



Research Article

Production of liquid fuel from co-pyrolysis of jatropha cake with tyre waste

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ARTICLE INFO

Article history

Received: 17 November 2021

Revised: 15 January 2022

Accepted: 09 March 2022

Key words:

Co-pyrolysis; Feedstock materials; Fixed bed reactor; Jatropha cake and tyre waste

ABSTRACT

This paper introduces the technique that can be used to produce liquid fuel in high quantity, and the technique is called co-pyrolysis technique. Co-pyrolysis is a process which involves two or more materials as feedstock. To the best of my knowledge, there have not been any research about the co-pyrolysis of Jatropha seed cake with tyre waste. The feedstock materials were Jatropha cake and tyre waste. The characterization of the feedstock materials were carried out based on proximate and ultimate analysis. The Jatropha cake with tyre waste were in particle form and were pyrolyzed in an externally heated fixed bed reactor with nitrogen as an inert gas. The reactor was heated by means of electrical furnace. The products of the experiment were: liquid fuel, char and gas. The ratio of jatropha cake to tyre waste materials were 1:1, 1:2 and 1:3 respectively. The parameters that has more influence on the production of liquid fuel yields includes: feed ratio, temperature and reaction time. The maximum liquid yield obtained from the co-pyrolysis of Jatropha cake with tyre waste was 68.0 wt% (that is at the parameters of: feed ratio 1:1, temperature 500°C and reaction time of 45 minutes).

Cite this article as: Babajo SA. Production of liquid fuel from co-pyrolysis of jatropha cake with tyre waste. Environ Res Tec 2022;5:2:111–118.

INTRODUCTION

Fossil fuels such as petroleum and natural gas are predicted to phased out after 2042, and only the coal reserves that will be available until at least 2112 [1]. This effect has made some researchers to put more effort in order to find solutions by utilizing alternative energy. Biomass is very abundant worldwide and can be easily found in diverse forms such as agricultural residues, wood residues, dedicated energy crops, and municipal solid waste [2]. Biomass based on its availability and its status as a waste product is one of the prime sources of renewable energy worldwide.

Biomass receives more attention in the utilization of energy source in Nigeria, contributing about 78% of Nigeria's primary energy supply [1, 3]. Biomass originates from plants or their by-products that are produced completely or partly by photosynthesis such as forestry residues, agricultural bi products or animal wastes that can be used as a source of energy [4]. The use of biomass as an energy fuel source also helps the environment because it has been recognized as a carbon neutral energy source. The conversion of biomass sources into energy fuels was achieved in several ways, such as thermal, biological, and physical methods. In thermal conversion, pyrolysis is the most

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promising processes that can convert biomass to various types of products such as liquid, char and gas. This technique is widely recognized as an environmentally friendly method, because no wastes are produced during the process. The process has also received more attention because it can produce liquid yield of up to 75 wt% with conditions of moderate temperature (500°C) and short hot vapor residence time [2]. The liquid fuel obtained from the pyrolysis process has the potential to be used as fuels or feedstock for many commodity chemicals. The research to produce liquid fuel via the pyrolysis of biomass has been conducted since the last four decades. In 1972, the energy crisis has pushed researchers to put more attention to maximize the production of pyrolysis oil by minimizing the byproducts of char and gases [5]. One of the best use of pyrolysis was achieved in the 1980s [6]. This co-pyrolysis technique has successfully led to several improvements, such as the high yield of liquid fuel production. Although the issue associated with liquid fuel quantity has been addressed, the improvement in liquid fuel quality still requires further research. Currently, many researchers put efforts in finding the suitable technique to produce high-grade pyrolysis oil and to explore more new variations of biomass that can be used as feedstock in the pyrolysis process. The liquid fuel produced from the pyrolysis of biomass contain high level of oxygen content and can cause many problems, such as low calorific value, high viscosity and corrosion problems. The current research finding showed that the technologies to eliminate the oxygen content are still expensive and can cost more than the oil itself [7]. Therefore, the sustainability of this research seems necessary to overcome this cost and to improve the quality of pyrolysis oil that is expected to compete with fossil-based liquid fuel.

Around 1.5 billion tires are produced worldwide every year, which will eventually be categorized or interpreted as waste tires [8, 9]. Waste or scrap tires are known to have a significant impact on the increasing the urban waste stream and it has become a major threat to the environment. Approximately 64% of waste tires are sent to landfill or illegally dumped or stockpiled, with only 13% of them being recycled [10]. In landfills, the waste or scrap tires are not easily degraded, but tend to float to the top over time due to the trapped gases contained, thus breaking landfill covers. The incineration of waste or scrap tires requires the more expensive control system of air emissions because this process produces toxic gases, which contain carcinogenic and mutagenic chemicals. More attention and treatment were needed to tackle waste or scrap tires, and pyrolysis has been found to be a technically feasible way to treat tires and recover valuable products. According to Abnisa et al. [11], palm shell and tyre waste materials were mixed in a ratio of 1:1. The palm shell material alone was heated at a temperature of 500°C, the liquid fuel and calorific value obtained from the palm shell alone was 46.13 wt% and 11.94MJ/Kg.

The second experiment was conducted by mixing the two materials and heated at same temperature of 500°C, the liquid fuel yield and calorific value obtained were 61.63 wt% and 38.01MJ/Kg respectively.

Alias et al. [12] carried out an experiment on pinewood and tyre waste materials, the pinewood material alone was heated at a temperature of 500°C. The liquid fuel and the calorific value obtained from the pinewood alone was 32.00 wt% and 21MJ/Kg respectively. While the mixture of pinewood and tyre waste materials were mixed in a ratio of 1:1 and were also heated at same temperature, the liquid fuel yield and the calorific value obtained were 53 wt% and 45MJ/Kg respectively.

According to Cao et al. [13], Waste tyres without wire steel were mixed with empty fruit bunches with a ratio of 1:1. The experiment was performed using a fixed bed reactor. Co-pyrolysis was carried out under a nitrogen atmosphere at a temperature of 500°C. Pyrolysis oils were collected in a condenser. The products of liquid obtained was 42.80 wt%. The liquid product was significantly decreased when the empty fruit bunches were pyrolyzed alone without being mixed with waste tyre. These showed that an increase of tyre waste in biomass during pyrolysis increases the liquid fuel yield and the calorific value of the fuel.

Wastes of tires are considered to be potential sources to use as a co-feed in co-pyrolysis to produce liquid fuel [14–16]. The materials having much energy content, the sources were specifically easy to find and readily available in huge amounts in all countries around the world. Pyrolysis of tire waste with other biomass (Jatropha cake) wastes will encourage the creation of innovative new concepts in waste management, energy security enhancement, and environmental concerns. Therefore, it is significant to note that, the development of the co-pyrolysis technique to produce liquid fuel is applicable in most countries of the world.

There is growing interest among researchers in the use of waste tyres as a fuel source through the process of pyrolysis. As a research output, several comprehensive reviews on various aspects of waste tyres pyrolysis for liquid production have been published [17]. In co-pyrolysis, many studies of pyrolysis of waste tyres mixed with other materials have been carried out. However, studies which focused on the pyrolysis of waste tyre/woodbased biomass blends are currently still limited. Therefore, some effort should be made to examine the existence of synergistic effects when employing various pyrolysis conditions for the pyrolysis of waste tyre/biomass.

The aim of this study is to produce liquid fuel from jatropha cake with tyre waste via co-pyrolysis technique. Jatropha normally grows in an arid land that means it can grow in area where there is no sufficient or lack of availability of water. Many researchers tried to find alternative fuels by converting jatropha oil to biodiesel for several purposes.

Jatropha cake is non edible materials which are produced in large quantities while producing biodiesel. The jatropha cakes were dumped as a waste after producing the biodiesel.

MATERIALS AND METHODS

Materials

The materials used in this research study includes;

- i. Jatropha Curcas Seed Cake: Jatropha curcas seed cake was selected for this study. It was obtained at Technology Business Incubation Center (TBIC), Kano, in Kano state. It was used as one of the feed stock in this research.
- ii. Shredded rubber tire was selected as tire waste in this study. It was used as one of the feed stocks in the co-pyrolysis process because of its availability, and are not easily degraded, which may become a major threat to the environment. It was obtained at Kofar Wambai, Kano market, Kano state. The waste or scrap tire was shredded into smaller sizes using an iron saw and the iron reinforcement content in the tire was removed. The sample was allowed to dry. The passenger car tire was used as scrap tire in this study.
- iii. Thermocouple: This is a sensor that is used to measure temperature of a sample. The thermocouple is a sensing device that detects the temperature inside the reactor produced by the electric heater. The thermocouple used for this research study was for measuring the temperature inside the reactor. The thermocouple comprises of a wire which was placed in the fixed bed reactor in order to detect the temperature inside the reactor. The model of the thermocouple used was PSI-TTM 1 model.
- iv. Temperature Controller: A temperature controller is a device that controls the temperature of the electric heater. It was used to control the temperature inside the fixed bed reactor. The model of the temperature controller used in this research was TEC201 Model.
- v. Test Sieves: The test sieve that was used for this study was a standard sieve that will sieve the particle of the material or feed stock of not more than 2 mm. It was used for sieving the feed stocks to a desired particle size.
- vi. Weight Balance: This is a device to measure weight. The type of weight balance that was used for this study was Digital of (TCS-100-ZE11) model. It was used for determining the weight of the material or feedstock.
- vii. Stop Watch: The type of stop watch that was used for this research was Digital Stop watch of N1280 model. The stop watch was used for measuring or determining the time that will take a feedstock materials to pyrolyzed.

Methods

Jatropha seed cake and tyre waste were sorted, sun dried and shredded into smaller size. Proximate and ultimate

analysis of a sample of the prepared feedstock was undertaken based on American Society for Testing and Materials (ASTM).

Proximate Analysis of the Feedstock Materials

The proximate analysis is defined as the loss in weight of the feedstock materials (Jatropha curcas seed cake and tyre waste) in terms of moisture content, ash content, volatile matter and fixed carbon. The proximate analysis was carried out in accordance with ASTM D3172–73 [18].

Determination of Ash Content (ASTM D 2939–07)

The samples (Jatropha curcas seed cake and tyre waste) were weighed and burnt in a furnace at 350°C and was left for some time in the furnace and the ash was later weighed. The ash content was determined on dried basis as follows:

$$\text{Ash content (wt \%)} = \left(\frac{W_3 - W_1}{W_2 - W_1} \right) \times 100 \quad \dots \quad (1)$$

Where W_1 =weight of empty crucible, W_2 =weight of crucible and sample and W_3 =weight of crucible and sample after heating.

Determination of Moisture Content (ASTM D 4643–18)

Percentage of moisture content (MC) of the samples was determined by calculating the loss in weight of sample using oven drying at temperature of 105°C–115°C until the weight of the sample was constant, after 60 minutes oven dried weight was obtained. Moisture content was then determined as thus;

Calculation;

$$\text{Moisture content (wt \%)} = \left(\frac{W_5 - W_6}{W_5 - W_4} \right) \times 100 \quad \dots \quad (2)$$

Where W_4 =weight of empty crucible, W_5 =weight of crucible and liquid fuel, and W_6 =weight of crucible and liquid fuel after heating.

Determination of Volatile Matter (ASTM E897–88)

Percentage of volatile matter (VM) of the samples was determined by calculating the loss in weight of sample using electrical furnace at temperature of 500°C–550°C, until the weight of the sample was constant, after 10 minutes to obtain dry weight, by weighing the dried sample after been cooled. Volatile matter was then calculated as thus;

$$\% \text{ VM} = \left(\frac{W_8 - W_9}{W_5 - W_7} \right) \times 100 \quad \dots \quad (3)$$

Where W_7 =weight of empty crucible, W_8 =weight of crucible and sample and W_9 =weight of crucible and sample after heating.

Determination of Fixed Carbon (ASTM E–870)

This was calculated by subtracting the sum of % of moisture content, volatile matter (VM) and % of ash content (AC) of the samples from 100.

$$\%FC = 100 - (\%MC + \%AC + \%VM) \quad \dots \quad (4)$$

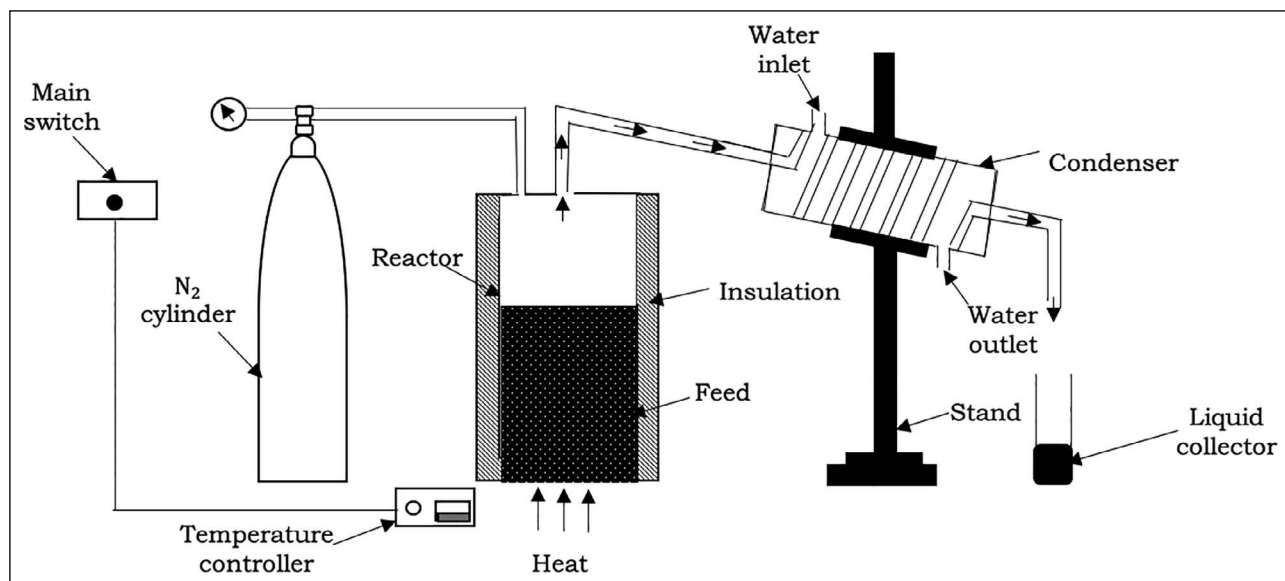


Figure 1. Conceptual representation of co-pyrolysis system.

Ultimate Analysis of the Feedstock Materials

The purpose of the test was to determine element percent of carbon, hydrogen, nitrogen, oxygen and sulphur content in the feedstock materials. The ultimate analysis was conducted according to ASTM standards E777–17a, E–778–15 and E–711–15. This is the estimation of the important chemical elements present in the feed stock materials of this study. The composition of the elements are percentage of carbon (C), hydrogen (H), Nitrogen (N₂), Sulphur (S).

Determination of Oxygen Content (ASTM D 7607)

Oxygen was determined by calculation

$$\text{Oxygen (\%)} = 100 - (\%C + \%H + \%N + \%S) \quad \dots \quad (5)$$

Where C=Carbon, H=Hydrogen, N=Nitrogen and S=Sulphur

Determination of Calorific Value (ASTM D2015–96)

The gross calorific value was determined using a bomb calorimeter model (CAB 101). Approximately 0.8 g fuel sample was weighed in a bomb crucible. The bomb cup was placed on its stand provided with the outfit. Firing nickel wire piece was stretched between the electrodes of the bomb (ASTM E–711). A wicking cotton thread, 9 cm long was tied to the stretched wire and the end dipped into the sample in the crucible. The crucible was then placed on a support ring. Exactly 1 ml of distilled water was pipetted out into the body of the bomb and then filled with $3.0 \times 10^6 \text{ N/m}^2$ of oxygen gas. Calorimeter was filled with distilled water until a weight of 3 kg (water plus the can) was obtained to submerge the bomb completely. The bomb was placed on the three supports in the calorimeter vessel and checked for leakage (it was confirmed by absence of bubbles). Cooling water was adjusted to flow at rate of 300 ml per minute. The water jacket was covered

with thermometers and stirrer was lowered and circuit completeness tested by firing circuit test plug. The temperature of the calorimeter vessel was allowed to stabilize and the initial temperature taken at $\pm 0.001^\circ\text{C}$, the fire switch was then pressed for 2 seconds to ignite the sample. The final temperature of the apparatus was taken after 10 minutes at $\pm 0.001^\circ\text{C}$. Readings were taken after sequential 3 minutes until the reading was found to be within a range of $\pm 0.002^\circ\text{C}$. The final temperature was recorded. Rise in temperature was calculated as a difference from initial to final temperature reading. Each sample was tested three times and the average values of weight of sample and temperature rise taken for the final determination of total heat released from the sample. The gross heat released was calculated using the equation 6:

$$\text{Calorific Value} = \frac{cv\Delta T - 0.12600}{M} \quad \dots \quad (6)$$

Where;

H=Calorific value, C_v =Heat capacity of apparatus (10.380 J/°C) and 0.12600 J=Constant heat gain. ΔT =Change in Temperature in °C, M=mass of the sample in (g).

The Co-pyrolysis System

Pyrolysis can be defined as the thermal decomposition of feedstock materials at higher temperatures in an inert atmosphere (absence of oxygen). The byproducts of pyrolysis process includes; liquid, gas and char. The main objective of this co-pyrolysis system is to produce liquid fuel. The main components of the system are the reactor and the condenser.

Figure 1 shows the conceptual representation of the pyrolysis system and Figure 2 depicts the co-pyrolysis system for the production of liquid fuel.



Figure 2. Co-pyrolysis system.

Experimental Procedure

Dried and weighted *Jatropha curcas* seed cake with tyre waste of (1:1) of particle size of 1.5 mm was fed into the fixed bed reactor for pyrolysis. The sample was taken in the stainless steel reactor for each run and placed in an electrically heated furnace. The experiments were conducted at various temperatures of 450°C, 500°C and 550°C with a heating rate of 20°C min⁻¹ and kept at constant till the reaction completes in 30 minutes. A nitrogen flow rate of 2 L/min was employed in other to purge air out of the system which prevent it from secondary cracking. The volatiles contents emanating from the reactor were channeled to the water cooled condenser and the non-condensable gases were flared into the atmosphere. The condensed oil was collected in a liquid collector, and weighted. The remaining residue (char) and the gases that escaped to the atmosphere were regarded as byproduct in this study. The experiments were conducted three times and the average values were recorded and reported. The liquid fuel yield was collected after it was condensed and was recorded carefully kept (separately) in well-sealed containers under room temperature. This procedure was repeated for the following feed ratios of 1:2 and 1:3 (i.e *Jatropha* cake with tyre waste) and the temperatures of 500°C and 550°C, with the reaction time of 45 and 60 minutes.

Optimization Using Taguchi Design of Experiment

The Taguchi method is a structured approach for determining the best combination of inputs to produce a product or service. Design of experiment is an important tool for designing processes and products. It is a method for quantitatively identifying the right inputs and parameters levels for making a high quality product or service. It is also a statistical or engineering methodology that aim at reducing the performance “variation” of a system [19]. In this study contains 3 parameters which are feed ratio, temperature and reaction time, and three levels for each. Meaning the feed ratio levels of 1:1, 1:2 and 1:3, the temperature has levels of 450°C, 500°C, and 550°C, and the reaction time has levels of 30 min, 45 mins and 60 min. By inputting the parameters and levels on taguchi design of experiment, Table 1 would be generated automatically.

Table 1. Co-pyrolysis of *jatropha* cake with tyre waste

Experiment no	Parameters			Liquid fuel (% wt)
	Feed ratio	Temp (°C)	Reaction time (min)	
1	1:1	450	30	63.0
2	1:1	500	45	68.0
3	1:1	550	60	60.0
4	1:2	450	45	57.3
5	1:2	500	60	58.2
6	1:2	550	30	53.0
7	1:3	450	60	52.0
8	1:3	500	30	53.0
9	1:3	550	45	50.3

Table 2. Proximate and ultimate analysis of *jatropha* cake

Proximate analysis (% wt)	
Moisture content	0.76
Volatile matter	78.30
Ash content	1.80
Fixed carbon	19.14
Ultimate analysis (% wt)	
Carbon	57.20
Hydrogen	8.30
Nitrogen	6.10
Sulphur	0.5
Oxygen	27.90
Calorific value (MJ/Kg)	25.47

¹Calorific value = $\frac{C_p \Delta T - 0.12600}{M}$, Where; C_v=Heat capacity of apparatus, ΔT=Change in temperature in °C, M=mass of the sample in (g).

It would come up with the minimum number of experiment to be conducted/performed. Both the means and delta values are automatically generated from the minitab version 18 software, and the delta is calculated as the difference between the maximum and the minimum of each column.

RESULTS AND DISCUSSION

Results

Table 2 shows the results of proximate and ultimate analysis of *Jatropha* Cake.

Proximate analysis (Table 2) shows that the sample (*Jatropha* cake) had high concentration of volatile matter (78.30%). The inherent moisture content of the sample was 0.76%. The ash and fixed carbon content contains 1.8% and 19.14% respectively.

Table 3. Proximate and ultimate analysis of tyre waste

Proximate analysis (% wt)	
Moisture content	0.76
Volatile matter	78.30
Ash content	1.80
Fixed carbon	19.14
Ultimate analysis (% wt)	
Carbon	83.20
Hydrogen	5.52
Nitrogen	1.50
Sulphur	0.20
Oxygen	9.78
Calorific value (MJ/Kg)	30.47

²Calorific value = $\frac{cv\Delta T - 0.12600}{M}$, Where; C_v = Heat capacity of apparatus, ΔT = Change in temperature in °C, M = mass of the sample in (g).

Ultimate analysis data (Table 2) indicates that the sample contains high proportion of carbon of 57.20%, with relatively low concentrations of sulphur (0.5%), nitrogen (6.10%), and hydrogen (8.30%). The oxygen content of the sample was found by calculation (27.90%). The calorific value or energy content of the sample was determined to be 25.47 MJ/kg.

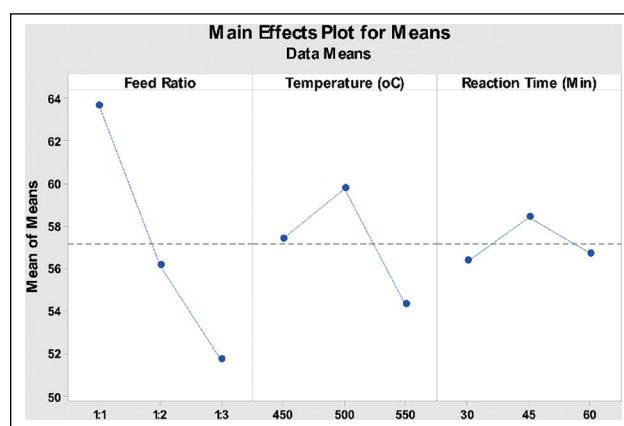
The Table 3 depicts the results of proximate and ultimate analysis of tyre waste.

Proximate analysis (Table 3) shows that the sample (tyre waste) has high content of volatile matter (55.95%). The inherent moisture content of the sample was 0.57%, however, the sample contains 15.30% of ash content. The fixed carbon obtained from the sample by calculation was 28.18%.

Ultimate analysis data (Table 3) indicates that the tyre waste contains high proportion of carbon of 83.0%, with low sulphur contents of (0.2%), nitrogen (1.50%), and hydrogen (5.52%). The oxygen content of the sample was found by calculation (9.78%). The calorific value or energy content of the sample determined was 30.47 MJ/kg.

The Table 1 depicts the results of liquid fuel produced via co-pyrolysis of Jatropa cake with tyre waste operating under various conditions using Taguchi's design of experiment.

It can be observed that from Table 1, at constant feed ratio of 1:1, as the temperature and the reaction time increases, the liquid yield produced decreases with the exception of the reaction temperature of 500°C and reaction time of 45 minutes, which had liquid yield of 68.0 wt%. It can also be observed that, at constant feed ratio of 1:2, as the temperature increases and the reaction time decreases, the liquid yield produced also decreases with the exception of the reaction temperature of 500°C and reaction time of 60 minutes, which had liquid yield of 58.2 wt%. It can also note that,

**Figure 3.** The graph of mean of means versus feed ratio, temperature and reaction time.**Table 4.** Response table for means

Level	Feed ratio (A)	Temperature (°C)	Reaction time (min)
1	63.67	57.43	56.33
2	56.17	59.73	58.53
3	51.77	54.43	56.73
Δ	11.94	5.30	2.20
Rank	1	2	3

at constant feed ratio of 1:3, as the temperature increases and the reaction time decreases, the liquid yield produced also decreases with the exception of the reaction temperature of 500°C and reaction time of 30 minutes, which had liquid yield of 53.0 wt%. The percentage weight of the liquid fuel obtained from pure jatropa cake and pure tyre waste were 22.6 wt% and 38.4 wt% respectively which are in consonance with the study of [1, 16–18]. As the waste tyre was added to the biomass as feedstock material, the liquid fuel yield increases so also the calorific value of the liquid fuel compared to that of pure jatropa cake or pure tyre waste.

Maximum Process for the Co-pyrolysis of Jatropa Cake with Tyre Waste

The Figure 3 shows the results of the optimization process for the co-pyrolysis of Jatropa cake with tyre waste.

It can be observed that from Figure 3, as the feed ratio of the samples decreases the liquid yield increases, and the liquid fuel yield was optimum at the feed ratio of 1:1. Furthermore, the reaction temperature and the reaction time that produces high liquid fuel yield was at 500°C and 45 minutes respectively. So the parameters that can yield maximum liquid fuel was at a feed ratio of 1:1, reaction temperature of 500°C and reaction time of 45 minutes. The result (liquid fuel) obtained at that conditions was 68.0 wt%. The Table 4 demonstrates the results of response table for means.

The results from the Table 4 shows the effect of each parameter or factor in the liquid fuel production. It showed that the feed ratio has the greatest effect on the production of liquid fuel, followed by temperature, and then reaction time.

CONCLUSION AND RECOMMENDATION

Conclusion

The findings of the characterization study using proximate and ultimate analysis indicate that, the Jatropha cake and tyre waste has a potential source of liquid fuel production. It was inferred from the Taguchi analysis that the combination of feed ratio of 1:1, reaction temperature of 500°C and the reaction time of 45 minutes was the optimal setting for obtaining maximum liquid fuel for the co-pyrolysis of Jatropha cake with tyre waste. The maximum liquid yield obtained from the co-pyrolysis of Jatropha cake with tyre waste was 68.0 wt%. It can also concluded that, the ratio of feed stock material has greatest influence on the production of the liquid fuel compared to reaction temperature and reaction time.

Recommendations

- i. Proximate and ultimate analyses showed that, the jatropha cake and the tyre waste materials has high content of carbon and hydrogen with low oxygen content and has potentials for the production of qualitative liquid fuel. Considering the results obtained, the liquid fuel from Jatropha cake and tyre waste can be used as an alternative fuel.
- ii. Other materials like HPDE (High density Polyethylene) and LDPE (Low density Polyethylene) and biomass mixtures should be researched upon to determine the synergy effect between them via co-pyrolysis technique. Because these materials has high carbon and hydrogen content. When mixed together with biomass will produce more content of pyrolytic liquid fuel.

DATA AVAILABILITY STATEMENT

The author confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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