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In-Situ Transesterification of *Jatropha Curcas* Seed Oil Using Cao Derived from Egg-Shell as Catalyst for Biodiesel Production

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ABSTRACT

Biodiesel production encountered many problems such as low energy balance and food verses energy problems. To solve these problems in-situ transesterification of *Jatropha curcas* seed oil was carried out, with 1:1 (v/v) of n-hexane – methanol co-solvent and calcium oxide derived from egg-shell as catalyst at 65℃ temperature for 2 hours. The soxhlet extraction apparatus was used and the effect of catalyst doses was studied, it was found that all the catalyst used in this research produced fatty acid methyl esters (biodiesel) with acceptable fuel properties with the exception of acid value which is higher than the ASTM biodiesel standard limit. GC/MS results revealed that Jatropha curcas oil biodiesel consists of 9-Octadecenoic acid (Z) - methyl ester, Octadecanoic acid, methyl ester, 9,12-Octadecadienoic acid,13-methyl, methyl ester, 8-Octadecenoic acid, methyl ester (E) and Hexadecanoic acid, methyl ester which indicated that this method is an alternative way of biodiesel production with lower energy input as compared to conventional method.

1. Introduction

Globally fossil fuels consumption increase owing to increased population and industrialization [1]. Depletion of these conventional fuel resources in the near future is a predictable fact [2]. Combustion of this type of fuels would increase greenhouse gases emission mainly carbon dioxide to the atmosphere [3], which may cause climate change [4]. Industrially, conservation of energy and environmental security become global issues [5]. One of the best renewable alternative source of energy is biodiesel [6]. Biodiesel is the mono alkyl ester of fatty acid with similar properties to that of petroleum diesel [7], it is potential alternative fuel to conventional fossil fuel [8]. Transformation of vegetable oil to corresponding fatty acid alkyl esters (biodiesel) would decrease its viscosity close to that of petroleum diesel fuel [9], with similar properties, which may use in compression ignition engine with or without changes [10], the main advantages of using biodiesel as fuel are reliable, low exhaust emissions, [11], biodegradable [12] and it is environmental friendly [13].

Transesterification of triglyceride is the main technologies involves in biodiesel production [14, 15], but conventional transesterification method is an energy-consuming process as compared to in-situ transesterification (which does not need oil extraction step). Biodiesel could be produced from plant seed oil, animal fat and some lipid rich microalgae species [16]. Currently edible oil is the major resources for biodiesel production [17]. Use of this kind fed stocks would make biodiesel expensive, do to food versus fuels competition [18] and contributed largely to the cost of biodiesel production [19]. Therefore, search for alternative method of low energy input for biodiesel production of non-edible seed oil like Jatropha curcas seeds oil could cut both the cost of production and biodiesel price.

Hence, at the same time ease the food vs. fuel crisis that retards the full development of the technology.

Jatropha curcas seeds are non-edible seed crop which has been considered as an oil crop for biodiesel industry. Jatropha curcas does not compete with food crop, and could be able to give competitive seed yields to a small and wasteland, whilst requiring little management [20]. The Jatropha curcas plant grows in harsh conditions and it is not suitable for human consumption [21]. It has a higher calorific value (HHV) and oil yield compared to other non-edible plants [22, 23]. The seeds grown-up in 3-4 months after flowering and once it becomes mature, it will continue producing seeds for 50 years [24]. The plant becomes highly adapted to harsh environment due to its leaf-shedding activity and it has the ability to tolerate a range of climate, but cannot grow in waterlogged land [24]. These have drawn the interest in both government and private sectors for biodiesel production solely due to those characteristics [21].

Homogeneous base catalyst is the common catalyst use for biodiesel industries do to its high activity [25], but it has several disadvantages which include production of wastewater that may pollute the environment and difficulty in products separation [26]. However, in recent year base solid heterogeneous catalyst is use in biodiesel production [27] do to its advantages over a homogeneous catalyst which includes eases product separation, catalyst recovery, less energy and water consumption [28]. The base heterogeneous catalyst is an efficient and active catalyst for biodiesel production [29], among base heterogeneous catalyst, calcium oxide is the most promising one due to its availability, higher base strength and solubility in methanol [30, 31]. Kawashima *et al.* [32] reported that CaO catalyst is economical and highly active catalyst for biodiesel

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production. From an economic point of view, CaO shows potential for biodiesel production as it could obtained from the $CaCO_3$ rich waste such as egg-shell, oyster shell, shrimp shell, mud crab shell, mollusk shell and fish scales. The use of these resources is a kind of innovation in economy of resources, thus making use of waste and improve the economic efficiency [33]. This research work investigated biodiesel production from *Jatropha curcas* seed using in-situ transesterification method using CaO derived from egg-shell as heterogeneous catalyst

2. Materials and Methods

2.1 Materials

The Jatropha curcas seeds were obtained from Ilorin, Kwara State, Nigeria. Seed were de-shelled, impurities were removed by hand-picking and kernel was grounded, using mortar and pestle and sieved with 2.00 mm mesh sieve. The egg shells were collected from Usmanu Danfodiyo University, mini market.

2.2. Experimental Procedure

2.2.1. Catalyst preparation

The egg shells were washed thoroughly and dried at 120 ℃ for 1 hour. It then grounded and sieved with 3″ (76 mm) sieve mesh. It was calcined at 900 °C for 2.50 hours, which later stored in a desiccator for further use.

2.2.2. Transesterification of Jatropha curcas seed oil

Jatropha seed powder (50 g) was weighed and added to the thimble, 100 ml of n-hexane and methanol each was measured and transferred to 250 ml round bottom flask. 5 g of prepared CaO derived from egg shell was added to the solvent mixture ([(v/v) of 1:1] n-hexane to methanol). The round bottom flask was fitted in a soxhlet extractor. The transesterification reaction was carried out at 65 ℃ for 2 hours with constant stirring. At the end of the reaction period, the round bottom flask was removed from soxhlet extractor apparatus allowing cooling to room temperature. The liquid phase was decanted from solid phase (catalyst). Liquid phase was then transferred to separating funnel and allowed to settle for an overnight. The two layers were formed, the lower layer was dark brown in colour contained glycerol and the upper layer was amber yellow in colour contained fatty acid methyl esters. The procedure was repeated for 10 g and 15 g of the prepared CaO catalyst.

The biodiesel produced from 5 g, 10 g and 15 g catalyst loading were identified as sample A, B and C respectively.

All fuel property analysis conducted using ASTM standard methods while the cetane index and HHV estimated using equations reported by [34] and [35] respectively.

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

GC-MS analysis was carried out using Agilent Technologies 5973 Network GC/MS System with 122-5533 capacity column (DB-5ms, 0.25 mm × 30 m × 1. 00 µm). 1µl of the sample was injected onto the column. The helium gas was used as the carrier gas at 1.2 ml/min flow rate. The inlet temperature was maintained at 230 ^{o} C and the oven temperature was programmed initially at 50 ^{o} C for 5 minutes, then programmed to increase to 300 ^{o} C at a rate of 10 ^{o} C ending with 25 minutes, this temperature is held for 15 minutes, total run time was 45 minutes. The MS transfer line was maintained at a temperature of 250 ^{o} C. The source temperature was maintained at 230 ^{o} C and the MS quad at 150 ^{o} C. The ionization mode used was an electron ionization mode at 70eV. Total ion count (TIC) was used to evaluate for compound identification and quantization. The spectrum of the separated compound was compared with the database of the spectrum of a known compound saved in the NIST02 reference spectra library.

Data analysis and peak area measurement was carried out using Agilent Chemstation Software.

3. Results and Discussions

3.1 The physicochemical properties of Jatropha curcas seed oil biodiesel are presented in Fig. 1 below:

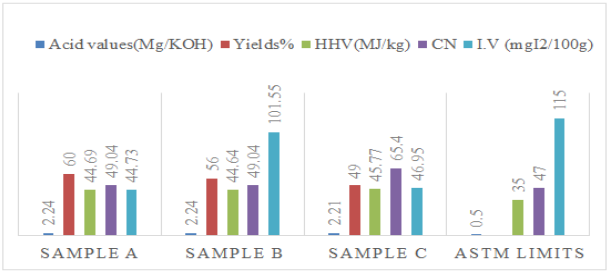

Fig. 1. Jatropha curcas seed oil biodiesel fuel properties.

3.1.1. Catalyst loadings

Catalyst dose is one of the factors affect the transesterification reaction [6] and it has significant impact on biodiesel yields [36]. 5 g loading catalyst (sample A) give the highest yield (60%) of the biodiesel follow by 10 g loading (sample B) with 56% yield while 15 g loading (sample C) give the lowest yield (49%) as shown in Fig., 1. Hence, from the result obtained, it can be deduce that the higher the CaO catalyst used in this study the lower the biodiesel yield and is in good agreement with some literature as reported by Koh and Ghazi. [37]

3.1.2 Acid value

Acid value is expressed as the amount of KOH in mg required to neutralize 1g of fatty acid methyl ester [38]. High amount of free fatty acids leads to high acid value. High acid content might cause severe corrosion in fuel supply system of compression ignition engine [39, 40, 41]. Acid values obtained in this research work were higher than the maximum standards of ASTM D6571 and EN 14214 as the results revealed, but less than the value reported by Ntaganda *et al.* [42]. This indicates that these biodiesel should undergo acid treatment which might decrease it high acidity before being used in compression ignition (C.I) diesel engine to avoid corrosion of fuel distribution system as reported by Silitonga *et al.* [25].

3.1.3 High heating value (HHV)

High heating value (HHV) is an important factor for estimating fuel consumption [1]. It determines the fuel performance in compression ignition engine [43]. In this study the highest catalyst loading (sample C) give highest HHV of 45.77 MJ/kg, while sample A and B has HHV of 44.69 and 44.64 MJ/kg. All the HHV recorded are higher than the value reported by Ong *et al*. [44] and Kartika *et al.* [45] and lower than the value reported by Belewu *et al.* [46]. Hence, the high calorific value of sample C might be due to the higher composition of saturated ester (Hexadecanoic acid, methyl ester). High heating value is not clearly specified in the biodiesel standards ASTM D6751 and EN 14214. However, a European standard for using biodiesel as fuels, EN 14213, specifies a least high heating value of 35 MJ/kg. Thus, HHV recorded in this work exceed the minimum standard recommended by EN 14213. This indicates that Jatropha biodiesel contained high calorific values and could give higher power out-put and small quantity of the fuel might cover long distance drive.

3.1.4 Cetane number

Cetane number is one of the important factor for assessing biodiesel quality, it associated with ignition delay

time, i.e., the time between fuel injection and beginning of ignition [47].

The higher the cetane number, the shorter the ignition delay time and the higher the tendency of the fuel to ignition [48]. Higher cetane number is desirable for ensuring good cold start properties and reducing the development of white smoke. Likewise, low cetane number may lead to knocking [49]. Biodiesel have higher cetane` number than petro-diesel which depend on it fatty acid alkyl esters content [7]. It increases with increasing carbon length and degree of saturated [49, 50]. In this study 15 g catalyst loading (sample C) has a higher cetane number of 65.40 while 5 g and 10 g catalyst loading i.e. sample A and B have the same cetane number of 49.04. According to ASTM D6751 specification, biodiesel should have a minimum cetane number of 47. All the samples A, B and C cetane number conformed to ASTM. Cetane number recorded in this work were higher than the value reported by Kartika *et al.* [45], Satar *et al*. [51] and Ong *et al*. [44]. It can also be deduced that higher cetane number of sample C (15 g catalyst loading) may due to high percentage of saturated compound.

3.1.5 Iodine value

Iodine value is an important fuel property which determines biodiesel quality. Iodine number used to measure the total level of unsaturation of biodiesel. High iodine value level of biodiesel may generate problems such as the cracking and polymerization during combustion, which may lead to formation of soot deposit on engine nozzles [46]. Iodine value also measures stability of biodiesel against oxidation, so high iodine value may lead to low biodiesel storage stability. Sample B (10 g loading catalyst) has the highest iodine value of 101.55 I_2g/100g followed by sample C (15 g loading catalyst) with 46.95 I_2 g/100g and the sample A (5 g loading catalyst) recorded the lowest iodine value of 44.73 I_2g/100g. These results are in good agreement with GC-MS results presented in Table 1. This indicates medium catalyst loading (sample B) has the highest degree of unsaturation which consist of (98.02%) unsaturated compounds followed by higher catalyst loading (sample C) with 56.04% unsaturated compounds, and the low catalyst loading (sample A) has the lowest (53.02%) unsaturated compounds. But one good thing about these results is sample A and B does not contain polyunsaturated compounds even sample C which content 9,12-octadecadienoic acid, 13-methyl methyl ester (polyunsaturated ester) is only 18.89% out of 98.02 % unsaturated compounds present in the biodiesel.This result indicates that the biodiesel produced may not cause severe problems with the compression ignition engine, sample A and B may have high stability period than sample C. The iodine value obtained in this study for different catalyst doses are within EN 14214 and ASTM D6571 less than the value reported by Satar *et al*. [51], Ong *et al.* [44], Kartika *et al.* [45] but higher than the value reported by Adebayo *et al*. [52].

3.2 GC/MS results of Jatropha curcas seed oil biodiesel are presented in Table, 1 and figures 2 – 4, shows GC-MS Chromatograms of fatty acid methyl esters of samples A, B and C.

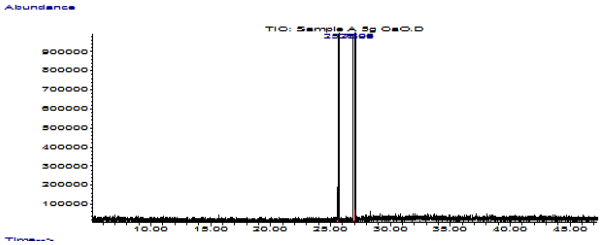

Fig. 2.0. GC-MS Chromatogram of fatty acid methyl esters of sample A.

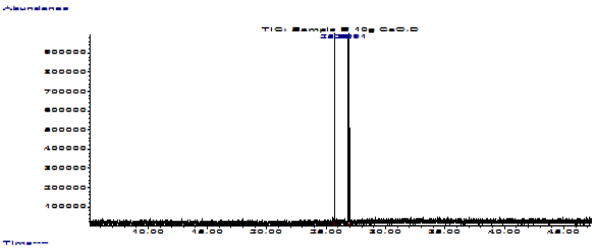

Fig. 3. GC-MS Chromatogram of fatty acid methyl esters of sample B.

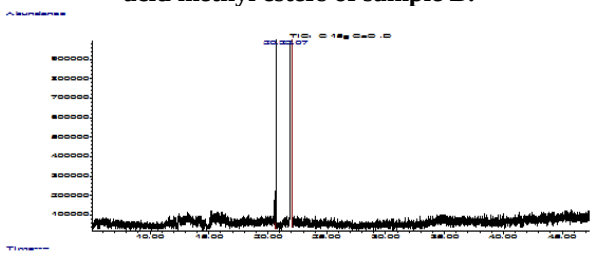

Fig. 4. GC-MS Chromatogram of fatty acid methyl esters of sample C.

3.2.1 GC/MS of Jatropha curcas seed oil biodiesel

The Jatropha curcas seed oil biodiesel were characterised using GC/MS. The fatty acid methyl esters obtained in this research work conformed to the results reported by Liu *et al.* [53]. Sample A (5 g catalyst loading) had the following fatty acid methyl esters composition; 27.28% hexadecanoic acid, methyl ester, 53.02% 9-octadecenoic acid, methyl ester and 19.70% octadecanoic acid, methyl ester. Sample B (10 g catalyst loading) had the following fatty acid methyl esters composition; 1.98% hexadecanoic acid, methyl ester, 18.89%

Table.1 GC/MS results of Jatropha curcas seed oil biodiesel

Sample A	Possible compounds	Peak Area (%)	Saturated FAME (%)	Unsaturated FAME (%)
	Hexadecanoic acid, methyl ester	27.28		
	9-octadecenoic acid (z)-methyl ester	53.02		
	Octadecanoic acid, methyl ester	19.70		
	Total FAME	∑100	46.98	53.02
Sample B				
	Hexadecanoic acid, methyl ester	1.98		
	9,12-octadecadienoic acid,13-methyl, methyl ester	18.89		
	9-octadecenoic acid, (z)-methyl ester	79.13		
	Total FAME	∑100	1.98	98.02
Sample C				
	Hexadecanoic acid, methyl ester	23.97		
	8-octadecenoic acid, methyl ester (E)	56.04		
	Octadecanoic acid, methyl ester	19.99		
	Total FAME	∑100	43.96	56.04

9,12-octadecadienoic acid methyl ester and 79.13% 9-octadecenoic acid, methyl ester while, sample C (15g catalyst loading) had the following fatty acid methyl esters composition; 23.96% hexadecanoic acid, methyl ester, 56.04% 8-octadecenoic acid, methyl ester and 19.99% to be octadecanoic acid, methyl ester. The GC/MS results revealed that biodiesel produced by 10 g catalyst loading (sample B) composed of the highest unsaturated fatty acid methyl ester compares to 5 g catalyst loading (sample A) and 10 g catalyst loading (sample C) and this might be responsible for the low cetane number and high heating value of 10 g catalyst loading (sample B) when compare to other samples as reported by Tariq *et al.* [54] and Sokoto *et al.* [55] Saturated hydrocarbon leads to higher cetane number and higher heating value. It contained good fatty acid methyl ester composition both unsaturated and saturated esters which are desirable for biodiesel fuels.

4. Conclusion

It was found that, it is possible to produce high quality fatty acid methyl esters (biodiesel) via in- situ transesterification of Jatropha Caracas with CaO derived from waste egg- shell catalyst using a soxhlet extraction method. All the tested catalyst dose produced good biodiesel with acceptable fuel properties excepted acid value, it could also be concluded that the biodiesel produced, recorded a good fatty acid methyl ester, in which sample A and B gives the highest percentage of biodiesel yield and fatty acid methyl esters which is desirable for biodiesel and could be used in internal compression engine. Hence, biodiesel produced from Jatropha curcas seed could be a potential replacement for fossil diesel. Therefore, the production and effective usage of Jatropha curcas seed biodiesel will help in protecting environment from the hazardous emissions due to combustion of fossil diesel and would boost the economy of the developed and developing countries.

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