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Production of Biodiesel from *Jatropha Curcas* Oil Using Potassium Silicate Catalyst Synthesised from Rice Husk

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A B S T R A C T

This work explored the application and efficacy of the green catalyst (potassium silicate) derived from rice husk for the production of biodiesel using non edible Jatropha curcas oil. The catalyst was developed by impregnation method using rice husk calcined at 600 oC for 4 h. X-ray Fluorescence (XRF), Fourier Transformed Infrared (FT-IR) and Brunauer-Emmett-Teller (BET) were employed for catalyst characterisation. Results showed that the catalyst contained potassium (17.766 %) and (38.937%) of silicon; and functional groups like siloxane, Si=O, silanol Si-OH were evident. Findings also showed that the synthesised catalyst had a pore size of 22.16 Å, pore volume of 0.003 cm3/g, and surface area of 13.85 m2/g. Further, Jatropha curcas biodiesel with (65%) yield was obtained. The physicochemical properties obtained were found to be within the limits of international standards. The prepared catalyst is an environmental-friendly, inexpensive and a highly efficient base heterogeneous catalyst. Therefore, the application of the synthesised catalyst for biodiesel production would contribute to advocacy on environmental sustainability in response to the global challenge of climate change, and thus to the achievement of Sustainable development goals.

Keywords: Biodiesel, Jatropha, Rice husk, Potassium Silicate

1. INTRODUCTION

The rapid increase in the world's population in the last century coupled with urbanization and industrialization has resulted in a surge in energy consumption, this is because energy is known to be the prerequisite for essential economic growth. This high rate of energy consumption has indirectly increased the rate of energy demand; this demand rate is approximately 10 times the quantity of energy used within the last centuries (Bugaje 2020; Basumatary *et al.,* 2021). Presently, petroleum is the most important source of energy supply and it provides over half of the world's energy supply. Petroleum-based fuels are non-renewable and non-degradable materials that contribute substantially to environmental challenges such as the emission of CO_2 and other poisonous gases into the atmosphere that causes ozone layer depletion and consequently global warming (Singh, *et al.,* 2021; Hangun-Bakir 2016; Basumatary *et al.,* 2020). The dearth of conventional fuels has led to economic inflation and the long-lasting pollution problems that have resulted in climate change have stimulated interest in exploring alternative energy sources of fuel that are renewable, biodegradable, environmentally friendly, and green energy sources (Ganesan *et al.,* 2020; Betiku *et al.,* 2017; Ebtisam *et al*., 2016; Noor *et al*., 2014).

Renewable energy is presently perceived as a vital source of environmentally friendly energy (Mitra *et al.,* 2021). Biofuel (bioethanol, biogas and biodiesel) has been identified as renewable energy that has the capacity for sustainable supply and reduction in greenhouse gas emissions (Bugaje 2020; Musa, 2016). Accordingly, biodiesel as one of the biofuels that is gaining attention in recent times as an environmentally friendly, clean and green source of energy (Basumatary *et al.,* 2021). This environmentally friendly fuel has numerous advantages over petroleum-based diesel. The merits of using biodiesel include better combustion performance, high Cetane number, non-toxicity, renewability, highly oxygenated, free of sulphur and aromatics, biodegradability and low emission of CO, hydrocarbon (unburnt), SOx and particulate matter (Singh, *et al.,* 2021; Basumatary *et al.,* 2018).

Biodiesel is the ester of several fatty acids obtained through the transesterification of vegetable oils with short-chain alcohol (methanol and ethanol) in the presence of a catalyst (Ganesan *et al.,* 2020; Basumatary et al., 2018). The use of edible oil such as Soybean, Rapeseed, Palm, sunflower, cottonseed, peanut and Canola has been previously employed (Kaisan *et al.,* 2016). However, the high cost of biodiesel production associated with the use of edible oil as well as food scarcity has suggested the need for using non-edible oil sources such as Castor, Tung, Neem, Cottonseed, Jojoba and Jatropha for biodiesel production. In addition to these edible and non-edible oils, animal fats, lipids from microalgae, and waste cooking oil have also been used and reported as viable feedstocks for biodiesel production. (Singh, *et al.,* 2021; Basumatary *et al.,* 2020; Ndiaye *et al.,* 2020; Cao *et al.,* 2019)

Biodiesel production using the catalytic transesterification technique is easier, cost-effective, as well improve reaction rate and yield (Balajii and Niju 2019). This catalyst could be homogeneous (acid and alkali), enzymes, or heterogeneous. However, homogeneous and enzyme-catalysed transesterification possess numerous disadvantages. (Sandova *et al.,* 2017; Mendonça *et al.,* 2018). Homogeneous catalysts, although characterised by high biodiesel yield, the catalyst used are not easily separated from the product. Enzyme-catalysed transesterification results in high-purity products (esters), and allows easy separation of products, but the reactions are extremely slow. In addition, these enzymes are expensive thereby making it not

feasible for cost-effective biodiesel production. (Basumatary *et al*., 2021). With the view of resolving the technical problems mentioned above, Heterogeneous catalysed transesterification processes are now been favoured. These catalysts are highly stable, mesoporous, possess strong active sites, result in a fast rate of reaction, are easily recoverable, less corrosive to equipment and are cheap (Basumatary *et al.,* 2018; Otori *et al*., 2018).

Most reported studies on heterogeneous catalytic transesterification of vegetable oil to methyl esters are limited to the use of synthetic/analytical grade chemicals such as CaO, ZnO, Al_2O_3, SiO_2, and zeolite. However, these chemical sources derived heterogeneous catalysts are not feasible for clean and green biodiesel production, this is due to their toxicity and the complexity associated with the production process. Therefore, the recent studies on biodiesel production indicate that researchers are consequently developing heterogeneous catalysts from agricultural wastes due to their availability and abundance in nature as well as their good catalytic properties.

The focus of this study is to synthesize a heterogeneous base catalyst (K_2SiO_3) from rice husk an agricultural waste that is loaded with silica, which is readily available and abundant in nature, and then to use the developed catalyst for the production of biodiesel via transesterification using Jatropha cucars oil.

2. MATERIALS AND METHODS

2.1. Materials

Rice Husk was obtained from a local rice mill at Kpebegi market in Bida, Niger State. Sodium hydroxide (NaOH 98wt%) and hydrochloric acid (HCl 37 wt.%) are purchased by Sigma-Aldrich. The distilled and deionized water used were obtained from the Chemical Engineering Laboratory Federal University of Technology Minna and were used when received.

2.2. Preparation of Rice Husk Silica and Potassium Silicate catalyst

The rice husk was washed with excess distilled water so as to remove all dirt, mud and other soluble materials that were present in the husk. The husk was then dried in an oven at 110 ^oC for 7 h. The dried husk was calcined at 600 ^oC for 4 h, after which the ash was washed with 0.1 M HCl for 2 h. This was then washed with deionized water, dried at 115 °C for 4 h and labelled RHS**.** Potassium silicate was produced using the wet impregnation method. Amorphous rice husk silica (RHS) obtained after the drying process was mixed with 2 M aqueous solution of alkali metal potassium hydroxide. The reactions were performed by mixing KOH and RHS (SiO_2). The obtained mixture was uniformly stirred and heated at 95 °C for 1h. Finally, the product(catalyst) was dehydrated at 120 °C for 7 h to reduce the water content and then calcined at 500 ^oC for 5 h.

2.3 Catalyst Characterization

The synthesized catalyst was characterized for X-ray fluorescence (XRF), Fourier Transformed Infrared (FTIR), and Brunauer-Emmett-Teller (BET). The XRF technique is mainly employed for regular, and non-destructive chemical analyses of rocks, sediments, minerals, and fluids. The chemical/elemental compositions for the synthesized catalyst were analyzed using XRF 1800, Shimadzu, Japan which was operated for 200 s with a current and voltage of 600mA and 50 kV respectively. Fourier transform infrared (FTIR) measurement was carried out using an FTIR spectrometer (Thermo Scientific Nicolet iS50, USA) that operated in the range of 4000–650 cm^{-1}. The Brunauer-Emmett Teller (BET) technique is a physical characterization technique that provides quantitative data on the specific surface area and porosity of the catalyst produced. The BET equipment used was the NOVA 4200e model; this comprises sample preparation and sample analysis stations. Nitrogen gas was utilized for degassing of samples while liquid nitrogen was utilized for analysing the sample. There is an exhaust pump in this equipment that suctions gases from the system into the outer environment.

2.4 Biodiesel Production

Fifteen millilitres (15ml) of jatropha oil was transferred into a round bottom flask mounted on a magnetic stirrer as shown in Figure 3.1. The oil was heated to 65 ^oC, and then 7.5 ml of methanol and prepared catalyst of 5 wt% of oil were added to the heated oil. The mixture was continuously stirred at this temperature (65 ^oC) for a reaction time of 1 hour. The content of the flask was then poured into a separating funnel where the mixture was allowed to stand for 24 hours to achieve proper separation. The biodiesel was decanted and washed with warm water and its yield was determined.

2..5 Characterization of Jatropha curcas oil (JCO) and Jatropha biodiesel.

Properties of Jatropha curcas oil (JCO) and jatropha biodiesel such as density, kinematic viscosity, moisture content, pour point, cloud point, and cetane number were determined by the procedure reported by Biniyam *et al.*, 2015. The FFA and acid values were obtained by the procedure outlined by Bilal *et al.*, 2013, while the Iodine value and saponification value were determined following reported procedures by Basumatary et al., 2014.

3.0 RESULTS AND DISCUSSION

Table 1: Elemental Composition of Developed Catalyst

		Percentage composition
S/No	Element	This Work
1	K	17.766
2	Mg	0.954
3	Si	38.937
4	O	41.273
5	Fe	0.201
6	Ca	0.221
7	Cl	0.015
8	Zn	0.005
9	Sn	0.007
10	Rb	0.002
11	Zr	0.004
12	Ti	0.014

3.1 XRF of Developed Catalyst

The results from Table 1 shows the elemental composition of the synthesized catalyst. It indicates that the major constituent of the synthesized catalyst is potassium (17.766 %), silicon (38.937%) and oxygen (41.273%). The increase in the composition of potassium from 0.32 to 17.766 % is a substantial improvement from the impregnation process. The observable increase in concentration shows the capacity of the RHA to hold potassium molecules within its pores, therefore establishing the potential of the RHA in catalyst developed for transesterification.

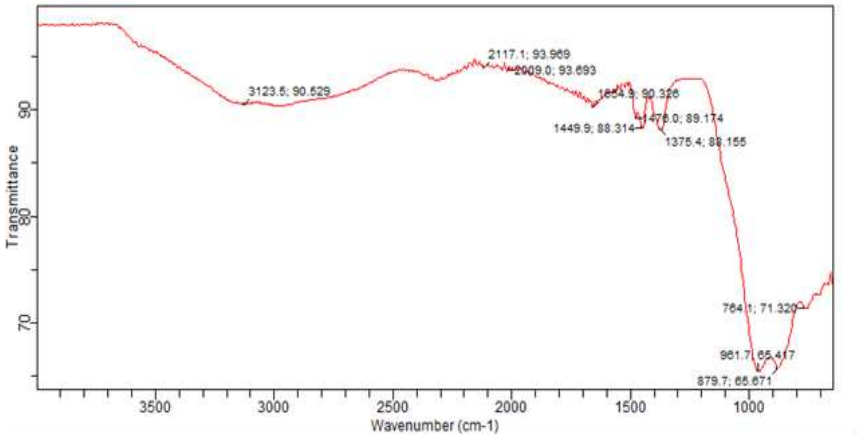

Figure 1: FT-IR for developed catalyst

3.2 FT-IR Characterisation

The FT-IR plot for the catalyst produced is shown in Figure 1. The Figure shows strong broad absorption bands at 794.1cm^{-1}, 879.7 cm^{-1} and 961.7 cm^{-1} that is attributed to asymmetric stretching vibrations of the siloxane bonds (Si-O). Absorption peaks show that the Si–Oi–Si bond, which is found in silica, has formed. (Dhaneswara et al., 2020; Supiyani et al., 2022). The works of Noor *et al.* (2014) and Wuttachai *et al.*, (2016) showed similar peaks for catalyst synthesized from rice husk. The peaks at 1375.4 and 1449.9 cm^{-1} respectively are attributed to Si-O-Si (siloxane) linking structure. Similarly observable peaks at 1478 and 1654.9 cm^{-1} respectively are attributed to -OH bending vibration of silanol groups (Si-OH). The stretching bond vibrations of K–O because of the presence of K_2O is assigned by the peak at 637 cm^{-1} as appeared in the FTIR of the catalyst. This work is in agreement with the reports of Dhaneswara et al., (2020); Supiyani et al., (2022); Wuttachai *et al.*, (2016); and Noor *et al., (*2014). Absorption band at 3123.5 cm^{-1} indicates

stretching frequency of –OH group of H_2O molecules adsorbed on the surface of the catalyst; the presence of water molecules may be due to the combination of OH^- from the KOH with atoms of H^+ present in the RHA.

3.3 Brunauer-Emmett-Teller (BET) Characterisation

The BET analysis result indicate that the pore size of the catalyst is 22.61 A with a pore volume of 0.00346 cc/g and a surface area of 13.85 m^2/g. This result shows that the catalyst produced has low pore volume, small pore size and a large surface area compared to RHA, the reduction in pore volume and pore size/diameter is attributed to K^+ filling the pores of the RHA and this in turn increases the surface area of the catalyst above that of RHA. This work is in agreement with that of Noor *et al*. (2014) who recorded pore size (18.74 A), pore volume (0.003 cc/g) and surface area (0.172 m^2/g) for K_2SiO_3 catalyst from rice husk. The surface area obtained from this work was much higher than that obtained by Noor *et al*. (2014); this may be due to the difference in the source of RH, catalyst preparation method and possibly the difference in catalyst synthesis condition. However, catalyst synthesized is of greater benefit as catalyst with large surface area are known to generally favour biodiesel production.

Wuttachai *et al*., (2016) reported a result somewhat different as the pore volume and surface area was obtained as 1.3 cc/g and 0.95 m^2/g respectively for Na_2SiO_3 catalyst. The difference observed in the works of Wuttachai *et al*. (2016) may be as a result of the metal (Na^+) bonded to the silica. Also, Pandit and Fulekar (2017) produced heterogenous calcium oxide (CaO) catalyst and reported a surface area for catalyst produced as 16.4 m^2/g. Although catalyst produced has larger surface area in relation to the works of Noor *et al*, (2014) and Wuttachai *et al* (2016), results obtained were lower than surface area for the catalyst produced by Pandit and Fulekar (2017) for the transesterification of vegetable oil to biodiesel.

4. Characterisation of Jatropha Biodiesel

The Jatropha oil and the biodiesel obtained were characterised for properties such as acid value, free fatty acid, saponification value, moisture content, cetane, calorific value, viscosity and density. Table 2 gives the summary of the physiochemical properties of jatropha curcas oil (JCO), produced Jatropha curcas biodiesel and its comparison.

Table 2: Physicochemical Properties of Jatropha oil, Jatropha Biodiesel and comparison

Parameter	Unit	Jatropha curcas oil	Jatropha biodiesel	ASTM D 6751	DIN EN 14214
Density	g/cm^3	0.927	0.870	0.875-0.90	0.87-0.90
Viscosity	mm^2/s	37.76	4.56	1.9-6.0	3.7-5.8
Mosture content	%	5	-----	< 0.03	<0.05
Acid value	mgKOH/ g oil	8.47	0.78	<0.80	<0.50
FFA	mgKOH/ g oil	4.24	0.39	-----	-----
Saponification value	mgKOH/ g oil	155	112.8	-----	-----
Iodine value	g/100g	100.2	85.3	------	60-135
Cetane		39	50	------	46-70
Calorific value	MJ/kg	40.37	42.18	------	39-41
Pour point	ºC	5	-2	0	-15 to 13
Cloud point	ºC	11	3	15	-11 to 16

The Specific gravity/density of fuel is an important parameter for injection system; this is because a high specific gravity results in excessive exhaust smoke thus causing environmental pollution and consequently global warming (Abubakar *et al.*, 2016). The density of the biodiesel produced was 0.870 g/cm^3 as shown in Table 2, this value is an indication that transesterification process resulted in a reduction in specific gravity/density of JCO from 0.927 to 0.870 g/cm^3. Although the value obtained for biodiesel falls within the range as recommended by DIN EN 14214, It was slightly lower than ASTM minimum value and in variance with 0.878g/cm^3 reported by Basumatary *et al*. (2021) and 0.89g/cm^3 by Olasheu *et al*. (2015). The variations may be due to the source of feedstock.

The viscosity of a fluid/fuel describes the resistance of a fluid to flow under gravity; it is also a significant property that determines the handling and behaviour fuel. Viscosity increases as molecular weight increase but decreases with increasing temperature. Also, low viscosity is required for favourable combustion characteristic and non-deposition in diesel engines. (Abubakar *et al*., 2016; Singh, *et al*.2021). The viscosity of the JCO was 37.76 mm^2/s

and much lower than 51 mm^2/s reported by Singh, *et al*. (2021). Similarly, Jatropha biodiesel produced in this work was found to be 4.56mm^2/s. The viscosity of biodiesel produced was not within range of acceptable value (1.9-6.0) for diesel fuel; however, it was higher than 3.624mm^2/s reported by Basumatary et al.2021.

The acid value is an indication of the level of free fatty acids (FFAs) present in the biodiesel produced from Jatropha curcus oil. It relates to the fuel's long-term stability and corrosion, thus, the higher the acid value the smaller the amount of the biodiesel produced and vice versa. In this work, an acid value of 8.47 mgKOH/g and 0.78 mgKOH/g oil was obtained for JCO and Jatropha biodiesel produced. The result of the acid value almost in agreement with 0.8 mgKOH/g oil reported by Abubakar *et al.,* (2016) for transesterification of WCO and also within the acceptable range by ASTM.

The saponification value represents the saponifiable unit per unit weight of the biodiesel. The saponification value for JCO and biodiesel were 155 and 112.4 mgKOH/g respectively. This value is in comparable with that reported by Basumatary *et al*. (2018) for Gmelina arborea biodiesel. However, the values were much lower than that reported by Marutani *et al*. (2018) for jatropha. The difference may be attributed to the source of the JCO used as a starting material. This high value obtained implies that the major fatty acids present in the Jatropha oil were all of high molecular mass triglycerides (Akintunde *et al.,* 2015).

Iodine value shows the level of unsaturation of fats and oils. The iodine value recorded for both JCO and biodiesel were 100.2 and 85.3 g/100g respectively. The EN 14214 test standard suggests a maximum value of120 g/100g of biodiesel. The value obtained for this work is lower than 69.11 g/100g reported by Basumatary et al.2021 and only slightly higher than 86.5g/100g reported by Singh et al 2021.

The pour point and he cloud point for this work was obtained as -2 ºC and 3 ºC respectively. Cetane number shows the combustion efficiency of fuel inside a compression engine (Akintunde et al., 2015). The cetane number obtained for this work as shown in Table 2 was 50; however, this is above the minimum for EN 14214 standards for diesel fuel.

5. CONCLUSION

Rice husk is found to contain high percentage of silica; this high quantity of silica makes it an excellent material for heterogeneous solid base catalyst synthesis. Impregenation method was used to synthesized potassium silicate. The characterization of catalyst for XRF, FTIR and BET showed that the catalyst produced contains high percentage of potassium (17.766 %) and silicon (38.937%) Functional groups such as siloxane, Si-OH, and Si=O were present, a pore size of 22.16A, pore volume 0.003cc/g, and surface area of 13.85m^2/g was obtained for BET analysis of the synthesized catalyst. This low-cost catalyst synthesized was used in the transesterification of non- edible Jatropha *carcus* oil to further reduce the cost of biodiesel production, the transesterification process produced a biodiesel yield of 65%. Biodiesel produced showed similar properties as compared to diesel fuel and other bio fuels. The physiochemical properties determined indicates that biodiesel produced had density (0.87g/cm^3), a high Calorific Value (42.18MJ/kg), high cetane number (50), good pour point (-2 ºC) and cloud point of 3 ºC.

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