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THE DETERMINATION OF HETEROGENEOUS CATALYTIC PERFORMANCE OF PALM OIL FUEL ASH (POFA) IN THE PRODUCTION OF BIODIESEL VIA TRANSESTERIFICATION OF JATROPHA OIL

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ABSTRACT
The high energy demand with the negative environmental impacts of using fossil fuel for energy generations raised question on over dependability on it for sustainable economic growth. Biodiesel tend to be one of the renewable alternative solutions towards the above problems. Biodiesel can be produced through various methods such as transesterification, micro emulsion and pyrolysis The heterogeneous catalytic performance of Palm Oil Fuel Ash (POFA) in the production of biodiesel via transesterification of jatropha oil was investigated. The morphological and chemical properties of POFA were studied using Scanning Electron Microscopy (SEM) and X-ray Fluoroscopy (XRF) respectively. The crude Jatropha oil was transesterified and 0.1 wt%, 0.2 wt%, 0.3wt%, 0.4wt% and 0.5wt% of POFA were used as heterogeneous catalyst. Fourier Transform Infrared (FTIR) has been used to determine the functional group of the samples. SEM indicates the presence porous structure, texture and irregular shape on POFA while XRF shows that it comprises mainly of SiO_2 (79.45 %). The maximum percentage of biodiesel yield was 92.30% at the application of 0.2wt% POFA. This shows that POFA can be used as a heterogeneous catalyst in the production of biodiesel.

Keywords: FTIR, Jatropha, POFA, SEM, XRF

INTRODUCTION

The declining reserves of fossil fuels and the growing environmental concerns have made renewable energy an exceptionally attractive future alternative energy source (Atabani *et al.,* 2012; Ismail *et al.,* 2022).

One of the renewable alternatives fuels to fossil fuels is biodiesel which received much attention worldwide as one of the first alternative renewable fuels that become known to the public. (Litchfied, 2011: Musa *et al.,* 2022).

Biodiesel is a non-toxic, biodegradable, renewable fuel that can be produced from a range of organic feedstock including fresh or waste vegetable oils, animal fats, and oilseed plants. Biodiesel has significantly lower emissions than petroleum-based diesel when it is burned, whether used in its pure form or blended with petroleum diesel. It does not contribute to a net rise in the level of carbon dioxide in the atmosphere and leads to minimize the intensity of greenhouse effect. In addition, biodiesel is better than diesel fuel in terms of sulfur content, flash point, aromatic content and biodegradability (Patil & Deng 2009; Muhammad *et al.,* 2023).

Common sources for biodiesel feedstock include soy bean, sunflower, safflower, pea nut, canola, and palm. Lately there was been growing controversy about the use of potential food sources for the production of fuel. In attempt to address these concerns, researchers have turned their focus from the popular feedstock and are currently investigating the use of alternative, non-food related feedstock such as oil from algae, neem, calabash, jatropha etc. (Jamo *et al.,* 2023; Nura *et al.,* 2023).

The Trans-esterification reaction is a chemical process that involves a number of consecutive reversible reactions between the triglyceride segment of vegetable oil and an alcohol (methanol, ethanol, propanol, butanol) in the presence of a catalyst (acid or base) to produce ester (i.e. biodiesel) and by-product (i.e. glycerin) (Ismail *et al.,* 2022).

Transesterification can be made using homogeneous catalyst (trifluoroacetic acid, sulfuric acid, sodium hydroxide, and potassium hydroxide) or heterogeneous catalyst. However, the removal of homogeneous catalysts is difficult, requiring lengthy phase separation and producing a large amount of wastewater. This problem is not a case for heterogeneous base catalysts, where catalyst separation can be simply filtered out and reused for many production cycles (Sundaramahalingam *et al.,*2021; Ismail *et al.,* 2022).

Jatropha plant (Physic plant) is a drought resistant plant found growing on uncultivated land in most parts of Africa which can be used as hedge plant. It is made up of a green epicarp, a fleshy mesocarp and hard endocarp. It is also known as a diesel fuel due to the facts that it could yield substantial quantity of oil (1000 barrels of oil per year per sq mile) that can be converted to biodiesel without refining (Kumar & Sharma, 2008; Belewu *et al.,* 2010). The oil from these seeds has low acidity, good oxidation stability, low viscosity and good cold properties (Shaaban *et al.,* 2016). Jatropha plant is a multipurpose tree which can serves as a commercial crop in the production of biodiesel due to the fact that it contains hydrocarbons with 16 to 18 carbon atoms per molecule which is greater than that of conventional fossil fuel. In Madagascar, Cape Verde and Benin, Jatropha oil has been used as mineral diesel substitute during the Second World War. This is practical evidence which shows that it can be used as a diesel substitute after transesterification (Ismail *et al.,* 2022; Perera *et al.,* 2022).

Ismail *et al.,* (2022) determine the performance of eggshell as heterogeneous catalyst using jatropha oil via transesterification and obtained 94.3% of biodiesel yield at the application of 0.2wt% egg shell as catalyst using 1:6 oil to methanol ratio in 1hr at 60-65℃. Therefore they concluded that eggshell has high catalytic potential to be used in the production of biodiesel via transesterification of Jatropha oil. The aim of this paper is to investigate the heterogeneous catalytic performance of palm oil fuel ash (POFA) in the production of biodiesel via transesterification of jatropha oil.

MATERIALS AND METHODS

Chemicals

The chemicals and materials used in carrying out this research are; crude Jatropha oil, sodium hydroxide (NaOH), POFA and methanol.

Equipment

The equipment used in carrying out this study are: magnetic stirrer with thermostatically controlled rotary hot plate (IKA C-MAG HS10), thermometer, measuring cylinder, Digital weight balance (AND model GT2000 EC), beakers, conical flask, 24 cm filter paper, funnel, Digital stop watch, sampling bottles, spatula, Fourier transform infrared spectroscopy (FTIR) machine SHIMADZU FTIR-8400S, X-ray fluoroscopy machine ARL QUANT'X EDXRF Analyzer (S/N 9952120) and scanning electron microscope (SEM) machine PHENOM PROX MVE01570775.

Methodology

Elemental Characterization of POFA (XRF)

XRF characterization of egg shell was done at Umaru Musa Yaraduwa University Katsina central laboratory using XRF machine where by X-rays has been directed towards the POFA which leads to the formation of diffraction. This diffraction is later send through focusing slit to analyze the POFA. Photons are diffracted with various wavelengths from the POFA crystal and collected by the detector which transfers them to the computer for analysis.

Morphological Characterization of POFA (SEM)

SEM characterization of POFA was done at Umaru Musa Yaraduwa University Katsina central laboratory using multipurpose Scanning Electron Microscope machine operated at 15 kV which employed secondary signals which revel the surface morphology of POFA.

Transesterification of Jatropha Oil without POFA

60g of the Jatropha oil was measured and pour in to a conical flask and then heated and stirred until it reaches a temperature of 60-65℃ using a hot magnetic stirrer plate, 0.6g of NaOH has been measured using the electronic weight machine and allowed to dissolve in 21ml of methanol and then added to the mixture and allowed it to heat for 60 minutes with the stirrer on the hot magnetic plate. After 60 minute of uniform stirring and heating on the hot magnetic plate maintaining a temperature of 65℃, then it has been poured into the separating funnel through a glass funnel. The mixture has been allowed to cool for about 40 minute. Afterwards, it has been observed to separate into two liquid layers. The upper layer is the biodiesel and the lower layer is triglycrol fatty acid.

Transesterification of Jatropha Oil Using POFA as Catalyst

60g of the Jatropha oil has been measured in 250ml of conical flask and then heated and stirred to a temperature of 60-65℃ on a hot magnetic stirrer plate, 0.6g of NaOH and 0.1wt% POFA was measured using the electronic weight machine and allowed to dissolve in 21ml of methanol and then added to the mixture and allowed it to heat for 60 minutes with the stirrer on the hot magnetic plate. After 60 minute of uniform stirring and heating on the hot magnetic plate maintaining a temperature of 65℃, then it has been poured into the separating funnel through a glass funnel. The mixture has been allowed to cool for about 40 minute. Afterwards, it has been observed to separate into two liquid layers. The upper layer is the biodiesel and the lower layer is triglycrol fatty acid. The same procedure was applied to 0.2wt%, 0.3wt%, 0.4wt% and 0.5wt% POFA.

Infrared Spectral Analysis of Transesterified Jatropha Oil

The FTIR spectral analysis was done at Umaru Musa Yaradua University Katsina central laboratory using FTIR machine which revealed the functional group of the sample.

During the analysis, the sample in a form of thin film was placed between two potassium bromide discs made from single crystals, then a drop of the liquid is placed on one of the disc and the other is placed on top it which leads to the spreads of the sample into a thin film.

The source which is located at the FTIR machine generates radiation which passes through the sample and interferometer and finally reaches the detector. Then the signal is amplified and then converted to digital signal by the amplifier and analog to digital converter respectively. Finally the signal is transferred to a computer in which Fourier transform is carried out.

Percentage of Biodiesel Yield

The percentage of biodiesel yield during the transesterification processes has been determined using the relation below.

$$Percentage\ of\ biodiesel\ yield = \frac{Mass\ of\ biodiesel\ produced}{Mass\ of\ oil\ used}\ X\ 100\% \qquad (1.1)$$

RESULTS AND DISCUSSION

XRF OF POFA

Table 1 shows the elemental composition of POFA which indicates that SiO_2has the highest elemental composition of (79.45 %) followed by K_2O with (7.64%) while Rb_2O with the lowest elemental composition of (1.16%). This indicates that POFA mainly comprises of silicon oxide. In a research conducted by Oyerinde & Bello (2016) similar result was obtained. Therefore it can be used as catalyst in the production of biodiesel.

Table 1: XRF Result

Elements	SiO_2	K_2O	CuO	Al_2O_3	SnO_2	CaO	Rb_2O	LOI
Percentage (%)	79.45	7.64	4.69	2.68	1.45	1.22	1.16	1.71

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SEM of POFA

Figure 1: SEM of POFA at the magnification of 500X.

Figure 1 shows the SEM of POFA at the magnification of 500X. The image shows that POFA has porous structure, texture in nature with irregular shape. Similar result was obtained by (Yaakob, 2013).

Percentage of Biodiesel Yield

The table 2 shows the percentage of biodiesel yield during transesterification process of Jatropha oil with and without POFA as heterogeneous catalyst using equation 1.

Table 2: percentage of biodiesel yield

Amount of catalyst used (wt. %)	0.00	0.10	0.20	0.30	0.40	0.50
Percentage of biodiesel produced (%)	43.30	68.50	92.30	84.40	80.30	74.50

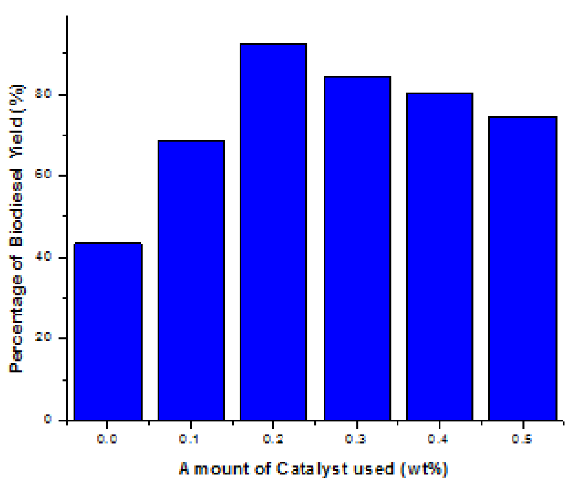

Figure 2: Percentage of biodiesel against amount of catalyst

Table 2 and figure 2 shows the relationship between the biodiesel yield and the catalyst yield during transesterification. It could be seen that the amount of biodiesel increases from 43.3% to 92.3% when the catalyst was added from 0.0wt% to 0.2wt% this was attributed as a result of the relationship between POPA and the jatropha oil. As the catalyst was increased from 0.3wt% to 0.5wt% the amount of biodiesel decreases from 84.4% to 74.5% which is equally attributed as a result of the relationship between the oil and the catalyst. This indicates that POFA can be used as heterogeneous catalyst in the production of biodiesel through transesterification at 0.2wt%.In Corresponding outcome was obtained by Singh & Jaiswal, (2019); Ismail *et al.,* (2022).

FT-IR Spectral Result

Shammeer & Nishath, (2019); Ismail *et al.,* (2022) described that the bands with ester (biodiesel) are the C-O (650 to 1400 cm^{-1}) and C=O (1500 to 1800cm^{-1}).

Figure 3 illustrates the FTIR spectrum plotted for transmittance against the wave number (cm^{-1}) based on the amount of light absorbed by specific molecules present in the transesterified Jatropha oil, the ester was at 1107, and 1740 peaks. This result tallys with the description given by Shammeer & Nishath, (2019); Ismail *et al.,* (2022).

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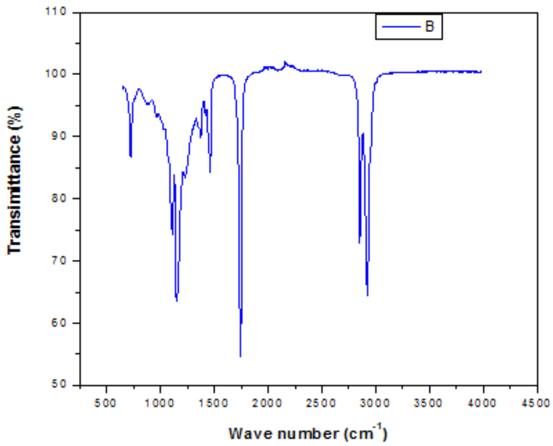

Figure 3: FT-IR Spectra of Transesterified Jatropha oil

Conclusion

This work investigate the influence of POFA as heterogeneous catalyst in the production of biodiesel via transesterification of jatropha oil. The mophorlogical characteristics of POFA studied using SEM shows that it has porous structure, texture in nature with irregular shape. The elemental analysis of POFA shows that it comprises mainly of SiO_2 (79.45 %). The use of POFA as hetrogeneous catalyst via transesterification of Jatropha oil has shown significan increase in the percentage of biodiesel yield from 0.0wt% to 0,2wt%. The maximum biodiesel yield was 92.30% at 0.2wt% . It can be concluded that POFA has heterogeneous catalytic behaviour in the production of biodiel.

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