Production And Characterization Of Neem Oil Methyl Ester

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Abstract

Feasibility of biodiesel production greatly depends upon the feedstock used. Because of the increasing edible oil prices, it is not viable to produce biodiesel from edible oils. Low cost and abundantly found non-edible oils such as Neem oil could be a better option for biodiesel processing. Neem oil could not be easily converted into biodiesel prior to its refining because of its higher moisture and FFA content. In this study, a soil to oil level study has been done to overcome the problems associated with biodiesel production from Neem oil. Oil extraction, refining of raw oils, optimization of variables of transesterification, process qualitative as well as quantitative aspects of Neem oil methyl ester (NOME) has been evaluated. With optimized production process, it was found that almost complete conversion of Neem oil to methyl ester has taken place. The fuel properties of NOME were found to be in accordance with the ASTM. IS and EN standards.

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1. Introduction

Due to the growing concern for environmental pollution caused by the conventional fossil fuels and their non-renewable nature search for more environment friendly and renewable fuels has become necessary now a days. Development of biodiesel which is a renewable alternative energy source, could easily reduce global dependence on petroleum, and could also help to reduce air pollution. Worldwide production of biodiesel is mainly done by utilizing edible oils such as soybean, sunflower and canola oils. Since, India is not self sufficient in edible oil production, hence, some non-edible oil seeds available in the country are required to be tapped for biodiesel production [1]. Edible oils cannot be utilized for the production of biodiesel due to their short supply and because of recent increase in their prices. This has given rise to a conflict of "fuel versus food". Therefore, it is required to search for a viable feedstock for production of biodiesel. The feasibility of biodiesel production lies with the abundant availability of oilseeds and other resources in any country. In fact, the price of raw oil is the most dominant factor in the cost of biodiesel fuel, and determines the competitiveness of biodiesel with fossil fuel in the fuel market.

In India, a large potential non-edible and other abundant sources of vegetable oil are untapped. These plant and seed oils have diverse physical and chemical properties. Some of them can be used as feedstock for the biodiesel. India has about 80 different types of oilseed bearing potential [2] trees. Abundance of forest and plant based non-edible oils such as *Pongamia pinnata* (karanja), *Jatropha curcas* (Jatropha), *Madhuca indica* (Mahua), *Shorea robusta* (Sal), *Azadirachta indica A Juss* (Neem) and *Hevea braziliensis* (Rubber), bears great potential for production of biodiesel in India.

Neem oil has the highest potential and production among the available wild oils. Only 20-25 % of the total potential of both the oils is being produced and utilized (mainly in soap industry and pharmaceutical based industries). Remaining 75-80% oil potential is available in surplus, which is not even being harnessed today and can be a good option for production of [2] biodiesel. Neem is scattered all over the country, except at high altitudes and thick forests. It grows mainly in states of Rajasthan, Uttar Pradesh, Madhya Pradesh, Andhra Pradesh, Tamil Nadu and Delhi.

In this work, biodiesel was produced from Neem oil using base-catalyzed transesterification. Neem oil bears higher amount of moisture content, responsible for inbuilt higher amount of FFA (free fatty acids), makes it unsuitable for biodiesel production. For production of biodiesel by base catalyzed transesterification method, oils or fats should be substantially anhydrous ($\leq 0.06\%$ w/w) and FFA of the vegetable oil should be below 0.5 %, else it will form soap with alkaline catalyst. The soap [3] can prevent separation of the biodiesel from the glycerine fraction.

Neem seed is consists of two seedcoats. The external seed-coat contains moisture content of about 2 % which is responsible for the inbuilt FFA right from the beginning i.e. just after oil extraction. Further Neem oil undergoes fast oxidation and thus results in a rapid increase in its acid value. Keeping this in mind study was done right from the beginning to analyze the problem from the grass root level. Studies were performed on cold filter press (oil expeller) at Akhadhya Tel Sabun Utpadan S.S. Ltd. (a local manufacturer of Neem oil), RIICO Industrial Area, Churu (Rajasthan). Methanol, potassium hydroxide, and all other chemicals used in this research work were of analytical grade (Merck Company). In this study, refining of crude Neem oil was done using caustic stripping method and optimization of base catalyzed transesterification process was done utilizing refined neem oil. Quality and quantity of biodiesel was analyzed according to the latest ASTM, IS and EN specifications for biodiesel.

2. Materials and methods

2.1. Preparation of crude neem oil

Taking into consideration, the appropriate requirement of feedstock for biodiesel production, neem oil was extracted from cold press oil expeller at temperature not exceeding 70-75 °C in two ways:

- (1) From seeds containing both external seed coat and internal seed coat.
- (2) From seeds after de-hulling external seed-coat of neem seeds.

The extracted crude neem oil was then filtered with the help of plate filter press. Acid number was determined according to the latest IS standard IS 1448 P:1/Sec.1 for getting an idea for further processing or refining of crude oil. Neem oil was then finally filtered with the help of cloth as well with the help of filter paper before degumming.

2.2. Degumming

The above filtered neem oil having relatively high levels of phosphatides were degummed prior to refining to remove the majority of phospholipid compounds. The process involved the treatment of the crude oil with a limited amount of water (1% - 3%) to hydrate the phosphatides and make them separable by mechanical agitation with the help of magnetic stirrer for 30 minutes at 70 °C.

2.3. Refining/Neutralization

The process of refining (sometimes referred to as "alkali refining") was performed on the degummed oil to reduce the free fatty acid content and to remove other impurities such as remaining phosphatides, proteinaceous, and mucilaginous substances etc. Caustic neutralization was carried out using 16° Baume sodium hydroxide (lye solution) in 10% excess. The lye solution was added drop by drop to the degummed oil in an open beaker with constant vigorous stirring with magnetic stirrer at 70 °C for 15 minutes, followed by 20 min settling time. The oil was then transferred to the separating funnel and washed to make it free of soap using $4 \times 15\%$ (v/v) hot deionised water washes, giving a pH of 7 on the final water wash. Each wash was done at 100 °C for 10 min followed by settling for 15 minutes. The soap water was then discarded. The oil was not bleached and deodorized but heated to 150 °C for 10 minutes on hot plate for the removal of moisture and odour to some extent.

2.4. Feedstock characterization for biodiesel production

The above refined Neem oil was analyzed for acid value using ASTM D 974. Unsaponifible matter and iodine number were determined by chemical titration method using IS: 548 and pr EN 14111 respectively. Moisture content was determined by oven method using IS: 548. Both the refined oils were also tested for phosphorus content using the ASTM D 4951 method with the help of UV-visible spectrophotometer.

The fatty acid compositions (%) of the saturated and unsaturated fatty acids of the neem oil were analyzed by a gas chromatography at FARE Labs, Gurgaon (Haryana). FAMEs were

injected into a gas chromatograph (Supeleo Model SP-2560 equipped with a flameionization detector (FID), a fused silica capillary column (0.32 mm internal diameter, 100 m length and 0.25 µm film thickness). Helium was used as the carrier gas, and nitrogen was used as the makeup gas. The initial column oven temperature was 115 °C, temperature programmed to 275 °C and held at this temperature until the analysis was completed. Fatty acid methyl esters were prepared for chromatographic analysis (GLC) using BF3methanol reagent [4]. FAME peaks were identified by comparison of retention times to a Supeleo component FAME mix (obtained from Sigma-Aldrich, St. Louis, US). The peak areas were computed, and percentages of FAME were obtained as area percentages by direct Data normalization. were expressed as normalized peak area percent of all identified FAME. Only the more abundant FA (>0.2 %) was considered. All analyses were carried out in triplicate.

2.5. Production of biodiesel (Transesterification)

The refined Neem oil was first filtered by filter paper mainly to remove the remaining dirt and other inert materials and then placed in a conical flask equipped with magnetic stirrer and water condenser. The base catalyzed transesterification process for biodiesel production was optimized. Under agitation the raw oil was heated closer to the boiling point to remove the water contaminant present in the oil. After that the oil was allowed to cool down under room temperature, and the treated oil alone was then taken for biodiesel production purpose. Again, under agitation, the above treated oil was heated up to a desired temperature on a hot plate. A fixed amount of freshly prepared sodium hydroxide-methanol solution was added to the oil, this moment was considered as the starting time of the reaction. When the reaction reached the preset reaction time, heating and stirring were stopped. The products of reaction were allowed to settle for three hours. During settling two distinct liquid phases were formed: crude ester phase at the top and glycerol phase at the bottom. The crude ester phase separated from the bottom glycerol phase was then washed by

warm de-ionized water several times until the washed water became clear. The NOME was dried using anhydrous sodium sulphate and then the remaining excess methanol and water in ester phase were removed by evaporation under atmospheric condition. In this way neem methyl ester was prepared. The final biodiesel was then filtered to remove remaining sediments with the help of filter paper. The reactions were investigated step by step. The optimal value of each parameter involved in the processes was determined while the rest of the parameters were kept constant. After each optimal value was attained, this value was adopted for the optimization of the next parameter.

2.6. FTIR and NMR testing

FTIR (Fourier Transform Infrared Spectroscopy) and ¹H NMR (nuclear Magnetic Resonance) were recorded at Department of Chemistry, University of Rajasthan, Jaipur. IR spectra were recorded on a FTIR model Shimadzu 8400S grating infrared spectrophotometer in KBr pellets and were in cm⁻¹. ¹HNMR spectra were measured on a JEOL-AL 300 MHz, using TMS (Tetramethylsilane) as an internal standard and CDCl₃ as a solvent and were in δ ppm.

2.7. Analysis of biodiesel quality

Neem oil methyl ester was then finally analyzed for moisture content using the Karl Fisher titrimeter according to ASTM D 6304 07. The acid and iodine indexes were evaluated using ASTM D 974 and pr EN 14111, respectively. The density of neem methyl ester was determined by pycnometer using EN ISO 3675. Kinematic viscosity was determined by Redwood viscometer according to ASTM D445 and flash point by Pensky – Martin's apparatus by ASTM D93 method. Calorific value was determined by Bomb calorimeter. Sulphated ash was determined by muffle furnace method according to IS 1448 P: 4, cloud point and pour point by cloud and pour point apparatus by IS 1448 P: 4. Phosphorus (mass %) was determined by spectrophotometer by ASTM D 4951.

3. Results and discussion

3.1. *Extraction and determination of initial acid number of crude neem oil*

The crude Neem oil obtained after extraction with the help of cold press oil expeller was found to consist of low levels of phosphatides and other components that have to be removed during pretreatment (refining). The crude Neem oil extracted from seeds after dehulling the external seed coat was found to have an acid value of 0.87. This amounts for FFA % equivalent to 0.435 (<0.5 %), which could be directly employed for biodiesel production without any pretreatment (refining of oil). While in the second step crude Neem oil which was extracted from Neem seeds containing both the seed coats (external as well as) was found to have an acid value of 7.01 (FFA % = 3.5), which requires pretreatment prior to biodiesel production.

3.2. Degumming of crude oils

The main reason for the removal of phospholipids was that some of the compounds, particularly the calcium and magnesium salts of phosphatidic and lysophatidic acids, are strong emulsifiers. If these compounds remains in the oil during the later alkali neutralization step, they may inhibit the separation of the soaps and lowers the yield of neutral oil. Phospholipids (also called phosphotides) will also react with water to form insoluble sediments. Phosphotides were efficiently removed from the crude oils.

Addition of phosphoric acid darkens the colour of neem oil which may prove to be problematic during the identification of phase separation of biodiesel and glycerol after transesterification. Therefore, phosphoric acid treatment was exempted for crude neem oil. The phosphotides and gums which were removed could be utilized for lecithin production.

3.3. Caustic stripping

Acid esterification was tried but it leads to the darkening of Neem oil and employs a huge amount of methanol which then creates a great problem for identification of clear phases of glycerol and methyl ester after transesterification. Therefore, caustic stripping method was utilized for the treatment of crude Neem oil.

A large reduction of free fatty acids was observed through their conversion into high specific gravity soaps. The remaining phosphatides and mucilaginous substances upon hydration with the caustic or refining solution were also readily separated. Desired % FFA of 0.1 % was gained for the refined Neem oil. A slight improvement in colour was also noticed for the Neem oil after treatment with caustic. Bleaching was not employed because; it further increases the acid value of the oil and may increase the overall cost of the treatment process. Moisture was efficiently removed by heating upto 150 °C.

3.4. Feedstock quality issues for biodiesel production

The characteristics of the feedstock oil which were analyzed are given in Table 1. FFAs (<0.5 %) and USM (<1%) were found to be in the required range. FFAs and USM can impact the production process by deactivating the catalyst, creating soap, and by releasing water when they are converted to esters, they do not seem to impact the final acid value of the biodiesel [5].

 Table 1: Feedstock characterization

Characteristics	Refined neem oil
FFA (%)	0.1
Moisture content (%)	No moisture
Unsaponifiable matter (%)	0.2
Phosphorus (ppm)	3.1
Iodine number	71.4

Higher amount of FFAs and USM could leads to emulsification and presents great difficulty during washing of biodiesel. It is important to remove water be prior to biodiesel production process because even a low level of water (1%) could increase soap production and measurably affect the completeness of the transesterification reaction.

Phosphorus must be removed because the ASTM specification for biodiesel limits the phosphorus content to 10 ppm. Fortunately, even if the oil is not degummed to remove this material, the usual biodiesel production process removes the phosphorus and it is either transferred to the glycerol fraction or removed by water washing. There does not seem to be any effect of the initial phosphorus content of the feedstock on the final phosphorus content of the biodiesel [5].

The iodine value is a commonly used indicator of the level of saturation of oil. While

saturation and fatty acid profile do not seem to have much impact on the transesterification process, they do affect the properties of the biodiesel. Saturated fats produce a biodiesel fuel with superior oxidative stability, a higher cetane number, but poor low temperature properties. Biodiesel from saturated fats is more likely to gel at ambient temperatures than biodiesel from vegetable oils [5].

The fatty acid composition of the Neem oil analyzed by gas chromatography is shown in Table 2. Very little amount of polyunsaturated compounds was observed. Biodiesel with polyunsaturated fatty acids with more than three double bounds is prone to deterioration in its oxidation stability, thus causing the precipitation of the biodiesel components in a fuel feeding system or combustion chamber [6].

 Table 2: Fatty acid composition (wt. %) of neem oil methyl ester

Types of fatty acids	Chemical structure	NOME
Palmitic acid	C 16: 0	17.79
Stearic acid	C 18: 0	15.25
Oleic acid	C 18: 1	46.73
Linolenic acid	C 18: 3	1.71
Eicosadienoic acid	C 20: 2	17.15

3.5. Transesterification

3.5.1 Effect of catalyst concentration

The above refined Neem oil was used for biodiesel production. The effect of sodium hydroxide concentration on the transesterification of the edible and non-edible oils was investigated with its concentration varying from 0.5 to 1.75 wt. % (based on the weight of raw oil). The operation conditions during the whole reaction process were fixed at the optimal level: reaction temperature of 60 °C, reaction time of 90 min. and 200 of methanol for 1000 ml of both the oils.

Experimental results showed changes in ester yield content with varied catalyst concentration. As the sodium hydroxide concentration increased, the conversion of triglyceride as well as the ester content also increased. Insufficient amount of sodium hydroxide resulted in incomplete conversion of triglycerides into the esters as indicated from its lower ester content. The ester content reached an optimal value when the sodium hydroxide concentration reached 1.0 wt. % for Neem oil. Increasing the catalyst concentration beyond these amounts showed decrease in the amount of methyl ester production as shown in Fig. 1. Large amount of soap was observed when sodium hydroxide was added in excess. This is because addition of excess alkaline catalyst caused more triglycerides participation in the saponification reaction with sodium hydroxide, resulting in the production of more amount of soap and reduction of the ester yield.

3.5.2 Effect of reaction time

The reaction time of the transesterification reaction conducted at 60 °C was optimized with the highest achievable mixing degree, an excess amount of alcohol (200 ml per liter of oil) and optimal sodium hydroxide concentration of 1.0 wt.% for Neem oil.

The changes in product composition with reaction time during the transesterification of the oils and the distribution of various components in the reaction system can be clearly seen. When the reaction time reached 90min, no triglyceride was left in the product mixture, indicating complete conversion. In this experiment, glycerol started to separate within 15 min. The ester content increased with reaction time from 15min onwards and reached a maximum at a reaction time of 90 min at 60 °C, and then remained relatively constant with increasing further the reaction time [Fig. 1]. The results indicated that an extension of the reaction time from 90 to 150 min had no significant effect on the conversion of triglycerides but leads to a reduction in the product yield. This is because longer reaction enhanced the hydrolysis of esters (reverse reaction of transesterification), resulted in loss of esters as well as causing more fatty acids to form soap.

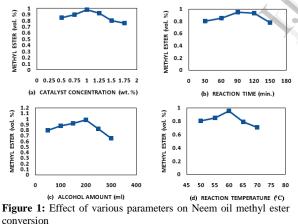
3.5.3 Effects of methanol amount

The effect of alcohol amount on yield of the transesterification experiments was conducted with different amounts of methanol to oil in the range of 50–300 ml. The optimized catalyst concentration and reaction time as obtained in the above sections were adopted. Maximum ester content was obtained at a methanol amount of 200 ml for Neem oil. With further increase in the methanol to oil amount above these amounts,

a very little effect on the biodiesel yield was observed [Fig. 1]. Moreover, it was observed that for high alcohol amount added the set up required longer time for the subsequent separation stage since separation of the ester layer from the water layer becomes more difficult with the addition of a large amount of methanol. This is due to the fact that methanol, with one polar hydroxyl group, can work as an emulsifier that enhances emulsion. Therefore, increasing the alcohol amount to oil is another important parameter affecting the biodiesel yield and biodiesel purity, apart from catalyst concentration and reaction time. Glycerol yield was found to be more in non-edible oil when compared to edible oil.

3.5.4 *Effects of reaction temperature*

To study the effect of reaction temperature on methyl esters conversation. the transesterification reaction was carried out under the optimal conditions obtained in the previous section (i.e.200 of methanol and 1.0 wt. % sodium hydroxide). The experiments were conducted at temperature ranging from 50 to 70 °C at 5 °C interval. The effect of reaction time is shown in Fig. 1.



Experimental results showed that the transesterification reaction could proceed within the temperature range studied but the reaction time to complete the reaction varied significantly with reaction temperature. It can be seen that a high product yield could be achieved at 60 °C. With the temperature increase above 60 °C, the product yield started to decrease. The reason for this is that higher temperature

accelerates the side saponification reaction of triglycerides.

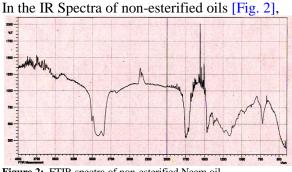


Figure 2: FTIR spectra of non-esterified Neem oil

3.6. FTIR and NMR testing

no absorption in the range of 3600-3300 cm⁻¹ was observed indicating the absence of any moisture in the feedstock oils [Table 3].

Table 3: FTIR an	alysis showing functional group	s and indicated compounds
of non-esterified t	riglyceride (Neem oil) and Neer	n oil methyl ester.
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Frequency range (cm ⁻¹)	Non-esterifie	d triglyceride	Neem oil met	hyl ester
	Functional group	Class of compound	Functional group	Class of compound
3190-3050	=C-H Stretching	alkene	=C-H Stretching	Alkene
2925-2850	Aliphatic C-H Stretching	alkane	Aliphatic C-H Stretching	Alkane
1750-1730	>C=O Stretching	Ester (strong)	>C=O Stretching	Ester (strong)
1265-1205	C-0	Ester, ether, alcohol	C-0	Ester, ether, alcohol
1147-1122	>C=C< Stretching	alkene	>C=C< Stretching	Alkene

Further the IR Spectra of NOME [Fig. 3] also showed absence of any absorption in the range of 3600-3300 cm⁻¹ indicating the absence of OH functional group. This also suggests that absence of any water molecule or unreacted methanol was present in the esterified and non-esterified Neem oil, possibly due to its removal by heating of esterified and non-esterified Neem oil before processing as well as post processing. Further, there is presence of ester absorption in the range of 1750-1730 cm⁻¹ in all the spectra recorded. Aliphatic C-H stretch of higher fatty acid ester was observed from 2925-2850 cm⁻¹ and that of =C-H stretching absorption in the range of 3190-3050 cm⁻¹, respectively.

Further confirmation of transesterification of triglycerides to fatty acid methyl ester was obtained from ¹HNMR spectra.

In the non-esterified neem oil [Fig.4] having a double doublet at δ 5.37 to 5.26 ppm due to H_C proton was observed. Protons H_a and H_b appeared as doublets of doublets in the region of δ 4.3-4.27 and δ 4.17-4.3 ppm, respectively. Other protons due to aliphatic R' groups were observed up-field in their usual absorption region.

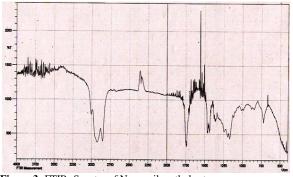


Figure 3: FTIR- Spectra of Neem oil methyl ester

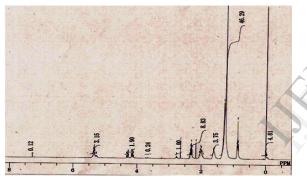


Figure 4: NMR- Spectra of Neem oil

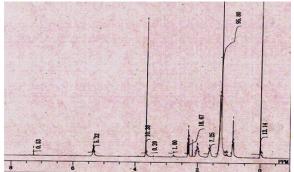


Figure 5: NMR- Spectra of Neem oil methyl ester

In [Fig.5] Neem oil methyl ester, disappearance of double doublets due to H_a , H_b , with H_c protons was observed. Appearance of a singlet due to CH₃ group of ester at δ 3.66-3.62 ppm was observed. These results suggest that almost

complete conversion of triglycerides to methyl esters have taken place.

3.7. Analysis of fuel properties of biodiesel

As shown in Table 4, the flash point of the Neem biodiesel was found to be much higher than that of petroleum diesel. Liquid fuel with a high flash point can prevent auto ignition and fire hazards at high temperatures during transportation and storage periods.

blend with diesel	Table	e 4: Comparision	of fuel	properties	of Neer	n oil	methyl	ester	and	its
biend with dieser	blend	with diesel								

Property (units)	NOME (B-100)	NOME (B-20)	DIESEL
Flash point (°C)	222	81	76
Kinematic viscosity at 40 °C (cSt)	4.8	4.1	3.1
Sulphated ash (mass %)	0		
Cloud point (°C)	-4	-10	-15
Water and sediment (vol. %)	0		
Acid value (mg KOH/gm)	0.12		
Phosphorus (mass %)	0.34		
Density @15 °C (Kg/m ³)	860	840	820
Calorific value (MJ/Kg)	40.25	43.56	44.34
Iodine value	49.12		

Viscosity is a significant fuel property that affects the flow and atomization characteristics of a liquid fuel. The kinematic viscosity of the biodiesel was found to be larger than that of the diesel, as shown in Table 4. Hence, it is inferred that the Neem biodiesel will have inferior injection and atomization performance, but offer lubrication and protection for the moving parts of an engine superior to those of the diesel.

Sulphated ash was absent in both the biodiesel fuels which indicates that there is no residual alkali catalyst as well as any other ashforming compounds that could contribute to injector deposits or fuel system fouling.

Cloud point of the neem biodiesel was found to be in the lower range than that of the cloud point of conventional diesel. According to the ASTM standards, cloud point must be reported to indicate biodiesel's effect on the final blend cloud point. Neem biodiesel was found to be free from moisture and sediments, which was possibly due to the heating and filtration activities performed after processing of biodiesel. Acid number for NOME was found to be negligible because of the use of NaOH as catalyst which served as a caustic stripper and removed the FFAs by converting them to soap that was removed during washing. Presence of phosphorus was also observed to be in the negligible quantity because of the degumming process performed prior to the biodiesel processing. Densities of the neem biodiesel was found to be in accordance with the ASTM, IS and EN standards. The calorific value of NOME was found to be lower than that of petroleum diesel. The higher the heating value, the lower is the fuel consumption rate.

Iodine number which is an indicator of degree of unsaturation was found to be in the limits prescribed by ASTM and IS standards for the neem biodiesel fuel. To some extent, presence of unsaturated fatty acid component in methyl esters is required as it restricts the methyl esters from solidification. However, with higher degree of unsaturation, methyl esters are not suitable for diesel engine fuel as the unsaturated molecules react with atmospheric oxygen and are converted to peroxide, cross-linking at the unsaturation site can occur and the material may get polymerized into a plastic like body. At high temperature, commonly found in an internal combustion engine, the process can get accelerated and the engine can quickly become gummed up with the polymerized methyl esters [7].

4. Conclusions

Problems related to the inbuilt FFAs could be easily removed by dehulling of external Neem seed coat. Oil extracted after dehulling of external seed coat could be easily employed for biodiesel processing because of its lower FFA content (0.435 %). Oil extracted from Neem seeds with both the seed coat contains a higher amount of initial FFAs, which could not be directly employed for biodiesel processing. Caustic stripping is the cheapest and convenient way for the reduction of FFAs from neem oil while acid esterification darkens the Neem oil. Base catalyzed transesterification is a faster and an economical way of biodiesel processing with its optimized parameters which leads to the achievement of desired quality of biodiesel fuel (within the limits prescribed by ASTM, IS and EN standards).

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