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Research article

Experimental and theoretical assessment of phenomena linked with separation and purification of biodiesel from *Ricinus communis* seed oil

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ABSTRACT

This study investigated the phenomena associated with the separation and purification of biodiesel produced from *Ricinus communis* oil seeds using experimental and theoretical approaches. The alkaline transesterification technique was used to produce the biodiesel from the *Ricinus communis* oil seeds which were later compared with standards based on EN and ASTM. Experimental investigation of the components in the mixture for separation and purification was conducted using the standard turbidimetric method for binodal solubility and tie-line compositions. The gas chromatographic technique was used to determine the composition of the homogeneous mixture. Novel components separation and purification of the *Ricinus communis* seed oil biodiesel was achieved using ternary diagrams highlighting the constituent components of the biodiesel at different temperatures for enhanced separation and purification. At the coexisting extract and raffinate phases, the orientation angle of the component compositions increases as the methanol concentration increases and temperature increases. The analysis of seed oil in terms of its physicochemical properties showed density, refractive index, acid value, and free fatty acid values of 905 kg/m^3, 1.486, 0.79 mg KOH/g, and 0.390 mg KOH/g respectively. The fatty acid composition of the seed oil and biodiesel revealed that the major characteristics of the oil and biodiesel were defined by the presence of linoleic acid ($C_{18:2}$) and a ricinoleic acid hydroxyl group ($C_{18:1}$, OH) with compositions of approximately 30% and 20% respectively. Fourier Transform Infra-Red (FTIR) spectrometry analysis of the oil and biodiesel showed that the absorption spectrum in terms of the wave number (cm^{-1}) ranged from 1000 to 4000 cm^{-1} with esters as the main functional group providing the main structural backbone. The presence of different fatty acids leads to lateral homogeneity of the biodiesel molecules which can serve to organize the molecules into discrete domains with different properties for enhanced separation and purification at the investigated temperatures. Optimal separation and purification were achieved at the different temperatures showing the castor oil biodiesel, glycerol, and methanol components system at the prevailing composition, time, and temperatures from the tie-lines and binodal solubility compositions. This approach provides a means for the design of a more efficient separation process for optimal biodiesel purification after production with knowledge of how the components are distributed in the ternary mixture after the transesterification reaction. This, leads to greater efficiency of the process, reducing material and operational costs and eliminating

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environmental issues linked with the biodiesel production process as the volume of wastewater generated would be hugely eradicated. The findings of this study will be useful in the setting up of a small-to-medium-size biodiesel production facility with improvement in the efficiency of product separation and purification.

1. Introduction

With the current global economic crises; inflation, shortage of resources for power generation, and environmental problems like climate change and global warming, the growing need to utilize green materials for a greener ecosystem and bio-economy as a substitute for crude oil products (Kerosene, diesel, and petroleum) used for power generations, which have a high share in the rate of environmental degradation is highly recommended [1–5].

The energy needs of mankind over the last century have largely been obtained from fossil sources resulting in the generation of harmful and hazardous gases such as hydrocarbons, oxides of lead, carbon, sulfur, and other metals during processing and use as fuels [1,6–11].

Consequently, there is a necessity to adopt a cleaner, green, and environmentally friendly tactic for a sustainable environment, and the extant and future generations of humans benefit that being devoid of any environmental pollution. Hence, the use of plant-based materials for the production of renewable fuels has been heralded by many researchers because of the several advantages they portend [12–17].

Biomass from agricultural wastes like grasses, animal guano, corn, seeds, and hay can be transformed into biochemical, bio-plastics, pharmaceutical products, and green energy. Gemar et al. [5] reported that biologically transformed products gotten from plant biomass could be utilized to reduce pollutants in the environment which stands as a sustainable tool for environmental remediation. Biodiesel produced from non-edible vegetable oil is a viable response to mitigating environmental issues associated with the use of fossil fuels because of its green fuel and its purity level and properties [18,19]. Due to the gradual decline in fossil fuel reserves, renewable energy alternatives are assuming attractive options [20]. Biodiesel as a renewable source of energy is made up of fatty acid long chains called mono-alkyl esters. It is biodegradable and environmentally friendly when used in diesel or biodiesel engines, it reduces the emission of toxic gases and other toxic hydrocarbons [21,22].

The most widely used method for the synthesis of biodiesel from edible and non-edible renewable biomass remains the trans-esterification reaction that involves the use of excess alcohol. A homogeneous mixture is formed during the reaction process consisting of specific biodiesel, alcohol, and glycerol [23–25]. Industrial production of biodiesel is usually hindered due to component separation challenges in the homogeneous mixture system [26,27]. Other challenges include understanding the phenomena associated with the equilibrium behavior of the homogeneous mixture when the alcohol transits from the raffinate glycerol phase to the extract biodiesel phase during alcohol regeneration [28,29]. Essentially, differences in density are mostly linked to the immiscibility of the biodiesel and glycerol phases and thus, separation of the two-phase components requires greater time, resources, energy, and capital investment for a continuous biodiesel production and processing plant [30,31]. In this regard, the separation and purification of biodiesel from glycerol become the limiting step in the manufacture of biodiesel which requires the development of viable options and far-reaching research [29,32,33]. These choices enhance the design options and optimization objectives for emerging novel biodiesel production processes [34–36].

Biodiesel separation and purification are based on several unit operation processes such as adsorption, distillation, and extraction [37,38]. Therefore, it is highly expedient to understand the limits inherent in the different purification methods of biodiesel and also be able to select an appropriate combination of feedstock and purification steps to achieve consistency in the quality of the biofuel produced. Adequate knowledge of phase equilibrium separation and purification is crucial to the understanding of the end-product of the transesterification process, improvement of the reaction rate, selectivity of the desired product, and the separation process for the product mixture. Most research on the manufacture of biodiesel is centered on the transesterification process and how to improve the final product yield through operating parameters optimization. However, and regrettably so, only a handful of experimental results on equilibrium phase separation and purification of biodiesel mixtures, the significance of the phases separated after transesterification, and the distribution of the components between the phases have been reported to the knowledge of the authors [1,39–41]. Several studies revealed that these data were needed to elucidate extensively on the separation and purification of the process [41–44]. The result would help in the proper understanding of the process of purification and separation of the biodiesel-rich phase from the glycerol-rich phase thereby reducing energy and material losses. Studies conducted by different researchers [2,45] on several bio-diesels, revealed that limited works presently exist on the equilibrium phase separation and purification of biodiesel mixtures because of the extremely high variations in feedstock used and composition of methyl esters in the different biodiesel fuels.

The phase separation and purification of the produced biodiesel is shown as a ternary diagram with each component in the mixture at the sides/vertices/tips of the triangle having the maximum composition and representing 100% of the pure component. Gradual decrease or increase along the sides of the ternary diagram represents a composition of a mixture of three components with different mole or mass fractions but all summing up to 100% or 1, affecting the purity of the resulting mixture [45]. The grid lines represent equilibrium compositions at which all components are equally represented at different mole or mass compositions. A shift in composition would imply that the constituents' composition of all three components' mole/mass composition has been altered which affects the purity of the components in the mixture. ASTM and EN standards provide maximum limits for the presence of glycerol and methanol in any biodiesel. In this regard, 100% pure biodiesel constituents' composition is theoretically implied. Hence, any pure and

quality biodiesel system is expected to have the three components (specific biodiesel, glycerol, and methanol) at the specified standard limit [45]. The use of ternary diagrams in highlighting the separation and purification of biodiesel from *Ricinus communis* seed oil would help in this regard to provide a basis at the onset to achieve that level of separation and purification efficiency.

Knowledge of the phase distribution of the components in the biodiesel system using ternary diagrams provides a visual and clearer separation representation of the components in the biodiesel system with enhancement in the purification of the final product to meet the different standards for the quality of biodiesels. It also helps in the designing of a more efficient separation process for optimal biodiesel purification after production with knowledge of how the components are distributed in the ternary mixture after transesterification reactions [45]. This would lead to greater efficiency of the process, reduce material and operational costs and eliminate environmental issues associated with the biodiesel production process as the volume of waste generated would be hugely eliminated.

Most papers on the production of biodiesel are generally centered on the technique of transesterification and its enhancement. These include commercially available micro-wave apparatus, ultrasound-assisted procedure, and hydro-dynamic cavitation methods [46–48] which are representative of new and improving methods. More so, kinetic models, theoretical studies, and various optimization techniques are also used [46,49]. Some recent studies on the characterizations of biodiesel by Refs. [50–52] and the valorization of biodiesel by Refs. [53,54] have been carried out. Phase components distribution and composition modeling have been carried out by different researchers [16,17] and the Fourier transform infrared (FTIR) spectrometry evaluation on plant-based materials had also been employed [50,55]. Nonetheless, studies on the utilization of *Ricinus communis* - castor seed oil mainly looked at the characterization and possible biorefinery for the production of ethanol, biogas, and biodiesel [50,52,56].

Ricinus communis commonly called castor oil plant is a bean seed from the Euphorbiaceae family. The plants grow in the tropics with heights of about 10–13 m or about 30–40 feet. This tropical environment has a dual season that encourages the young plant to sprout year in and out. The leaves of the plants are fanlike with a beautiful lobed palmate feature consisting of 12 leaflets with spined-bristle red-bronze group bean fruits [57].

In Africa, the plant is grown for industrial, pharmaceutical, and commercial use because of its oil which has beneficial properties and functions. Fig. 1(a - d) show a typical plantation of the plant and the subsequent processes undertaken to obtain the seed from which the oil is extracted. However, Britannica, The Editors of Encyclopedia [57] reported that the oil contains ricin a noxious substance that has lethal properties when chewed. The oil by weight is about 30–55% depending on the variety and breed. Amongst vegetable oils, it has the highest viscosity with a molecular mass of 298 mg/ml [58] Fig. 1(a - d).

In the reaction to produce biodiesel and the successive separation and purification of the product mixture, it is imperative to

Fig. 1. a) Castor seed plant, b) Castor seeds, c) Crushed castor seeds, and d) Bare endosperm of *Ricinus communis* L. (castor seeds).

investigate the nature of the individual components' distribution in the homogeneous mixture at the investigated temperatures. This study investigates; using experimental and theoretical assessment, the phenomena linked with the separation and purification of biodiesel produced from *Ricinus communis* seed oil; elucidating the biodiesel components distribution in the homogeneous mixture after transesterification reaction at different investigated temperatures; evaluating the properties of the oil as a suitable substrate for biodiesel production and assessment of the produced biodiesel in comparison with global standards; and finally, evaluating the potential environmental benefit (s) of the biodiesel as a renewable energy source for greenhouse pollution reduction as against fossil energy utilization.

2. Materials and methods

2.1. Materials

The materials used for the experimental procedures in this study include distilled water; castor seed oil, castor seed oil biodiesel, glycerol >99%, and methanol >99%. The equipment used was gas chromatography/mass spectroscopy equipped with a flame ionization detector (GC/MS FID), beakers, stopwatch, conical flasks, temperature control water bath, pipette, diagnostic balance, power-driven agitator, and burette.

2.2. Methods

2.2.1. Preparation of the castor seed before extraction

The castor seed was prepared for oil extraction by removing the endocarp, sun-dried to lessen the moistness content, winnowed to detach the shell from the cotyledon to attain high production of oil, and finally pulverized to powder form using a crushing machine. The extraction of the castor oil from the castor seed was done using a hydraulic press equipped with a temperature controller. On completion of the oil extraction, the oil from the castor oil-bearing seeds was centrifuged severally at 1200 rpm for about thirty (30) minutes to eliminate any remnants in the oil. The castor oil was thereafter subjected to physicochemical characterization and biodiesel production [40].

2.2.2. Reaction mechanism in castor seed oil biodiesel production

Castor seed oil was utilized in the manufacture of castor oil biodiesel. The castor seed oil was extracted from the seed plant using a hydraulic press machine. Thereafter, the castor oil was subjected to a transesterification reaction with excess methanol in the presence of potassium hydroxide as the catalyst [59–61] after determining the extant properties of the oil as a suitable candidate for the production of biodiesel.

A 500 ml glass batch reactor was used to prepare the biodiesel from castor oil sourced from the castor seed using oil and methanol at a ratio of 1:6 correspondingly. 5%w/v catalyst was utilized with a temperature set at 60 °C. Reaction temperature greatly affects biodiesel yield and production. At higher temperatures (below the boiling point of the particular alcohol used; in this case B.P of methanol: 64.5 °C, the reaction rate becomes faster in the mixture and the rate of reaction is more, thus reducing the reaction time needed to overcome the process [62] which leads to increase methyl ester formation [63]. On the other hand, if the reaction temperature is above the boiling point of the alcohol used (Methanol), alcohol evaporation will occur and this would lead to a reduction in the volume of the mixture solution thereby negatively affecting the yield of the methyl ester obtained [64]. Most research revealed a high yield of biodiesel at reaction temperatures of 50 °C, 53 °C, and 60 °C using methanol, basic and acid catalysts with optimal methanol-to-oil molar ratios [65,66]. Most studies confirmed that higher reaction temperature within optimal values (50 °C–62 °C) do not increase biodiesel yield [67,68] for homogeneous catalysts and is also dependent on the feedstock. Research by Ref. [69] also showed that the activation energy barrier is usually overcome with increased temperature due to the nature of the feedstock. In this context, using an agitator or stirrer, the reaction condition would then be treated as a pseudo-homogeneous system with uniform composition with no mass constraint thereby allowing for chemical control of the entire process via kinetics [70,71]. All chemicals used were of high analytical grade [60,61]. The oil was first put into the chamber and later heated to the chosen temperature then the methanol/catalyst blend was introduced to get a desirable temperature. The temperature was maintained at 60 °C using a stirrer adjusted at 1200 rpm. A persistent agitation of the mixture was kept throughout the reaction. Potassium hydroxide at a concentration of 5 %w/v was used as the catalyst [60,61]. Potassium hydroxide (KOH) catalyst was used because of its superior catalyst properties; dissolves more easily in methanol and is less sensitive to water. Additionally, the glycerol by-product of processing with potassium hydroxide remains liquid and is easier to dispose of. With catalyst concentration increases beyond a certain limit or ratios in the transesterification reaction, the yield associated with the production of the biodiesel would reduce and the quality and purity of the produced biodiesel would also be affected due to the generation and formation of soap and glycerol above optimized level [61]. A typical reaction mixture consisted of 0.5 L of oil and 0.2 L of methanol giving a molar ratio of 6:1. This molar ratio was kept constant throughout the study while the reaction time was kept at 60 min. Knowing the reasonable methanol-to-oil ratio influences the final amount of methyl ester yield that would be obtained. Thus, if the volume of the methanol-to-oil is insufficient for the transesterification reaction, the biodiesel yield will be negatively affected (decrease in biodiesel yield) because the glycerides will not be converted into fatty acid methyl esters [72,73]. This situation is likely due to the high mass ratio of reactants maximizing contact between the alcohol and the oil molecules [74]. Similarly, with a high amount of methanol in the reaction solution, it is also likely that the polarity of the mixture may rise and this can enhance the solubility of the glycerol in the biodiesel phase which ultimately negatively affects the yield of the biodiesel [55,75,76]. Generally, an excessive amount/volume of alcohol is not recommended as it will reduce the methyl ester

yield [77], reduce or inhibit the recovery of glycerol and affect the economies of the process [45,78]. Hence, most studies maintain a methanol-to-oil ratio of 6:1, optimal catalyst concentration, and optimal reaction temperature. Then the reaction chambers were opened and the contents were drained to signify the end of the reaction. The mixture and the separating funnel were permitted to isolate into 2 layers with the lower stage consisting of methanol and glycerol, and the upper stage consisting of the desired biodiesel. Later, the lower stage was allowed to drain off. Then the biodiesel formed at the top stage was washed away with 0.01 M concentrated H_2SO_4 which was used to counterbalance any residue of the catalyst. Thereafter, 1 L of ultra-distilled water was used to wash off any soap and glycerol residues. Later, the samples from the castor seed oil biodiesel were centrifuged using a Mark IV, Auto Bench, Baird centrifuge at a temperature of 5 °C run for 10 min at 1200 rpm. This was done to eliminate any hidden soaps and water with the samples of the biodiesel. Later, the samples were preserved in small bottles [60,61]. Generally, biodiesel yield reduces when there is an increase in the catalyst concentration (an excessive amount of catalysts) because of soap formation. This also affects the quality of the produced biodiesel. Excess amounts of catalyst concentration would result in the formation of emulsion that can enhance the formation of gels [79]. More importantly, basic catalysts used in transesterification reactions are very sensitive to water. When water is present in the reaction medium, esters saponification may be induced in such basic conditions. Thus, with the presence of the excess amount of catalyst concentration, an induced shift of equilibrium of the reaction mixture towards the backward reaction will/or may cause a decrease in the conversion of the oil to biodiesel and glycerol [80]. The biodiesel seed oil obtained after separation from the glycerol layer was then rinsed off carefully with ultra-distilled water to wade any traces of scum or impurities. Thereafter it was dried using an oven (Thermotech TIC – 4000E) to remove any trace of methanol and water. The biodiesel seed oil obtained from the castor oil was later analyzed with a mass spectrometer/Auto-Sampler gas chromatography (Thermo Scientific Trace GC Ultra AS 300) linked with an FID (flame ionization detector) [60,61].

2.2.3. Castor seed oil biodiesel and castor seed oil characterization

The oil and biodiesel were exposed to several physicochemical analyses based on relevant and current standards applicable to oil and biodiesel analysis (AOCS, IS0/TR 210, IS0 212, and ISO/TR 21092). The various properties such as viscosity, calorific value, FTIR, cloud, flash, fire, and smoke points, the content of the ash, density, content of the moisture, RF (refractive index), SP (specific gravity), the acid, fatty, peroxide, iodine, and saponification values were investigated [81].

Using the GC (gas chromatography) method and following the FAME norm EN 14214, EN 14103, and EN 14105 standards, the content of seed oil of the castor/biodiesel was investigated. The FID and the PSP (polar stationary phase) were used to analyze the biodiesel/seed oil of the castor plant using the procedure of [81–83].

The GC configuration (Trace GC Ultra) used was fused with a 5MS capillary silica column close-fitting with an FID and an SP (Splitless injector). Elite-Famewax was used as the major analytical column [82,83]. The temperature used for the inlet was kept at 250 °C with a flow rate at the column of 1 ml/min. In addition, a split rate of flow (50 ml/min) was also maintained at an injection concentration in the volume of 0.5 μl [82–84]. The final and initial temperature of the furnace was maintained at 230 and 210 °C correspondingly with a hold time of 1 and 2 (13.00 and 15.00 min) respectively at an equilibria rate of 0.00 min. The column film Elite – Famewax was used with a dimension of 30 m × 320 μm × 0.25 μm with a ramp1 set of 5 °C/min. The gas used was Helium and the FID was set at 250 °C. The air and hydrogen flow rates were 450 and 45 ml/min respectively with a range of −5 to 1 [82–84]. This was used to profile the castor biodiesel seed oil and the castor seed oil for feedstock production.

2.2.4. Components composition phase analysis

The component's composition at the different temperatures was investigated using cloud-point titration techniques as employed by Refs. [85–88].

2.2.5. Determination of the composition of castor seed oil biodiesel extract phase

A micro-burette was used to obtain the initial volume of glycerol as 25 ml. Using an analytical balance (XP 3000), the mass of glycerol was determined with an instrument precision of ±0.00006 g [89]. Thereafter, 10 ml of castor seed oil biodiesel and 15 ml of methanol were independently measured, weighed, and placed in different beakers. The measured quantities of castor seed oil biodiesel and methanol were afterward thoroughly mixed in a 250 ml beaker. Titration was performed using a micro-burette containing glycerol into a mixture of castor seed oil biodiesel and methanol. The mixture was mechanically agitated using a magnetic stirrer (Fisatom 752A) [89]. The point of termination of titration was when there was a color change in the blend from crystal clear to turbid or cloudiness which was a well-thought-out saturation point of the glycerol in the castor seed oil biodiesel-methanol mixture. The technique was done at different compositions of glycerol in the castor seed oil biodiesel/methanol mixture to provide the component compositions for the castor seed oil biodiesel extract phase system using GC analysis [89].

2.2.6. Determination of the composition of glycerol raffinate phase

The initial volume of castor seed oil biodiesel was determined using a micro-burette of 25 ml. Thereafter, the mass of the biodiesel was determined using a Denver instrument XP 3000 analytical balance with a precision of ±0.00006 g. Weighing independently 10 ml of glycerol and 15 ml of methanol, the liquids were placed into different beakers and thoroughly mixed in a 250 ml beaker. Titration of castor seed oil biodiesel was initiated using a micro-burette into the mixture of glycerol and methanol in the 250 ml beaker and stirred with a Fisatom 752A magnetic stirrer. Observing the mixture change from transparent to turbid, the titration procedure was terminated. This was considered to be the saturation point of biodiesel in the glycerol-methanol mixture. The final volume and mass of biodiesel left in the burette at the saturation point of biodiesel in the glycerol-methanol solution were determined. The technique was repeated at different compositions of biodiesel, glycerol, and methanol in the glycerol/methanol mixture to obtain the binodal

solubility curve data in the glycerol lower–rich phase system using GC [90].

2.2.7. Determination of component compositions (tie lines compositions)

The three components in the homogeneous mixture namely, castor seed oil biodiesel, glycerol, and methanol were prepared in a 250 ml conical flask such that the proportion of castor seed oil biodiesel - glycerol was constant while the volume of methyl alcohol was progressively amplified. Using a Fisatom 752A magnetic stirrer and a vortex touch mixer, the solution was thoroughly agitated for 4 h to ensure homogeneity. The mixture was thereafter maintained in a thermostatic water bath with a temperature regulator at the investigated temperatures. After twenty-four (24) hours of idleness, equilibrium was observed to have been achieved as the homogeneous mixture had separated into two liquid phases as also reported by Refs. [17,41,85,87,91]. These were castor biodiesel's seed oil extract phase and glycerol raffinate phase. Using calibration curves obtained by the gas chromatography technique, the mass fractions of the various phase compositions were determined.

3. Results and discussion

3.1. Physicochemical analysis of the investigated castor seed oil

Table 1 presents the physicochemical results of the analysis of castor seed oil. From the results of the table, the values of the properties fall within the range of the standard used for this study (AOCS (American Oil Chemists Society), EN14214, ASTM D6751, and ASTM D9751). The properties of the oils such as refractive index, peroxide value viscosity, free fatty acid, iodine value, and saponification values are utilized as specifications of the oil characteristics. Whereas the rest parameters are only empirical in nature, however, they are important in the identification of oil in the plant seed [81,92]. It could be seen from Table 1 that castor seed oil with a density value of 905 kg/m^3 was lower than that reported by Ref. [59] (948 g/cm^3) and [93] (958.7 g/cm^3) for same plant seed under this current study. The percentage moisture content of plant seed oil was 0.3 as stated in Table 1. The low content in moisture content was an indication of an effective distillation process in the recovery process and also a sign of the good storage capacity characteristics of the oil [94]. The refractive index obtained for the castor seed oil was 1.486. The refractive index is an indicator of the optimal clarity level of the plant seed oil and oil biodiesel relative to water. The value of the refractive index of the castor seed oil was a sign of the point of saturation of the oils. The saponification value of any oil is an indication of its suitability of such oil for industrial applications [95–97]. It also has an inverse proportionality to the molecular weight of such oil [12]. The saponification value increased with an increase in moisture content. The iodine rate measures the proportion of unsaturated acids present in the oils. It measures the degree of unsaturation of the oil [12]. Oils having iodine values of more than 130 are referred to as drying oils while oils with iodine values ranging from 100 to 130 are known as semi-drying oils. Non-drying oils have iodine <100. Therefore, the castor seed oil is within non-drying and semi-drying oils. According to EN14214 (European committee for standardization), the iodine values of oils to be used for biofuel (biodiesel) production must be less than 120 I_2/100 g. Oils having high unsaturation of fatty acids when subjected to heat are prone to polymerization of the glycerides. This then leads to the formation of deposits and could cause oxidative instability [12]. The susceptibility of the oxidation of oils is linked to the amount of iodine present in the plant sample. This also implies that a lesser amount of H_2 would be required for industrial purposes to convert the unsaturated oil. Meanwhile, the value of the peroxide gotten in this study could be used to estimate the primary oxidation phase of the oil whose basic materials or products are hydrocarbons which are later converted to water via oxidation. This can result in rancidification of the oil at the point of storage and this defines the amount of oxidation that will happen before the final examination.

The value obtained for the plant seed oil was 0.4. The low amount implied that the oil was stable with a high level of oxidative

Table 1
Physicochemical scrutiny of castor seed oil.

Analysis conducted	Results obtained
Color	slightly pale golden
Density (g/cm^3)	0.905
Moisture content (%)	0.300
Refractive index	1.486
Saponification value (mg KOH/g Oil)	171.10
Iodine value	85.61
Peroxide value (mleq. oxy/Kg)	0.40
Acid value (mg KOH/g)	0.790
Free Fatty Acid (%)	0.390
Viscosity (mm^2/s)	4.80
Smoke point (°C)	186
Fire point (°C)	>260
Flashpoint (°C)	>260
Cloud point (°C)	−10
Pour point (°C)	−2
Titre temperature (°C)	−14
pH	5.80
Turbidity	5.00
Yield (%)	38.321

stability and less prone to rancidity in storage. A low peroxide value is indicative of the low amount of oxidative rancidity of the oil and also a strong or high presence of antioxidants [95]. The free fatty and acid value are excellent indicators of the age and quality of the oil or fat. Low acid values enhance the stability of most oils while low values of the free fatty acid implied that the oils contain acids that are not combined with glycerol and thus do not easily break down nor become rancid, an indication of little or no lipase action. The acid value of castor seed oil was 0.79 mg KOH/g while the free fatty acid value was 0.39%. The value was within the ranges for the free-fatty acid of oils which was anticipated to be flanked by 0.00–3.0% before the oil could be used for industrial application. However, the FFA could be modified by subjecting it to the refining process to improve its quality for industrial purposes. According to Ref. [98], fatty acids are frequently used as an indication of the edibility and conditionality of oils. The pour, cloud, flash, fire, and smoke points showed that the plant seed oil had huge potential as fuel for industrial use or application. The smoke, flash, and fire points of the oils showed a linear association with the free-fatty acid content found in the oil. This is an indication of the combustion capacities of the various oils [99,100]. Adejumo et al. [98] stated that the smoke point tended to be higher for oil extracted at lower seed moisture content than that at higher seed moisture content while [95] described the smoke point as the temperature at which the first smoke was detected.

3.2. The results of the fatty acid composition of the castor seed oil

The fatty acid composition of the castor seed oil is presented in Table 2. From the result, castor seed oil has five major fatty acid groups. The major characteristics of the oil were defined by the presence of the ricinoleic acid hydroxyl group which influenced the behavior of the oil. The oil was observed to consist mainly of highly monounsaturated acids and therefore very useful for biodiesel production due to its high level of mono-unsaturation [81,101,102]. The values of the fatty acid composition of the plant seed oil were in consonant with what was obtained by Refs. [41,59,103–105]. Ramos et al. [105] stated that fatty acid with double bonds (=) has the best quality as a first-class choice for the production of biodiesel because the components of biodiesel and oil are practically the same even after the process of transesterification.

3.3. The properties of the produced castor seed oil biodiesel

Fig. 2 presents the results of the examination of the properties of castor plant seed oil biodiesel. The mean and standard error values show the saponification values were very high at 117.51 mgKOH/g. Meanwhile, the free fatty acid and acid values were low at 0.00% and 0.00 respectively.

3.4. Evaluation of parameters of castor seed oil biodiesel with the diverse standards

Densities, as well as specific gravities, are important properties of consideration for diesel fuel injection and combustion systems. From Table 3, it can be observed that the values obtained were somewhat higher than the set limits of ASTM D9751, EN 14214, and ASTM D6751. Though, the value for castor seed oil biodiesel was slightly higher than the standards. The free fatty acid value obtained was observed to be consistent with the various standards of ASTM D9751, EN 14214, and ASTM D6751. The acid value of the castor oil biodiesel obtained after the transesterification process of the castor seed oil was considered to be good when compared with similar oils in fuel production [106,107]. The ash content of castor seed oil biodiesel was above the limits of the different standards (ASTM D and EN) which could likely be due to the presence of mineral elements in the biodiesel [108]. The conductivity of the castor seed oil biodiesel is quite high which could be ascribed to the influence of the OH^- group in its fatty acid backbone structure. The viscosity of castor seed oil biodiesel falls within the range of values as defined by ASTM D and EN and therefore met the minimum requirement. The viscosity parameter helps in the determination of the operational, storage, and optimal handling conditions of the biodiesel to improve flow characteristics thereby ensuring an adequate supply of fuel in the injector at the different operating temperatures. Interestingly, the viscosities of seed oils generally decreased after the transesterification reaction and this is important in enhancing the efficiency of engines as many diesel engines use highly technological injection pumps which are intolerant to viscous fluids due to fuel filter clogging. High-viscosity fuels could form oil droplets on injection and could result in poor atomization of the fluid. Similarly, low-viscosity oils could produce biodiesels with low viscosity resulting in insufficient lubrication for a precision fill of fuel injection pumps [109,110]. The iodine value (I.V) indicated the number of double bonds (=) in the fatty acid mixture in the biodiesel. It indicated the total degree of unsaturation of the biodiesel. The iodine value of the castor seed oil biodiesel falls within the ASTM D and EN standard specifications though the value was within the ASTM D9751 and EN 14214 limits.

Table 2
Fatty acid composition (FFA) (wt. %) of castor seed oil.

Analysis	Results obtained
Palmitic ($C_{16:0}$)	0.76
Oleic ($C_{18:1}$)	2.82
Stearic ($C_{18:0}$)	0.96
Linoleic ($C_{18:2}$)	4.61
Linolenic ($C_{18:3}$)	0.33
Ricinoleic ($C_{18:1,}$ OH)	89.58
Dihyroxylstearic ($C_{18:2,}$ OH)	0.94

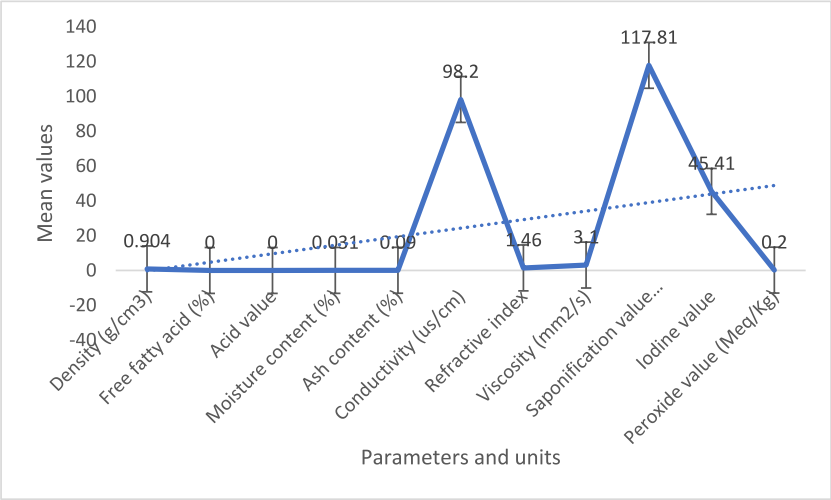


Fig. 2. Properties of the produced castor seed oil biodiesel.

Table 3
Comparison of obtained parameters of the castor seed oil biodiesel with the diverse set limits.

Parameters	Produced castor seed oil biodiesel	ASTM D9751	ASTM D6751	EN 14214
Density (Kg/m^3)	904	850	880	860–900
FFA (%)	0.003	0.31	0.25	0.25
Acid value (mg KOH/g)	0.012	0.062	0.50	0.50
Moisture content (%)	0.031	–	–	–
Ash content (%)	0.09	0.01	0.02	0.02
Conductivity (us/cm)	98.2	–	–	–
Refractive index	1.4600	–	–	–
Viscosity (mm^2/s)	3.10	2.6	1.9–6.0	3.5–5.0
Saponification value (mg KOH/g)	117.81	–	–	–
Iodine value (Wij's)	45.41	42–46	–	120 max
Peroxide value (Meq/Kg)	0.20	–	–	–
Flashpoint (°C)	130	60–80	100–170	120
Fire point (°C)	33	–	–	–
Cloud point (°C)	−15	−20	−3 to 12	–
Pour point (°C)	−12	−35	−15 to 16	–
Smoke point (°C)	20	–	–	–

ASTM: American Society for Testing and Materials EN: European Committee for Standardization.

The cold flow properties of the castor seed oil biodiesel determined were smoke, pour, cloud, fire, and flash points settings. These properties were essentially low-temperature fuel parameters that were needed to specify the biodiesel fuel usage and application. Standards for the pour, cloud, and flash points are contained in the ASTM D9751, ASTM D6751, and EN 14214 while that for fire and smoke points are not in the biodiesel standard though they are still required to be reported due to climatic conditions of the different countries of the world. The pour point value of the castor seed oil biodiesel was observed to be very low. This was advantageous because the pour point described the lowest temperature at which frozen oil could flow and was used to describe the cold temperature instability of fuel oils. In this regard, the values obtained for the pour point of the castor seed oil implied that the biodiesel produced

Table 4
Ricinus communis biodiesel fatty acid profile and other parameters.

Component	Retention time	Concentration (ppm)	% Concentration
Laurate (lauric) ($C_{12:0}$)	5.100	5.5454	9.949
Heptadecanoate (heptadecanoic) ($C_{17:0}$)	10.070	0.9641	1.729
Myristate (myristic) ($C_{14:0}$)	15.093	4.5861	8.229
Arachidate (arachidic) ($C_{20:0}$)	20.313	4.3316	7.772
Linoleate (linoleic) ($C_{18:2}$)	25.350	16.8251	30.188
Palmitate (palmitic) ($C_{16:0}$)	30.073	7.0597	12.667
Stearate (stearic) ($C_{18:0}$)	35.106	5.3446	9.590
Ricinoleate (ricinoleic) ($C_{18:1,}$ OH)	40.060	11.0772	19.875
Total		55.7337	

would perform excellently in temperate as well as tropical regions of the world.

3.5. Fatty acid constituents of the produced castor seed oil biodiesel

The result of the fatty acid profile of the *Ricinus communis* oil biodiesel, retention time, concentration in parts per million, and percentage concentration is shown in Table 4. It could be seen that linoleate (linoleic) ($C_{18:2}$) and ricinoleate (ricinoleic) ($C_{18:1,}$ OH) had the strongest influence on the produced biodiesel with the composition of approximately 30% and 20% respectively of the fuel property. This result aligns with a study conducted by Refs. [56,81] on castor oil biodiesel and other associated issues. Thus, the main character of the biodiesel was defined by the high concentration of the methyl linoleic and ricinoleate esters which was in agreement with what was obtained in the fatty acid constituents of the corresponding castor seed by Ref. [105]. Ramos et al. [105] also reported that the composition of fatty acids from the seed oil and biodiesels remained fairly the same with minimal variations even after the process of transesterification. This behavior was observed for the castor seed oil and the castor seed oil biodiesel. The main fatty acid for the biodiesel and castor oil in this study was observed to be linoleic acid and ricinoleic acid respectively and this influenced the character of both oil and the biodiesel.

Table 5 shows the data of the FAME(s) composition of the *Ricinus communis* biodiesel as determined by GC-MS. The highest retention time (40.060) was in the C18:1, OH components, having a concentration of 11.0772 ppm in the castor plant. Meanwhile, the lowest retention time (5.100) was in the C12:0 component having a concentration of 5.5454 ppm in the castor plant.

3.6. Fourier Transform Infra-Red (FTIR) spectrometry analysis of castor seed oil biodiesel

From Fig. 3, twenty (20) identified peaks were observable in the FT-IR spectrum for castor seed oil biodiesel sample analysis. The absorption spectrum in terms of the wave number (cm^{-1}) ranged from 1000 cm^{-1} to 4000 cm^{-1} with esters as the main functional group providing the main structural backbone. At the lowest peak position of 718.327, the vibrational type causing the IR absorbance was a C–O stretching at a peak area of 10.844. The absorbance range was about 650 cm^{-1}. However, the absorbance band was centered at 718.327 cm^{-1}. The highest absorbance band was centered at 3826.837 cm^{-1} which represented the lowest transmittance while the lowest absorbance band was centered at 2957.001 and 1853.126 respectively which also represented the highest transmittance. The castor seed oil biodiesel spectrum in Fig. 3 showed the molecular absorbance and transmittance of the castor seed oil biodiesel creating the molecular fingerprint of the castor seed oil biodiesel that was unique only to the sample [55,111]. Some of the bonds of the atoms constituting the castor seed oil biodiesel sample were vibrational stretching (C–O) and bending type with the main functional group being the ester functional group. It was observed that the peak area and size were indications of the characteristics of the biodiesel and the functional group. The functional groups present included = C–H, C–O, CH_2, C═O, C≡C, and O–H with types of vibration causing the IR absorption ranging from bending, stretching, split rocking, bending/rocking, and symmetrical/stretching at the different wave numbers (cm^{-1}).

3.7. Spectrometric analysis of castor seed oil using Fourier Transform Infra-Red (FTIR) technique

Fig. 4 shows the FTIR spectrum of the castor seed oil which similarly had twenty (20) identified absorbance peaks. The FTIR spectrum was quite different from that of the corresponding castor seed oil biodiesel owing to the occurrence of a fatty acid component in the biodiesel. The wave number (cm^{-1}) was from 1000 to 3800 cm^{-1} with the lowest absorbance being at the center point of 3500.854. The type of vibration at that position was bending with = C–H functional group. Other prevailing functional groups in the CASO sample were C–*O*–C, C–O, CH_2, C═O, C≡C, and O–H groups with different vibration types such as bending, bending/rocking, stretching, and symmetrical/stretching. The highest absorption band was centered at 3913.418 cm^{-1} which represented the lowest transmittance while the lowest absorbance band was centered at 3503.65413 (−1.5 abs) and 3500 cm^{-1} with a peak area of 28.16557 which also represented the highest transmittance and showed the molecular fingerprint of the castor seed oil thereby creating a unique identity for the sample [55,111].

Table 5
Data of the FAME(s) composition of the *Ricinus communis* biodiesel as determined by GC-MS.

Component	Retention time	Area	Height	Concentration	Unit
$C_{12:0}$	5.100	16374	913.097	5.5454	Ppm
$C_{17:0}$	10.070	7159.22	406.391	0.9641	Ppm
$C_{14:0}$	15.093	10257.0332	580.238	4.5861	Ppm
$C_{20:0}$	20.313	11146.4474	626.675	4.3316	Ppm
$C_{18:2}$	25.350	7906.5730	448.311	16.8251	Ppm
$C_{16:0}$	30.073	3571.0450	203.072	7.0597	Ppm
$C_{18:0}$	35.106	10327.8121	568.664	5.3446	Ppm
$C_{18:1}$,OH	40.060	5552.5835	315.551	11.0772	Ppm
Total		72294.8239		55.7337	

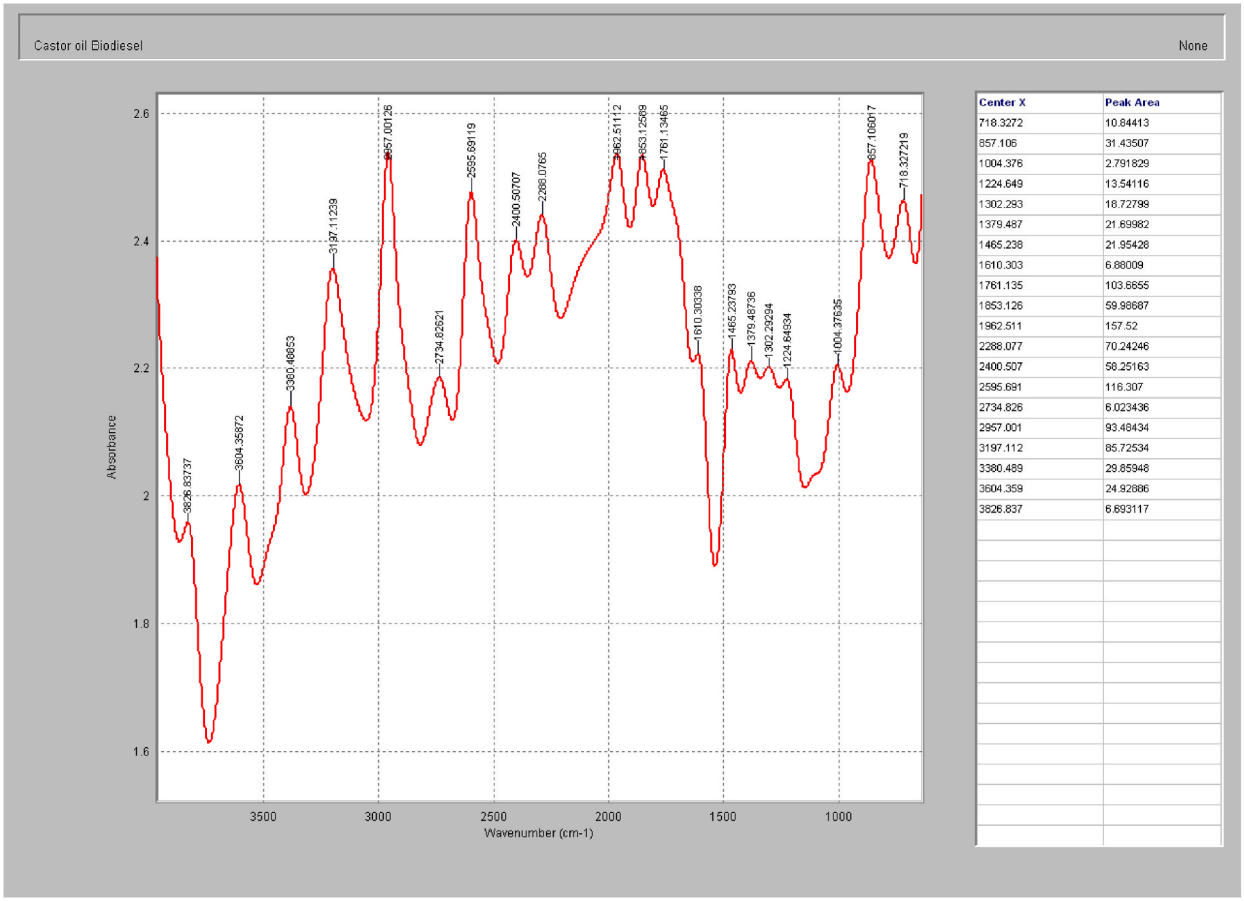
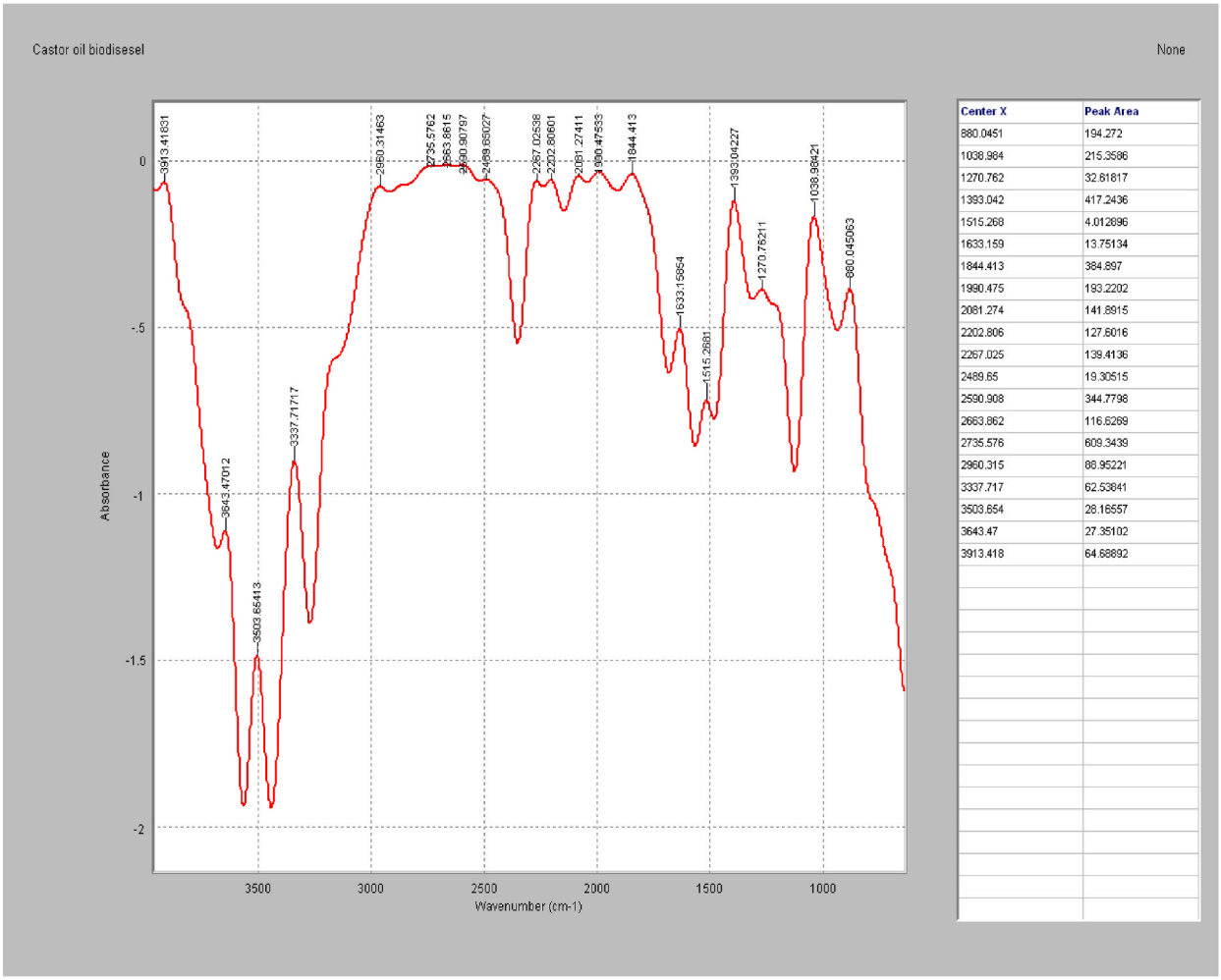


Fig. 3. Castor seed oil biodiesel FTIR spectra.

3.8. Components composition analyses for a homogeneous mixture of castor seed oil biodiesel system at investigated temperatures

The component compositions in Fig. 5(A–D) revealed higher solubility of the castor seed oil biodiesel in the glycerol raffinate phase. This phenomenon was more pronounced at temperatures of 20 °C, 30 °C, and 60 °C as evident in the plot. The higher solubility was because the biodiesel component of the ternary mixture was composed primarily of methyl linoleic and ricinoleate which formed the backbone of the fatty acid and thus, influenced the solubility in the mixture composition. Investigation of the fatty acid profile of the castor seed oil biodiesel as seen in Tables 4 and 5 and Fig. 4 showed that the main fatty acid component was methyl linoleic and ricinoleate which accounted for about 50.1% of the character of the biodiesel. This was observed to align with the work of [16,17,41,112]. This ester is composed of an acid and a hydroxyl group that readily forms hydrogen and dipole-dipole bonds with methanol and glycerol. This bonding system enhances the system's mutual solubility. The components composition plots of Fig. 5(A–D) also reveal a gradual increase in solubility with increasing temperature. However, these increases in solubility did not adversely affect the solubility and phase characters at the different investigated temperatures for the biodiesel homogeneous mixture. Further observations show that the basic parameter defining the character and solubility of the castor seed oil biodiesel phase composition was the amount of methanol in the solution. The amount of methanol in the solution affected the stability and solubility of the biodiesel extract phase. Generally, the solubility of glycerol in the biodiesel extract phase was slightly higher than that of the biodiesel in the glycerol raffinate phase. This was observed at all the investigated temperatures. In addition, the composition plots in Fig. 4 showed that the homogenous region increases with an increase in temperature which has an inverse relationship with the two-phase region. This relationship is true because, with an increase in temperature, the region of a uniform composition containing more castor seed oil biodiesel increases while the region of a composition containing more of the glycerol decreases; hence the shrinkage in the two-phase region with increasing temperature observed in the castor seed oil biodiesel ternary mixture. Similarly, since the methanol readily solubilizes in the raffinate phase, the homogeneity of the extract phase was generally enhanced as a result of an increase in temperature. The mixable nature of the extract and raffinate phases were quite high with the methanol observed to be dispersed between the extract phase but with a larger amount of the methanol resident in the raffinate phase in agreement with the results obtained by other studies by Refs. [16,113]. Furthermore, while methanol was seen to be a very good solvent for glycerol separation in the homogeneous mixture, at a high methanol-to-biodiesel mass ratio, the density difference between the top and lower phases of the mixture tended towards zero.



Center X	Peak Area
880.0451	194.272
1038.984	215.3586
1270.762	32.61817
1393.042	417.2436
1515.268	4.012696
1633.159	13.75134
1844.413	384.897
1990.475	193.2202
2081.274	141.8915
2202.806	127.6016
2267.025	139.4136
2489.65	19.30515
2590.908	344.7798
2663.862	116.6269
2735.576	609.3439
2960.315	88.95221
3337.717	62.53841
3503.654	28.16557
3643.47	27.35102
3913.418	64.68892

Fig. 4. Castor seed oil FTIR spectra.

Consequently, because of this phenomenon, the residence time for phase separation increased to very high values and phase separation became practically impossible. This could only be achieved with the aid of a centrifuge. Adding methanol thereafter led to phase inversion and the residence time was found to be very high approaching approximately seventy-two (72) hours. The inversion of phases was because the density of the raffinate phase decreased by adding more methanol to the mixture of glycerol, biodiesel, and methanol. Essentially, to have an optimum operation, the composition of the homogeneous mixture must be at a methanol-to-biodiesel ratio of approximately 1–3 or less to prevent phase inversion. According to Refs. [16,113], although methanol can successfully enhance separation and be used as a solvent in the purification of biodiesel from a homogeneous mixture of biodiesel, methanol, and glycerol, its lengthy residence time required for separation could be improved upon to enhance its practical industrial use. In the laboratory operation, its use was efficient and effective. Due to the phenomenon of the formation of methanol in the single-phase liquid and inversion phase, it should be advised that the use of methanol as a solvent in the separation and purification process of glycerol from biodiesel must be cautiously handled. Biodiesel and glycerol are immiscible in the absence of methanol. Biodiesel, methanol, and glycerol mixture systems are polar mixtures and highly non-ideal systems. They are blends of constituents with significantly diverse solubility parameters and as such, their activity coefficients were much greater than unity. Since the solubility parameter was sufficiently different, immiscibility was found to be prevalent and this confirmed the general trend that polar molecules like methanol and glycerol tend to have high solubility parameters while non-polar molecules like fatty acid alkyl esters (biodiesel) have low solubility parameters. According to Refs. [114,115], substances with solubility parameters different by certain empirical values will generate two liquid phases. Hansen [114], and Spange et al. [115] postulated that the solubility parameter was related to the solvent polarity parameter. This parameter was a measure of the molecule's ability to participate in dispersion forces, dipole-dipole interactions, hydrogen bonding, and dielectric interactions. The homogeneous mixture of castor seed oil biodiesel, glycerol, and methanol in our current research is generally separated into two liquid phases. The castor seed oil biodiesel in this work was composed primarily of six different methyl esters with close solubility parameters thus, forming one single phase. The values of the solubility parameter of methyl oleate were taken as representative of the solubility parameter for all other types of methyl esters in the biodiesel for ease of analysis.

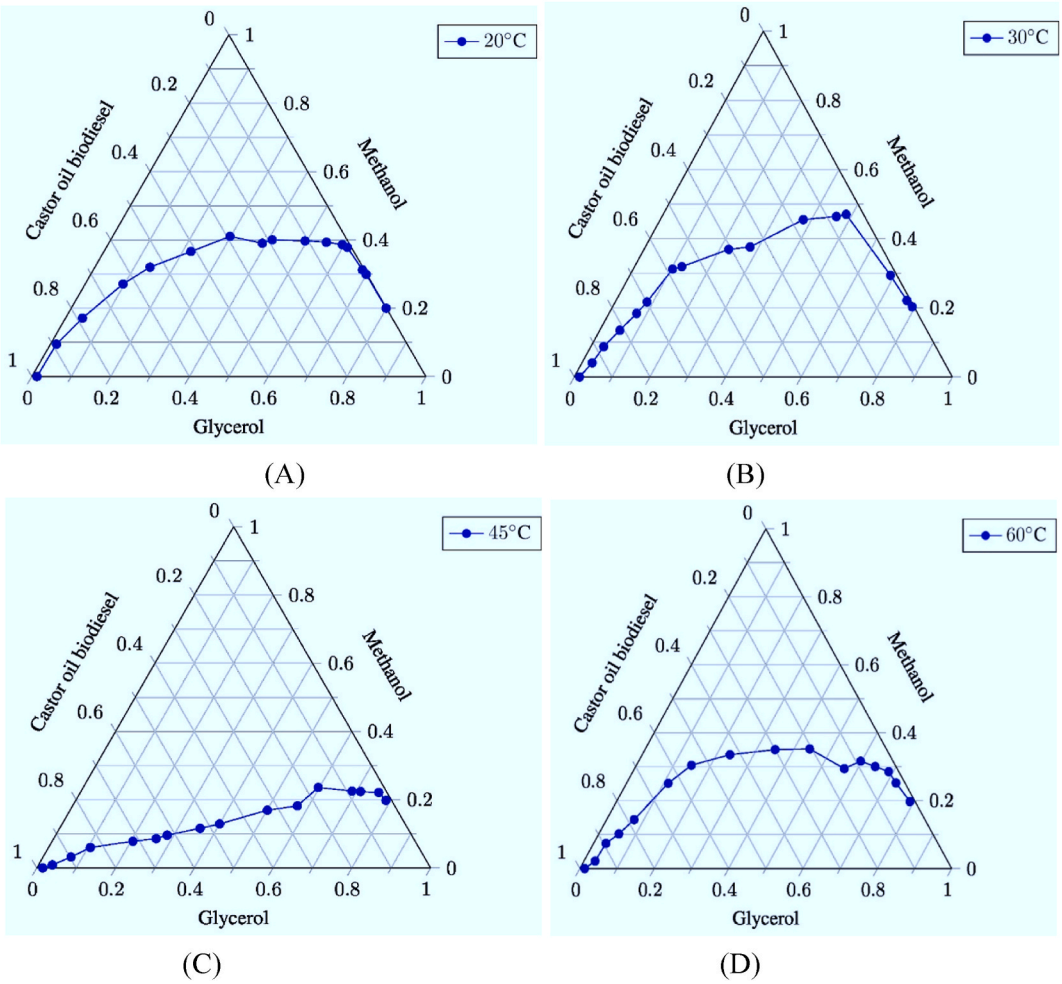


Fig. 5. Components composition at investigated temperatures (A) 20 °C (B) 30 °C (C) 45 °C (D) 60 °C.

3.9. Examination of component composition for castor seed oil biodiesel extract and raffinate phases at the investigated temperatures

Fig. 6(A–D) shows the component compositions (tie line compositions) between the conjugate phases involving the equipoise biodiesel extract phase and the glycerol raffinate phase. It was observed that at the coexisting extract and raffinate phases, the orientation angles of the composition increase as the methanol concentration increases and with temperature increases. The presence of different fatty acids leads to lateral homogeneity of the molecules which serves to organize the molecules into discrete domains. The lateral molecular homogeneity is caused by differences in the effective interaction of free energies between the different molecules in the biodiesel system. Compositional analysis showed that castor seed oil biodiesel and glycerol were partially miscible and soluble in methanol. However, there was a high level of the immiscibility of castor seed oil biodiesel and glycerol in each other which was in accord with other studies conducted [16,17,116]. The solubility of glycerol in castor seed oil biodiesel was very low and the same was that of castor seed oil biodiesel in glycerol at the investigated temperatures as evident in the plots of Fig. 6(A–D). Although the solubility in both cases was quite low, the solubility of glycerol in the castor seed oil biodiesel extract phase was higher than that of castor seed oil biodiesel in the glycerol raffinate phase at all temperatures. The lower solubility of castor seed oil biodiesel in the raffinate phase showed that it was possible to achieve clean, clear, and total separation and purification of glycerol from castor seed oil biodiesel. Furthermore, the solubility of methanol in the raffinate phase was higher than in the extract phase.

3.10. Practical and theoretical implications of the research

The separation and purification of the castor oil biodiesel components were conducted and the results were presented as ternary diagrams with each component in the mixture at the vertices or sides of the triangle having the maximum composition representing 100% pure component. Movement; either upward or downward along the sides and inside the ternary diagram represents a change in composition mixture of the three components with different mole or mass fractions in the biodiesel but all three components still

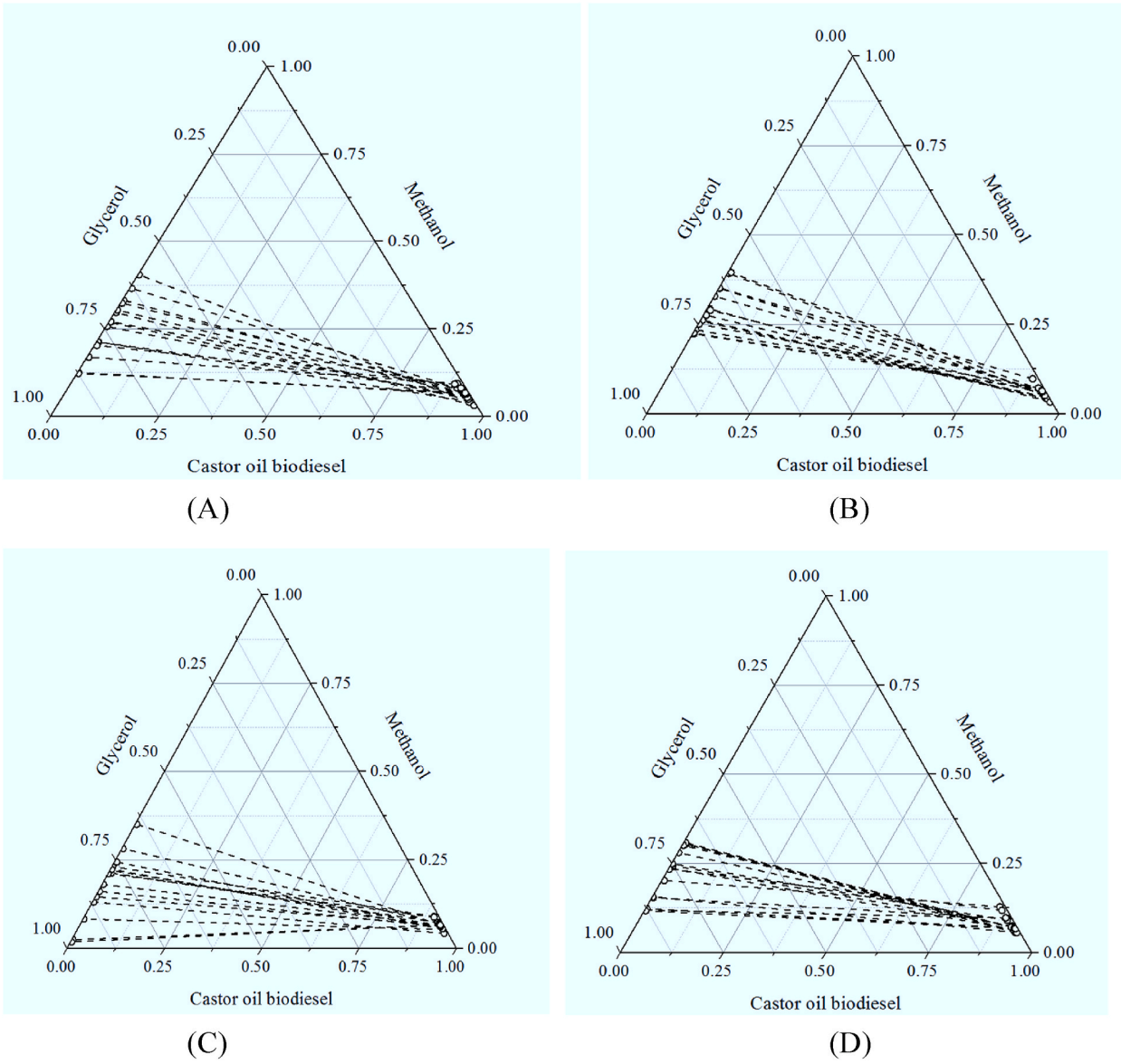


Fig. 6. Compositions of the homogeneous mixture at investigated temperatures (A) 20 °C (B) 30 °C (C) 45 °C (D) 60 °C.

summing up to 100% or 1 mol; thus, affecting the quality and purity of the resulting final biodiesel product either positively or negatively based on the individual constituent composition at any particular temperature, and time.

The temperature and time variations are important parameters of the investigation to ascertain and established an optimum operating condition. In this instance, the temperature of investigation of the separation and purification was 20 C, 30C, 45 C, and 60 C. These were equipment monitored temperatures of the investigation process. The ternary diagrams show grid lines that represented equilibrium compositions at which all components in the biodiesel phase are equally represented at a different mole or mass composition at the specific temperature. A shift in composition would imply that the composition of all three components' mole/mass composition has been altered either positively or negatively which would have an effect on the purity of the components in the mixture as the final product. ASTM and EN standards provide maximum limits for the presence of glycerol and methanol in any biodiesel. In this regard, obtaining biodiesel with strictly 100% biodiesel without glycerol and methanol is unrealistic and impracticable. Hence, any biodiesel system is expected to have the three components (the specific biodiesel, glycerol, and methanol) as the primary constituents with trace side products depending on the international standard applied.

The use of the ternary diagram concept for the separation and purification of the biodiesel helped in this regard to provide a basis at the onset of production, to achieve efficiency in the separation and purification. The temperature variation parameters provided for selectivity of operation of the process based on location, feedstock variety, climatic condition, oil plant maturation, and differences in plant species resulting in yield and variation in oil and biodiesel composition. Essentially, the benefits of the separation and

purification process using the ternary diagram is that it provided a visual representation of the components in the castor oil biodiesel system with further enhancement in the purification of the final product to meet global best practices. A further implication of the study is that it can be used to design a more efficient separation process for optimal biodiesel purification after production with knowledge of how the components distribute themselves in the ternary mixture after the completion of the transesterification reaction. This would lead to greater effectiveness of the process, reduce material and operational costs and eliminate environmental issues associated with the biodiesel production process as the volume of wastewater generated would be drastically reduced.

Additionally, high amounts of esters in the produced biodiesel indicated the good quality of the biodiesel. This showed that the amount of side products was minimal based on the GC-MS analysis which established the masses of the fragmented compounds and increased the ease of detecting isomers in the biodiesel based on international standards. Furthermore, the results for the range of hydrocarbons in the biodiesel obtained proved that mostly straight-chain saturated and unsaturated hydrocarbons were found in the transesterified liquid products which were also in agreement with several studies conducted on different biodiesel feedstocks [29,41, 45,81]. Only small amounts of oxygenated intermediates detected such as alcohol and glycerides. The properties of the produced biodiesel showed a good correlation concerning the various standards. Viscosity, acid value, and free fatty acid being essential parameters of biodiesel were relatively good. High viscosity presents a setback in fuel atomization by clogging injector nozzles, resulting in poor engine performance. High viscosity also harms biodiesel engine performance as it results in increased fuel consumption.

Thus, the results and parameters of this study will be useful in the setting up of a small-to-medium-size biodiesel production facility with improvement in the efficiency of product separation and purification.

4. Conclusion and recommendation

Biodiesel produced from non-edible vegetable oil seeds is a viable response to mitigating environmental issues associated with the use of fossil fuels because of its green nature and purity level of inherent properties. Due to the gradual decline in fossil fuel reserves, renewable energy alternatives are assuming attractive options. Experimental and theoretical assessment of phenomena linked with the separation and purification of biodiesel from *Ricinus communis* seed oil was investigated in this study. The approach was based on oil extraction and characterization, biodiesel production and subsequent property evaluation, and constituent components composition analyses to determine the properties of the biodiesel extract and glycerol raffinate phases respectively at different temperatures.

The physicochemical results of the analysis of the castor seed oil revealed that the values of the properties fall within the range of the standard used for this study (AOCS (American Oil Chemists Society), EN14214, ASTM D6751, and ASTM D9751). The properties of the oils such as refractive index, peroxide value viscosity, free fatty acid, iodine value, and saponification values are utilized as specifications of the oil characteristics. Whereas the rest parameters are only empirical in nature, however, they are important in the identification of oil in the plant seed.

The findings from the results of the fatty acid composition of the castor seed oil showed that the major characteristics of the oil were defined by the presence of the ricinoleic acid hydroxyl group which influenced the behavior of the oil. The oil was observed to consist mainly of highly monounsaturated acids and therefore very useful for biodiesel production due to its high level of mono-unsaturation. Meanwhile, the properties of castor plant seed oil biodiesel. Showed that the saponification values were very high at 117.51 mgKOH/g.

Densities, as well as specific gravities, are important properties of consideration for diesel fuel injection and combustion systems. The evaluation of parameters of castor seed oil biodiesel with the diverse standards showed that they were somewhat higher than the set limits of ASTM D9751, EN 14214, and ASTM D6751. Though, the value for castor seed oil biodiesel was slightly higher than the standards. The free fatty acid value obtained was observed to be consistent with the various standards of ASTM D9751, EN 14214, and ASTM D6751. The findings here are that the castor seed oil was considered to be good when compared with similar oils in fuel production.

The findings from the fatty acid profile of the *Ricinus communis* oil biodiesel, retention time, concentration in parts per million, and percentage concentration showed that linoleate (linoleic) (C18:2) and ricinoleate (ricinoleic) (C18:1, OH) had the strongest influence on the produced biodiesel with the composition of approximately 30% and 20% respectively of the fuel property.

The FT-IR spectrum for castor seed oil biodiesel sample analysis showed that the absorption spectrum in terms of the wave number (cm^{-1}) ranged from 1000 cm^{-1} to 4000 cm^{-1} with esters as the main functional group providing the main structural backbone. At the lowest peak position of 718.327, the vibrational type causing the IR absorbance was a C–O stretching at a peak area of 10.844. The absorbance range was about 650 cm^{-1}.

The influence of methanol concentration, time variation, and temperature on the separation and purification processes was determined via composition diagrams at four temperatures. Spectroscopic analysis using Fourier Transform Infra-Red (FTIR) was employed as a means of identifying the true nature of the oil and biodiesel in addition to the fatty acid profile. Meanwhile, the FTIR spectrum was quite different from that of the corresponding castor seed oil biodiesel owing to the occurrence of a fatty acid component in the biodiesel. The wave number (cm^{-1}) was from 1000 to 3800 cm^{-1} with the lowest absorbance being at the center point of 3500.854. The type of vibration at that position was bending with = C–H functional group.

Findings from the components composition analyses for a homogeneous mixture of castor seed oil biodiesel system at investigated temperatures revealed higher solubility of the castor seed oil biodiesel in the glycerol raffinate phase. This phenomenon was more pronounced at temperatures of 20 °C, 30 °C, and 60 °C as evident in the plot. The higher solubility was because the biodiesel component of the ternary mixture was composed primarily of methyl linoleic and ricinoleate which formed the backbone of the fatty acid and thus, influenced the solubility in the mixture composition.

The examination of the component composition for castor seed oil biodiesel extract and raffinate phases at the investigated temperatures showed different tie line compositions between the conjugate phases involving the equipoise biodiesel extract phase and

the glycerol raffinate phase. It was observed that at the coexisting extract and raffinate phases, the orientation angles of the composition increase as the methanol concentration increases and with temperature increases. The presence of different fatty acids leads to lateral homogeneity of the molecules which serves to organize the molecules into discrete domains.

An examination of the practical and theoretical implications of the research showed that the separation and purification of the castor oil biodiesel components were conducted and results were presented as ternary diagrams with each component in the mixture at the vertices or sides of the triangle having the maximum composition representing 100% pure component. Movement; either upward or downward along the sides and inside the ternary diagram represents a change in composition mixture of the three components with different mole or mass fractions in the biodiesel but all three components still sum up to 100% or 1 mol; thus, affecting the quality and purity of the resulting final biodiesel product either positively or negatively based on the individual constituent composition at any particular temperature, and time.

The ternary diagrams show grid lines that represented equilibrium compositions at which all components in the biodiesel phase are equally represented at a different mole or mass composition at the specific temperature. The use of the ternary diagram concept for the separation and purification of the biodiesel helped in this regard to provide a basis at the onset of production, to achieve efficiency in the separation and purification. The temperature variation parameters provided for selectivity of operation of the process based on location, feedstock variety, climatic condition, oil plant maturation, and differences in plant species resulting in yield and variation in oil and biodiesel composition. Essentially, the benefit of the separation and purification process using the ternary diagram is that it provided a visual representation of the components in the castor oil biodiesel system.

A further implication of the study is that it can be used to design a more efficient separation process for optimal biodiesel purification after production with knowledge of how the components distribute themselves in the ternary mixture after the completion of the transesterification reaction. This would lead to greater effectiveness of the process, reduce material and operational costs and eliminate environmental issues associated with the biodiesel production process as the volume of wastewater generated would be drastically reduced. Additionally, high amounts of esters in the produced biodiesel indicated the good quality of the biodiesel. This showed that the amount of side products was minimal based on the GC-MS analysis which established the masses of the fragmented compounds and increased the ease of detecting isomers in the biodiesel based on international standards.

In conclusion, this study underpins the fact that the *Ricinus communis* seed oil has great potential for the production of biodiesel because of the rich properties of the seed oil as seen in the physiochemical characterizations, fatty acid profile, and spectroscopic analysis of the oil and biodiesel which showed high conformity with the relevant standards. The study further revealed that separation and subsequent purification of glycerol from biodiesel after methanol removal can be easily achieved due to the high immiscibility nature of the biodiesel and glycerol in each other and also, their low solubility.

Author contribution statement

Kenneth Kennedy Adama: Conceived and designed the experiment; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Osikemekha Anthony Anani: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

Data will be made available on request.

Additional information

No additional information is available for this paper.

Declaration of competing interest

We the authors declare no competing and financial interests in this research work.

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