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Full Length Research Article

INFLUENCE OF EGG SHELL AS HETEROGENEOUS CATALYST ON THE VISCOSITY OF TRANSESTERIFIED JATROPHA OIL

*[1]H.U. Jamo, [2]U.I. Ismail , [2]K. Yunusa , [2]Sani G. Durimin Iya , [3]E. Tolufase, [3]Olaseni M. Bello, [4]I.Y. Getso

[1]Department of Physics, Kano University of Science and Technology Wudil, P.M.B. 3244, Kano
[2]Department of Physics, Sule Lamido University Kafin Hausa, P.M.B. 3244, Jigawa
[3]Department of Physics, Nigeria Police Academy Wudil, P.M.B 3479, Kano, Nigeria
[4]Department of Physics, Federal College of Education, Kano, Nigeria

*Corresponding Author Email Address: abufatimaassalafi@gmail.com

ABSTRACT
The use of renewable sources of energy such as biodiesel has become one of the solutions towards the problems generated as a result of the use of fossil fuels. Biodiesel can be produced through various methods such as transesterification, micro emulsion and pyrolysis. The influence of egg shell as heterogeneous catalysts on the viscosity of transesterified jatropha oil was investigated. The physical properties of the catalyst were studied using Xray diffraction (XRD) characterizations. The crude Jatropha oil was purified; 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. Brookfield viscometer was used to measure the viscosity of crude, purified, transesterified and transesterified with 0.1 wt%, 0.2 wt%, 0.3wt%, 0.5wt% and 0.5wt% of egg shell as catalyst were measured for comparison. XRD characterizations indicate that the catalyst is Calcite with hexagonal crystal system and $CaCO_3$ as its chemical formula. The FTIR indicates the present of ester (biodiesel) on the samples. The viscosity of the samples decreases as the temperature increases but at 0.2wt% shows significant variation at equal range of temperature. This indicated that the egg shell has influence on the viscosity of transesterified jatropha oil.

Keywords: Egg shell, Jatropha oil, SEM, XRD, Viscosity

INTRODUCTION
Energy is the basic requirement for economic development in any country. Due to this, energy demands have been increasing along with the growth of human population and industrialization. Common sources of energy are petroleum, natural gas and coal from fossil fuels. The negative global environmental impacts of using fossil fuel brings a question on dependability over fossil fuel for sustainable economic growth. One of the solutions towards this problem is the use of renewable sources of energy. Biodiesel is one of the alternative renewable sources of energy that has advantages over fossil diesel such as technological production processes, biodegradability, non-toxicity, release of less greenhouse gases, free from sulfur free, from aromatics among others (Tiwari, 2017; Gebremanian & Marchetti, 2018; Kanwal *et al.,* 2022).
The U.S. Department of Energy defines "biodiesel" as "renewable, biodegradable fuel manufactured from vegetable oils, animal fats of recycled restaurant grease. It is a liquid fuel often referred to as B100 or neat biodiesel in its pure form" (Balasubramanian & Steward, 2019). Biodiesel is an alternative fuel for diesel engines that is produced by chemically reacting a vegetable oil or animal fat with an alcohol such as methanol in the presence of catalyst (usually strong base such as sodium hydroxide or potassium hydroxide) to produces new chemical compounds called methyl esters (biodiesel) (Van, 2005; Çamur & Al-Ani, 2022). Vegetable edible oils (pea nut, clove, sesame, palm cannel etc.) and non-edible vegetable oils (Jatropha, castor, calabash, neam etc.) are more favorable and draw higher attention than animal fats. This is due to the facts that animal fats with high saturated fatty acids which normally exist in a solid form at room temperature cause problems in the biodiesel production process (Koh & Ghazi, 2011; Gebremanian & Marchetti, 2018). Biodiesel production depends mainly on edible vegetable oils due to their high potentiality, but its frequent utilization may prompt some undesirable impacts such as starvation and rise in food prices in developing countries. Therefore, non-edible plant oils such as Jatropha turn out to be the optional feedstock for biodiesel production (Shaaban *et al.,* 2016).
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; Tsedeke *et al.,* 2022;).
Viscosity is the resistance of liquid to flow which is determined by measuring the amount of time taken for a given measure of oil to pass through an orifice of a specified size. Kinematic viscosity is the most important property of biodiesel since it affects the operational performance of fuel injection equipment, particularly at low temperatures. It has been shown that the viscosity oil methyl esters decrease sharply after transesterification processes of biodiesel (Abbas *et al.,* 2010; Atabani *et al.,* 2013). In research conducted by Raja *et al.,* (2011) found out the viscosity of Jatropha biodiesel decreases from 57 to 4.73. The kinematic viscosity in biodiesel is determined using ASTM D445 (1.9 – 6.0 $\mathrm{mm^2 s^{-1}}$) (cSt) (Atabani *et al.,* 2013).
Transesterification as the most recognized method of producing biodiesel can be made using homogeneous catalyst (trifluoroacetic acid, sulfuric acid, sodium hydroxide, and potassium hydroxide) or heterogeneous catalyst. The use of homogeneous base catalysts becomes common during the transesterification process because it yields high percentage of biodiesel in short reaction times. But they are unrecyclable after the reaction and cause alkaline waste. These cause high corrosion on the equipment which eventually leads to the high operational prices. Therefore, there is considerable number of studies on heterogeneous catalysts for the transesterification process in order to solve the problems

associated with homogeneous catalyst. Heterogeneous catalysts exhibit other advantages such as high recyclability, simple recovery, easy separation, lower energy consumption and cleaner operational conditions among others (Lam *et al.,* 2010; Torres *et al.,* 2016).

The use of egg shell as heterogeneous catalyst during transesterification of Jatropha oil has shown significant 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 indicates a potential catalytic behavior of egg shell as heterogeneous catalyst. Therefore eggshell can be used as heterogeneous catalyst (Joshi *et al.,* 2015; Ismail *et al.,* 2022). The aim of this research is to determine the influence of egg shell as heterogeneous catalyst on the viscosity of transesterified Jatropha oil.

MATERIALS AND METHODS

Chemicals

The chemicals, reagent and materials used in carrying out this research are; crude Jatropha oil, sodium hydroxide (NaOH), egg shell, methanol, 64 % citric acid (C 6H8O7, purity: 99.7%), Silicon reagent, activated carbon, acetone and distilled water (H_2O).

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, digital viscometer, sampling bottles, spatula, FTIR machine, XRD machine and SEM machine.

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.

XRD Characterization of Catalyst (Egg shell)

X-ray diffraction characterization of egg shell was done at Umaru Musa Yaraduwa University Katsina central laboratory using XRD machine ARL'XTRA (serial number 197492086) based on constructive interference of monochromatic X-rays and a crystalline sample of egg shell. These X-rays are generated by a cathode ray tube filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the egg shell sample. As the sample and detector are rotated, the intensity of the reflected X-rays is recorded. After the geometry of the incident X-rays impinging the sample of egg shell satisfies Bragg's law ($n\lambda = 2d\sin\theta$), constructive interference occurs and a peak intensity appears. A detector records and processes this X-ray signal and converts the signal to a count rate, which is finally display at the computer monitor.

Purification of Jatropha Crude oil

The crude Jatropha curcas oil has been purified through the following procedure; 200 ml of the Jatropha oil has been measured using measuring cylinder which is then freely-heated to $70^0\ C$ using hot magnet stirrer with thermometer. 0.5g of citric acid has been measured and dissolved into 1.5ml of distilled water and then added to the heated oil sample which is continuously heated and stirred for 15 minutes at $70^0\ C$. 4 ml of 8 % NaOH (by dissolving 8g NaOH in 100 ml of distilled water) has also been added to the oil and continuously heated and stirred for 15 minutes at $70^0\ C$.The mixture has been transferred to the vacuum oven where it has been heated at $85^0\ C$ for 30 minutes.Then it has been taken back to the hot magnetic stirrer and heated at $70^0\ C$ after which a 2g of silicone reagent has been added while it was being heated and stirred for 30 minutes. Then the temperature has been increased to $85^0\ C$ and 4 g of activated carbon has been added to each 100 ml of the oil sample, heated and stirred for 30 minutes. Then the mixture has been separated using separating funnel.

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°C 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 minutes of uniform stirring and heating on the hot magnetic plate maintaining a temperature of 65°C, then it has been poured into the separating funnel through a glass funnel. The mixture has been allowed to cool for about 40 minutes. 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°C 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 minutes of uniform stirring and heating on the hot magnetic plate maintaining a temperature of 65°C, then it has been poured into the separating funnel through a glass funnel. The mixture has been allowed to cool for about 40 minutes. 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%, 0.3wt%, 0.4wt% and 0.5wt% egg shell.

Infrared Spectral Analysis of Transesterified Jatropha Oil

The Fourier infrared spectral analysis was done at Umaru Musa Yaradua University Katsina central laboratory using Fourier transform infrared SHIMADZU FTIR-8400S Spectroscopy 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 discs 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.

Measurement of Viscosity

Viscosity was measured using Brookfield viscometer DV-II+PRO (S/N 621-216) with an operational speed range of 50 rpm with spindle size of 2. The crude Jatropha oil was poured into a beaker then the viscometer was started and angular speed was selected on it. The Viscometer revel the viscosity of the crude Jatropha oil which has been read and recorded. The same procedure has been applied to the purified, transesterified and transesterified with egg shell catalyst Jatropha oil has been measured.

Results and Discussions

XRD of Egg Shell

Figure 1 shows the XRD pattern of egg shell which indicates that it is Calcite with hexagonal crystal system, R-3C space group, 167 space number, 2.71gcm^{-3} density, 367.61 71$X10^6m^3$ volume of cell $CaCO_3$ as its chemical formula. In research conducted by (Kavitha *et al.,* 2019; Qadriyah *et al.,* 2019) similar result has been obtained.

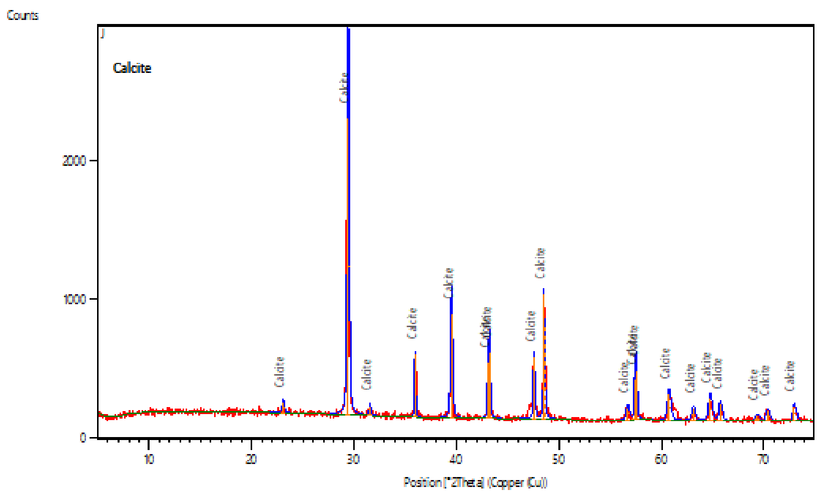

Figure 1: XRD Pattern of Egg Shell

FT-IR Spectral Analysis

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).

Figure 2, 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 is at 723.10354, 961.65316, 1107.01934 4, 1155.47473, 1233.74882, 1375.38766, 1461.11643 and 1740.66677 peaks.

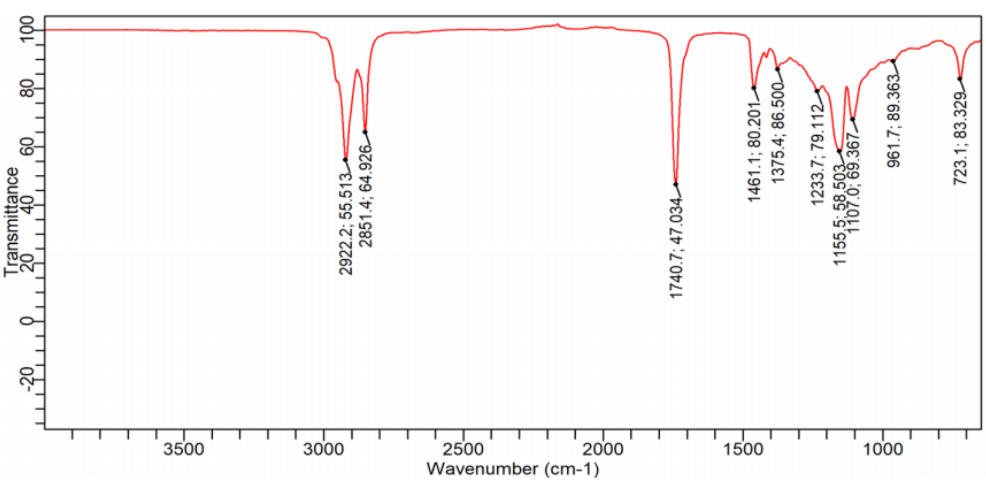

Figure 2: FT-IR Spectra of Transesterified Jatropha Oil with 0.2wt% egg shell catalyst

Viscosity

Table 1 shows the Viscosity of crude Jatropha oil, purified Jatropha oil, transesterified Jatropha oil in centistokes (cSt). The viscosities decrease with the increase in temperature.

Table 1: Viscosity of crude, purified and transesterified Jatropha oil

Temp. (°C)	Viscosity of Crude Jatropha Oil (cSt)	Viscosity of Purified Jatropha Oil (cSt)	Viscosity of Trans-esterified Jatropha Oil (cSt)
20	57.3	40.5	7.5
30	54.2	35.4	6.9
40	51.4	30.1	6.3
50	48.5	25.5	5.7
60	45.9	20.3	5.1
70	42.5	15.0	4.5
80	39.3	10.8	3.9
90	36.2	6.3	3.6
100	33.6	4.1	3.3

The transesterified viscosities of table 1 are within the ASTM limit and also similar to the results obtained by (Raja *et al.,* 2011).
Figure 3 is the graphical representation of table 1 which is the viscosities (cSt) of crude, purified and transesterified Jatropha oil against temperature (°C**).** It equally shows that as the temperature increases the viscosity of the oil decreases.

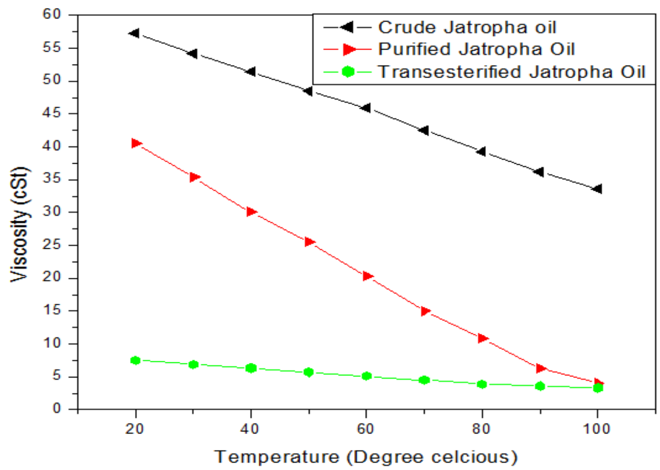

Figure 3: Graph of Viscosity of crude Jatropha oil, purified Jatropha oil and transesterified Jatropha oil

Table 2 shows the Viscosities (cSt) of transesterified Jatropha oil with 0.1wt%, 0.2wt%, 0.3wt%, 0.4wt%, and 0.5wt% of eggshell as catalyst. The viscosities decrease with the increase in temperature.

Table 2: Viscosity of transesterified Jatropha oil with 0.1wt%, 0.2wt%, 0.3wt%, 0.4wt%, and 0.5wt% of eggshell as catalyst

Temp. (°C)	Viscosity of Trans-esterified Jatropha Oil with 0.1 wt% egg shell catalyst (cSt)	Viscosity of Trans-esterified Jatropha Oil with 0.2 wt% egg shell catalyst (cSt)	Viscosity of Trans-esterified Jatropha Oil with 0.3 wt% egg shell catalyst (cSt)	Viscosity of Trans-esterified Jatropha Oil with 0.4 wt% egg shell catalyst (cSt)	Viscosity of Trans-esterified Jatropha Oil with 0.5 wt% egg shell catalyst (cSt)
20	7.3	7.1	7.4	7.2	7.0
30	7.0	6.8	7.0	7.0	6.6
40	6.7	6.5	6.6	6.8	6.2
50	6.4	6.3	6.2	6.6	5.8
60	6.1	6.0	6.0	6.4	5.4
70	5.9	5.7	5.8	6.1	5.2
80	5.7	5.5	5.6	5.8	5.0
90	5.5	5.2	5.4	5.5	4.8
100	5.3	4.9	5.2	5.2	4.6

The viscosities of table 2 are within the ASTM limit and equally similar to the results obtained by (Raja *et al.,* 2011).
Figure 4 represent table 2 in graphical form (viscosities (cSt) of transesterified Jatropha oil with 0.1wt%, 0.2wt%, 0.3wt%, 0.4wt%, and 0.5wt% of eggshell as catalyst against the temperature (°C**)).**

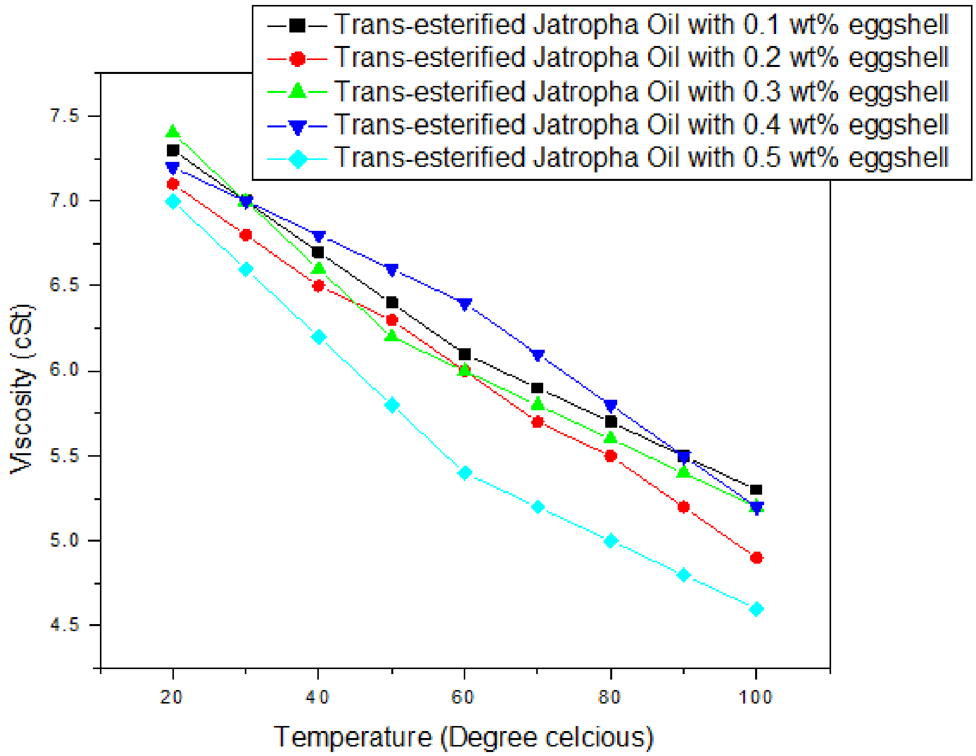

Figure 4: Graph of Viscosity of transesterified Jatropha oil with 0.1wt%, 0.2wt%, 0.3wt%, 0.4wt%, and 0.5wt% eggshell catalyst against temperature

Figure 4 shows that the viscosity varies inversely with the temperature, but at 0.2wt% it varies within a standard range of value. This indicate that the biodiesel produced using Jatropha oil as a feedstock and eggshell as homogeneous catalyst at 0.2wt% can be used as diesel fuel at different parts of the world (temperate and tropic regions).

CONCLUSION

This work investigate the influence of egg shell as heterogeneous catalyst on the viscosity of transesterified jatropha oil. The crystal properties of the egg shell studied through Xray diffraction revealed that it is Calcite with hexagonal crystal system and $CaCO_3$ as its chemical formula. 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 viscosity of the samples decreases as the temperature increases, but at 0.2wt% shows variation at standard interval for the change in temperature. This indicate a potential catalytic behaviour of egg shell as heterogeneous catalyst at 0.2wt%. Therefore biodiesel produced using jatropha as feedstock and egg shell as heterogeneous catalyst at 0.2wt% via transesterification can be used in dieasel engine. The research shows that egg shell shell has influence on the viscosity of transesterified jatropha oil at 0.2wt%.

REFERENCES

Abbas, K.A., Abdulkarim, S.M., Saleh, A.M. and Ebrahimian, M., 2010. Suitability of viscosity measurement methods for liquid food variety and applicability in food industry-A review. *Journal of Food Agriculture and Environment, 8*(3-4), pp.100-107.

Atabani, A. E., Silitonga, A. S., Badruddin, I. A., Mahlia, T. M. I., Masjuki, H., and Mekhilef, S, 2012. A comprehensive review on biodiesel as an alternative energy resource and its characteristics. *Renewable and sustainable energy reviews*, *16*(4), pp.2070-2093.

Balasubramanian, N. and Steward, K.F., 2019. Biodiesel: history of plant based oil usage and modern innovations. *Substantia, 3*(2), pp.57-71.

Belewu, M., Adekola, F., Adebayo, G., Ameen, O., Muhammed, N., Olaniyan, A., Adekola, O. and Musa, A., 2010. Physico-chemical characteristics of oil and biodiesel from Nigerian and Indian Jatropha curcas seeds. *International Journal of Biological and Chemical Sciences, 4*(2). pp.524-529.

Çamur, H. and Al-Ani, A.M.R., 2022. Prediction of oxidation stability of biodiesel derived from waste and refined vegetable oils by statistical approaches. *Energies, 15*(2), p.407.

Gebremariam, S.N. and Marchetti, J.M., 2018. Biodiesel production through sulfuric acid catalyzed transesterification of acidic oil: Techno economic feasibility of different process alternatives. *Energy Conversion and Management, 174*, pp.639-648.

Ismail, U.I., Jamo, H.U., Usman, A., Magama, A.A., Baban, K.Y., Nura, I., Turaki, S., Musa, F.U. and Gwadabe, S.H., 2022. Influence of egg shell as heterogeneous catalyst in the production of biodiesel via transesterification of Jatropha oil. *Science World Journal, 17*(2), pp.191-195.

Joshi, G., Rawat, D.S., Lamba, B.Y., Bisht, K.K., Kumar, P., Kumar, N. and Kumar, S., 2015. Transesterification of Jatropha and Karanja oils by using waste egg shell derived calcium based mixed metal oxides. *Energy Conversion and Management, 96*, pp.258-267.

Kanwal, S., Mehran, M.T., Hassan, M., Anwar, M., Naqvi, S.R. and Khoja, A.H., 2022. An integrated future approach for the energy security of Pakistan: Replacement of fossil fuels with syngas for better environment and socio-economic development. *Renewable and Sustainable Energy Reviews, 156*, p.111978.

Kavitha, V., Geetha, V. and Jacqueline, P.J., 2019. Production of biodiesel from dairy waste scum using eggshell waste. *Process Safety and Environmental Protection, 125*, pp.279-287.

Koh, M.Y. and Ghazi, T.I.M., 2011. A review of biodiesel production from Jatropha curcas L. oil. *Renewable and sustainable energy reviews, 15*(5), pp.2240-2251.

Kumar, A. and Sharma, S., 2008. An evaluation of multipurpose oil seed crop for industrial uses (Jatropha curcas L.): a review. *Industrial crops and products, 28*(1), pp.1-10.

Lam, M.K., Lee, K.T. and Mohamed, A.R., 2010. Homogeneous, heterogeneous and enzymatic catalysis for transesterification of high free fatty acid oil (waste cooking oil) to biodiesel: a review. *Biotechnology advances, 28*(4), pp.500-518.

Raja, S. A., 2011. Biodiesel production from jatropha oil and its characterization. *Res J Chem Sci, 1,* pp.81-87.

Shaaban, W., El-Shazly, A.H., Elkady, M.F. and Ohshima, M., 2016. Biodiesel production from jatropha oil in a closed system. In *MATEC Web of Conferences* (Vol. 69, p. 02002). EDP Sciences.

Shameer, P.M. and Nishath, P.M., 2019. Exploration and enhancement on fuel stability of biodiesel: A step forward in the track of global commercialization. In *Advanced biofuels* (pp. 181-213). Woodhead Publishing.

Tiwari, A. K., Kumar, A., and Raheman, H., 2007. Biodiesel production from jatropha oil (Jatropha curcas) with high free fatty acids: an optimized process. *Biomass and bioenergy, 31*(8), pp.569-575.

Torres-Rodríguez, D. A., Romero-Ibarra, I. C., Ibarra, I. A., and Pfeiffer, H., 2016. Biodiesel production from soybean and Jatropha oils using cesium impregnated sodium zirconate as a heterogeneous base catalyst. *Renewable Energy, 93*, pp.323-331.

Tsedeke, R.E., Minale, M. and Tedila, A., 2022. Seed Oil Content and Variability of Some Provenance of Jatropha Curcas Population in Ethiopia. *Advance in Environmental Waste Management & Recycling, 5*(3), pp.284-287.

Van Gerpen, J., 2005. Biodiesel processing and production. *Fuel processing technology, 86*(10), pp.1097-1107.