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Biodiesel production by two step process from an energy source of *Chrysophyllum albidum* oil using homogeneous catalyst

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ABSTRACT

Due to the exhaustion of fossil diesel, it has become necessary to discover alternative diesel fuel from non edible sources and suitable technology to obtain biodiesel as alternative fuel. In this research work, two step processes was carried out for biodiesel production from a non edible source of *Chrysophyllum albidum* seed. The lipids were extracted using a mixed solvent by the superheated extractor and the fatty acids compositions were characterized by gas chromatography (GC). *Chrysophyllum albidum* seed oil first undergoes an esterification reaction using H_2SO_4 homogeneous catalyst and transesterification parameter effects such as reaction temperature, catalyst loading, methanol-oil molar ratio, reaction time, and stirring rate were studied. The highest conversion of oil to biodiesel was achieved as 99.2 wt% at the satisfying conditions of 1:9 molar ratio of oil to methanol, 1 wt% of KOH, and 500 rpm of mixing intensity with 40 min of reaction time at 65 °C. The conversion of oil to biodiesel was analysed and confirmed by ^{1}H NMR Spectroscopy and physic-chemical properties were analyzed and compared with the ASTM standards. According to our experimental investication, recommend that Two step process of esterification followed by transesterification process highly suitable for biodiesel production from *Chrysophyllum albidum*.

1. Introduction

Fuel is a chief supply mainly for industries, agricultural and transportation segments. Right now, petroleum fuel outflow has been enhanced continuously, which expedite the reduction of limited petroleum deliver and necessarily increase petroleum prices (Kasirajan et al., 2011). It is dreadfully essential to discover alternative energy, predominantly the fuel for transport diesel engines, indirect to extend the petroleum delivery. Current state of affairs renewable & sustainable energy has received vast consideration as a replacement for present fossil fuels (Jain and Sharma, 2010).

Fuel enslavement in the transport segment is even of superior concern because it is the smallest amount diversified fuel end use segment worldwide, with 92% of transport ultimate fuel demand consisting of oil products (IPCC 2018). Transport is the segment which largely fuel consumes, and CO2 is the most important contributor to climate alteration (Greenpeace 2019). Most important challenges for infinite decarbonization are patently a must in this segment; this is not the only objective that renewable energies should accomplish. Policy-makers must have into relation other criteria in the triple dimension (environmental, economic, and social) so that renewable fuel use might be sustainable (Iea 2019).

Abila (2010) state that potential for the development of energy crops and production of bio-energy is enormous (Abila, 2010). The critical assessment, as the production of bio-energy in the nation is concerned, engage assessing all the essential considerations, especially the feedstock accessibility and value, considered to be the most significant success determinant for the production as it covers 60–70% of the production feed costs (Shehu and Clarke, 2020). Raw material supply takes 75% of the whole biodiesel production cost (Ghazali, 2015). Chief feedstock considerations include the narration of pricing, production pattern, profusion and existing quantity, haulage and storage alternatives in the potential processing and production spot (Shehu and Clarke, 2020).

Biodiesel has been used truthfully or miscellaneous with petroleum diesel at various proportions in many nations (Zhang et al., 2003). It is derived from a renewable and motherland resource, in this manner relieving dependence on petroleum trade-in (Zeng et al., 2009). Weigh against petroleum fossil fuel; biodiesel has a most encouraging combustion emission outline such as less emission of SO2, carbon monoxide,

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unburned hydrocarbons, and particulate matters. Besides, biodiesel has suitable viscosity, higher flash point, higher cetane number, and the engine alterations are non-compulsory (Yang et al., 2014). The cost of feedstock is accounted for as a major expenditure for biodiesel production. The exploitation of low prize substrates is the most excellent unconventional source for the production of biodiesel (Sharma and Singh, 2010). Transesterification is a catalytic chemical reaction involved the renovation of a carboxylic acid ester into a dissimilar carboxylic acid ester. It is a progression of exchanging the organic alkyl groups of non-edible and edible oil – an ester with the methyl alcohol. When an alcohol comes in make contact with free fatty acids, they bond to form alkyl ester as a biodiesel (Vandkata et al., 2012).

Some technologies counting one-step reaction process (Loong and Idris, 2016) and two-step reaction process (Dubey et al., 2015) have been urbanized to trim down biodiesel production cost from various resources. Firstly, one-step reaction is a transesterification reaction, this process suitable for the feedstock oils with a low FFA (free fatty acid) content, which is very complicated to take place if FFA content present in feedstock. Higher utilization of catalyst and alcohol content as compare to the two-step transesterification is major shortcoming of the one step reaction (Pisarello and Querini, 2013). Secondly, the two-step reaction process includes esterification followed by second step transesterification (Cai et al., 2015). Some researchers have used additional two-step esterification process (Photaworn et al., 2017). The two-step reaction process can be applied to any feedstock oils, chiefly in case of high FFA content non-edible oils. If the FFA substance is more than 1 wt %, then the first step esterification followed by the second step transesterification processes were highly suitable. The aim of the esterification step is to diminish the FFA content as much as feasible and to become suitable feed for the transesterification step. This two-step reaction process has been demonstrated the most effective tools for any feedstock oils utilize in biodiesel production process (Cai et al., 2015).

There are numerous sources accessible for biodiesel production such as non-edible oil, waste greases, used restaurant oil residues (Alptekin and Canakci, 2011), waste animal fats, waste marine algae, and industrial residues (Kasirajan et al., 2017). But, the Chrysophyllum albidum seed is having more than 15 wt% of lipids. The Chrysophyllum albidum (African Star apple) is one of the fruits of great economic value in tropical India due to its diverse industrial and medicinal applications. It is mostly a forest tree species, its natural episodes have been reported in diverse ecozones in India, Nigeria, Uganda, Niger Republic, Cameroon, and Cote d'Ivoire (Ewansiha et al., 2011). The seed of this plant has been rarely exploited for the production of oil for commercial purposes. Most often the seed is thrown away after the consumption of its juicy pulp. It's highly available in the forestry region of southern Ethiopia and India (mostly in the southern part of India).

Therefore, the *Chrysophyllum albidum* oil is considered as a best source for biodiesel production. To the best of our knowledge, the researchers no one investigated about *Chrysophyllum albidum* for biodiesel production. In commercial process homogeneous catalyst only provide more conversion then other catalysts. The base catalyst will persuade the soap formation during the reaction, when the acid values are more than 2 mg of KOH/gm of oil. However, presence of free fatty acids in the *Chrysophyllum albidum* is higher than the limited value. The acid catalyzed esterification followed by transesterification reaction only the possible way to achieve higher conversion of oil to biodiesel (Deng et al., 2010).

In this research study, evaluated the potential of the *Chrysophyllum albidum* source used for biodiesel production and optimized some of key parameters distressing the conversion of acid- base catalyzed production of biodiesel such as the reaction temperature, reaction time, molar ratio of oil to methanol, amount of catalyst and mixing intensity. Furthermore, analyzed the most important fatty acid gears of the *Chrysophyllum albidum* oil and analyzed the physic-chemical properties of the resultant *Chrysophyllum albidum* based biodiesel to the ASTM standard.

2. Materials and methods

2.1. Materials

Chrysophyllum albidum seed has been collected in Jimma, ethiopia and Kerala states forest station in India. The 98% of sulphuric acid have been purchased from Merck India ltd, Chennai. Chloroform (>99.9% purity) was purchased from SRL Chemicals India Ltd. Methanol (purity of 99.9%), Hexane (purity of 99%) were obtained from SRL Chemical India Ltd, Chennai. KOH used as alkali catalysts for the straight transesterification reaction was purchased from Sisco Research Laboratory Ltd, Mumbai, India.

2.2. Extraction procedure and characterization of lipids

The *Chrysophyllum albidum* seed was consumed as a raw material for biodiesel production process experimental investigation. Initially, *Chrysophyllum albidum* seed was dried in a hot air oven at a constant temperature of 50 ± 0.5 °C over night to obtain equilibrium weight loss. The dried *Chrysophyllum albidum* seed were grounded to shrink the particle size. Subsequent to size reduction, the desired particle size was separated using a vibrator sieve-shaker (mesh type) into a range of 0.25 to 0.5 mm. The yield of oil content of the *Chrysophyllum albidum* was determined using the conventional method. 25 gm of powder particle loaded in to the superheated solvent extraction apparatus and the extraction process was carried out with the mixed solvents of hexane, chloroform, and methanol at the ratio of 3:2:1 (Kasirajan et al., 2014). After the completion of extraction process, mixed solvents was recovered using batch distillation then reused and the traces quantity of solvents has been separated by rotary evaporator and the extracted oil was quantified by weighing method.

The properties such as average molecular weight, density (AOCS (American Oil Chemical Society) 1998), acid value, FFA (free fatty acid), saponification value, and iodine value (Vicente et al., 2004)of extracted *Chrysophyllum albidum* oil were analyzed by using standard procedures. Fatty acid composition present in *Chrysophyllum albidum* oil was analyzed by gas chromatography (GC). One gram of oil was taken and the analysis was carry out using GC, which consisted of CHEMIT GC 8610 flame ionization feeler in the column BPX-70. Nitrogen and hydrogen gas were used as carrier gasses and oxygen was used for detonation purpose. The data's were documented with the use of Winchrom software.

2.3. Two-step biodiesel production procedure

Earliest step, H_2SO_4 catalyst was used for esterification pretreatment in the reactor (The three-neck round bottom flask was connected with a water-cooled reflux condenser was used as a reactor) to convert the free fatty acids into biodiesel. In this process, 2 wt% of H_2SO_4 catalyst (with respect to oil wt%), 1:12 molar ratio of oil-methanol, 400 rpm stirring speed and 20 min pre-tratment time with 65 °C were used to study their influence on diminution of the FFAs of *Chrysophyllum albidum* oil. 25 gm of *Chrysophyllum albidum* oil was taken in the reactor to carryout the pre-treatment process. The reaction mixtures were actively stirred and refluxed up to 60 min. After the reaction completion, the un-reacted methanol was separated from the liquid phase using rotary evaporation (Thein et al., 2019).

At the second step, KOH was used as catalyst for the reason that this had a higher catalytic motion for the transesterification reactions. The *Chrysophyllum albidum* was considered as the pre-treated material for transesterification process. The suitable process conditions such as, molar ratio of oil-methanol, reaction temperature, reaction time, catalyst loading quantity and mixing intensity for transesterification reaction were examined. After the completion of reaction, the mixtures were allowed to settle definite length of period to remove by-product of glycerol. Further, the traces quantities of methanol and catalyst presence

with the biodiesel were washed out using distilled water (Supaporn and Ho Yeom, 2016).

2.4. Biodiesel characterization

Biodiesel produced from *Chrysophyllum albidum* oil by the two step techniques characterized by ^{1}H NMR spectra were acquired using a Bruker 500 MHz Avance III device with tetramethylsilane (TMS) as an internal standard solution and the Chloroform-d ($CDCl_3$) as solvent. A ^{1}H spectrum was evidenced with pulse duration of 45 °C and 16 scan. From the ^{1}H NMR spectra, the conversion was calculated using the following Eq. (1).

$$C = \frac{2A_{ME}}{3A_{CH2}} \times 100 \quad (1)$$

Where C - the percentage conversion of *Chrysophyllum albidum* oil to biodiesel, A_{ME} - assimilation value of the methoxy protons of the methyl ester, A_{CH2} - assimilation value of the methylene protons. Factors 2 and 3 were forecasted from the fact that the methylene carbon possesses 2 protons, while the methyl alcohol carbons have 3 attached protons (Knothe and Kenar, 2004). The physic-chemical properties such as density, viscosity, cetane number, flash point, fire point, moisture content, sulfur content, and etc., of biodiesel were investigated as per the ASTM standard.

3. Result and discussion

3.1. Lipid extraction and characterization

The bio-oil was extracted using superheated solvent extractor; the maximum oil was obtained as 18.65 wt% at constant parameter conditions of 90 °C at 15 bar pressure, the mixed solvent flow rate of 1 Lt/hr, mixture of hexane, chloroform & methanol volume ratio of 3:2:1 and the mean particle size of 0.25 mm for 120 min of extraction time (Kasirajan et al., 2014). The extracted oil from *Chrysophyllum albidum* has found as non edible oil and it's highly suitable for biodiesel production.

The physic-chemical properties of extracted oil were analysed as per the ASTM stsndard. The acid value as 5.4 mgKOH/g, free fatty acids as 2.7%, saponification value as 219.6 mgKOH/g, density of oil as 928 kg/m^3 and iodine value as 46 gI_2/100 g. Vegetable oil with iodine value below 100 is generally classified as non drying oil (Adebayo et al., 2012). These classes of oil are generally appropriate for soaps, lubricating oils and lighting candles productions (Anang et al., 2019). From the gas chromatography, the fatty acid compositions of *Chrysophyllum albidum* oil were found that Linoleic Acid(C18:2) – 41.7%, Oleic Acid (C18:1) – 20.3%, Palmitic Acid (C16:0) – 18.9%, Stearic Acid (C18:0) – 14.2%, Arachidic Acid(C20:0) – 2.4%, Lignoceric Acid(C24:0) – 0.84%, Linolenic Acid (C18:3) – 0.81%, Bechenic Acid (C22:0) – 0.52%, Myristic Acid(C14:0) – 0.19%, Lauric Acid (C12:0) – 0.069% and Palmitoleic Acid(C16:1) – 0.061% and some traces amount of inert are present. Average molecular weight of the *Chrysophyllum albidum* oil was calculated as 873.65 g/mol by using the fatty acid compositions.

3.2. Esterification reaction studies (Pre-treatment process)

Esterification reaction was used in order to pre-treat the *Chrysophyllum albidum* oil by converting the free fatty acids into biodiesel (Methyl Ester) using an acid catalyst. The free fatty acids content of *Chrysophyllum albidum* oil was found as 2.7% (acid value as 5.7 mgKOH/g), which is not be favourable for biodiesel production through direct transesterification process. Transesterification reaction will not occur if the FFAs content in oil is more than 0.5% (acid value as 1 mgKOH/g), it will induce the soap formation during the transesterification reaction. Therefore, the limits of FFAs were set to a maximum of 0.5% for all transesterification experimentation.

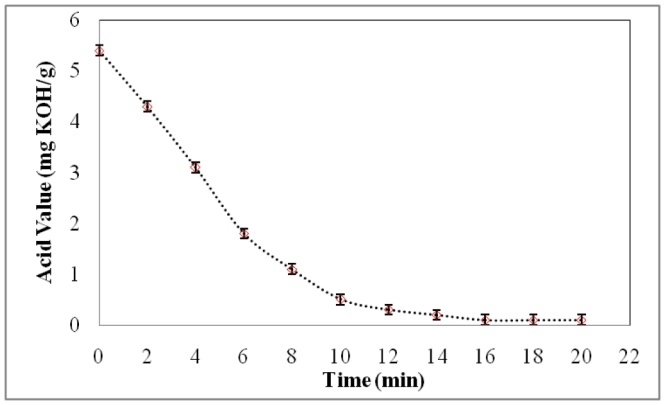

Fig. 1.. Esterification process at constant temperature of 65 °C, 2 wt% of H_2SO_4 catalyst (with respect to oil wt%), 1:12 molar ratio of oil-methanol, 400 rpm stirring speed with respect to different reaction time period.

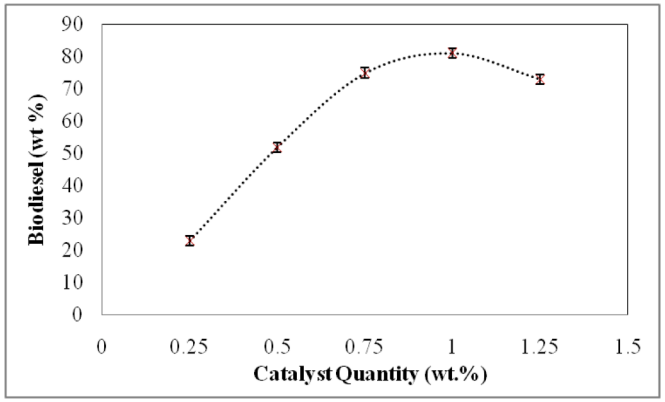

Fig. 2.. Catalyst effect on Transesterification process at constant temperature of 65 °C, 1:12 molar ratio of oil-methanol, 600 rpm stirring speed and 60 min of reaction time.

It was instigate that the pre-treatment step significantly reduced the acid value from 5.4 to below 1 mg KOH/g. The effect of acid value respect to reaction time was shown in Fig. 1. The investigational results suggested that the H_2SO_4 acid catalysis pre-treatment (esterification) have effect in three phases i.e. rapid, slow and static. The rapid phase, reaction rate was speedy in favor of which the acid value concentrated from 5.4 to 1.1 mg KOH/g within 8 min of reaction period and the dropdown of acid value was found as 79.63%. After 8 min the reaction rate decelerate up to 16 min, the dropdown of acid value was 98.15%. In the stationary phase the esterification reached equilibrium after 16 min. The acid value was observed less than 1 mg KOH/g at 10 min, but it was striking to keep a longer reaction time of 20 min to get an inferior target of < 0.5 mg KOH/g. Thein et al., (2019) investigated the esterification of two different brand of crude rice brand oil with 8 and 10% of FFA content, obtained similar result of FFA reduced to 0.61 and 0.7% under the optimum condition of 60 oC with 62.1 and 70:1 methanol to FFa molar ratio, 90 and 48 min reaction time, 22.5 and 20% of H2SO4, respectively. Comparatively, except temperature all other operating conditions were very less for our experimental work due to the lesser amount of FFA present in *Chrysophyllum albidum* oil (Thein et al., 2019).

4. Transesterification process

4.1. Catalyst effect on transesterification of Chrysophyllum albidum

In most cases, from the journalism it was accomplished that alkali catalyst deliberation of less than or equal to 1 wt% is required for a

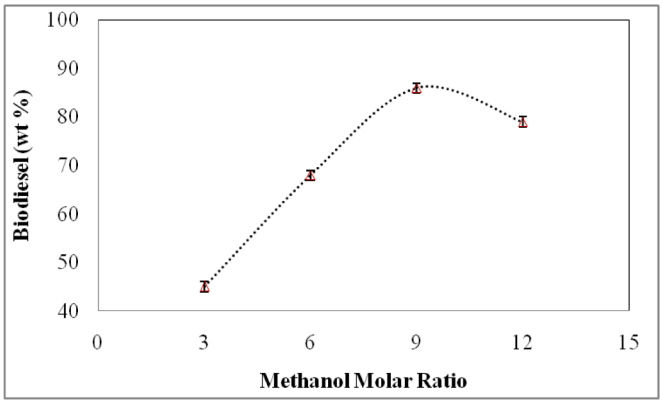

Fig. 3.. Methanol-oil molar ratio effect on Transesterification process at constant temperature of 65 °C, 1 wt% of catalyst (with respect to oil wt%), 600 rpm stirring speed and 60 min of reaction time.

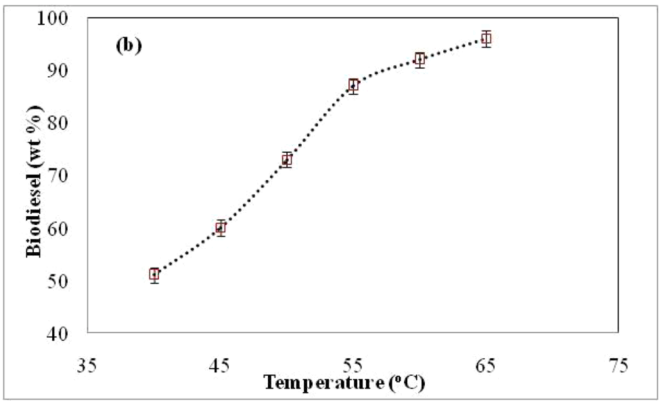

Fig. 4.. Temperature effect on Transesterification process at constant quantity of 1 wt% of catalyst, 1:9 molar ratio of oil-methanol, 600 rpm stirring speed and 60 min of reaction time.

successful conversion of triglycerides to biodiesel depending on the physic-chemical properties of oil used. In peace with the results shown in Fig. 2, the oil conversion to biodiesel was minute for the catalyst quantity of 0 – 0.75 wt%. This is due to the scarce magnitude of potassium hydroxide used in the reaction. From this investigation we observed that 1 wt% of KOH is most favorable and enough quantity to achieve superior conversion of 81 wt%. The surplus quantities of catalyst count were resultant in the soap creation, which shrinks the yield by giving supplement to emulsification of biodiesel and glycerol phase. Ramachandran et al., (2017) was examined that 1 wt% of KOH catalyst attentiveness was found to be most favorable quantity for the transesterification of *Solanum nigrum L* oil in an open system reactor (Kasirajan et al., 2017). Most of the journalism was examined that alkali catalyst measure of $\leq$ 1 wt.% is essential for a victorious conversion of oils to biodiesel depending on the nature of oil used as a supply for biodiesel production (Hanh et al., 2007).

4.2. Oil-Methanol molar ratio effect on transesterification of *Chrysophyllum albidum*

Mole ratio of oil-Methanol is one of the key aspects distress the conversion of *Chrysophyllum albidum* oil to biodiesel, as well as the general processing expenditure of biodiesel. The transesterification reaction requires more methanol to succeed the process. Conversely, in tradition the oil-methanol mole ratios have to be superior to that of the stoichiometric relative amount in order to strength the reaction towards complete conversion. In this investigation, the mole ratio of *Chrysophyllum albidum* oil to methanol was studied in the range of 1:3 to 1:12. Fig. 3 represents the consequence of oil-methanol mole ratio in the conversion of biodiesel; it was found that the *Chrysophyllum albidum* oil to biodiesel conversion increases with increase in mole ratio. The optimal conversion of 86% was achieved at 1:9 mole relative amount of *Chrysophyllum albidum* oil to methanol with 1wt% KOH catalyst. Dukare et al. (2010) also observed that supplementary increases in mole relative amount of methanol the rate of biodiesel conversion were decreased due to the mass transfer constraint (Dukare et al., 2010).

4.3. Temperature effect on transesterification of Chrysophyllum albidum

The conversion of triglycerides to biodiesel was considerably controlled by the temperature in the transesterification process. The reaction temperature is the key variable parameter which is comprehensively affects the yield of biodiesel. The reaction was carried out for 30 min with various temperature ranges from 40 °C to 65 °C. The conversion of biodiesel was achieved only 51 wt.% at 40 °C. Conversely, the highest conversion achieved as 96%, when the reaction temperature was augmented to 65 °C. Hence, the temperature has a more extensive cause

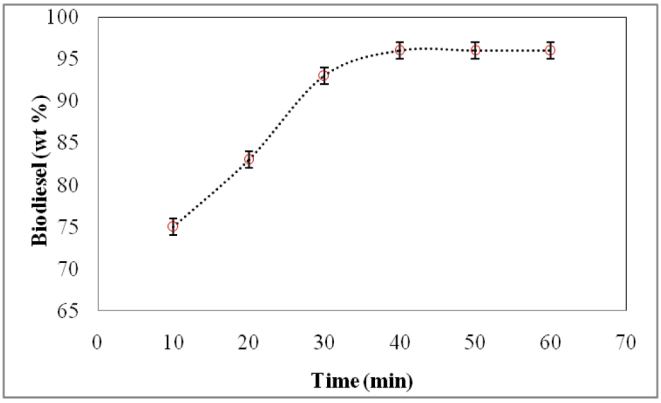

Fig. 5.. Effect of reaction time on Transesterification process at constant quantity of 1 wt% of catalyst, 1:9 molar ratio of oil-methanol, 600 rpm stirring speed and 65 °C of reaction temperature.

on the conversion at higher reaction temperature than lower temperature. Fig. 4 demonstrates the cause of reaction temperature on *Chrysophyllum albidum* oil to biodiesel conversion. At lower reaction temperature, there was an inadequate energy supply to sustain wide collisions between *Chrysophyllum albidum* oil and methanol with catalyst. Alternatively, at higher reaction temperature, the possibility of collision between the reactants of *Chrysophyllum albidum* oil and methanol became enhanced and uncomplicatedly achieved the required activation energy to succeed the reaction (Al-Widyan and Al-Shyoukh, 2002).

4.4. Reaction time effect on transesterification of Chrysophyllum albidum

Tolerable contact time is required to complete the transesterification reaction. The effects of reaction time was studied at diverse time intervals ranging from 0 to 60 min. Fig. 5 shows that cause of reaction time on the conversion of *Chrysophyllum albidum* oil to biodiesel. It was observed that the conversion of biodiesel increased with an increase in transesterification reaction time. The maximum conversion of *Chrysophyllum albidum* oil to biodiesel has been achieved as 96 wt% at 40 min of reaction time. Further increase of reaction time it was observed that, there is no considerable changes on the conversion of *Chrysophyllum albidum* to biodiesel. Albishri et al., (2013) stated that excess reaction moment in time will lead to decrease the product yield as a result of the backward reaction of transesterification, resulting in a thrashing of esters as well as causing further fatty acids to form soaps (Albishri et al., 2013).

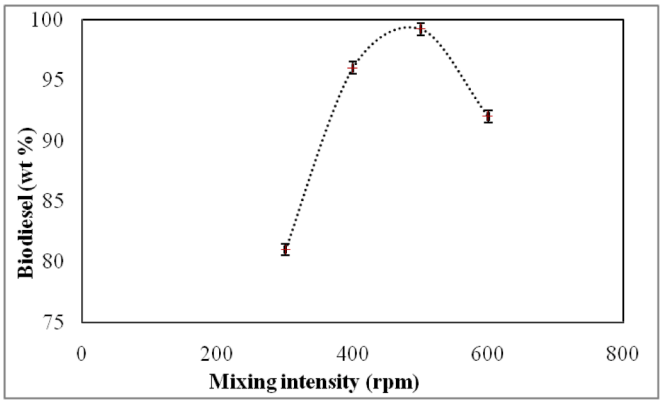

Fig. 6.. Effect of reaction mixing intensity on Transesterification process at constant quantity of 1 wt% of catalyst, 1:9 molar ratio of oil-methanol, 40 min of reaction time and 65 °C of reaction temperature.

4.5. Mixing intensity effect on transesterification of Chrysophyllum albidum

The mixing strength is a vital cause for the immiscible reactants in order to enhance the rate of diffusion between the reactants. Mixing rate will increases the contact surface area between oil and catalyst in methanol solution to initiate the transesterification reaction. In this investigation, mixing intensity was considered in the range of 300 to 600 rpm. Fig. 6 exhibits the cause of mixing intensity on the conversion of *Chrysophyllum albidum* to biodiesel. The result exposed that the rate of mixing of 500 rpm for this particular reaction were sufficient to succeed the biodiesel conversion as 99.2 wt%. On the other hand, the conversion for 500 rpm was higher than that of 600 rpm and above mixing rate. Poor mixing may possibly lead to lower reaction rate, thus affects the conversion rate. Therefore, 500 rpm was selected as suitable stirrer speed in the transesterification of *Chrysophyllum albidum* oil. Further enhancement of the mixing rate decreases the conversion rate; this clearly indicates that diffusion controlling steps have effect at a higher mixing rate. Aldo Okullo Temu et al., (2010) investigated that the continuation of two phases will diminish the reaction rate as a result of the strong mass transfer boundaries (Aldo Okullo Temu et al., 2010).

4.6. Characterization of biodiesel

Fig. 7 represents the ^{1}H NMR spectra of *Chrysophyllum albidum* biodiesel. The quality wave produced from the protons of methylene cluster adjacent to the ester assembly of a molecule in TG (a-CH_2) was eluted at 2.3 ppm and the protons in the methoxy cluster of the methyl ester were distinguished at 3.66 ppm. The conversion of biodiesel was accomplished based on these attribute proton gestures in the NMR spectra. The conversion of the methyl ester from *Chrysophyllum albidum* oil was resolute as 99.2 wt%. Table 1 represents the physic-chemical properties such as kinematic viscosity, density, flash point, cetane number, moisture content and etc.

5. Conclusion

Two step esterification and transesterification process completely control the soponification of free fatty acids which leads the soap formation and minimize the biodiesel conversion efficiency; instead FFA completely converted into biodiesel. The results have shown that

Table 1
Physic-chemical properties of *Chrysophyllum albidum* biodiesel with ASTM standards.

Content	*Chrysophyllum albidum Biodiesel*	ASTM D 6751	Units
Density at 15 °C	0.86	0.82–0.9	g/cc
Kinematic viscosity (40 °C)	3.5	1.9–6.0	mm^2/s
Cetane number	60	>47 min	–
Flash point	158	>130 min.	°C
Water content	0.01	0.05% max.	v/v
Sulfated ash	0.003	0.02% max.	w/w
Acid value	0.1	0.5 max.	mg KOH/g
Copper strip corrosion	1	No. 3 max.	–

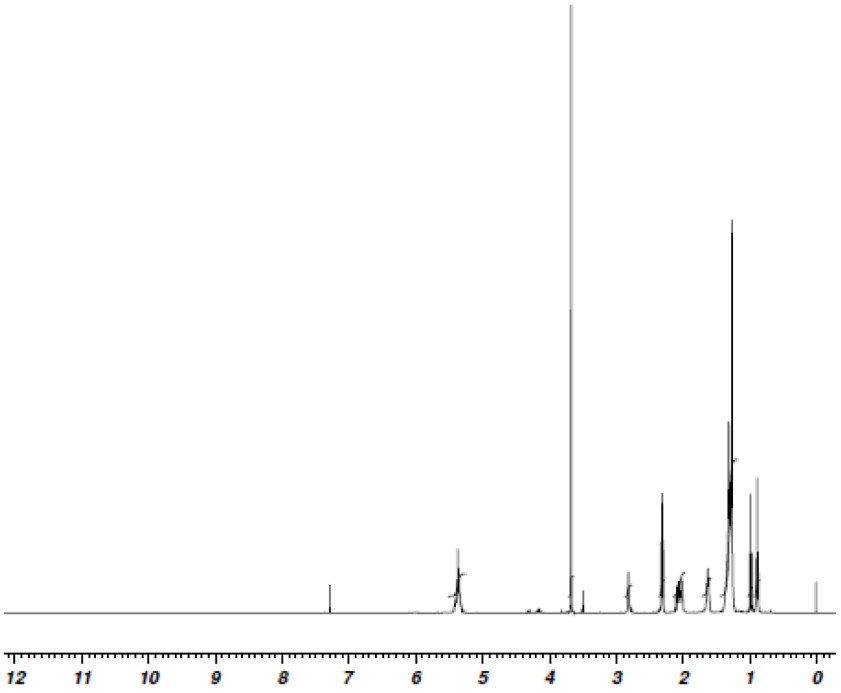

Fig. 7.. ^{1}H NMR Spectra of *Chrysophyllum albidum* Biodiesel obtained under optimized condition.

successful conversion of oil from *Chrysophyllum albidum* seeds results high quality biodiesel. The seeds which are considered as waste contains 18.65% oil and can supply as alternative feedstock for biodiesel production. The two step process result has shown the yield of 99.2% with optimum parameters conditions using H_2SO_4 as acid catalyst for esterification of FFA and KOH as a base catalyst for transesterification of rest. The biodiesel yield is affected by the quantity of catalyst, temperature, Oil-Methanol ratio, reaction time and mixing intensity which has been optimized. The produced biodiesel was confirmed by ^{1}H NMR spectra. The physic-chemical properties of the *Chrysophyllum albidum* biodiesel were within ASTM standards limits. It has been recommended that *Chrysophyllum albidum* might be used as a potential feed source for biodiesel production by two step process. This process reduces the waste, minimize the cost by utilizing entire resource conversion and control the pollution.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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