Quality Sensing of Degummed and Neutralized Calophyllum Inophyllum Oil Using Ultraviolet Absorption Spectra

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ABSTRACT

Non edible oils are purified in various stages in the production of biodiesel process. The Calophyllum inophyllum oil is investigated in this paper for the purification stages sample oil are, Crude Calophyllum Inophyllum Oil (CCIO), Degummed Calophyllum Inophyllum Oil (DCIO) and Neutralized Calophyllum Inophyllum Oil (NCIO) to enrich the knowledge of quality of the oil. The stagesare confirmed and compared with local available commercial diesel fuel by a reliable and affordable technology for the identification and detection of carbon groups using ultraviolet (UV) absorption spectroscopy. Since the absorbances of undiluted samples are too high to measure reliably, samples are diluted with n- Hexane. And for all three stages of sample oil, commercial local available diesel is considered as reference. The presence of hydrocarbons at the stages of degummed and neutralization shows, it can be used in internal combustion diesel engines and also the presence of unsaturated fatty acids and esters groups in the oil indicates further needed of process like esterification and transesterification for extracting better quality of fuel ie., biodiesel, for replacing the diesel fuel.

Keywords. Calophyllum inophyllum oil, degummed oil; diesel; spectroscopy UV - visible.

1. INTRODUCTION

In the usage of non edible oils as alternate source of fuel for fossil fuels, the determination of quality of fuel plays an important role for replacing diesel fuel.Zawadzki et al [1], illustrated that biodieselabsorption in the nearUV range of 245 to 305 nm was unaffected by biodiesel from different feedstocks diluted withnheptane. A linear relation was observed for the absorbancesof various blend levels from 245 to 305 nm, and the correlationcoefficient was greater than 0.99 for the wavelengths from 254 to 281 nm. The highest correlation coefficient was observed for the absorbance around 260 nm. When the absorbance of the diesel fuel was known, asingle wavelength could be used to detect the blend level. Sylvester O'Donnell et al [2] pointed that Ultraviolet – Visible (UV) spectroscopic analysis is fast becoming a common technique for providinganalytical data on degradation of biodiesel. Changes in peroxidevalues were studied using iodometry and UV-Vis spectroscopy to check for oxidative degradation. Marcos Roberto Monteiro et al [3] stated that UV spectroscopy procedure for the determination of methanol in biodiesel samples. According to the authors, this method was reasonable and it had good reproducibility and accuracy.

Armando Guerrero et al [4] illustrated that the biodiesel spectrum has absorbance bands in the wavelength range between 400 to 550 nm of the visible region. These bands are attributed to energy absorption by the double bond in unsaturated carbonyl compounds. The spectrum of the blends petrodiesel/biodiesel has multiple absorbance bands in the wave- length range between 200 to 235 nm in the UV region, these bands the may be related to the presence of aromatic compounds that contain unsaturated double bonds in its molecular structure. David D. S. Fernandes et al [5]proposed the use of UV-Vis spectrometry as a simpler alternative for detection of vegetable oil adulterations in biodiesel/diesel blends for soybean oil adulterations were investigated because this is the cheapest and most common vegetable oil found in Brazilian market. The proposed UV-Vis spectrometric method can be considered a useful complement to the methods usually employed by the regulatory agents. Vincent Baeten et al [6] stated that the most widely used and accepted physical technique for oil and fat authentication is ultraviolet (UV) spectrometry. Other promising physical techniques which have been investigated for oil and fat authentication include mass spectrometry, pyrolysis mass spectrometry, GC-electron ionisation mass spectrometry, nuclear magnetic resonance and infrared spectrometry.K.S. Shobha et al [7] illustrated that the UV-Visible absorption spectra of the metabolite was determined with a (Elico SL 159, India) spectrophotometer at 300-400nm to determine the λ maximum of the band. Sohannateressa, G. et al [8] stated that coconut, groundnut, castor and olive oils showed transmission above 70% in the UVB region. Olive oilshowed the least absorption among the four. Neem-seed, sunflower, sesame and cod - liver oil sowed transmission below 60% UVB region. Chikh Sabrina et al [9] used UV-vis for analysis of commercial diesel; diesel in the absence and presence of the antioxidant additive at different times of oxidation: 0, 1, 2, 3, 4, 5 and 6 hours. The absorbance at 256 nm of diesel showed at different times of oxidation. Comparing the spectrum of diesel with 2000 ppm additive and the blank we will notice a clear reduction in the peroxide value but the comparison of diesel with 2000 ppm antioxidant and the commercial one show that they have the same results. The results showed that the samples have absorption pick at 256 nm. The UV-vis spectrum of diesel in absence of additive resulted with absorbance at 232 nm from 0, 65962(0 h of oxidation) to 1, 24540 (6 h of oxidation).

H. E. Hassan et al [10] stated that the transmission light percentages were decreased by using infrared lights from 700 to 900 nm, visible lights from 400 to 700 nm, and ultraviolet lights from 200 to 400 nm wavelengths. The highest light transmission percentages were found of 94.89, 88.57 and 33.17 % for using wavelengths of 900 nm (IR), 700 nm (VIS) and 400 nm (UV), respectively. it was possible to use 900 nm (IR) to 700 nm (VIS) wavelengths as measuring quality of heated corn oil but, it is preferred to use about 700 nm wavelength as visible light. Allouis, C et al [11] pointed that the spectrum of the fine particulate sample collected in the exhaust is very similar to the parent oil one in the UV range (up to 350 nm), but in the visible a broad signal extending up to 550 nm appears, testifying thepresence of heavy aromatic structures absorbing in the visible. Moreover, these structures are not fluorescing atall in UV-visible range both using a fixed excitation wavelength in UV and in the visible range and alsoacquiring a synchronous fluorescence spectrum. Fernando H. N. Souza et al [12] stated that according to the UV-vis spectroscopy data, it is noteworthy that this technique can supply indications about quality of sunflower biodiesel and its oxidation products derived from accelerated oxidation test. This UV-vis spectroscopy determination constitutes a different approach from the measure of primary and secondary oxidation products, for example, peroxides, ketones, acid, aldehydes and others, and actually, it has been adopted more and more. In this direction, the authors used a specific absorbance at 232 nm to monitor the appearance of conjugated dienes in consequence of peroxidation products of sunflower biodiesel. The results showed that the samples presented absorption maxima at 232 nm. This behavior occursbecause the oxidation of polyunsaturated fatty acids, is accompanied of the displacement of isolated double bonds for conjugated double bonds. UV visible spectrum of sunflower biodiesel with absorbance at 232 nm from 0,207 (0 h of oxidation) to 1,359 (6 h of oxidation). Ravi KiranChekuboyina et al [13] illustrated that if no absorption peaks between 200 - 400 nm were detected, there is no conjugate double bond and C=O group, demonstratingthat this is most probably a saturated compound. If there is a weak peak (=10 - 100) between 270 - 350 nm, and no other peaksdetected over 200 nm it may contain >C=O, >C=C - O- or >C=C - N< etc. If there are many peaks in the UV region, some of them are even within the visible region, and then the compounds may have long conjugation bonds. When λ max is over 250 nm, and is between 1000 -10000, the compound may contain aromatic structure. If the peaks appear at the wavelengths 425, 455 and 480 nm in addition to 525, 570 and 590 nm chromophores, they may belong to carotenoids and flavonoids. Vorobyova O. A et al [14] stated that the shape and position of the absorption bands in UV-Vis spectra were the same for all hexane solutions afteradsorption, but the optical density in the visible region of carotenoids (400-500 nm) increased. Results showed a typical visible spectrum of a pumpkin seed oil hexane solution after passing it through a columnwith MgO, where bands have λmax equal to 424 and 434 nm. The same absorption bands were observed

ifcarotenoids were desorbed from the column by chloroform, but the optical density had different values at $\lambda max = 424$ and 434 nm. Thus, it may be possible to analyze the content of carotenoids in hexane or chloroform solution using appropriate calibration curves for each solvent. Besides, the shoulder in the region of 474 nm and the bands of 531 nm, 572 nm, and 630 nm, characteristic for protophilic compounds, e.g., chlorophyll were observed in the spectra of hexane extracts after saponification. The structures of carotenoids were investigated according to data of well-known UV-Vis spectra. Carotenoids of vegetable oils occur only in stable form where all double bonds are in trans configuration ("all-trans-form"). Maria P. et al [15] pointed that Jatropha oils had two main absorbance peaks in the UV-C region (272 and 282 nm; 10% oil in hexane). The absorbances at 272 nm were 0.84 ± 0.02 , 1.24 ± 0.01 , and 1.72 ± 0.04 for J. curcas, J. cinerea and J. platyphylla, respectively. The second peak (282 nm) followed absorption trend similar to that at 272 nm with 0.90 ± 0.02 , $1.26 \pm$ 0.01, and 1.61 \pm 0.04 for J. curcas, J. cinereaand J. platyphylla, respectively. The high absorption of J. platyphyllaoil suggests cisconfigurations and higher diene conjugations than those of J. cinereaand J. curcas. Mean absorptivity at 245 nm were 0.031, 0.034, and 0.036 for J. cinerea, J. curcas, and J. platyphyllaoils respectively, inferring presence of yellow pigment in the oils.B. Dave Oomaha et al [16] illustrated that Crude raspberry seed oil showed some absorbance in the UV-C (100 - 290 nm) and UV-B (290 - 320 nm) range. Thus, raspberry seed oil may act as a broad spectrum UV protectant and provide protection against both UV-A, an exogenous origin of oxidative stress to the skin, and UV-B. The optical transmission of raspberry seed oil, especially in the UV range (290 - 400 nm) was comparable to that of titanium dioxide preparations with sun protection factor for UV- B (SPF) and protection factor for UV-A (PFA) values between 28 - 50 and 6.75 - 7.5, respectively.

Numbers of papers are published in the quality sensing of non – edible oil such as jatropha, pungamia, corn, neem and pumpkin etc. As knowledge of the authors so far no paper has been published on the quality sensinganalysis of degummed calophyllum inophyllum oil and neutralized calophyllum inophyllum oil using UV visible absorption spectra. So objective of this paper is to study the quality sensinganalysis of degummed calophyllum inophyllum oil and neutralized calophyllum inophyllum oil using UV visible absorption spectra.

2. MATERIAL AND METHODS

The samples were prepared from the seed of calophyllum inophyllum according to the stages of purification of oil. The collected seeds are first removed outer shell, and by mechanical extraction method the oil was extracted and the oil is allowed for degumming to remove the gums, lectins and phosphatides and then the oil is neutralized.

2.1 Extraction of CCIO:

The seeds of calophyllum inophyllum were collected at the Nagerkovil district in the south side of Tamilnadu (India). The outer shells of the seed were removed and inner parts (kernel) collected. By means mechanical expeller, the kernels pressed and oil

was collected in storage vessel. About 65% of the oil is collected by means mechanical extraction method. The photographic view of collected Crude calophyllum inophyllum oil is shown in Figure 1.



Figure 1. Crude Calophyllum Inophyllum Oil

2.2 Degumming:

Oybek Zufarov et al [17] proceeded that Crude Calophyllum Inophyllum Oil (CCIO) was heated to 80 °C and water solution of citric acid (30 %) was then added in amount of 2 % (by volume of the oil). The mixture was stirred for 20 minutes. The oil/acid mixture was kept at 80 °C up to 15 min, cooled down to 25 °C, mixed with water (1 %) and transferred to a holding vessel. After settling for 60 min the mixture was centrifuged for 20 min to separate acid Degummed Calophyllum Inophyllum Oil (DCIO) from its by-products. The photographic view of collected Degummed calophyllum inophyllum oil is shown in Figure 2.



Figure 2. Degummed Calophyllum Inophyllum Oil

2.3 Neutralization:

Mustapha S I et al [18] illustrated that the Degummed Calophyllum Inophyllum Oil was neutralized by adding an alkali (sodium hydroxide solution). The mixture was then stirred and then heated at a regulated temperature of 75°C for 20 min to break the emulsions formed. Two layers were formed after centrifuging, oil and soap. The soap stock was then filtered off. The remaining collected oil was Neutralized Calophyllum Inophyllum Oil (NCIO). The photographic view of collected Neutralized calophyllum inophyllum oil is shown in Figure 3.



Figure 3. Neutralized Calophyllum Inophyllum Oil

UV – VIS Spectrophotometer:

A spectrophotometer is a device to measure light intensity at different wavelengths. It produces light with a light source, and after the light passes through a subject, the light is diffracted into a spectrum which is detected by a sensor and interpreted into results we can use.

The output of a spectrophotometer is usually a graph of light intensity versus wavelength. The data collected to generate this graph can typically be saved as a table of wavelengths and intensities. The y values of the graph can be represented as either transmittance or absorbance.UV spectroscopy is most commonly used for liquids, but can also be used for gases and even solids. Samples are placed incuvettes, a small rectangular container, usually 1cm in width. These are can be made of plastic, glass, or quartz (listed in increasing expense). Plastic and glass absorb UV, so they can only be used for visible light spectroscopy.

An Elico model SL 159 UV-Visible Single beam spectrophotometer with 1cm matched quartz cells at 200 to 500 nm were used for recording spectra and absorbance measurements. The dimensions are 520 X 395 X 180 cm with light source Deuterium (D) & Tungsten (W) Halogen Lamps and C z e r n y - Turner type with 1200 lines/mmHolographic grating monochromator used, with accuracy of \pm 0.005 Abs. at 1.0 Abs.

The amount of aromatic compounds varies with the stages of purification of oil, three stages samples of calophyllum inophyllum oil were collected from each stage. The three stages feedstocks chosen for oil were CCIO, DCIO and NCIO. The samples were prepared in 1 mL volumetric flasks and pipettes. The selected samples of oil have very high absorbance in the UV range. In order to bring the absorbance to

the measurable range of a spectrophotometer, the samples were diluted with n-hexane. In order to reduce the amount of n-hexane and to reduce the error, the dilution was completed in fivesteps. In each step, 0.2 mL of sample was accurately mixed with 0.8 mL of n-hexane. This dilution reduced the absorbance in the 240 to 350 nm wavelength range to within 1.2 for all samples. This concentration was chosen by trial and error to reduce the absorbance to the desired range of less than 2. Samples were prepared with commercial local available Diesel as reference. The photographic view of UV — Spectrophotometer is shown in Figure 4. The sample preparation for each purification stage is listed in Table 1.

- 1. Stage 1: Crude calophyllum inophyllum oil mixing with solvent n-hexane
- 2. Stage 2: Degummed calophyllum inophyllum oil mixing with solvent n-hexane
- 3. Stage 3:Neutralized calophyllum inophyllum oil mixing with solvent n-hexane

S.No	STAGE 1 (CCIO)		STAGE 2 (DCIO)		STAGE 3 (NCIO)	
	CCIO in ml	Diesel in ml	DCIO in ml	Diesel in ml	NCIO in ml	Diesel in ml
1	1.0	0	1.0	0	1.0	0
2	0.8	0.2	0.8	0.2	0.8	0.2
3	0.6	0.4	0.6	0.4	0.6	0.4
4	0.4	0.6	0.4	0.6	0.4	0.6
5	0.2	0.8	0.2	0.8	0.2	0.8

Table: 1 Sample preparation stages



Figure 4. Photographic view of UV - Spectrophotometer

3. RESULTS AND DISCUSSION

Liquid fuel is predominantly a mixture of hydrocarbons with differing molecular structure. The carbon to hydrogen ratio which is one of the important parameters and their nature of bonding determine the energy characteristics of the hydrogen fuels. The differences in physical and chemical properties between the different types of hydrocarbon depend on their chemical composition and affect mainly the combustion process. In this analysis, the UV spectrum shows the presence of hydrocarbons in the samples considered for analysis of calophyllum inophyllum oil.

The analysis was done by keeping diesel as reference for all the three stages of the purification of calophyllum inophyllum oil. The spectrums are showing the presence of hydrocarbons like Benzene, naphthalene, aniline, pyridine, thiophene, isoquinoline, acetopheone, 2-acetyl thiophene etc. Sample 4 gives more predominant peaks when compare with the other samples.

3.1 Quality sensing of CCIO:

Figure 5 presents typical UV-Vis spectra of CCIO.For the crude calophyllum inophyllum oil the predominant peaks are 190 nm,204.5 nm, 222.5 nm,237.0 nm 245 nm and 259 nm. Usually 180- 260 nm values are for C-X groups. The hydrocarbons present are C=N, CH₃-(CH=CH)n, Benzene, Pyridine, Pyrazine etc.

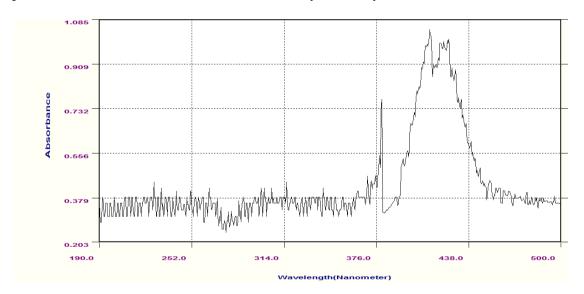


Figure 5.UV – Vis spectra of CCIO

3.2 Quality sensing of DCIO:

Figure 6 presents typical UV-Vis spectra of DCIO.Predominant peaks for the degummed calophyllum inophyllum oil are 244.5 nm, 247.5 nm, 250 nm, 278.0 nm 374.5 nm, 377.5nm, 450nm, 470nm and 485.5 nm. Usually 180- 380 nm values are for poly unsaturated and aromatic groups. 420-450nm range is for Nitrous oxide. The degumming process has changed the CCIO hydrocarbons. This is clearly visible in the spectrum. The hydrocarbons present are acetophenone, benzaldehyde etc.

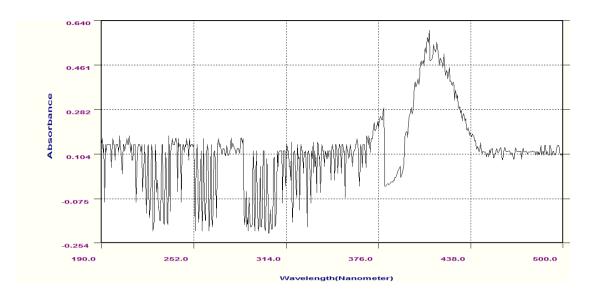


Figure 6.UV – Vis spectra of DCIO

3.3 Quality sensing of NCIO:

Figure 7 presents typical UV-Vis spectra of NCIO.Predominant peaks for the NCIO are 193 nm, 214 nm, 223 nm, 258 nm, 250 nm, 269nm, 285nm, 343nm, 470nm and 494.5 nm. Usually 180- 380 nm values are for poly unsaturated and aromatic groups. 420-450nm range is for Nitrous oxide. The neutralized process has changed the degummed crude oil hydrocarbons. This is clearly visible in the spectrum. The hydrocarbons present are Benzene, CH₃-(CH=CH)n, CH₃NH₂, 2-acetylfuran, 2-acetyl thiophene, pyridine etc.

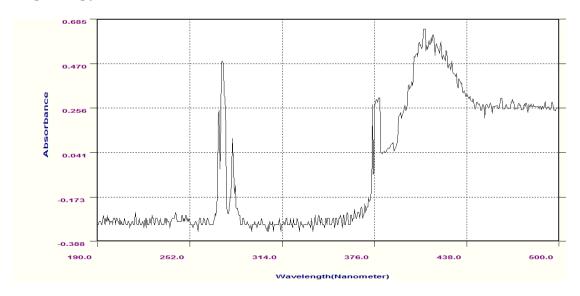


Figure 7.UV – Vis spectra of NCIO

Normallyparaffin's are exhibit the poorest antiknock quality when it used in petrol engines. But the antiknock quality improves with the increasing number of carbon atoms and the compactness of the molecular structure. The aromatics offer the best resistance to knocking in spark ignition engines. The aromatic series are presence in all the samples of purification stages. For diesel engines, it is vice versa i.e., the normal paraffin's are the best fuels and aromatics are the least desirable. Similarly, the paraffin series are presence in all the samples of purification stages. The heating value generally increases as the proportion of hydrogen atoms to carbon atoms in the molecule increase due to the higher heating value of hydrogen than carbon. Thus paraffin's are having the highest heating value and the aromatics the least. The above spectrum taken for the various stages for purification of CCIO identify the hydrocarbons present. This identification confirms the efficiency of crude calophyllum inophyllum oil.

4. Conclusion

The method of purification at each stage was evaluated, for the quality sensing of calophyllum inophyllum oil samples, sensedbased on the absorbance of samples diluted using n-hexane in the UV absorption range of 200 nm to 500 nm. The absorbance pattern, rather than the absolute absorbance of the aromatic, olefins and paraffin's content of the calophyllum inophyllum oil was used to distinguish hydrocarbons present in the purification stages of CCIO, DCIO and NCIO. In all spectrums, paraffin, olefin, Naphthene and Aromatic series are present. In paraffin series hydrocarbons like Isobutane the valency of all the carbon atoms is fully utilized by single bonds with hydrogen atoms. They are saturated and characteristically very stable. Olefins like butadiene are also straight chain compounds similar to paraffin's but are unsaturated because they contain one or more double bonds between carbon atoms, hence they are not stable. So these products are kept low by specification. Aromatic compounds are stable, though the presence of double bond indicates the unsaturation.

When the absorbance of the CCIO was known, a single wavelength could be used to detect the blend level. However, since the amount of hydrocarbons in the samples varied, it was shown that a single wavelength was insufficient to detect the blend level with an unknown source of diesel. Hydrocarbons presence was confirmed by UV visible spectrophotometer and this ensures this can be used for the synthesis of biodiesel.

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