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Full Length Research Article

INFLUENCE OF EGG SHELL AS HETEROGENEOUS CATALYST IN THE PRODUCTION OF BIODIESEL VIA TRANSESTERIFICATION OF JATROPHA OIL

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ABSTRACT
The increase in energy demand together with the negative global environmental impacts of using fossil fuel for energy generations brings a question on dependability on it for sustainable economic growth. The way out is the use of renewable sources of energy such as biodiesel which has significant advantages over its counterpart (fossil fuel). Biodiesel can be produced through various methods such as transesterification, micro emulsion and pyrolysis. The influence of egg shell as heterogeneous catalysts in the production of biodiesel via transesterification of Jatropha oil was investigated. The physical and chemical properties of the catalyst were studied using scanning electron microscopy (SEM) and Xray fluoroscopy (XRF) characterizations. The crude Jatropha oil was transesterified and 0.1 wt%, 0.2 wt%, 0.3wt%, 0.5wt% and 0.5wt% of egg shell were used as heterogeneous catalyst during transesterification process. Fourier Transform Infrared (FTIR) was used to determine the functional group of the samples. SEM and FTIR characterizations indicate the presence of dispersed particles on the catalyst and ester (biodiesel) on the samples respectively. The maximum percentage of biodiesel yield is 94.3% at the application of 0.2wt% egg shell as catalyst using 1:6 oil to methanol ratio in 1hr at 60-65°C. This indicated that the egg shell has high potential to be used as catalyst in the production of biodiesel via transesterification of Jatropha oil.

Keywords: Egg shell, Jatropha oil, SEM, Transesterification, XRF

INTRODUCTION

The world energy demand is increasing mainly due to economic growth and population expansion. Such high demand together with the negative global environmental impacts of using fossil fuel brings a question on dependability over it for sustainable economic growth. As a way out, the world has to depend more on renewable sources to secure its extensive energy demand (Gebremanian & Marchetti, 2018). Biodiesel is one of the promising renewable sources of energy that has significant advantages over its counterpart (fossil diesel) such as, biodegradability, non-toxicity, release of less greenhouse gases, free from sulfur free from aromatics among others (Gebremanian & Marchetti, 2018; Wang*et al.,*2020).

Biodiesel is the name given to fuel for diesel engines created by the chemical conversion of animal fats or vegetables oil (Indhumathi*et al.,* 2014). Biodiesel can be produced from renewable resources like edible vegetable oils (pea nut, clove, sesame, palm cannel etc.), non-edible vegetable oils (Jatropha, castor, calabash, neam etc.), animal fats, and waste cooking oil (Gebremanian & Marchetti, 2018). Animal fats with high saturated fatty acids which normally exist in a solid form at room temperature may cause problems in the production process, which makes its processing cost to be significantly higher than that of vegetable oils. Thus, vegetable oils are more favorable and have attracted much attention than animal fats (Koh& Ghazi, 2011; Kumar*et al.,*2021).

Currently, edible vegetable oils are the main resources for world biodiesel production (more than 95%). However, the broad utilization of edible oils may prompt some undesirable impacts such as starvation and higher food prices in developing countries. Hence, non-edible plant oils turn out to be exceptionally encouraging option feedstock for biodiesel production (Shaaban *et al.,* 2016; Yap, 2021).

The most widely recognized approach used in delivering biodiesel is transesterification; which 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) (Hoque& Gee, 2013; Pandya*etal.,* 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 (Sudsakorn*etal.,*2017; Sundaramahalingam*etal.,*2021).

Jatropha is a species of flowering plant in the spurge family, Euphorbiaceaea and it is a drought resistant tree. It is a native of tropical America, however now flourishes in numerous parts of the tropics and subtropics in Africa/Asia including Nigeria (Shaaban *et al.,* 2016). It has few nuisances and diseases and will grow under a wide range of rainfall regimes from 200 to over 1500 mm per annum. 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 (Openshaw, 2000; Jamo *et al.,* 2019; Perera *et al.,* 2022).

Buasri *et al.,* (2013) show that the use of cost-effective and environment-friendly catalysts is particularly useful for the production of biodiesel. They used egg shell as catalyst for their research which contains $CaC0_3$after calcination at temperatures of 900°C for 4hours it is then converted to CaO. The optimum conditions yielded a conversion of palm oil of nearly 92 and 94% for duck and chicken eggshell waste-derived catalyst, at a reaction time of 4hours, at a temperature of 65°C, methanol/oil molar ratio 9, and catalyst loading 20 wt.% with pressure 1 atm in glass reactor. The fuel properties of the biodiesel so obtained meet all biodiesel standards. Therefore they concluded that it has potential industrial application in the transesterification of palm oil to FAME.

The aim of this paper is to investigate the influence of egg shell as heterogeneous catalyst in the production of biodiesel via transestrerification of Jatropha oil.

MATERIAL AND METHODS

Chemicals

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

Catalyst preparation

The waste egg shell has been collected from restaurant and washed with tap water in order to remove the contaminated impurities. It is then allowed to dry over night at a temperature between 29°C to 37°C for 2 days and grinded using mortar and pestle.

SEM Characterization of Catalyst (Egg shell)

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

XRF Characterization of Catalyst (Egg shell)

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

Transesterification of Jatropha Oil without Catalyst

60g of the Jatrophacurcas 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 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 Egg Shell as Catalyst

60g of the Jatrophacurcas 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% egg shell 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. The same procedure has been applied to 0.2wt% and 0.3wt% egg shell.

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.

$$Percentage\ of\ biodiesel\ yield = \frac{Mass\ of\ biodiesel\ produced}{Mass\ of\ oil\ used}\ X\ 100\% \qquad (1)$$

RESULTS AND DISCUSSION

Surface Morphology of Egg shell

Figure 1 reveled the SEM of the egg shell at. It indicates the presence of dispersed particle and cloud structure at the magnification of 300X (200μm and 10KV), 500X (100μm and 10KV), 1000X (80μm and 10KV) and 1500X (50μm and 10KV) respectively. This is similar to the result obtained by Kavitha *et al.,* (2019).

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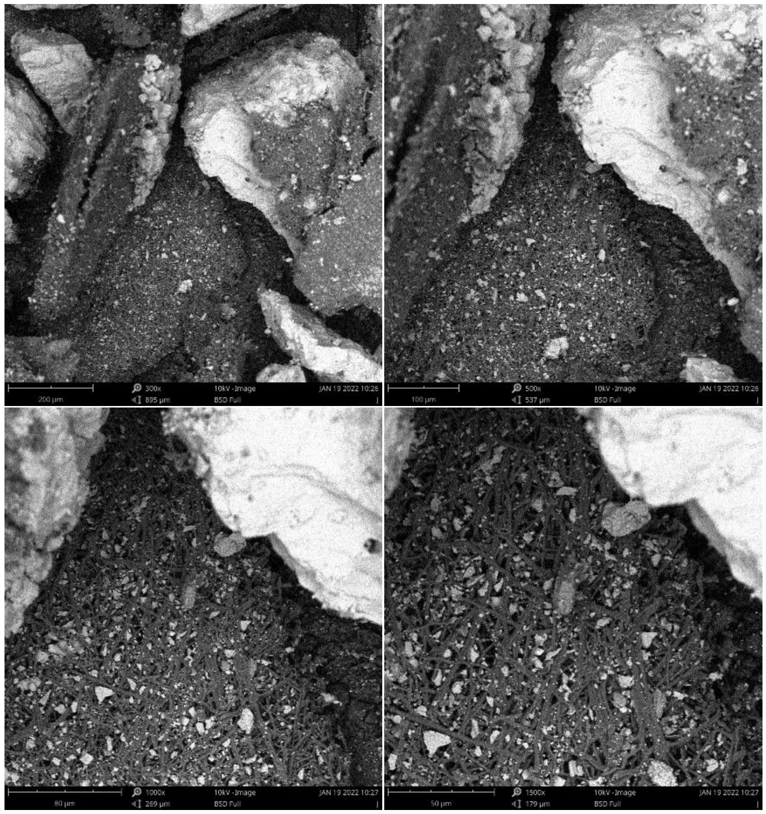

Figure 1: SEM of egg shell at the Magnification of 300X, 500X, 1000X and 1500X

Elemental Analysis of Egg shell

The XRF elemental percentage concentration is presented in table 1. Based on the results presented on table 1, O and Ca have the highest percentage in terms of the elemental concentration on the egg shell, follow by C and Sr. This indicate that the major elemental concentration in the egg shell is Calcite i.e. Calcium carbonate. In a research carried out by (Boro *et al.,* 2012; Mosaddegh & Hassankhani, 2013: Buasri *et al.,* 2013). Therefore it can be used as catalyst in the production of biodiesel and other biomaterials.

Table 1: Percentage Concentration of Element

S/N	Elements	Percentage concentration (%)
1	**O**	43.01
2	**Ca**	40.92
3	**C**	8.41
4	**Sr**	2.12
5	**S**	0.75
6	**Al**	0.54
7	**Pb**	0.61
8	**Ni**	0.31
9	**Fe**	0.25
10	**Cu**	0.24
11	**Mg**	0.23
12	**P**	0.22
13	**Zn**	0.11
14	**Cl**	0.04
15	**Others**	1.23
16	**Loss of Ignition**	1.01

Percentage of Biodiesel Yield

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

Table 1: Percentage of biodiesel yield

Amount of catalyst use (wt%)	0.00	0.10	0.20	0.30	0.40	0.50
Percentage of biodiesel yield (%)	63.00	80.50	94.30	73.50	70.10	66.70

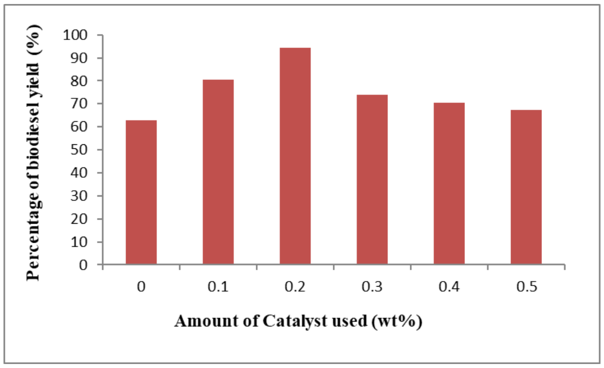

Figure 3: Percentage of biodiesel yield

Based on the results above the percentage of biodiesel yield increased as the catalyst is increased until it riches 0.20wt%, any further increased in the percentage of catalyst it will leads to the decreases in the percentage of biodiesel yield. This indicates that the egg shell can be used as heterogeneous catalyst in the production of biodiesel via transesterification at 0.2wt%. In a research conducted by Boro *et al.,* (2012) and Yusuf *et al.,* (2020) similar result was obtained.

FT-IR Spectral Result

The band with peaks 650 to 1400 cm^{-1}describes C-O bond then 1500 to 1800cm^{-1} described C=O bond while 2700 to 3000 cm^{-1} described C-H stretching and finally from 3000 to 3700cm^{-1}described OH bond. Here C-O and C=O signify the presence of ester or ether group in the sample (Shammeer&Nishath, 2019).

Table 2 is the result of transesterified Jatropha oil FT-IR which revealed the peak number, wave number and the intensity of light absorbed by specific molecules present in the transesterified Jatropha oil.

Table 2: FT-IR of transesterified Jatropha oil

Peak Number	**Wavenumber (cm-1)**	**Intensity**
1	723.10354	83.32947
2	961.65316	89.36337
3	1107.01934	69.36702
4	1155.47473	58.50345
5	1233.74882	79.11228
6	1375.38766	86.50037
7	1461.11643	80.20105
8	1740.66677	47.03421
9	2851.41345	64.92558
10	2922.23286	55.51293

Figure 4: 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 purified Jatropha oil, the ester is at 723.10354, 961.65316, 1107.01934 4, 1155.47473, 1233.74882, 1375.38766, 1461.11643 and 1740.66677 peaks. These indicate that there is an improvement compare to purified Jatropha oil.

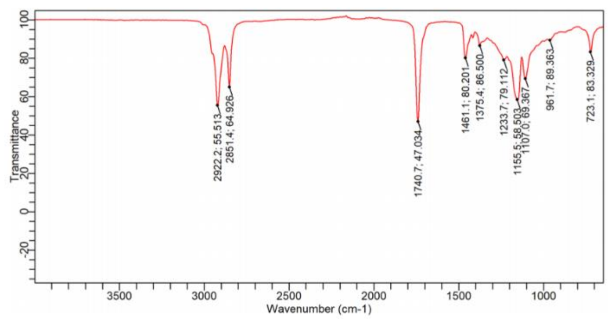

Figure 4: FT-IR Spectra of Transesterified Jatropha oil

CONCLUSION

This work investigate the role of egg shell as heterogeneous catalyst in the production of biodiesel through transesterification of jatropha oil. The mophorlogical characteristics of egg shell studied using scanning electron microscope indicate the presence of dispersed particle and cloud structure. The chemical consituent of egg shell studied through Xray flouroscopy revealed that the egg shell comprises of 43.01% of O, 40.92% of Ca and 8.41% of C which indicate that it is calcium carbonate.The FT-IR result of crude, purified and transesterified Jatropha oil shows that the ester was produced between 723.10354cm^{-1} and 1740.66677cm^{-1} peaks.The use of egg shell as hetrogeneous catalyst during transesterification of Jatropha oil has shown significan increase in the percentage of biodiesel yield from 0.0wt% to 0,2wt%. The maximum biodiesel percentage yield is 94.30% at 0.2wt% egg shell using 1:6 oil to methanol ratio in 1hr at 60-65°C. This indicate a potential catalytic behaviour of egg shell as heterogeneous catalyst. Therefore egg shell can be used as heterogeneous catalyst.

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