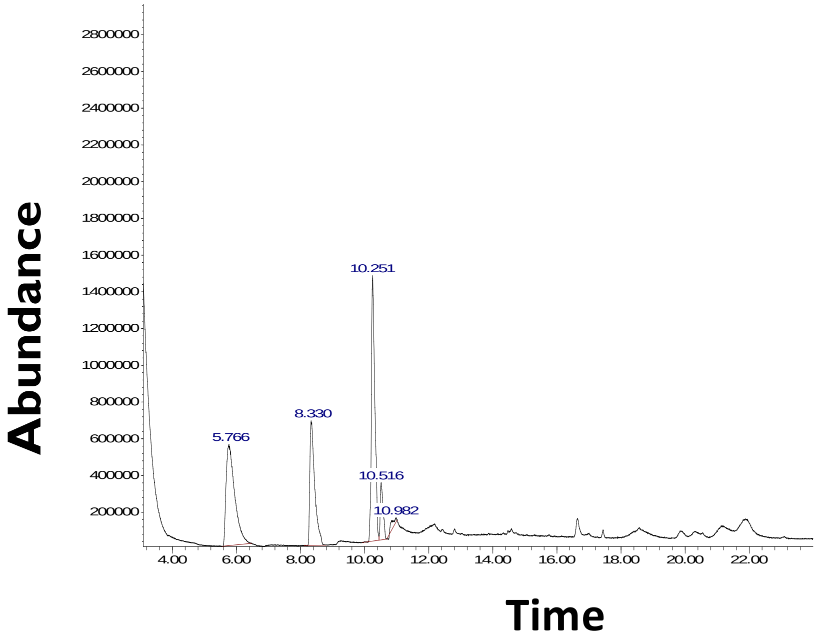


Figure 5: Chromatogram of the observed fatty acid methyl esters at 5 h reaction time.

Table 6: Detected fatty acid methyl esters at 5 h reaction time

Names of fatty acid methyl esters (FAMEs)	Retention time (mins)	FAMEs yield (%)	Molecular Weight	Molecular formula
Methyl tetradecanoate	5.766	33.012	242	$C_{15}H_{30}O_2$
Hexadecanoic acid, methyl ester	8.330	22.999	270	$C_{17}H_{34}O_2$
(E)-9-Octadecenoic acid, methyl ester	10.251	34.406	296	$C_{19}H_{36}O_2$
Cis-13-Octadecenoic acid, methyl ester				
11-Octadecenoic acid, methyl ester				
Methyl stearate	10.516	6.262	298	$C_{19}H_{38}O_2$
Linoelaidic acid methyl ester	10.562	0.864	294	$C_{19}H_{34}O_2$
9,12-Octadecadienoic acid (Z,Z)- methyl ester				
9,12-Octadecadienoic acid (Z,Z)- methyl ester	10.982	0.310	294	$C_{19}H_{34}O_2$
% Total		97.853		

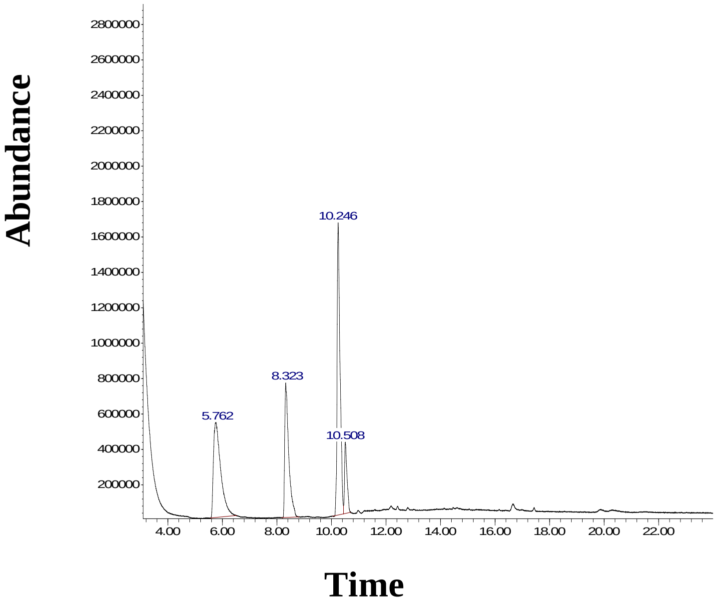


Figure 6: Chromatogram of the observed fatty acid methyl esters at 6 h reaction time.

Table 7: Detected fatty acid methyl esters at 6 h reaction time

Names of fatty acid of methyl esters (FAMEs)	Retention time (min)	FAMEs yield (%)	Molecular weight	Molecular formula
Methyl tetradecanoate	5.762	30.526	242	$C_{15}H_{30}O_2$
Hexadecanoic acid, methyl ester	8.323	23.808	270	$C_{17}H_{34}O_2$
11-Octadecenoic acid, methyl ester	10.246	37.870	296	$C_{19}H_{36}O_2$
(E)-9-Octadecenoic acid, methyl ester				
Cis-13-Octadecenoic acid, methyl ester				
Methyl stearate	10.508	7.797	298	$C_{19}H_{38}O_2$
% Total		99.938		

Conclusions

An *in-situ* transesterification process can be successfully used to generate biodiesel from palm kernel biomass with fuel properties that are within ASTM standards. The yield of biodiesel from palm kernel seed was observed to be significantly influenced by catalyst loading, reaction time and reaction temperature. Optimal conditions for biodiesel yields in this investigation were about 2 wt% catalyst loading and 4 h reaction time.

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