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Synthesis and gas chromatography based characterisation of biodiesel prepared from waste soybean oil

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Petroleum resources are limited on a global scale. Due to non-renewability, these fossil-fuels are projected to be depleted in next 40-60 years if consumption continues at the current pace. Moreover, the price instability of crude oil poses a significant threat to nations with constrained financial and economic resources. To address these challenges, alternative energy sources are being explored, and there is ongoing emphasis on further progress in these areas. As an alternative fuel, biodiesel can be used in neat form or mixed with petroleum-based diesel. Therefore, we need to move towards alternative fuel like bio-diesel. Several alternative fuels have been studies among which biodiesel from waste soybean oil having great importance. Bio-diesel could be easily prepared from waste soybean oil, by the process of transesterification with alcohol in presence of sodium hydroxide or potassium hydroxide. India is the fourth largest producer of soybean in the world, and from last few decades, soybean oil is exclusively use as edible oil. In different food industries, several million tonnes of soybean oil uses every year. Therefore, huge amount of waste soybean oil is produced every year. In the present work, we attempt to synthesize biodiesel using waste soybean oil via transesterification, and characterize by physical, chemical, and spectroscopic instruments, and found the characteristics properties of this biodiesel is quite similar to petroleum diesel . Therefore, this synthesis may use biodiesel as an alternative for petroleum diesel.

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KEYWORDS

Waste soybean cooking oil; transesterification; biodiesel; alternate fuel.

Introduction

Minerals Energy is the prime requirement for the development of a country. Global energy consumption increases two times between 1971 and 2001, and the global energy demand will increase 53% by the year 2030 [1-2]. In such way, day by day the demand of petroleum diesel also increases, which is a non-renewable resource of energy which may end within 40-60 years if the consumption remain constant [2].

Diesel engines contribute in economic value in developing countries. Such engines run with petroleum diesel, this petroleum diesel produces smoke, particulate matters, oxides of nitrogen, oxides of carbon, un-burnt hydrocarbons, etc. and they have limited resources. Therefore, we need to move towards alternative fuel like biodiesel [1-2]. Several alternative fuels have been studies among which biodiesel from waste soybean oil having great importance. Biodiesel could be easily prepared from waste soybean oil, by the process of transesterification with alcohol in presence of sodium hydroxide or potassium hydroxide.

India ranks 5th in global production of soybean, following the US with 118.68 MT, Brazil produces 102 MT, Argentina's share is 57 MT, and China at 12.5 MT, recording a production of 12.30 MT in 2015-16. On international stage, India stands to be the top consumer and buyer of edible oils. In the 2013-14, India had an oilseed cultivation area spanning 28.051 million hectares, leading to a yield of 32.75 MT [1].

Biodiesel is proving to be increasingly significant as an alternate fuel option given the dwindling reserves of fossil fuels. As a versatile alternative, biodiesel can be blended with traditional diesel [1, 6] and can be easily used in diesel engines. In simple terms, biodiesel is a chemical compound having the long-chain fatty acids found in soybean oil. Its production involves a process called transesterification, where the oil is allowed to react under some conditions with a specific type of alcohol in the presence of a basic catalyst [3].

In different food industries, several million tonnes of soybean oil uses every year. Therefore, huge amount of waste soybean oil is produced every year. Waste cooking oil is essentially the oil left over after the meal preparations. After being reheated multiple times in cooking, this oil then becomes unsuitable for consumption because of its higher levels of free fatty acids. Discarding the waste oil leads to many serious issues such as water and soil contamination, health risks, and disturbances to aquatic ecosystems. As a substitute of contributing to these environmental challenges, it makes better sense to use this readily available waste oil, as a cost-effective feedstock for biodiesel production. This repurposing transforms the waste oil into a valuable resource that powers the engine [7]. Synthesizing bio-diesel from leftover soybean oil is a win-win situation for the environment. Not only is this bio-diesel ecosystem-friendly, but it also avoids emits less pollutants like nitrous oxides, carbon oxides, and unburned hydrocarbons [2].

For the vast majority of nations, providing enough reliable energy to meet their needs remains a top priority. Promoting biodiesel as an energy source in both developed and developing nations has been driven by technological improvements, rising global petroleum costs, the potential to decrease local and worldwide environmental emissions, and the attainment of objectives. Diesel oil and other fossil fuels may be replaced with biodiesel. Biodiesel is a renewable fuel that may be used in place of conventional diesel [11]. Because of their increased viscosity compared to diesel, biodiesels made from vegetable oils and animal fats may be used as fuel in diesel engines [12-13]. The first-generation biodiesel feedstock include oils derived from soybean oil, palm oil, and sunflower oil, all of which are edible vegetable oils (Bowyer et al., 2018) [15]. Soybean oil is a kind of vegetable oil that is extracted from soybean seeds. Soybean oil contains 16 grams of saturated fat, 23 grams of monounsaturated fat, and 58 grams of polyunsaturated fat per 100 grams [16-17]. Triglycerides from soybean oil include three different types of unsaturated fatty acids: 7-10% polyunsaturated alpha-linoleic acid, 51% linoleic acid, and 23% monounsaturated oleic acid [18]. It also has 4% stearic acid and 10% palmitic acid, both of which are saturated fatty acids. Micro-emulsion, pyrolysis, and transesterification are just a few of the methods that may be used to produce biodiesel. Transesterification is the best option among these technologies because of its high conversion yield and low cost [19-20]. In most cases, a catalyst such as sodium hydroxide or potassium hydroxide is used. Mostly, the cooking oil is being reused in the food industry and restaurants for a variety of economic reasons; however, its repeated use leads to the oil oxidation [21]. The dissolution of oxygen in vegetable oil due to repeated heating causes a reaction with unsaturated glycerols; thereby leading to the production various oxidative products [22]. In recent years, it has been found that reuse of waste cooking oil increases the risk several fatal diseases related to disproportionate growth of cells and diseases related to heart [23]. Recently, it has been found that the waste cooking oils are inappropriately disposed which causes extreme harm to the ecosystem and the aquatic organisms [24]. Due to the strict government policies regarding the reuse of WCO, it is estimated that a huge amount approximating 16.5 million tons is produced annually [25] which is presently either disposed as municipal waste [26] or into some sewage system [27]. To circumvent, the ill effects of WCO, many strategies had been adopted like using it for animal feed that was later banned [28], it was further used for soap production, but it was also stopped for ecological reasons [29]. A large number of studies have also been done for the environmental impact and the life-cycle analysis of WCO. It has been observed that the energy generation from glycerol and other organic wastes had resulted in a net reduction of the environmental impacts in comparison to the conventional methods of landfilling or incarnations [30]. In Portugal, the emission of carbon particles due to biodiesel production from WCO resulted in a net savings [31]. It has been found that in respect to diesel the biodiesel obtained from WCO is eco-friendly and is environmentally safe [32]. WCO biodiesel production has been further done with recovery of methanol was achieved and was found that production by alkaline catalysis with acid pre-treatment was eco-friendlier than vice-versa [33]. Few researchers [34] have found that using WCO as oil feedstock instead of Jatropha oil in biodiesel production led to a massive reduction in carbon dioxide emissions.

The objective of the study is to generate biodiesel through the transesterification process using waste soybean oil gathered from a local cafeteria in the presence of NaOH as the alkali catalyst and ethanol as the alcohol reagent. This will be done to accomplish the goal of the study. When the biodiesel has been made, its physical parameters, including density, kinematic viscosity, cloud point, flash point, and pour point, are evaluated and analysed. An additional gas chromatography test is carried out to do further analysis on the biodiesel that has been generated.

Experimental

Materials and methods

Pure refined sunflower oil, used cooking oil acquired from street vendors, ethanol, potassium hydroxide (KOH), sodium hydroxide (NaOH), distilled water, and petroleum diesel were among the materials gathered for this investigation. Beaker, measuring cylinder, thermometer, scale, wire mesh filter, magnetic stirrer, heating chamber, filter paper, and a weighing machine were all needed.

Preparation of Sample

The used cooking oil was purchased from a local seller who sells meals on the street. First, the used cooking oil that had been acquired was passed through a filter made of wire mesh, and then collected in a beaker. After that, it was run through filter paper with a pore size of 15 microns to remove any and all types of physical contaminants and pollutants that may have been found in the WSCO. The filtered sample is acquired in a beaker, and then two samples of 400 ml and two samples of 200 ml are created from it in beakers. All of the samples are taken from the filtered sample.

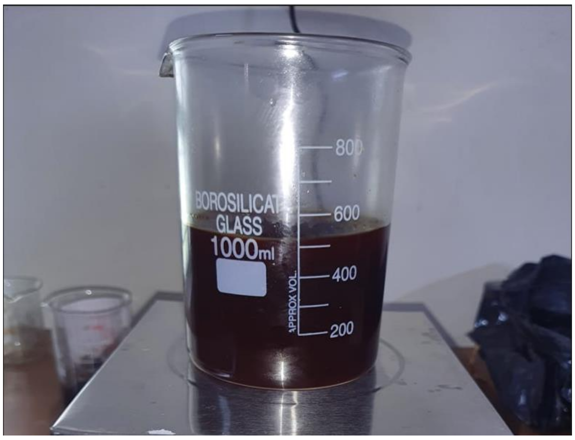

FIGURE 1 Sample preparation and collection in a beaker

Transesterification

After filtering, 500 mL of soybean waste cooking oil is poured into a beaker and heated to 65 °C in a hot air oven. After weighing on a digital machine, 5 g of sodium hydroxide (NaOH) was taken (1% of the sample of heated waste cooking oil) and combined with 100 ml of ethanol in a beaker. To produce the combination, a solution of NaOH and ethanol was progressively added to a heated sample of waste cooking oil and agitated for one hour, as displayed in Figure 1, using a magnetic stirrer.

After stirring for an hour, the ingredients are left alone. After 6 hours, 24 hours, and 48 hours, significant changes in the combination were noticed. To separate the glycerol and biodiesel, the mixture was kept undisturbed. After 6 hours, a tiny layer of glycerine was seen forming at the bottom of the beaker. After 24 hours, the glycerine layer was more apparent in the bottom of the beaker, and after 48 hours, the glycerine had developed fully and settled in the bottom of the beaker, as depicted in Figure 2, with the biodiesel floating on top.

FIGURE 2 Observation of sample after 24 hours

Cleaning of the product

When the glycerol had separated and dropped to the bottom of the beaker, the biodiesel was filtered out of the beaker and into a new beaker. The resulting biodiesel included small amounts of glycerol, ethanol, and potassium hydroxide (NaOH). To get pure biodiesel, it was processed further and rinsed with distilled water. The biodiesel in the beaker was first heated to a temperature of 50 °C to 60 °C, and then allowed to cool to room temperature. The biodiesel was water washed to get rid of any remaining glycerol and contaminants, as demonstrated in Figures 3 and 4. Due to its greater density than the product generated, water was separated from the sample using a separating funnel.

FIGURE 3 Biodiesel obtained after cleaning

Production of biodiesel

The ratio of waste cooking oil to ethanol was 1:5 for sample 3, giving the lowest biodiesel yield of 74%. It was found that a volumetric ratio of 1:4, waste cooking oil to ethanol produced the best results for biodiesel yield and was 74%.

From the proposed research, it was found that biodiesel when mixed with diesel fuel, can significantly improve the properties of the resulting fuel. This breakthrough has the potential to revolutionize the transportation industry by offering an environmentally-friendly and sustainable alternative to traditional fossil fuels.

Biodiesel can be viewed as a versatile and efficient fuel that can power any conventional diesel engine without any such modifications. In addition, it offers substantial advantages over traditional diesel fuel by increasing its lubricity. This upgrade is essential for the longevity and efficiency of diesel engines, reducing maintenance costs and extending their lifespan. Thus, biodiesel is a practical and cost-effective solution for enhancing the performance and efficiency of diesel-powered vehicles and equipment. Figure 4 shows a sample of the biodiesel produced in the process

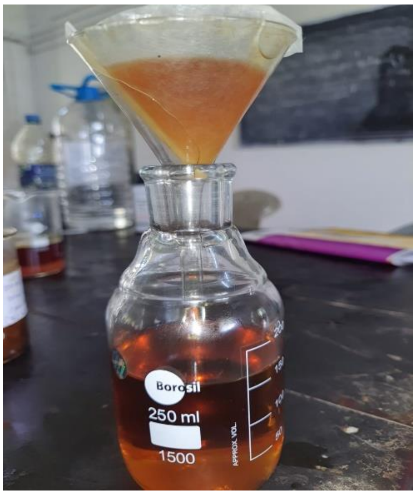

FIGURE 4 Sample of produced biodiesel

Characterisation of biodiesel (obtained from WSO)

While analysing its properties, the viscosity, density, flash point, fire point, pour point, and calorific value of the biodiesel that was derived from WCO were taken into consideration. The earlier research work has demonstrated that increasing the molar ratio of ethanol to oil results in an increase in the amount of biodiesel that can be produced. The molar ratio of oil to ethanol that produced the highest amount of biodiesel was one that read "1 to 1." The current study comes to the conclusion that the 1:3 ratio of waste soybean oil to ethanol will yield more biodiesel than any other combination of waste soybean oil and ethanol. The yield of biodiesel was 71.2% when the oil-to-ethanol molar ratio was 1:1.

Density is a key biodiesel characteristic. A lengthy chain of hydrocarbons is the primary component of the fuel known as biodiesel. The density of the diesel-biodiesel combination may be made denser by increasing the quantity of biodiesel used as a ratio to diesel. It was determined that pure biodiesel produced from edible waste oil has a density of 0.872. Increases in the concentration of biodiesel in the combination of biodiesel and diesel both result in increases in the density of the mixture. The viscosity of motor fuel is an extremely important quality. It has a significant impact on the fuel injection, mixing, and combustion processes. When the viscosity of the fuel is too low, the fuel injection system will not operate correctly, which will lead to inappropriate combustion. When the viscosity of the fuel is too high and is thick enough, the premature failure of fuel injection system is inevitable, which will cause the engine to freeze up. Temperature at which a volatile chemical produces vapours to produce an ignitable mixture with the oxygen in air is referred to as the flash point of the substance. Flash point is an important criterion to determine the safety of biodiesel and fuels, and also it is connected with the associated risks in handling of fuels while they are being transported.

Gas chromatography

The technique of gas chromatography involves injecting a gaseous or liquid sample into a solvent system, which is typically referred to as the carrier gas, and then passing the gas through a stationary phase to separate the compounds that are present in a mixture. The mobile phase is often an inert gas or a gas that does not react with other gases, such as hydrogen, helium, argon, or nitrogen. The GC is used in the process of separating and measuring gases and organic compounds.
Our GC analysis of the biodiesel and diesel samples revealed that there are common components between them. This indicates that the waste soybean oil used to produce biodiesel can be a viable fuel source and a potential alternative to diesel fuel. By offering similar chemical characteristics, waste soybean biodiesel presents a promising option for reducing reliance on traditional fossil fuels and transitioning towards more sustainable energy solutions.

Results and discussion

Characterisation of waste Soybean cooking oil (WSO)

Measurements were taken of the qualities of used cooking oil, including its viscosity, density, and flashpoint, amongst others. The details are presented in Table 1.

TABLE 1 Properties of Waste Soyabean Cooking Oil (WSO)

Properties	Experimental Value
Physical state	Liquid
Colour	Deep oily
Viscosity (mm^2/s at 40 °C)	54.53
Molecular Weight of oil (gm/mol)	864.5
Flash point (°C)	63

Characterisation of biodiesel and petroleum diesel

Biodiesel and petroleum diesel are compared with respect to their respective viscosity, density, flash point, etc. The details are provided in Table 2.

TABLE 2 Properties of biodiesel and petroleum diesel

Properties	Biodiesel	Petroleum Diesel
Density (27 g/cm^3)	0.52	0.42
Viscosity (mm^2/s at 20 °C)	5.89	4.26
Flash point (°C)	165	85

Characterisation of biodiesel

Biodiesel (made from WSO) has its physical properties tested, including its viscosity, density, flash point, fire point, and calorific values. Pure biodiesel produced from edible waste cooking oil has a density of around 0.52, whereas petroleum diesel has a density of about 0.42. Hence, refined biodiesel made from leftover cooking oil has a greater density than diesel. Pure biodiesel made from recycled cooking oil has a viscosity of roughly 5.89, compared to 4.26 for petroleum fuel. Since biodiesel made from recycled cooking oil has a higher viscosity than diesel, it is often blended with diesel to improve performance. The point at which a volatile substance evaporates into an ignitable mixture in the air is called its flash point. Biodiesel's and other fuels' flash points are crucial indicators of how safe the fuel is to use, store, and transport. Between 160 and 200 °C is where you'll find biodiesel's flash point. Pure biodiesel produced from edible waste cooking oil has a flashpoint of 165 °C, whereas diesel's flashpoint is 85 °C. As a result, refined biodiesel has a greater flash point than diesel.

Characterisation of biodiesel obtained from different sources

Measurements were taken to determine the properties of Biodiesel produced from fresh cooking oil and waste cooking oil, including viscosity, density, flash point, and other relevant characteristics. The details are presented in Table 3.

TABLE 3 Properties of biodiesel obtained from different sources

Properties	Biodiesel from Waste Oil	Biodiesel from Fresh Oil
Density (15 g/cm^3)	0.888	0.9
Viscosity (mm^2/s at 40 °C)	4.75	4.458
Flash point (°C)	170	170

Gas chromatography

Gas chromatography is a versatile analytical technique widely utilized across various domains due to its ability to perform qualitative and quantitative analysis. It is a rapid method with a high peak capacity, making it a valuable tool for analytical purposes.

The waste soybean oil and its biodiesel product was provided multiple rounds of washing and purification prior to conversion, and the yield was analysed using GC-MS and is shown in Figures 5-7. The technique provided scope for the identification of major constituents present in the waste soybean oil, thereby providing insight into the effectiveness of purification process prior to biodiesel production. Table 4 provides the details of integration results.

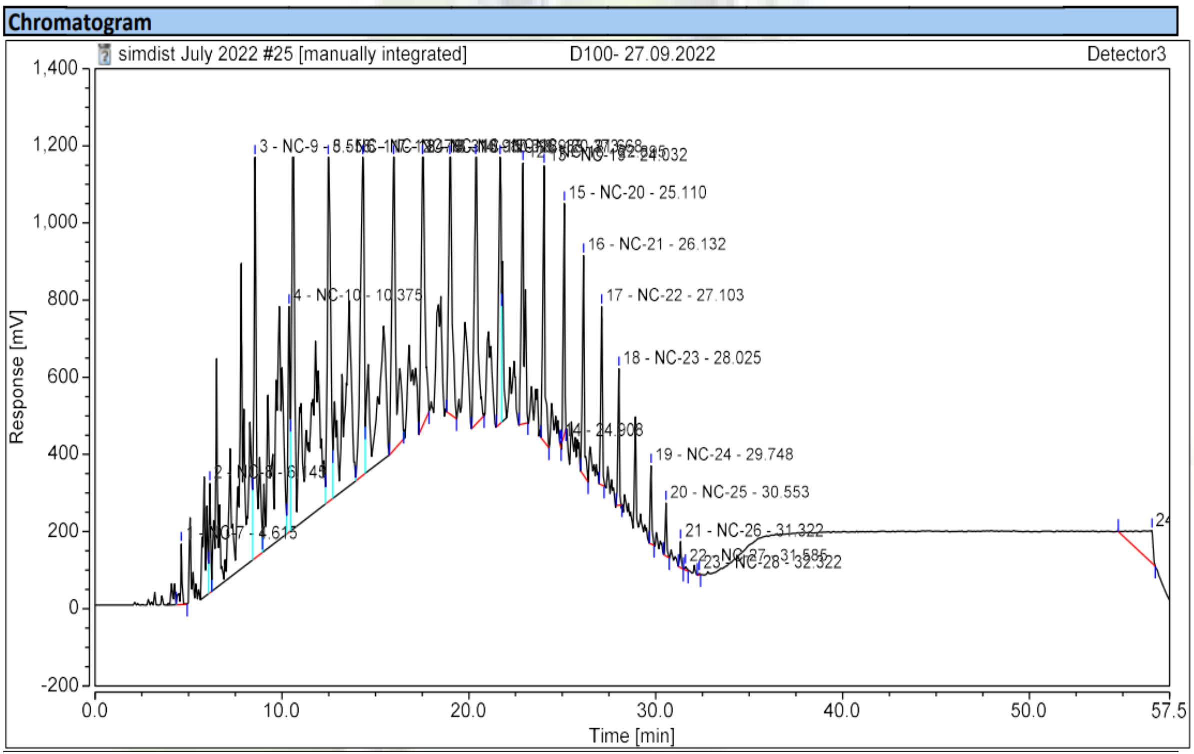

FIGURE 5 Graph representing petroleum diesel

TABLE 4 The details of integration results

Integration Results							
No.	Peak name	Retention Time min	Area mV*min	Height mV	Relative area %	Relative height %	Amount total %
1	NC-7	4.615	14.921	156.615	0.77	1.40	n.a.
2	NC-8	6.145	27.373	283.451	1.41	2.54	n.a.
3	NC-9	8.558	166.815	1038.567	8.59	9.29	n.a.
4	NC-10	10.375	70.997	584.394	3.66	5.23	n.a.
5	NC-11	12.478	181.018	892.909	9.32	7.99	n.a.
6	NC-12	14.318	191.439	824.538	9.86	7.38	n.a.
7	NC-13	15.980	210.150	758.892	10.82	6.79	n.a.
8	NC-14	17.522	163.187	696.428	8.40	6.23	n.a.
9	NC-15	18.983	150.939	665.453	7.77	5.95	n.a.
10	NC-16	20.373	145.862	692.059	7.51	6.19	n.a.
11	NC-17	21.668	105.930	690.178	5.45	6.18	n.a.
12	NC-18	22.895	116.350	676.161	5.99	6.05	n.a.
13	NC-19	24.032	94.388	715.485	4.86	6.40	n.a.
14		24.908	0.406	12.016	0.02	0.11	n.a.
15	NC-20	25.110	55.237	597.915	2.84	5.35	n.a.
16	NC-21	26.132	60.222	569.036	3.10	5.09	n.a.
17	NC-22	27.103	40.494	462.624	2.09	4.14	n.a.
18	NC-23	28.025	30.402	353.726	1.57	3.16	n.a.
19	NC-24	29.748	17.402	202.736	0.90	1.81	n.a.
20	NC-25	30.553	11.019	137.375	0.57	1.23	n.a.
21	NC-26	31.322	5.431	67.386	0.28	0.60	n.a.
22	NC-27	31.585	1.124	9.513	0.06	0.09	n.a.
23	NC-28	32.322	0.401	4.706	0.02	0.04	n.a.
n.a.	NC-29	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
n.a.	NC-30	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
24		56.563	80.549	84.583	4.15	0.76	n.a.
Total			**1942.055**	**11176.747**	**100.00**	**100.00**	

A total of 23 distinct components were identified during the GC-MS analysis of waste soybean oil, based on their chemical composition. This analytical method allowed for the separation and detection of individual components present in the mixture, which can include various fatty acids, glycerides, and other lipid-related compounds. Identification of components provides valuable insight into the quality and purity of the waste soybean oil sample, as well as any potential contaminants or impurities that may be present. The results of the GC-MS analysis can be used to optimize the purification process and ensure the production of high-quality biodiesel from the waste soybean oil. The identified components with their percentage, retention times, and area height, are listed in Table 5.

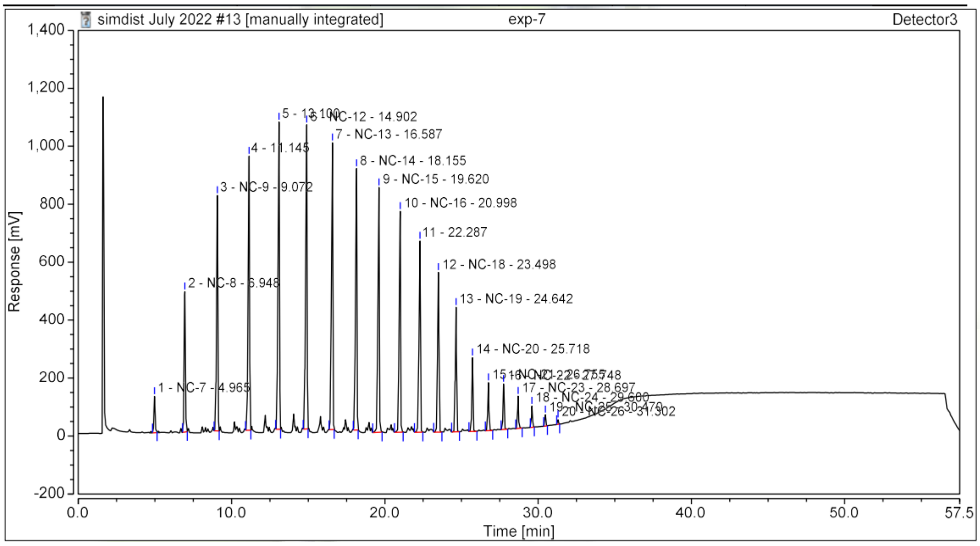

FIGURE 6 Graph representing biodiesel

TABLE 5 The details of integration results

Integration Results							
No.	**Peak name**	**Retention Time min**	**Area mV*min**	**Height mV**	**Relative area %**	**Relative height %**	**Amount total %**
1	NC-7	4.965	10.930	125.191	1.05	1.20	n.a.
2	NC-8	6.948	42.194	484.639	4.05	4.64	n.a.
3	NC-9	9.072	77.457	812.732	7.44	7.79	n.a.
n.a.	NC-10	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
4		11.145	98.412	946.188	9.46	9.07	n.a.
n.a.	NC-11	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
5		13.100	110.083	1060.888	10.58	10.17	n.a.
6	NC-12	14.902	112.396	1049.856	10.80	10.06	n.a.
7	NC-13	16.587	107.606	989.816	10.34	9.49	n.a.
8	NC-14	18.155	96.616	902.132	9.28	8.65	n.a.
9	NC-15	19.620	89.738	844.714	8.62	8.10	n.a.
10	NC-16	20.998	77.512	762.912	7.45	7.31	n.a.
n.a.	NC-17	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
11		22.287	65.072	659.625	6.25	6.32	n.a.
12	NC-18	23.498	52.170	551.168	5.01	5.28	n.a.
13	NC-19	24.642	36.971	429.329	3.55	4.11	n.a.
14	NC-20	25.718	20.841	254.238	2.00	2.44	n.a.
15	NC-21	26.755	12.730	165.654	1.22	1.59	n.a.
16	NC-22	27.748	12.004	156.408	1.15	1.50	n.a.
17	NC-23	28.697	8.626	111.593	0.83	1.07	n.a.
18	NC-24	29.600	5.346	73.561	0.51	0.70	n.a.
19	NC-25	30.470	2.769	38.419	0.27	0.37	n.a.
20	NC-26	31.302	1.131	15.661	0.11	0.15	n.a.
n.a.	NC-27	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
n.a.	NC-28	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
n.a.	NC-29	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
n.a.	NC-30	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Total			**1040.605**	**10434.723**	**100.00**	**100.00**	

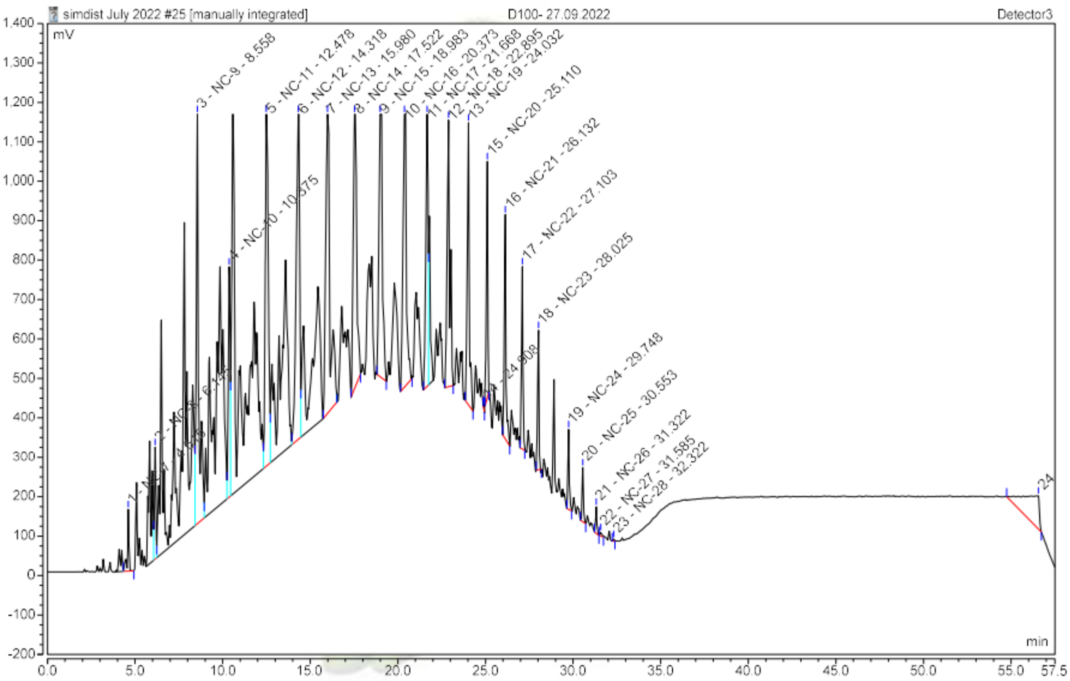

FIGURE 7 Comparison of petroleum diesel and biodiesel

Gas chromatography (GC) analysis conducted on Biodiesel (B100) and Diesel (D100) samples had similar spectra reflecting a similar composition. This study suggests that B100 and D100 share some common chemical features, indicating that B100 could be potentially used as a quality fuel as a replacement to traditional diesel fuel.

This ability biodiesel to act as substitute for diesel fuel can have a significant impact on the environment, as biodiesel is produced from renewable resources and emits lower levels of harmful pollutants compared to traditional diesel fuel. The results of GC analysis help to confirm that B100 can function as an effective alternative fuel source without compromising the quality or performance of the engine.

In addition, the similarity of composition between B100 and D100 observed through GC analysis may provide significant help to simplify the transition to biodiesel use in diesel engines, as the same fuel storage and transportation infrastructure used for diesel fuel can be used for biodiesel. Overall, these findings suggest that biodiesel produced from renewable sources such as vegetable oils can be a promising alternative fuel source to reduce the environmental impact of traditional diesel fuel.

Conclusion

After conducting an extensive study on the laboratory scale for production, synthesis, and characterization of biodiesel prepared from waste soybean oil, some pertinent conclusions can be made and they are as follows:

The research demonstrated that biodiesel produced from waste soybean oil has a great potential to serve as a viable and sustainable alternative to conventional fossil fuels.

a. Not only does this process reduce the dependence on non-renewable resources, but it also offers a practical solution to the problem of waste disposal in an effective manner thereby reducing the health risk and pollution.

b. The production of biodiesel from waste soybean oil is a cost-effective solution as the waste cooking oil can be sourced from various eateries and restaurants at almost zero cost. Furthermore, it reduces greenhouse gas emissions and provides a means of reusing a valuable resource that would otherwise go to waste.

c. In this research, we synthesized biodiesel by using waste soybean oil via transesterification using alcohol, and characterized by physico-chemical and spectroscopic instruments, after that we found the characteristics properties of this bio-diesel is quite similar to petroleum diesel.

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Conflict of Interest

The authors do hereby declare that there exists no conflict of interest in any form among them or with any other person.

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