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Evaluation of the effectiveness of the optimization procedure with methanolysis of waste oil as case study

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ABSTRACT

Local white stone (LWS) identified as Brette Pearl Spar Mable was used to catalyze the transesterification of waste used oil (WUO) to waste used oil biodiesel (WUOB) in the presence of methanol acting a solvent. The conversion of WUO to WUOB was monitored by calcinated LWS (CLWS). Analysis of CLWS demonstrated that potassium (K) is the major active component responsible for the activity of the catalyst in CLWS synthesis, and the transesterification of WUO to WUOB in the presence of methanol shows the catalyst to be suitable for biodiesel production from WUO. To model and optimize the process condition, response surface methodology and Artificial Neural Network was employed in the conversion of WUO to WUOB using CLWS as heterogeneous base catalyst. The modeling was carried out by considering three factors, reaction time (RT), catalyst amount (CA) and the ratio of methanol/oil (M/OR). The optimum conditions that achieved 92.45 (%w/w) for RSM and 98.46 (%w/w) for ANN were RT of 40 min, CA of 6 g and M/OR of 5.5:1. Characterization of the produced WUOB shows that the WUOB can replace conventional diesel when blends. Nevertheless, statistical analysis showed that the conversion using presoaked CLWS, proved to be a suitable heterogeneous catalyst for transesterification of biodiesel.

1. Introduction

Biodiesel has long been brought to the limelight as a stepping stone to the world's source of renewable energy, it was based on this knowledge that researchers have thrived in their search for optimum conversion of agricultural waste into biodiesel as a renewable energy. According to American Society for Testing and Materials (ASTM), biodiesel is best described as the production of long-chain mono-alkyl esters of fatty acids from renewable lipid feedstock, such as animal fats, waste used oil or vegetable oil, by transesterification reaction (Carlos et al., 2011). It is worthy to know that biodiesel significantly represents approximately 78% of CO_2 because it is majorly derived from renewable biomass sources; this fuel diesel produced from renewable lipid feedstock is known for its low emission of pollutants and its biodegradability (Ibrahim and Abubker, 2015).

Over the years, waste used oil (WUO) which is one amongst the many lipid feedstocks has posed a lot of challenges to human existent in terms of its disposal. Indiscriminately discarding waste oil has caused environmental problems which severely contaminate the groundwater, plants, and animals, who in turn can possibly suffocate from the depletion of oxygen and wear and tear of sewer pipes (Conrad and R, 2015). It is as a result of these harmful effects, that there is a necessity for the recycling of WUO and maximally converting it to renewable energy (biofuel). The most efficient and economical means of production of biodiesel is the use of lipids feedstock with a high percentage of free fatty acid and the addition of an excess amount of alcohol (Betiku and Adepoju, 2012).

Optimization and modeling of the biodiesel produced from WUO will further enhance its yield of the biodiesel (Adepoju and Eyibio, 2016), as well as its optimum conversion. However, research has shown that there is a glitch in the results obtained from using a single variable method for modeling and optimization; hence there is the need to combine two or more methods in modeling and optimization of the process variables needed for biodiesel production (Seramen et al., 2010).

Various researchers have used waste oil or waste used oil for biodiesel production and obtained good conversion yields (Chesterfield et al., 2012; Wanodya and Arief, 2013; Kao-Chia et al., 2014; Maurizio et al., 2014; Atilla et al., 2015; Ricky et al., 2015). Since the use of WUO for biodiesel production can conveniently solve the problem of WUO disposal, this paper explores the application of WUO to produce a biodiesel using heterogeneous based catalyst. The catalyst, LWS

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identified as Brette Pearl Spar Mable was pre-soaked in methanol and calcined before it was used as a biobase for biodiesel production. For modeling and its optimization, an integrated (response surface methodology (RSM) and artificial neural network (ANN)) approach was adopted to determine the effects of variables on the optimum conversion of oil to biodiesel.

2. Materials and methods

2.1. Materials

The WUO (*groundnut oil and palm oil*) used for this research was obtained from Akwa Ibom State University Cafeteria, Nigeria. The obtained WUO was dark and dirty because of it used and re-used over times for frying; this oil was dumped at the back of the cafeteria, used to ignite fire and also littered the ground, hereby polluted the soil because of lack of proper disposal. This oil was collected, pre heated to reduce the viscosity and then filtered to remove the dirt's. The physical, chemical and fuel properties of the WUO were determined using standard AOAC methods. The local white stone (LWS), used as a base catalyst was also collected from the Akwa Ibom University Senate building. The LWS was washed with ionized water, oven dried to a constant weight, crushed and then sieved into a 0.5 cm particle size powder. The powder was stored in a cleaned crucible for further processing.

2.2. Method

2.2.1. Characterization of waste used oil (WUO)

The qualities of the oil determine the nature of reaction (esterification/transesterification or both) to which the process route will take. Among these qualities are the dependable factors that determine the reversibility of the reaction, such factors could be the moisture content, the specific gravity, the acid value and the free fatty acid value. These qualities were determined using recommended AOAC, 2000 standard methods.

2.2.2. Gas chromatography-mass spectrometry analysis of WUO

Gas chromatography-mass spectrometer (GCMS), model 19091S-433HP-5MS was employed to determine the fatty acid composition of the WUO before transesterification. The analytical conditions of GCMS for fatty acid detection were 30 mm × 250 µm × 0.25 µm, composed of 5% phenyl methyl silox), operating in Electron Multiplier Volts 1329.412 eV, Helium (99.99%) was used as carrier gas at a constant flow of 1.5 ml/min and an injection volume of 1 µl was employed (Split ratio of 10:1), Injector temperature of 150 °C and Ion-source temperature of 250 °C. The oven temperature was programmed from 35 °C (Isothermal for 5 min), with an increase of 4 °C/min, to 150 °C, for 2 min, then 20 °C/min to 250 °C, for 5 min (Isothermal at 250 °C). Mass spectra were taken at an average velocity of 44.297 cm/s with hold up time of 1.1287 min, a pressure of 11.604 psia and frequency of 50 Hz. The overall total GCMS running time was observed for 45 min.

2.2.3. Catalyst calcination and elemental characterization

Four samples labeled A, B, C, D of LWS grounded powder, each weighs 50 g was measured into 250 ml conical flask, and 100 ml of methanol (BDH Analar: 95%) was added to each flask, shake for 10 min and then filtered. The filtrate was discarded, while the residual cake were calcinated in a Carbolite AAF1100 furnace at 700°C for 4 h for sample A, 5 h for sample B, 6 h for sample C and 7 h for sample D. Furthermore, samples analysis were carried out using a recommended silver standard XRF Spectrophotometer (EDX 3600B) calibrated with ore standard calibration curve. The calcined presoaked powdered CLWS with the highest potassium (K) was used for base catalysed transesterification process.

2.2.4. Base catalyst transesterification of WUO

The properties of WUO showed that the acid value of 0.34 mg KOH/g oil which corresponds to a free fatty acid value (FFA value) of 0.17% was estimated. These observations showed that the oil could be converted directly to biodiesel using catalytic base (known as transesterification). Hence, biodiesel production was carried out using transesterification step as follow. A 500 ml reactor flask was placed on a hot plate with the magnetic stirrer and 200 ml of oil was charged into the reactor for preheating prior to transesterification to remove unwanted moisture content. A mixture of a known weight of base catalyst and a known volume of an alcohol was added to the preheated oil on the hot plate with the magnetic stirrer and the flask was covered with a stopper to prevent alcohol escaping as the reaction proceeded at the desired temperature for complete reaction at a particular time. At the end of the reaction, the resulting mixture was transferred to a separating funnel and allowed to stand for 24 h for glycerol and biodiesel clarity separation. Glycerol was then tapped out of the bottom of the funnel while the untapped biodiesel left in the separating funnel was washed with ionized water to remove the impurities and excess methanol left in the biodiesel. The washed biodiesel was then dried over the heated salt of calcium, allowed to cooled and then filtered to obtained waste used oil biodiesel (WUOB). The final product of the biodiesel yield was then expressed using equation (1).

$$WUOB\ \%\ (w/w) = \frac{Weight\ of\ biodiesel\ produced\ (g)}{Weight\ of\ oil\ sample\ (g)} \times 100 \quad (1)$$

2.2.5. Design of experiment for transesterification of WUO to WUOB

For the purpose of experimental design, variable factors need to be considered in other to model and optimize the transesterification step. Some of the factors are controllable while others are uncontrollable variables (Adepoju and Olawale, 2015). Factors such as reaction time, catalyst amount, reaction temperature and methanol/oil molar ratio are controllable variables that predict the rate determining step as the reaction proceed. In this study, these factors were selected as reaction time (RT): 40–60 (min): X_1; catalyst amount (CA): 4–6 (g): X_2; and methanol/oil ratio (M/OR): 3–8 (v/v): X_3. Meanwhile, a 2^3 full factorial design and central composite design will produce 20 experimental runs (Box and Behnken, 1960), hence to reduce the number of experimental runs, Box-Behnken design (BBD_{RSM}) was employed to generate 17 experimental runs used to study the effects of selected factors on biodiesel yield. To ascertain the visibility of using the design, an artificial neural network was also used and genetic algorithms network (GA_{ANN}) was tested for the interaction of selected factors on the yield. Table 1 showed the selected factors and their levels. For the coefficient of the quadratic model of the response fitting, multiple regressions model was adopted using Statistical software 10 (Stat Inc., Tulsa, OK, USA). Regression analysis and test of significance are the computational intensive process that is best carried out via statistical software version 10; hence the quality of the fitted model was evaluated using test of significance and regression analysis of variance (ANOVA) via equation (2).

$$R_F = \tau_0 + \sum_{i=1}^{k} \tau_i X_i + \sum_{i=1}^{k} \tau_{ii} X_i^2 + \sum_{i<j}^{k} \tau_{ij} X_i X_j + e \quad (2)$$

Table 1
Coding of experimental factors and level.

Selected factors	Symbol	Coded factor levels		
		$-^{\beta}$ (−1)	0	$+^{\beta}$ (+1)
Reaction time	X_1: RT (min)	40	50	60
Catalyst amount	X_2: CA (g)	4	5	6
Methanol/oil ratio	X_3: M/OR	3	5.5	8