International Journal of u- and e- Service, Science and Technology
Vol.9, No. 12 (2016), pp.11-18
http://dx.doi.org/10.14257/ijunesst.2016.9.12.02

# Quantitative Analysis of Degummed Calophyllum Inophyllum Oil

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***Abstract***

*The chemical properties of biodiesel fuel are much depends upon the fatty ester composition. The Calophyllum inophyllum seed oil without purification in crude stage and purified Calophyllum inophyllum oil in degummed stage were studied. The Crude Calophyllum Inophyllum Oil (CCIO) and Degummed Calophyllum Inophyllum Oil (DCIO) was the test samples considered for fatty acid quantitative analysis. The composition of fatty acids was identified by using Gas Chromatography (GC) to quantitative the test samples. In the results, the higher percentage presence of straight chain saturated fatty acid (palmitic) and unsaturated fatty acid (linoleic and oleic) has identified in the CCIO sample. By Degumming process, the percentage level of saturated fatty acid Palmitic diminished and unsaturated fatty acid Linoleic increased respectively; also the lesser amount formation of saturated fatty acid Capric, but unsaturated fatty acid Oleic and saturated fatty acid Myristic was completely removed.*

***Keywords:*** *Calophyllum Inophyllum Oil, Bio diesel, Degummed oil, Non-edible oil, Gas chromatography, Fatty acid*

## 1. Introduction

The non edible vegetable oils are a better alternate fuel for diesel due to its renewable and potentially inexhaustible source of energy with energy content close to diesel fuel. The nature of bio-degradability, no sulfur and aromatic contents, make bio-diesel as an attractive source of alternate diesel fuel because of its environmental sensitive properties [1]. The engine performance and emission characteristics by using vegetable oil are closer to the diesel fuel. Vegetable fuel has some unfavorable properties as fuel, such as high density, drying with time and gumming, lower cetane number, heavier exhaust smoke, *etc.*, Fuel characteristics of vegetable oils to be improved [2]. HPLC is a technique that gives good results, but the cost of the analysis is a little higher than the GC. Thus, the GC is preferred to

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ISSN: 2005-4246 IJUNESST

quantify biodiesel [3]. The first report on the use of capillary gas chromatography discussed the quantitation of esters as well as mono–, di–, and triacylglycerols [4]. The majority over (90%) of the fatty acid in the Canola, Linseed and Sunflower oils were those containing 18 carbons and included Stearic, Oleic, Linoleic and Linolenic acids in the order of increasing unsaturation. The primary fatty acid in the rape seed oil / ester was erucic acid [5].

Vegetable oils are dominated by five fatty acid species: Palmitic (16:0), Stearic (18:0), Oleic (18:1), Linoleic (18:2), and Linolenic (18:3). The physical and chemical properties of a biodiesel are determined by its chemical composition [6]. Raw Pungamia oil contains Palmitic acid (51%), Stearic acid (32.23%), Linoleic acid (8.93%), Arachidic acid (7.32%) and Oleic acids (0.18%). Saturated fatty acid content is about 91%, which is a favorable trait as a fuel property because it contains a high cetane number and stability. In purified oil, the value of the saturated fatty acid decreases, which is responsible for high viscosity [7].

Oleic and linoleic acids are the main fatty acids in sunflower oil [8]. The percentage of biodiesel productivities from vegetable oils rich in Linoleic acid (18:2), such as Safflower and Soybean, were slightly higher than those of oils rich in Oleic acid (18:1), such as Olive and Hazelnut [9]. The identified components of the GC result that the dominant fatty acid in Safflower, Soybean, Sunflower, waste Sunflower oil, Canola, and Corn oil esters was Linoleic acid (18:2) [10]. The fatty acid composition is variable based on the purification process of the oil considered for the test analysis. The result of fatty acid composition plays a vital role in characterizing of the oil and also in the Physio – chemical properties which are essential in the determining the engine performance evaluation [11]. Crude Rapeseed and sunflower oils were heated to 80 °C and water solution of citric acid (30 %) was then added in the 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 oil from its by-products [12].

Numbers of papers are published in the quantitative analysis of non – edible oil such as Jatropha, Pungamia, Sunflower, Canola, Olive, Castor, Pumpkin, Ground nut and Neem *etc.*, As knowledge of the authors so far no paper has been published on the quantitative analysis of Degummed Calophyllum Inophyllum Oil. So objective of this paper is to study the quantitative analysis of Degummed Calophyllum Inophyllum Oil.

## 2. Materials and Methodology

The seeds of Calophyllum inophyllum were collected at the Kanyakumari district in the south region of Tamilnadu, India. The outer shells of the seed removed and kernel pressed by mechanical expeller, and Crude Calophyllum inophyllum oil collected in the storage vessel. Crude Calophyllum Inophyllum Oil (CCIO) heated to 80 °C and water solution of citric acid (30 %) added in the amount of 2 % (by volume of the oil). The mixture stirred for 20 minutes. The oil/acid mixture 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 Degummed Calophyllum Inophyllum Oil (DCIO) from its by-products. The GC analysis is performed on a PerkinElmer Clarus 500, Turbo mass version 5.2.0 software program is used.

## 3. Result and Discussion

### 3.1. Quantitative Analysis of CCIO

 

The identified fatty acid components and other compounds in the Crude Calophyllum Inophyllum Oil are listed in Table 1, and chromatogram graph shown in Figure 1, respectively.

**Table 1. Major Chemical Compounds in the CCIO**

| S.No | Peak Name | Retention Time | Peak Area | % Peak Area |
|---|---|---|---|---|
| 1. | Name: 1,5-Heptadiene, 3,3,6-trimethyl-<br>Formula: C10H18 MW: 138 | 6.31 | 5288258 | 0.0927 |
| 2. | Name: Hexanoic acid<br>Formula: C6H12O2 MW: 116 | 6.79 | 10398792 | 0.1824 |
| 3. | Name: Bicyclo[2.2.1]heptane, 7,7-dimethyl-2-methylene-<br>Formula: C10H16 MW: 136 | 7.11 | 1636987 | 0.0287 |
| 4. | Name: 2-Hexenoic acid<br>Formula: C6H10O2 MW: 114 | 7.77 | 41258504 | 0.7236 |
| 5. | Name: Nonanal<br>Formula: C9H18O MW: 142 | 8.48 | 2865792 | 0.0503 |
| 6. | Name: Octanoic Acid<br>Formula: C8H16O2 MW: 144 | 10.30 | 2110952 | 0.0370 |
| 7. | Name: 2-Decenal, (E)-<br>Formula: C10H18O MW: 154 | 11.48 | 4264323 | 0.0748 |
| 8. | Name: Nonanoic acid<br>Formula: C9H18O2 MW: 158 | 12.02 | 1243800 | 0.0218 |
| 9. | Name: 2,4-Decadienal<br>Formula: C10H16O MW: 152 | 12.14 | 2475514 | 0.0434 |
| 10. | Name: 2,4-Decadienal, (E,E)-<br>Formula: C10H16O MW: 152 | 12.60 | 4622246 | 0.0811 |
| 11. | Name: 2-Undecenal<br>Formula: C11H20O MW: 168 | 13.26 | 5148062 | 0.0903 |
| 12. | Name: 3,5-Dimethyl-4-octanone<br>Formula: C10H20O MW: 156 | 13.59 | 1222706 | 0.0214 |
| 13. | Name: 3,4-Hexanedione, 2,2,5-trimethyl-<br>Formula: C9H16O2 MW: 156 | 15.23 | 377305 | 0.0066 |
| 14. | Name: 2-Propenoic acid, 3-phenyl-<br>Formula: C9H8O2 MW: 148 | 15.66 | 165079344 | 2.8951 |
| 15. | Name: 1H-2-Benzopyran-1-one, 3,4-dihydro-8-hydroxy-3-methyl-<br>Formula: C10H10O3 MW: 178 | 16.83 | 9140232 | 0.1603 |
| 16. | Name: 5-Methyl-(E)-2-hepten-4-one<br>Formula: C8H14O MW: 126 | 18.00 | 1087279 | 0.0191 |
| 17. | Name: 3,5-Dimethyl-4-octanone<br>Formula: C10H20O MW: 156 | 18.31 | 1692250 | 0.0297 |
| 18. | Name: Tetradecanoic acid<br>Formula: C14H28O2 MW: 228 | 19.71 | 1556955 | 0.0273 |
| 19. | Name: 2-Propenoic acid, 3-phenyl-, 2-methylpropyl ester<br>Formula: C13H16O2 MW: 204 | 20.26 | 2772373 | 0.0486 |
| 20. | Name: Hexadecanoic acid, methyl ester<br>Formula: C17H34O2 MW: 270 | 21.76 | 5804019 | 0.1018 |
| 21. | Name: Hexadecanoic acid, ethyl ester<br>Formula: C18H36O2 MW: 284 | 23.01 | 24343042 | 0.4269 |
| 22. | Name: n-Hexadecanoic acid<br>Formula: C16H32O2 MW: 256 | 24.52 | 2007138432 | 35.2009 |

| | | | | |
|---|---|---|---|---|
| 23. | Name: 9,12-Octadecadienoic acid, ethyl ester<br>Formula: C20H36O2 MW: 308 | 27.05 | 51638280 | 0.9056 |
| 24. | Name: (E)-9-Octadecenoic acid ethyl ester<br>Formula: C20H38O2 MW: 310 | 27.21 | 79490040 | 1.3941 |
| 25. | Name: 9-Octadecen-1-ol, (E)-<br>Formula: C18H36O MW: 268 | 28.61 | 531094560 | 9.3143 |
| 26. | Name: 9,12-Octadecadienoic acid (Z,Z)-<br>Formula: C18H32O2 MW: 280 | 29.08 | 768845440 | 13.4839 |
| 27. | Name: 9,12-Octadecadienoic acid (Z,Z)-<br>Formula: C18H32O2 MW: 280 | 29.54 | 1333272064 | 23.3827 |
| 28. | Name: Oleic Acid<br>Formula: C18H34O2 MW: 282 | 29.89 | 136115232 | 2.3872 |
| 29. | Name: Octadecanoic acid<br>Formula: C18H36O2 MW: 284<br>Stearic acid | 30.29 | 499971328 | 8.7684 |

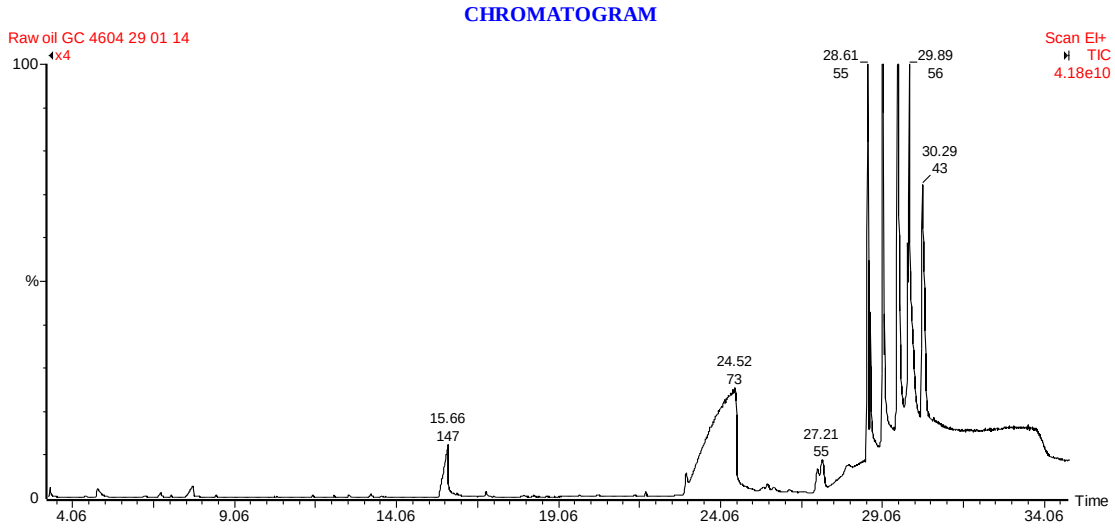


**Figure 1. Chromatogram of CCIO**

### 3.2. Quantitative Analysis of DCIO

The identified peaks and compounds in the Degummed Calophyllum Inophyllum Oil are shown in Figure 2, and the identified fatty acid components and other compounds are listed in Table 2, respectively.

**Table 2. Major Chemical Compounds in the DCIO**

| S.No | Peak Name | Retention Time | Peak Area | % Peak Area |
|---|---|---|---|---|
| 1. | Name: 1,6-Octadiene, 2,7-dimethyl-<br>Formula: C10H18 MW: 138 | 6. 29 | 4708881 | 0.1006 |
| 2. | Name: Hexanoic acid<br>Formula: C6H12O2 MW: 116 | 6.75 | 9299471 | 0.1987 |
| 3. | Name: Cyclobutane, 1,2-bis(1-methylethenyl)-, trans-<br>Formula: C10H16 MW: 136 | 7.09 | 1148588 | 0.0245 |
| 4. | Name: 2-Hexenoic acid<br>Formula: C6H10O2 MW: 114 | 7.76 | 4869339 | 1.0406 |
| 5. | Name: Nonanal<br>Formula: C9H18O MW: 142 | 8.46 | 2052285 | 0.4386 |

| | | | | |
|---|---|---|---|---|
| 6. | Name: Octanoic Acid<br>Formula: C8H16O2 MW: 144 | 10.33 | 1886981 | 0.0403 |
| 7. | Name: 2-Decenal, (E)-<br>Formula: C10H18O MW: 154 | 11.46 | 1740916 | 0.0372 |
| 8. | Name: Nonanoic acid<br>Formula: C9H18O2 MW: 158 | 11.99 | 906823 | 0.0194 |
| 9. | Name: 2,4-Decadienal<br>Formula: C10H16O MW: 152 | 12.12 | 1452294 | 0.0310 |
| 10. | Name: 2,4-Decadienal, (E,E)-<br>Formula: C10H16O MW: 152 | 12.56 | 2689505 | 0.0575 |
| 11. | Name: 2-Octenal, (E)-<br>Formula: C8H14O MW: 126 | 13.23 | 902051 | 0.0193 |
| 12. | Name: 3,4-Hexanedione, 2,2,5-trimethyl-<br>Formula: C9H16O2 MW: 156 | 13.57 | 394084 | 0.0084 |
| 13. | Name: 2-Propenoic acid, 3-phenyl-<br>Formula: C9H8O2 MW: 148 | 15.59 | 1615923 | 3.4534 |
| 14. | Name: 1H-2-Benzopyran-1-one, 3,4-dihydro-8-hydroxy-3-methyl-<br>Formula: C10H10O3 MW: 178 | 16.78 | 8210086 | 0.1755 |
| 15. | Name: 7-Tetradecene, (Z)-<br>Formula: C14H28 MW: 196 | 17.88 | 762393 | 0.0163 |
| 16. | Name: n-Decanoic acid<br>Formula: C10H20O2 MW: 172 | 19.52 | 3081698 | 0.0659 |
| 17. | Name: 2-Nonadecanone<br>Formula: C19H38O MW: 282 | 21.15 | 1962500 | 0.0419 |
| 18. | Name: Tridecanoic acid, methyl ester<br>Formula: C14H28O2 MW: 228 | 21.45 | 4114849 | 0.0879 |
| 19. | Name: n-Hexadecanoic acid<br>Formula: C16H32O2 MW: 256 | 23.18 | 1372402 | 29.3295 |
| 20. | Name: 9-Octadecenoic acid (Z)-, methyl ester<br>Formula: C19H36O2 MW: 296 | 25.07 | 4523047 | 0.0967 |
| 21. | Name: Octadecanoic acid, methyl ester<br>Formula: C19H38O2 MW: 298 | 25.70 | 5002653 | 0.1069 |
| 22. | Name: 9,12-Octadecadienoic acid, ethyl ester<br>Formula: C20H36O2 MW: 308 | 26.56 | 2761356 | 0.5901 |

**CHROMATOGRAM**

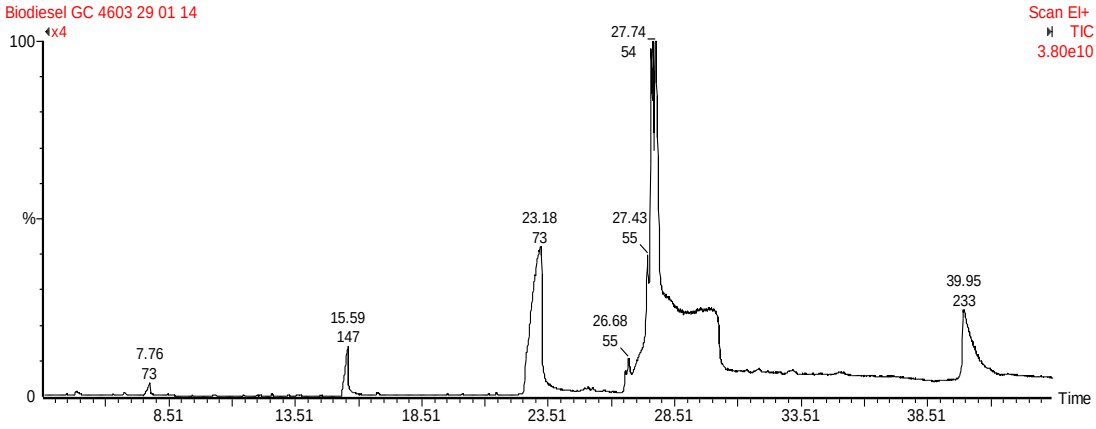


**Figure 2. Chromatogram of DCIO**

### 3.3. Saturated Fatty Acid of CCIO and DCIO

Caproic acid, Peak 2 of CCIO boiling point, melting point and flash point are 205.8 deg C, -3.4 deg C and 90 deg C, respectively. Caprylic acid, Peak 6 of CCIO boiling point,

melting point and flash point are 239 deg C, 16.5 deg C and 107.4 deg C, respectively. Its density is 0.910 at 20 deg C. Pealorganic acid, Peak 8 of CCIO crystallizes when cooled. Its boiling point, melting point and flash point are 254.5 deg C, 15.4 deg C and 115 deg C. Its density is 0.9052 g/cm$^3$ at 20 deg C. Myristic acid, Peak 18 of CCIO boiling point, melting point and density are 362.2 deg C, 53.9 deg C and 0.8622 at 54 deg C, respectively. Palmitic acid, Peak 22 of CCIO boiling point and melting point are 351.5 deg C and 62.5 deg C, respectively. The density is 0.8527 g/cm$^3$ at 62 deg C. Stearic acid, Peak 29 of CCIO boiling point and melting point are 350 deg C and 69.3 deg C, respectively. Its density is 0.9408 at 20 deg C/4 deg C. Capric acid, Peak 16 of DCIO boiling point and melting point are 268.7 deg C and 31.5 deg C, respectively.

The major portions of CCIO are Linoleic acid of unsaturated acid of 36.8 % and saturated fatty acid of Palmitic acid 35.2 % and stearic acid 8.7%.

### 3.4. Unsaturated Fatty Acid of CCIO and DCIO

Linoleic acid, Peak 26 and 27 of CCIO boiling point and melting point are 230 deg C at 16 mm Hg and – 8.5 deg C, respectively. Its density is 0.9022 g/cm$^3$ at 20 deg C. Oleic acid, Peak 28 of CCIO is insoluble in water and alcohol. Its boiling point, flash point and melting point are decomposed at 360 deg C and 1 atm, 270 deg C and 16.3 deg C, respectively. The comparable properties of fatty acid for the selected CCIO and DCIO samples, measured values listed in the Table 3.

**Table 3. Comparison of Fatty acid Properties of CCIO and DCIO**

| Fatty acid | Class | CCIO | DCIO |
|---|---|---|---|
| Caproic | C6:0 | 0.18 | 0.19 |
| Caprylic | C8:0 | 0.03 | 0.04 |
| Pelargonic | C9:0 | 0.02 | 0.019 |
| Capric | C10:0 | - | 0.06 **a** |
| Lauric acid | C12:0 | - | - |
| Myristic | C14:0 | 0.02 **b** | - |
| Palmitic acid | C16:0 | 35.2 | 29.32 |
| Stearic acid | C18:0 | 8.7 | 14.8 |
| Oleic acid | C18:1 | 2.3b | - |
| Ricinoleic acid | C18:1 | - | - |
| Linoleic acid | C18:2 | 36.8 | 38.2 |
| Linolenic acid | C18:3 | - | - |
| Eicosanoic acid | C20:0 | - | - |
| Arachidic | C20:0 | - | - |
| Behenic | C22:0 | - | - |
| Erucic | C22:1 | - | - |
| Lignoceric | C24:0 | - | - |

[a] Present only in DCIO ( Not detected in CCIO)
[b] Present only in CCIO (Not detected in DCIO)

## 4. Conclusion

The dominant fatty acid in the Crude Calophyllum Inophyllum Oil in terms of unsaturated fatty acid is Linoleic and saturated fatty acid is Palmitic, respectively. Another higher percentage of straight chain saturated fatty acid, Stearic and unsaturated fatty acid Oleic identified. By degumming process, the level of saturated fatty acid, Stearic increased to 14.8 % and Palmitic much reduced to 29.32%, also Caproic and Caprylic increased minor amounts of 0.19 % and 0.04 %, respectively, but the unsaturated fatty acid, Oleic and saturated fatty acid, Myristic were completely removed.

 

Simultaneously Capric formed in minor amount of 0.06 % whereas Pelargonic remains same about 0.02 %.

The biodiesel productivity percentage of oils rich in Linoleic acid (18:2), were slightly higher than those of oils rich in Oleic acid (18:1). The results showed that the DCIO, Linoleic acid has a higher percentage of 38.2 %. Hence, the quantitative analysis of degummed stage confirmed that increase of Linoleic acid, which is favorable for biodiesel production.

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