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Analysis of bio-oil production and characterization from duckweed biomass

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Abstract

In this study, the production and characterization of bio-oil from duckweed was carried out through a pyrolysis process. The method used in the study aim is to produce and characterize bio-oil from duckweed through a pyrolysis process. The method used in the production of bio-oil from duckweed in this study is the pyrolysis method where a fix-bed reactor was connected to a condensers and heat was applied from a gas burner and the temperature was monitored using a thermocouple connected to the reactor and time monitored by a stopwatch until the last drop of product was observed from the system. Analysis of produced bio-oil shows that it is a heavy oil with API gravity of °8.73, density value of 1.006 g/cm3 at 22 oC, specific gravity of 1.009 kg/m3 and a high viscosity of 9.32 mm2.s-1. Further analysis resulted in a high flashpoint of 93 oC and a pour point of 14 oC respectively. GC/MS analysis carried out on the bio-oil sample shows that it contains Polyaromatic hydrocarbon (PaH) at different concentrations. The study also showed that the produced bio-oil contains carbon chain compounds; C8-C28. It was concluded that bio-oil produced from duckweed is a potential green alternative to fossil fuels and efforts should be made by relevant bodies to convert the laboratory scale production of duckweed to industrial scale as it will be a good and possible alternative for fossil fuel.

Keywords: Duckweed; Pyrolysis; Bio-oil Production; Renewable Energy; Biomass

1. Introduction

A sustainable economy has considerable challenges regarding energy security, which calls for the creation of alternative renewable energy sources that can satisfy future demand. All of humanity has been reliant on non-renewable fuels such as petroleum-based fuel since their discovery more than 4,000 years ago. Owing to the negative environmental effects, expanding global demand, and limited availability of petroleum-based fuel, scientists are seeking for cleaner and sustainable alternatives. A proposed alternative energy source and hydrocarbon replacement is bio-oil.

Due to its high CO₂-sequestering capacity, high lipid productivity, and ease of growth in open ponds and waste/marine/brackish water, duckweed appears to be a promising step towards sustainability and cleaner fuels among renewable energies [1]. Relying on conventional fuels as the main source of energy is no longer unsustainable. The estimations for the remaining oil reserves are pessimistic and suggest that they will last for decades rather than millennia [2]

Furthermore, the use of fossil fuels has been linked to numerous negative environmental effects and with the development of the global climate change concerns due to the rising atmospheric greenhouse gas (GHG) concentrations, there is rising interest in environmentally friendly alternatives including the production of bioenergy to lessen reliance on fossil fuels and cut out GHG emissions.

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Particularly, there is a great need for alternative liquid fuel for transportation, which is why bio-oil has seen tremendous growth and commercialization in recent years. Bio-oil can be produced using a range of feedstocks, such as animal oils, biomass made from organic waste, plant oils, sugars, starches, and lignocellulose biomass, as well as cultivated microbes. Numerous biofuel feedstocks have come under fire for competing with food crops for nutrients, water, and agricultural land, including sugarcane, wheat grain, and maize starch. Future biofuel production could make use of "energy crops" other than food, such as switchgrass and *Jatropha curcas*. Conflict over agricultural resources, though, can still provide some challenges [3]

Bio-oils are a viscous or free-flowing, dark brown liquid with a distinct odor. Different physical qualities of bio-oils emerge from the composition of the produced bio-oil, which is dependent on the thermochemical process, the kind of feedstock, and operating circumstances. A mixture of hundreds of different complex organic chemicals, aldehydes, esters, phenols, ketones, acids, alcohols, and guaiacols, make up bio-oil. Some of these substances have a direct connection to the unfavorable characteristics of bio-oil. Wood-derived bio-oil from pyrolysis was described by Garcia-Perez et al. [4] The mixture of sugars, non-polar and heavy polar molecules, MeOH-toluene insoluble, volatile organic chemicals, and moderately volatile polar compounds were the ones they identified as making up bio-oil.

Duckweed is the tiniest angiosperm known to science. It is a little aquatic plant that floats freely and has the traits of rapid multiplication, simplicity in growth, and bacterial resistance. Duckweed can multiply quite quickly, and under the right circumstances, its bulk can double in 24 hours. Duckweed is a water filter of low-cost because it has developed the capacity to quickly collect the organic nutrients and minerals, they need from the water in which they float. Utilizing nutrients from animal waste lagoons uptake for intake of nutrients could be one method to create copious volumes of duckweed at a low cost [5]. Since they get their nutrients from the wastewater, they do not need any energy to thrive. It produces 7 to 20 tons of dry weight of biomass per year per hectare of water surface. Duckweed is a promising choice for a feedstock in biofuels due to its high production. Therefore, one option to increase the value of land while having little impact on the environment is to use duckweed as a biomass for the manufacture of bio-oil.

Duckweeds are tiny, free-floating vascular plants that grow on freshwater surfaces all over the world (or sometimes brackish). Due to their high amount protein content, their quick accumulation of biomass when compared to other terrestrial plants, and capacity to absorb fertilizers and other substances, duckweeds have received a great deal of attention since the 1970s [7]. A possible protein source for people and livestock, particularly chicken and fish, duckweed can grow swiftly in ditches, small ponds or swamps where it can take huge amounts of nutrients [8]. Duckweeds are used to filter waste waters and remove toxins from them because they have high capacity to absorbed mineral and can withstand heavy organic loading. Many different climatic zones and geographic supports different duckweed specie with most species living in subtropical regions and tropical [6]. Deserts without water and places that are always freezing are barren of their growth. Most of the time, duckweeds are hardy survivors with a wide tolerance for high temperature, conductivity, pH, phosphorus and nitrogen [7].

The objective of this study was to produce and characterize bio-oil from duckweed biomass using pyrolysis. A sample of duckweed was pyrolyzed gradually without the use of a catalyst in this investigation. The pyrolysis system used fixed bed reactor, and a condenser was used to collect the oil into a measuring cylinder. The system temperature was monitored by a thermocouple, and the reactor was heated by a gas burner. The time spent heating was monitored using an electronic stopwatch. The product yield was noted along with the temperature and time. The produced bio-oil was evaluated for its API gravity, viscosity, specific gravity, pour point, pH, fixed carbon content, flash point and volatile matter. A GC/MS analysis was also performed on the sample to identify the oil's contents.

2. Material and methods

2.1. Materials

Duckweed was be harvested from stagnant water in Port Harcourt, Rivers State. The materials used for the study are heater, Fixed-bed reactor, condenser, Thermocouple, water for cooling, lagging materials, measuring cylinder, weighing balance, retort stand, beaker, separating funnel, round bottom flash and gas burner. A reactor unit was constructed with mild steel, a hopper and thermocouple which are connected to a glass condenser. The reactor was properly lagged to allow for appropriate gaseous product condensation. The gas burner provided the reaction's heat. 26 °C water was employed as the cooling medium. Condensed oil was delivered in a measuring cylinder so that product creation could be tracked. The reactor was wired with a thermocouple to track the temperature of the reaction, and a timer was used to clock the reaction.



Figure 1 Duckweed collection site

2.2. Method

A sample of duckweed was also collected, dried for 7 days to eliminate the moisture, and weighed before being fed through the reactor's hopper. The hopper was coverd properly. Adequate precautions were also put in place



Figure 2 Dried Duckweed biomass

the experiment. To cool the condensing vapor from the reactor, a glass condenser was firmly fastened to it. The vapor was cooled using water that was 26°C and attached to a counter. There was no catalyst utilized. When the final drop of oil was seen in the measuring cylinder, the gas burner was turned on and the pyrolysis process continued. Temperature and time measurements were used to track the amount of bio-oil generated. The created bio-oil was promptly examined, and the outcome was noted. Additionally, different temperatures were used throughout the pyrolysis to see how they affected the end products.

2.3. Bio-oil analysis

2.3.1. Oil density

Density of oil the mass per volume of the oil. Less frequently than kg/m3, it is in quantities of grams per milliliter for oils. Temperature has an effect on density; rising temperatures cause it to decrease. While bitumen and other fuel oils may have densities more than 1.0 gm/ml, all new crude oils and the majority of fuel oils have densities less than this value. Pycnometer was used to determine the oil's mass. The weight of the empty pycnometer and the oil's mass were both recorded, and the pycnometer was heated after being filled with 25ml of the bio-oil. To calculate water's density, the mass of the liquid was also measured. The density of the oil was calculated using equation (1):

$$Density = \frac{mass \ of \ oil - mass \ of \ empty \ pycnometer}{Volume \ of \ oil} \qquad \dots \dots \dots \dots \dots \dots (1)$$
$$Density = \frac{M}{V} \qquad (2)$$

Density water = $\frac{mass of water-mass of empty pynometer}{2}$(3) Volume of oil Specific gravity= $\frac{\rho_{oil}}{\rho_{water}}$ (4)

2.3.2. Specific aravity

Specific gravity, also referred to as relative density, is used to relate the weight or density of liquids to that of water. Specific gravity is a unitless measurement that is derived as a ratio of either the weight of another liquid or the density of another liquid divided by the weight or density of water. Temperature was considered when determining specific gravity, since density changes in relation to temperature. The specific gravity of the bio-oil was determined by eq. (4).

2.3.3. API gravity

The API gravity, or American Petroleum Institute gravity, is a measurement of how heavy or light a petroleum liquid is in relation to water: if the API gravity is greater than 10, the petroleum liquid is lighter and floats; if the API gravity is lower than 10, the petroleum liquid is heavier and sinks.

Thus, API gravity is a measure of a petroleum liquid's density in relation to water's (also known as specific gravity). It is employed to compare the liquid petroleum densities. Specific gravity of oil (Spgr.) and American Petroleum Institute (API) gravity are two qualities that relate to densities. The API gravity is determined as follows:

API gravity
$$= \frac{141.5}{SG} - 131.5....(5)$$

2.3.4. pH measurement

pH is a measure of the alkalinity or acidity of a solution. The hydrogen ion (H+) content of a solution is indicated by the pH value. The solution becomes more acidic, and its pH decreases as the concentration of H+ increases.

Using pH paper, the biofuel's pH was calculated. The pH strip's tip was submerged in the bio-oil. After a brief period, the paper was removed, and the pH strip's color was compared to a color chart included in the pH paper package.

2.3.5. Viscosity

The resistance of a substance to motion when a force is applied is measured by its viscosity. The outcome is often expressed in centipoise (Cp), or 1 mPa s. (millipascal second). Shear stress is the amount of force per unit area required to move one fluid layer relative to another. The viscosity of the biofuel was determined using a Redhood viscometer.

2.3.6. Fixed Carbon

The solid combustible residue that is left after a coal particle is heated and the volatile stuff is released is known as fixed carbon. Subtracting the percentages of moisture, volatile matter, and ash from a sample yields the fixed-carbon content of a coal. Eq was used to determine the oil's fixed carbon content (3.7)

2.3.7. Chemical Composition

A mass spectrometer and gas chromatography (GC) (CHEM32) were used to analyze the chemical composition of the generated bio-oil (MS). The injection volume was 1 l, the temperature was 270 °C, and the carrier gas, helium, flowed at a rate of 1 mL min-1. The starting temperature for the analysis was 40°C, and the heating rate was kept constant at 1.5°C min-1 until 46°C before being raised to 4°C min-1 until the temperature reached 209°C. The chemical makeup of the pyrolysis sample was assessed in real time using a single-shot pyrolyzer connected to a GC-MS (Agilent 7890A/5975C, USA) with an inert XL mass spectrometry detector and a capillary column (30 m in length, 0.25 m in internal diameter, HP-5 MS). The concentration level and proportion of different compounds in the duckweed's pyrolysis products were evaluated using online Py-GC/MS under a ramping temperature gradient from 350 °C to 400 °C at a temperature interval of 100 °C. Total ion current (TIC) diagrams of the duckweed pyrolysis products were obtained under varied temperature conditions. The outcomes were evaluated using Agilent MSD Productivity Chem Station for GC and GC/MS System data Analysis application software, by contrasting the NIST 2011 Database with the retention durations and peak area proportions of several compounds in the pyrolysis products. The concentrations of each substance were correctly matched to the percentage of the corresponding peak region.

3. Results and discussion

3.1. Pyrolysis Results of bio-oil produced from Duckweed

The effect of temperature of on the pyrolytic system is shown in figure 3. At temperatures between, 0 °C-20 °C, no drop of product was observed from the pyrolysis process, the first drop of product was observed at temperature of 30 °C.



Figure 3 Effect of Temperature and Time on Bio-Oil yield from Duckweed

The heating time of the reaction was carefully observed during the production of bio-oil from pyrolysis of duckweed. It was discovered that from time 0 mins – 20 mins heating time, no product was formed from the pyrolysis process, the first drop of product was seen at heating time of 30 mins, the volume of product formed from the pyrolysis process 46mins after the first drop was 2ml as shown in figure 4.



Figure 4 Effect of Heating Time on Bio-Oil yield from Duckweed

3.2. Characteristics of Bio-oil produced from duckweed

The bio-oil has a specific gravity of 1.009, according to Table 1. According to Emery [9], the specific gravity of bio-oils from woody biomasses typically ranges from 0.80 to 1.21, whereas pine sap has a specific gravity of 0.889 to 0.989. The specific gravity of the bio-oil generated is greater than that of gasoline which varies from 0.68 To 0.74. This demonstrated that, when evaluated from the value of its specific gravity, bio-oil produced by the pyrolysis of duckweed may be used as a combination of biodiesel. The API gravity at 22 °C was 8.73, which suggests that it is a very heavy oil. The viscosity of the bio-oil at 22 °C is 9.32 mm2.s-1; This suggests that the fluid has a very high viscosity and is more concentrated than other biomass sources. Due to the intense heat used during the pyrolysis process, the viscosity of the bio-oil made from duckweed suggests that the oil will have a good consumption rate when used since it controls the sealing effect of oils, the rate of oil consumption, as well as how easily machines may be started or operated under various temperature circumstances.

The kind of raw material, pyrolysis temperature, water content in pyrolysis oil, and the pyrolysis process are all variables that might affect the viscosity of different pyrolysis oils. The pH level of the duckweed bio-oil is (4.2).

According to Mohan et al. [10], only a small number of biomass feedstocks, such as almond shells (5.5) and rice straw (4.2), have pH values above 3.0. (2006).

Physical properties	Quantity	Unit	
Density at 22°C	1.076	g/cm3	
Specific gravity	1.009	-	
API gravity at 22°C	8.73	-	
Viscosity	9.32	mm2.S-1	
рН	5.4	-	
Pour Point	14	°C	
Flash Point	93	°C	
Fixed carbon	86.56	%	

Table 1 Physical Properties of the Produced Bio-Oil from duckweed

The pH value is a crucial parameter for boiler operations because if it is too low, boiler corrosion damage may result. The pH of bio-oil fluctuates depending on the pyrolysis temperature. It was evident that when the pyrolysis temperature rose, the pH of the bio-oil rose as well. This shows that the pH of bio-oil tends to be closer to neutral the higher the temperature process. Furthermore, Boscagli et al. [11] state that the temperature and kind of raw material have an impact on the acidity (pH) value (2018). The pour point was (14°C), which is within the range for pour points of waxy crude oil. According to ASTM Standard D5949, a high pour point is frequently correlated with a high paraffin content, which is typically present in crude that is generated from a greater percentage of plant material. The bio-oil point was 93 °C, which falls within the range of flash points for heavy crude with densities larger than 0.9 g/cm3 in temperature.

Heavy fuel oil typically comprises fractionator bottoms, reduced crude, or heavy product from the cracking coil that is combined (cut back) to a certain viscosity with cracked gas oils. As a result, heavy crude typically has a high flash point. The bio-oil generated has 0.53 volatile matter and 73.45 fixed carbon.

3.3. GC/MS analysis of the produced bio-oil

Different temperatures were used to get the product after the pyrolysis of duckweed at various degrees of sample degradation. It was proposed that the sample's pyrolyzed products may be pyrolyzed once more before the re-pyrolyzed products were collected. The proportion of key chemicals in the pyrolysis products obtained from the duckweed sample was determined using GC-MS analysis, which would serve as crucial benchmarks for biomass thermochemical conversion, industrial use, and bio-oil upgrading.

The chemical make-up of the oil produced by the pyrolysis of duckweed is shown in Table 2. Table 2 displays the concentration and retention duration of the carbon chain hydrocarbons found in the bio-oil. The table demonstrates that, at various concentrations and retention times, the bio-oil formed 19 carbon chain compounds (C8-C28), Pristine, and phytane. Retention time (RT) and peak area were two crucial considerations while employing the GC-MS (PA). The PA might frequently change from run to run because of human and mechanical mistake. Due to the sample's physical characteristics, RT should continue to be constant as long as we used analytical method. Prior to doing a GC-MS analysis on a sample, it is crucial to comprehend the sequence in which the mixture's constituent chemicals elute. To do this, the GC-MS analytical technique was employed. The procedure must be ramped up high enough for the compounds to elute off the column and exhibit an exact retention period, which is another benefit of using a specific sequence for elution. A bio-oil sample made from duckweed that had been pyrolyzed was examined using a TIC diagram. Three peaks with retention times of 16.926, 18.196, and 12.700 minutes were found when the oil's contents were examined in GC-MS, as shown in Figure 5.

RetTim	е Тур	pe	Area Amt/Area		Amount Grp	Name	
[m	nin]		[pA*s]		[ppm]		
2.689	BH	Т	249.79150	9.97110e-2	996.27806	n-C8	
4.306			-	-	-	n-C9	
5.514	Н	Т	155.69240	59240 1.21694e-1 757.87322		n-C10	
6.765	Н	Т	88.94972	7.27325e-2	258.78154	n-C11	
7.672	Н	Т	278.32986	1.36239e-1	1516.77837	n-C12	
8.219	Н	Т	69.32565	1.13181e-1	313.85411	n-C13	
9.149	Н	Т	96.99173	3.02811e-1	1174.80824	n-C14	
10.138	Н	Т	124.35025	2.73556e-1	1360.67168	n-C15	
10.969	Н	Т	76.32633	2.42366e-1	739.95746	n-C16	
11.711	Н	Т	83.93609	4.12039e-1	1383.39734	n-C17	
11.929	Н	Т	96.11134	1.70143e-1	654.10680	Pristine	
12.650	Н	Т	78.72800	2.11064e-1	664.66610	Phytane	
12.700	Н	Т	157.60229	3.27137e-1	2062.30188	n-C18	
13.209	Н	Т	71.93558	2.83073e-1	814.52204	n-C19	
13.245			-	-	-	o/-Terphenyl	
13.767	HH		112.76443	2.55489e-1	1152.40127	n-C20	
14.342	HH		83.95947	2.48838e-1	835.69374	n-C21	
14.931	HH		79.11005	2.51184e-1	794.84740	n-C22	
15.496	BB		91.31449	2.67679e-1	977.71892	n-C23	
16.037	HB		73.65247	3.02580e-1	891.43112	n-C24	
16.561	BB		56.62801	2.77501e-1	628.57213	n-C25	
16.926	HB		403.51221	3.17105e-1	5118.23274	n-C26	
17.654			-	-	-	n-C27	
18.196	BB		133.38660	3.64184e-1	n-C28	n-C28	
18.577				-		n-C29	
19.015				-		n-C30	
19.441				-		n-C31	
19.853				-		n-C32	
20.251				 		n-C33	
20.641					n-C34		
21.036						n-C35	
21.479					n-C36		
21.984							
22.563							
23.234				-			
24.013				-			
Total:			2.50400e4				

Table 2 GC results for retention time and component of bio-oil produced from duckweed



Figure 5 TIC Diagrams of for Bio-Oil Produced from Pyrolysis of Duckweed

The highest amount of was observed in n-C26 (5118.23274) at retention time of 16.926 mins.

Com	pound Dev (Min)	R.T.	QIon	Response	se Conc		Units	
Syste	System Monitoring Compounds							
7)	o-Terphynyl	7.090	230	1073	0.16	ppm		0.23
Targ	Target Compounds						Qvalue	
1)	Naphthalene	3.148	128	731	1.11	ppm		100
2)	Acenaphthylene	4.567	152	823	1.72	ppm		100
3)	Acenaphthene	4.744	153	354	1.50	ppm		100
4)	Fluorene	5.357	166	237	1.23	ppm		96
5)	Phenanthrene	6.427	178	774	4.99	ppm		100
6)	Anthracene	6.524	178	285	1.78	ppm		100
8)	Fluoranthene	7.880	202	999	9.60	ppm		100
9)	Pyrene	8.092	202	556	4.99	ppm		100
10)	Benzo(a)anthracene	9.579	228	1028	8.80	ppm		100
11)	Chrysene	9.642	228	407	3.41	ppm		100
12)	Benzo(b)fluoranthene	10.770	252	1286	13.17	ppm		100
13)	Benzo(k)fluoranthene	10.815	252	860	8.54	ppm		100
14)	Benz(a)pyrene	11.124	252	639	10.35	ppm	#	38
15)	Dibenz(a,h)anthracene	12.337	276	236	7.08	ppm	#	50
16)	Indeno(1,2,3-cd) pyrene	12.292	276	227	7.98	ppm		100
17)	Benzo(g,h,i)pyrene	12.646	276	268	8.36	ppm		100

After further analysis, it was discovered that the pyrolyzed products of duckweed at 350 °C contained mainly 17 different compounds such as acids, alcohols hydrocarbons and other organic substances as outlined in Table 3.

Table 3 shows the polyaromatic compounds present in the produced oil from pyrolysis of duckweed. The concentration and retention time of the compounds which are acids, alcohols, hydrocarbons, and other organic substances present in the bio-oil was shown in the table. The table shows that the bio-oil produced contains 17 PaH compound at different concentration which are show at different retention time. Upon analyzing the constituents of the oil in GC-MS, we observed peaks with retention time of 7.880, 10.770 and 11.124 minutes respectively as illustrated in Figure 6.



Figure 6 TIC Diagram for MS Analysis of Bio-Oil Produced from Duckweed

3.4. Fractions of the Produced Bio-oil and Uses

Based on the number of carbon atoms in the bio-oil molecules, Table 4 displays the composition of the bio-oil fraction. These general fuel fractions, which include kerosene, diesel, biodiesel, and heavy fuel fractions, are referred to as "bio-oil fractions." Similar to the work by, the gasoline portion is made up of hydrocarbons with carbon atoms ranging from C5 to C10 [7]. One form of fuel oil used in internal combustion engines is gasoline. Gasoline has fewer carbon atoms than other fuels made of hydrocarbon molecules. In addition to the gasoline component, bio-oil produced from the pyrolysis of microalgae and duckweed also contains a fraction of diesel. Hydrocarbon fuel known as diesel is utilized in diesel engines. Carbon atoms in diesel range in number from C10 to C15. Additionally, the biooil included heavy fuel components. Heating fuels having carbon atoms ranging from C18 to C25 are known as heavy fuels [7].

Table 4	Bio oil	fraction	composition
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Number of Carbon Atoms	Fuel Fraction
C6-C10	Gasoline
C10-C16	Kerosene
C16-C20	Diesel
C20-C30	Lub Oil

From the GC result, it shows that the bio-oil produced from duckweed can be refined to produce gasoline, kerosene, diesel, Lubricant oil and Fuel oil

4. Conclusion

The alternative and direct conversion of duckweed biomass to biofuel is made possible by analysis of the bio-oil production system. Duckweed is among the most promising sources for third-generation bio-oil production is duckweed. Although extraction methods and processing steps are still being studied, bio-oil production in laboratory scale is still sufficient to project for commercialization in the next decades. Due to the worldwide emphasis on alternate and more sustainable oil sources, the development of appropriate duckweed processing processes for scaled applications is anticipated in the near future along with the advancement of extraction techniques.

In this investigation, a sample of duckweed underwent gradual pyrolysis without the aid of a catalyst. The pyrolysis procedure was place in a fixed bed reactor, and the oil was collected into a measuring cylinder using a condenser. A thermocouple was used to measure the system's temperature, while a gas burner provided heat to the reactor. An electronic stopwatch was used to keep track of the heating duration. The product yield was tracked over time and at various temperatures. The viscosity, pH, pour point, specific gravity, API gravity, flash point, and fixed carbon of the generated bio-oil were all measured by analysis.

The obtained bio-oil sample was subjected to GC/MS analysis, and the TIC diagrams of duckweed from the pyrolysis bio-oil were displayed. The 211 distinct chemicals, including acids, hydrocarbons, alcohols, and other organic materials, made up the majority of the pyrolyzed products. The study also demonstrates that duckweed bio-oil is appropriate for upgrading since it comprises a chain of hydrocarbons from C8 to C26 that may be processed to generate fuel oil, gasoline, kerosene, and diesel.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare that there is no competing interest in connection with this paper.

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