



Original Research Paper

## Production and characterization of biodiesel using palm kernel oil; fresh and recovered from spent bleaching earth

Abiodun Aladetuyi<sup>1</sup>, Gabriel A. Olatunji<sup>1</sup>, David S. Ogunniyi<sup>2</sup>, Temitope E. Odetoeye<sup>2\*</sup>, Stephen O. Oguntoye<sup>1</sup>

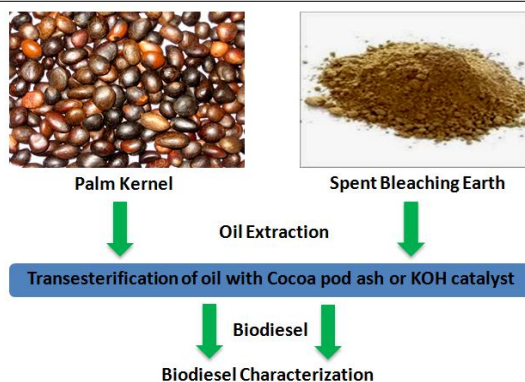
<sup>1</sup>Department of Chemistry, University of Ilorin, P.M.B 1515, Ilorin, Nigeria.

<sup>2</sup>Department of Chemical Engineering, University of Ilorin, P.M.B 1515, Ilorin, Nigeria.

### HIGHLIGHTS

- Utilization of agricultural residue; cocoa pod ash, in place of KOH catalyst for biodiesel production.
- Palm kernel oil was recovered from spent bleaching earth.
- Cocoa pod ash as catalyst led to higher yields of methyl esters from palm kernel oil and spent bleaching earth oil.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Palm kernel oil (PKO) was recovered from spent bleaching earth with a yield of 16 %, using n-hexane while the fresh oil was extracted from palm kernel with n-hexane and a yield of 40.23% was obtained. These oils were trans-esterified with methanol under the same reaction conditions: 100 °C, 2 h reaction time, and oil-methanol ratio of 5:1 (w/v). The cocoa pod ash (CPA) was compared with potassium hydroxide (KOH) as catalyst. The percentage yields of biodiesel obtained from PKO catalysed by CPA and KOH were 94 and 90%, respectively. While the yields achieved using the recovered oil catalysed by CPA and KOH were measured at 86 and 81.20 %. The physico-chemical properties of the biodiesel produced showed that the flash point, viscosity, density, ash content, percentage carbon content, specific gravity and the acid value fell within American Society for Testing and Materials (ASTM) specifications for biodiesel. The findings of this study suggest that agricultural residues such as CPA used in this study could be explored as alternatives for KOH catalyst for biodiesel production.

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### 1. Introduction

Fossil fuel depletion, concern for the environment and unstable crude oil prices have led to intensified search for alternative non-fossils fuels. Oil recovered from spent bleaching earth (SBE) and palm kernel (PK) has been

found to be an alternative source of energy (Knothe et al, 2005). These oils cannot be used directly in internal combustion engine due to two main reasons: low volatility and high viscosity (Knothe et al, 2005; Atabani et al.,

\* Corresponding author at: Tel.: +2347064356000  
 E-mail address: [totetoeye@yahoo.com](mailto:totetoeye@yahoo.com)

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2012). To overcome these problems, vegetable oils or animals fats are reacted with simple alcohol (methanol, ethanol, propanol) to produce fatty acid methyl esters (FAME), fatty acid ethyl esters (FAEE) and fatty acid propyl esters (FAPE) which are also known as biodiesel. The methods to achieve such conversion include pyrolysis, micro-emulsion and trans-esterification (Knothe *et al.*, 2005). Among them, trans-esterification was found to be the best route with minimal engine complication (Knothe *et al.*, 2005; Gupta *et al.*, 2007; Shahid and Jamal, 2011).

Biodiesel is found to be the best substitute for petro-diesel, not only because of its comparable calorific value but also for its several other advantages such as: low toxic emissions, biodegradability, high flash point, excellent lubricity and environmental compatibility (Balat and Balat, 2010; Knothe *et al.*, 2005). Biodiesel has been promoted and reported as a promising long term renewable energy source which has the potential to address net emission of carbon dioxide (CO<sub>2</sub>) to the atmosphere, security concerns and the fluctuating prices of fossil fuels (Alamu *et al.*, 2007a, 2007b ; Balat and Balat, 2010).

Triglycerides used for the production of biodiesel come from various sources: edible oils, non-edible oils, waste/used oils, animal fats and from microorganisms (Meng, *et al.*; 2009). However, there is an economic sense in the use of waste oil feedstocks such as recovered oil from SBE or PKO. Bleaching earth is used in vegetable oil refinery to remove colouring matters, soap, gums, metal (iron, nickel), oxidized compounds and polymers. Bleaching earth retains 20 – 40 % of oils and importantly, the adsorbed oil represents a considerable part of the bleached oil (Ong, 1983; Nursulihatmarsyila *et al.*, 2010). It is estimated that about 600,000 metric tons or more of bleaching earth are utilized worldwide in the refining process based on the worldwide production of more than 60 million tons of oils (Kaimal *et al.*, 2002).

SBE is usually disposed as waste by dumping in landfills without any attempts to recover the oil. Its disposal by incineration, inclusion in animal feeds and concrete manufacturing are sometimes practiced. Overall, SBE due to the substantial oil content in the earth, poses a serious threat to the environment by causing fire and releasing pollution hazards. Hence, it has been suggested that the oil in SBE be recovered and re-used as an alternative energy source which could also reduce the cost associated with refining processes (Nursulihatmarsyila *et al.*, 2010).

Palm kernel oil (PKO) in its fresh form or recovered from SBE can be used as biodiesel. Various studies have been carried out on the conversion of PKO and oil recovered from SBE to biodiesel using conventional homogenous catalysts (i.e. sodium hydroxide or potassium hydroxide) or heterogeneous catalysts e.g. calcium oxide through the trans-esterification reaction (Alamu *et al.*, 2007a; Boey *et al.*, 2009; Huang and Chang, 2010). In the present work, we report the use of cocoa pod ash (CPA) as catalyst in the trans-esterification of PKO and oil recovered from SBE to obtain biodiesel.

## 2. Materials and methods

### 2.1. Materials

The two types of oil used included oil recovered from SBE and neat PKO. The SBE was obtained from Unilever Nigeria Plc., Agbara, Ogun State, Nigeria while the palm kernel seeds used were purchased from the Ajobamidele market at Odo-Oro Ekiti in Ikole local government area, Ekiti State, Nigeria. All solvents including n-hexane, methanol and ethanol were of analytical grade and purchased from Sigma Aldrich (England). The KOH catalyst used for the conversion of the oils to biodiesel was a commercial product obtained from BDH Chemical Limited, Poole, England. Cocoa pod was sourced for in a local cocoa farm at Odo-Oro Ekiti, Ekiti State, Nigeria and was subsequently converted to CPA.

### 2.2. Methods

#### 2.2.1. Cocoa pod ash (CPA) preparation

The cocoa pods were washed with distilled water, air dried and burnt. The burnt cocoa pods were converted into ash in a muffle furnace at a temperature of 550 °C for 50 min. The ash obtained was sieved to obtain a fine powder. The ash was analysed for its mineral content using an atomic absorption

spectrometer (AAS) at International Institute of Tropical Agriculture (IITA) laboratory, Ibadan, Nigeria.

#### 2.2.2. Oil extraction

The oil was recovered from the bleaching earth by cold-extraction with n-hexane solvent. One kg of fresh SBE was extracted with 2 L of n-hexane in a 4-L extracting jar, i.e. 2 L of n-hexane was poured into a 4-L jar containing 1kg of fresh SBE. The jar was covered and allowed to stay at room condition for a period of 72 h. After which the resulting solution was decanted off the jar and filtered using Whatman filter paper. The filtrate (oil + n-hexane) was then concentrated by distillation with the aid of a vacuum pump at room temperature to recover the SBE oil. 160 g of oil was recovered from the 1kg SBE giving an oil yield of 16%. Fresh n-hexane solvent was used for the extraction. The solvent recovered could be re-used, but in this work it was not re-used since the extraction was a one-batch experiment.

In the same way, dried PK was mechanically ground to fine granules. Then, 1.2 kg of the ground sample was cold-extracted with 2 L of n-hexane for 3 d; this was then filtered and concentrated in-vacuo to give 482.67 g palm PKO i.e. 40.23 % extraction yield.

#### 2.2.3. Determination of total alkali in the ash

Determination of the total alkali in the CPA was achieved by titration with acid solution. 0.1M HCl acid solution was firstly standardized using 0.1M Na<sub>2</sub>CO<sub>3</sub>. The standardized 0.1M HCl acid solution was also titrated against 0.1M KOH as a reference basic solution. The standardized 0.1M HCl acid solution was then titrated against the CPA solution. Hence, the molar concentration of equivalent alkali in the CPA which corresponds to that of 0.5 g potassium hydroxide (KOH) used for the conversion catalyst was determined by stoichiometry.

#### 2.2.4. Transesterification procedure

The method described by Alamu *et al.* (2007a) was adopted with some modifications. Fifty g of the oil recovered from SBE was heated for 20 min at 45 °C in a 100 ml, two-neck quick-fit round bottom flask on a water bath. Then, 0.5 g KOH, dissolved in 14 mL methanol was added through the second neck to the heated oil. The reaction mixture (oil-methanol ratio of 5.62:1 mol/mol (5:1 w/v)), was refluxed and stirred at 100 °C for 2 h. Preliminary experiments indicated poor yields at lower temperatures (especially below 60 °C)(data not shown). Therefore, trans-esterification was carried out under reflux at 100 °C that was higher than methanol boiling point. The reaction product was transferred into a separating funnel and allowed to stand for 12 h. After glycerol separation, the obtained FAMES were washed three times with water and then dried over anhydrous MgSO<sub>4</sub>.

While using CPA as catalyst, 1.09 g of CPA (equivalent amount of alkali) was leached in 14 mL of methanol, filtered with Whatman filter paper and also added through the second neck of the flask to the heated oil to produce biodiesel from SBE as described above. Similarly, the same procedure was followed as of PKO. The experiments were carried out in triplicates and the average methyl ester yield was determined. Biodiesel yield was estimated using the following equation (Eq. 1):

$$Y = V_p/V_s \times 100 \quad \text{Eq. 1}$$

Where, Y is the yield (%), V<sub>p</sub> represents the volume of product obtained (ml) and V<sub>s</sub> is the volume of starting material (ml).

#### 2.2.5. Biodiesel fuel characterization

The fuel-related properties of the extracted oil from the SBE, PKO and their corresponding biodiesel products, obtained with KOH and CPA catalysts, were determined according to ASTM standard methods. Comparisons were also made considering the European biodiesel standard EN14214, commercial petroleum diesel, and the ASTM specification for pure biodiesel (B100). The viscosity and specific gravity measurements were made using the Ostwald viscometer thermostatted at 40 °C and thermal-Hydrometer apparatus following the ASTM standards D445 and D1298, respectively. All

measurements were performed in triplicates and the mean values were reported.

The flash points and percentage carbon contents of the oils and their corresponding biodiesels were determined at LUBCON Nigeria Ltd, Ilorin, using flash point apparatus and by pyrolysis following the ASTM standard D93 and D4530, respectively. The density measurements were obtained according to the ASTM standard test method D4052. The oxidative ash contents of the oils and biodiesel samples were determined through sulphated acid test in a muffle furnace at 700 °C until oxidation of carbon was complete in accordance with the ASTM standard test method D874. Acid value measurements were carried out using a modified method of the ASTM D664. The refractive index was determined using a refractometer at the Laboratory of Chemistry Department, University of Ilorin, Nigeria. The FAMES compositions of the biodiesel were determined using a QP2010 PLUS, GC interfaced with a BG mode analytical mass spectrometer. Helium gas was used as carrier gas at 1.2 ml/mm. The MS operating conditions were: ionization voltage 70 eV, and ion source 230 °C. The expected and actual compound on each line of a given retention time was identified by choosing the hit number that corresponded with the mass spectra of the authentic samples and the most frequent retention index number.

### 3. Results and discussion

The percentage yield of the residual oil recovered from EBE with n-hexane was 16%. The oil had a light yellow colour. Also, the percentage yield of the golden yellow oil extracted from PK was 40.34%. These results are comparable to those reported by other researchers (Boey *et al.*, 2009; Huang and Chang, 2010). Table 1 shows the concentrations of the metals present in the CPA compared to the values reported in the literature (Osinowo and Taiwo, 2001; Ayeni, 2011).

**Table 1.**  
Metal contents present in CPA and the values reported in the literature.

Metals	AAS result obtained (%)	Literature value (%)*
Potassium (K)	11.33	16.07
Sodium (Na)	0.09	0.2
Magnesium (Mg)	0.57	1.00
Calcium (Ca)	0.04	5.40
Iron (Fe)	Trace	1.22

\* (Osinowo and Taiwo, 2001; Ayeni, 2011)

The relatively high concentration of potassium (11.3 %) might be due to the use of CPA as potassium based fertilizer in the soil. The metal content values of the CPA obtained were compared with literature values (Osinowo and Taiwo, 2004; Ayeni, 2011). The difference in metal contents may be due to the difference in samples origin. Generally, the study revealed that the use of CPA as a catalyst gave the highest methyl ester yield of 94% compared to the 90% yield obtained through the KOH-catalysed reaction (Table 2).

**Table 2.**  
Percentage yield of methyl ester obtained using KOH and CPA.

Oil type	Catalyst	Biodiesel Sample	Yield (%)
PKO	KOH	PKO-KOH	90.0
	Cocoa pod ash	PKO-CPA	94.0
SBE Oil	KOH	SBE-KOH	81.2
	Cocoa pod ash	SBE-CPA	86.0

The relatively high yield of biodiesel from the use of CPA catalyst is attributed predominantly to high content of potassium (K) as given by the AAS result analysis (Table 1). The oxide/hydroxide of potassium has the ability to co-catalyse the trans-esterification process. Oil recovered from SBE resulted in a lower yield of biodiesel with the use of both catalysts. This might be as a result of impurities introduced to the extracted oil from the SBE for the earth was initially used for refining purpose. Despite this hindrance, CPA catalyst led to a better biodiesel yield of 86% compared to 81.20 % obtained

from KOH-catalysed process. The results obtained demonstrated that CPA is better catalyst than KOH for the production of biodiesel from SBE and PKO. The yield value of 90% for KOH-catalysed PKO biodiesel was quite comparable to the yield of 90 – 92% achieved by Alamu *et al.* (2007a) from the same oil feedstock.

#### 3.1. Fuel-related properties

##### 3.1.1. PKO and SBE oil

The fuel-related properties of PKO and oil recovered from SBE are indicated in Table 3. These properties such as viscosity, density, specific gravity, ash content and refractive index values were higher for PKO compared to those of the oil recovered from SBE. However, the percentage carbon residue, flash point and the acid value of PKO were lower than those obtained for SBE oil (Table 3).

**Table 3.**  
Fuel-related properties of PKO, SBE oil, and their corresponding biodiesels obtained using KOH and CPA catalysts. The ASTM specifications for B100 are also presented.

Properties	PKO	SBE OIL	SBE KOH	SBE CPA	PKO KOH	PKO CPA	Petro-diesel	ASTM method	Biodiesel standard limits
Kinematic Viscosity @ 40°C (cSt)	29.60	27.45	3.97	4.03	4.76	5.48	2.85	D445	1.9 -6.0
Flash point (°C)	-	90	70	130	100	170	64	D93	100 min
Density (g/cm <sup>3</sup> )	0.9720	0.7064	0.7064	0.7367	0.7679	0.8624	0.850	D4052	-
Ash content (w%)	0.1160	0.0154	0.0156	0.0172	0.0040	0.0137	-	D4530	0.80max
Refractive index	1.447	1.338	1.327	1.326	1.443	1.443	-	-	-
Carbon content (%)	27.69	41.08	41.08	33.11	53.03	55.23	-	D874	0.05max
Acid value (mgKOH/g)	3.37	8.90	6.21	4.88	0.80	0.81	-	D664	0.80max

##### 3.1.2. PKO and PKO-biodiesel

Table 3 tabulates the fuel properties of the extracted PKO and its biodiesel products. The results show that all the investigated fuel properties for the PKO had higher values than the corresponding biodiesel product. This is an indication of the conversion of the oil to methyl esters. The PKO methyl esters fuel properties were comparable to those of petroleum diesel and agreed with the ASTM standard values for B100, thereby making the biodiesel product suitable as a substitute for petroleum diesel. The viscosity of PKO (29.60 cSt) reduced to 4.76 cSt for PKO biodiesel catalysed with KOH and 5.48 cSt for CPA-catalysed biodiesel. These reductions correspond to percentage decrease of 83.92% and 81.49%, respectively which will also enhance the biodiesel fluidity in the engine. The flash point (170 °C) of CPA-catalysed PKO biodiesel was greater than that of PKO biodiesel catalysed with KOH. Both these values were greater than the PKO flash point and were in agreement with the ASTM flash point specification for B100.

Ash contents of KOH-catalysed PKO and CPA-catalysed PKO biodiesel products were lower than that of PKO but fell within the ASTM specification for B100. The densities of the PKO methyl esters catalysed with CPA and KOH were lower than the density of PKO. Only PKO CPA-catalysed methyl ester had a density of 0.8624gcm<sup>-3</sup>, greater than that of petroleum diesel with (0.850gcm<sup>-3</sup>). Also, the acid value of PKO methyl ester catalysed with KOH (0.80 mgKOH/g) was lower than that of the corresponding CPA-catalysed biodiesel (0.81 mgKOH/g), but acid value of PKO (3.37 mgKOH/g) was higher than the values obtained for the corresponding biodiesels. High percentage carbon residue was obtained for both CPA-catalysed PKO biodiesel and KOH-catalysed biodiesel.

### 3.1.3. SBE oil and SBE oil-biodiesel

All the examined fuel properties for the oil recovered from SBE had higher values than their corresponding biodiesel products. The viscosity of SBE oil (27.45 cSt) was reduced to 3.97 cSt and 4.03 cSt for KOH and CPA-catalysed biodiesels, respectively. These correspond to a percentage decrease of 85.54% for KOH-catalysed biodiesel and 85.32% for CPA-catalysed biodiesel. High specific gravity value for the SBE oil was obtained compared to its biodiesel product. Flash point of SBE oil (90 °C) was greater than its biodiesel product catalysed with KOH (70 °C) but was less than 130 °C obtained for CPA-catalysed methyl ester. Only SBE oil biodiesel catalyzed with CPA fell within the ASTM specification for B100. The ash content of the SBE oil (0.0154 w%) was less than those of the KOH and CPA-catalysed biodiesels at 0.0156 w% and 0.0172 w%, respectively. SBE oil and KOH-catalysed SBE oil biodiesel possessed the same density value of 0.7064 gcm<sup>-3</sup>. This value was lower than that obtained for CPA-catalysed biodiesel. The density values of SBE oil biodiesels (0.7064 and 0.7367 gcm<sup>-3</sup>) were measured lower than that of petroleum diesel (0.850 gcm<sup>-3</sup>).

### 3.1.4. PKO biodiesel and SBE oil biodiesel

From Table 3, higher viscosity values were obtained for PKO biodiesel with the use of both KOH and CPA catalysts compared to SBE oil biodiesels. Also, high flash point values were obtained for PKO biodiesel with the use of both catalysts compared to SBE oil biodiesel catalysed with KOH. CPA-catalysed SBE oil biodiesel possessed a flash point greater than that of PKO biodiesel obtained by using KOH catalyst. Nevertheless, the flash point value of the latter also fell within ASTM specification for B100 and was higher than the flash point of petroleum diesel. The densities of PKO biodiesels obtained using KOH and CPA catalysts were higher than SBE oil biodiesels. The specific gravity of PKO methyl ester obtained using CPA catalyst was measured higher than that of the SBE oil biodiesels catalysed by KOH and CPA. All specific gravity values obtained fell within the ASTM (D1298) specification for B100.

With the use of both catalysts, the ash contents of the PKO biodiesels were lower than that of the SBE oil biodiesels and were in line with the ASTM (D4530) specification for B100, thereby marking the biodiesel product a suitable substitute for petroleum diesel. The percentage carbon contents of the PKO biodiesels were higher than those of SBE oil biodiesels. Relatively higher acid values of SBE oil methyl ester compared to the values obtained for the PKO biodiesels catalysed with KOH and CPA could be attributed to the high free fatty acids content of the SBE oil and probably due to the impurities picked up by the oil in the course of refining process.

From the economic point of view, the biodiesel raw materials used in this study i.e. CPA and SBE oil could make the biodiesel production more affordable and economical for CPA is an agricultural residue and SBE is an industrial waste. Utilization of these wastes in the production of biodiesel could also have a double waste management effect on the environment while the biodiesel produced is preferred over its petroleum counterpart owing to its environmental advantages.

Notwithstanding, more effort needs to be directed towards optimizing the extraction process of SBE oil to make it more economically viable including recovery and re-use of the solvent used in extraction.

### 3.2. GC-MS analyses

Figures SI to S3 shows the gas chromatogram of the SBE oil biodiesels obtained using CPA. The GC-MS results also confirmed that higher conversion was achieved in biodiesel production from the PKO compared to biodiesel production from SBE oil when the CPA catalyst was used (Tables 4 and 5).

**Table 4.**  
Chemical compositions (%) of the transesterified PKO using CPA catalyst.

Compounds	Composition (%)	Ret index
Methyl octanoate	2.52	1083
Methyl nonanoate	0.34	1183
Methyl decanoate	3.10	1282
Methyl decanoate	0.44	1282
Methyl dodecanoate	19.49	1481
Methyl tetradecanoate	9.97	1680
11-Hexadecenal	1.67	1808
14-methyl pentadecanoate	10.40	1814
Hexadecanoic acid	4.93	1968
Methyloctanoate	0.60	2077
12-Methyl octadecanoate	17.90	2085
Octadecanoic acid	9.74	2167
6-Octadecenoic acid	14.69	2175
2-hydroxyl-1-Methylhexadecanoate	2.18	2498
3-hydroxyl-9-Methyl octadecenoate	2.62	2527

**Table 5.**  
Chemical composition (%) of the transesterified SBE oil using CPA catalyst.

Compounds	Composition (%)	Ret index
Hexanoic acid	0.78	974
Heptanoic acid	1.13	1073
Octanoic acid	2.06	1173
Nonanoic acid	3.68	1272
Decanoic acid	0.80	1380
Decenoic acid	0.29	1380
Heptylhexanoate	5.41	1481
Dodecanoic acid	0.82	1570
Dodecenoic acid	0.30	1578
Methyltetradecanoate	0.02	1680
Tetradecanoic acid	1.75	1769
Pentadecanoic acid	0.27	1869
Methylhexadecanoate	2.00	1878
Hexadecanoic acid	15.63	1968
12-methyloctadecenoate	5.50	2085
9-octadecenoic acid	30.40	2175
2, 3-dihydroxylhexanoate	4.05	2482
Tetradecylhexadecanoate	4.74	3171
2-hydroxyl-1,3-propanoate	12.22	3997
9-methyloctadecenoate	8.17	6149

## 4. Conclusion

This study shows that, CPA as trans-esterification catalyst led to higher yields of methyl esters with both PKO and SBE oil as feedstock. This demonstrates the alkaline strength of the potassium contained in the ash. Hence, biodiesel produced from PKO and SBE oil using CPA could be a potential substitute for petroleum diesel and may be blended with petroleum diesel. All fuel properties examined fell within the ASTM specification for B100. This investigation also suggests that agricultural residues could be explored and utilized in place of conventional catalysts e.g. NaOH/KOH which in turn could improve the economic features of biodiesel production processes.

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## Supplementary Data

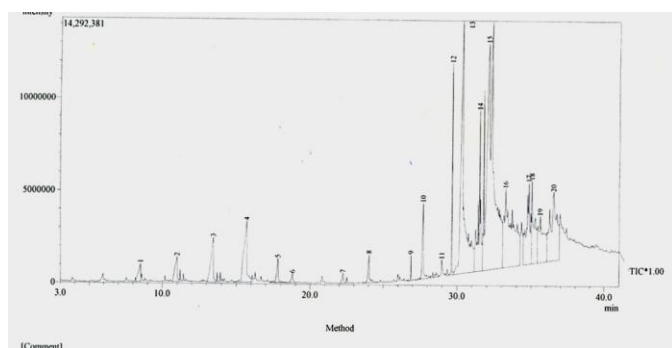


Fig. S1. Gas chromatogram of SBE oil biodiesels obtained using CPA catalyst.

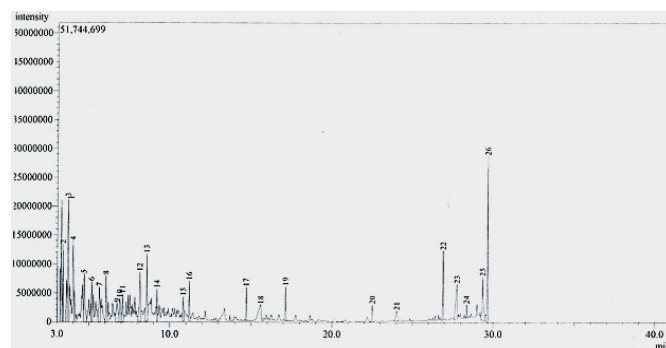


Fig. S2. Gas chromatogram of SBE biodiesels obtained using KOH catalyst.

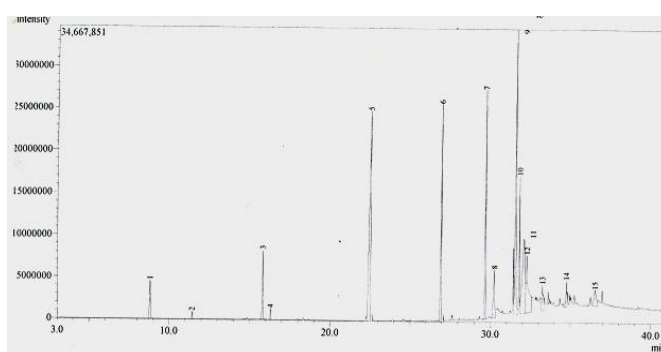


Fig. S3. Gas chromatogram of PKO biodiesels obtained using CPA catalyst.