Elicitation and Characterization of Free Fatty Acids and Low Cost Biodiesel Production from the Seeds of Hevea Brasiliensis

Michael Donatus S, Madonna Shalma S, Vijayalakshmi S, Ranjitha J*
CO₂ Research and Green Technologies Centre, VIT University, Vellore-14
j.ranjithaammu@gmail.com
+919952223180

Abstract—The present paper describes about the elicitation and characterization of fatty acids and biodiesel production from the seeds of *Hevea brasiliensis*. In this study, soxhlet extraction had been used to extract, the rubber seed oil which contains high percentage of fatty acids. The extracted rubber oil was characterized using GC-MS spectrophotometer. Based on the spectral data, totally five fatty acids were identified using NIST GC-MS library. Further, the crude rubber oil was converted into biodiesel using acid-catalyzed trans-esterification reaction. Finally, the produced biodiesel was analyzed for their physicochemical properties using ASTM Standard.

Index Terms—Bio-diesel; *Hevea brasiliensis*; Fatty acids; GC-MS; Soxhlet’s extraction; Transesterification.

1 INTRODUCTION

The major problem is the increase use of fossil fuels, it cause damage to the environment increase in greenhouse gas (GHG) emissions. So the researcher interest in identifying alternative renewable sources of fuel that are potentially carbon neutral (Hill et al., 2006). In recently focused on renewable sources that were and eco-friendly sources of energy (Chisti, 2007). Thus, the production of biodiesel has been attempted as alternative sources using various materials, such as plants, microalgae, and animal fat, has been (Vasudevan and Briggs, 2008; Setapar et al., 2014) the rubber oil have high fatty acid for the biodiesel production, and the rubber oil not edible due the bitter taste. Lipids are main feed stock for the biodiesel production. Lipids mainly used for the biodiesel production such as plant oils and animals Fats (Prabakaran et al., 2011).

Many researchers used edible oils for the biofuel production that was increase the demand and prices of the edible oil, choice the rubber as raw materials. More than 90 percent of rubber was produced in India is from Kerala, rubber seeds are the one important by product. Rubber was used as a feed stock for the biodiesel production. The gases emitted by petrol, diesel driven vehicles causes harmful effect to the environment as well as human health (Imaekhai, 2003). Biodiesel has more environmental benefits than fossil fuels, biodiesel produce less smog forming, sulphur emissions, sulfuroxides, than diesel fuel those component cause damage to the environment such as ozone’s hole, acid rain (Senthil Kumar et al., 2014). (Ramadhas et al., 2005) demonstrated that rubber seeds oil contain the methyl esters that could be successfully used in existing diesel engines without any modifications. Then Lower concentrations of biodiesel blends improved thermal efficiency. At higher concentrations of biodiesel in the blend, there was a reduction of smoke density in exhaust gas. *Jatropha curcas* and rubber seed are both non-edible plants, their seed oil can be used to alternative fuels that can run diesel engines without requirement changes in design or modification in diesel engines. *Jatropha curcas* and rubber seed oil have similar chemical and performance characteristics with that of petroleum diesel (Hairsh et al., 2008). This research work focus on potential biodiesel production from the non-edible crude rubber oil from rubber seed. In southern India rubber production high and the biodiesel produce from the rubber oil can be used diesel engines without any changes. It has the important benefits to the environment to reduce the greenhouse effect, CO₂ effect.

2. MATERIALS AND METHODS

2.1. Collection of sample:
The rubber seeds *Hevea brasiliensis* were collected from southern part of Tamil Nadu in Kanyakumari district. The rubber shell was removed by manually and the seeds were cleaned. Approximately, take 500 grams of rubber seeds and kernels were heated in an oven at 110°C for about 5 hours to remove moisture content. The dried seeds were powdered and used for further processing.

2.2. Elicitation of Rubber Oil using Soxhlet’s extraction process: Weigh 100grams of *Hevea* seed powder material and transferred into soxhlet’s extraction tube. About 250 ml of n-hexane was poured into a round-bottomed flask and then, the Soxhlet's apparatus was connected with LB condenser. Finally the organic solvent was heated using a heating mantle for about 48 hours at a temperature of 70°C. The rubber oil was collected from extraction process using distillation process.

2.3. Yield of oil in percentage: After the oil was extraction by Soxhlet's extraction process and then the bio-oil was transferred into a measuring cylinder which was placed over water bath for 30 minutes at 70°C, to ensure complete evaporation of...
2.4. Fatty acid analysis: The qualitative and quantitative analysis of fatty acids was analyzed using GC-MS spectrophotometer.

Gas Chromatography: An Agilent 6890 gas chromatograph was equipped with a straight deactivated 2 mm direct injector liner and a 15m All tech EC-5 column (250μ I.D., 0.25μ film thickness). A split injection was used for sample introduction and the split ratio was set to 10:1. The oven temperature program was started to start at 35°C, hold for 2 minutes, then ramp at 20°C per minute to 300°C and hold for 5 minutes. The helium carrier gas was set to 2 ml/minute flow rate (constant flow mode).

Mass Spectrometry: A JEOL GC mate II benchtop double focusing magnetic sector mass spectrometer operating in electron ionization (EI) mode with TSS-20001 software was used for all analyses. Low-resolution mass spectra were acquired at a resolving power of 1000 (20% height definition) and scanning the magnet from m/z 25 to m/z 700 at 0.3 seconds per scan with a 0.2 second inter-scan delay. High resolution mass spectra were acquired at a resolving power of 5000 (20% height definition) and scanning the magnet from m/z 65 to m/z 750 at 1 second per scan.

Mass spectrometry library search: Identification of the components of the purified compound was matching their recorded spectra with the data bank mass spectra of NIST library V 11 provided by the instruments software.

2.5. Transesterification of Rubber oil: The transesterification of rubber oil was carried out using 5% solution of H2SO4. Take the Lipid/Methanol in the molecular ratio of 1:10 ratio and the reaction was performed at 95°C under constant stirring at 6000 rpm for about 5 hours. After completion of the reaction, it was transferred into a separating funnel allowing for about 2 hours to settle down and form a two-layer.

3. Results and Discussion
3.1. Moisture Content of Rubber seed: In this study the Rubber seed (Hevea brasiliensis) was used as a raw material for the production of Biodiesel. Since it contains moisture, to remove the moisture content the seed was dried using oven at 110°C for about 5 hours still constant weight was obtained. The moisture content was calculated and it was found as 8%. It has been reported in many studies that the optimum range of moisture content in oil seed processing was between 5 to 13%. (Ebeweke et al., 2010) have reported that the extracted rubber seed oil contain moisture content 10% at 70°C.

3.2. Rubber seed oil extraction: According to (Setapar et al., 2014), seed oil was extracted by using n-hexane by temperature at 68°C. He also reported that the oil yield from the rubber seeds was more than 50%. In this study hexane was used for the extraction of lipid from the rubber seeds at 70°C for 48 hours. The maximum oil yield was achieved more than 60%, due to the high temperature and high process duration. (Say-yar et al., 2012) was extracted oil from the Jatropha seeds the amount of oil yield from non-polar solvents did not change significantly after 6 hours, but the in this study increase in temperature and hours had increased the yield of oil content. (Setapar et al., 2014) also reported that increase in temperature and hours also increases the yield oil content.

3.4. Fatty acid analysis by Gas Chromatography and Mass Spectrometry (GCMS) analysis: Gas Chromatography and Mass Spectrometry (GCMS) analysis has been performed on the extracted bio-oil sample. The spectrum of the sample spread from 450 to 850 m/z values and the relative abundance of the resulted components varies from 10000000 to 110000000. The results revealed that the presence of fatty acids. The total compositions of the isolated components are under the peak reflection of different components. The mass spectroscopy of different obtained fatty acids and fatty acid methyl esters have been shown and their chemical structures are reported by (Vila et al., 2008). Based on the spectra, we can find that the chemical and bonding structure of the fatty acids and fatty acid-methyl or ethyl esters. This shows that the obtained sample of Hevea brasiliensis consist of plenty of fatty acids, which are the important features in producing the bio diesel. Based on the GC-MS spectrum of the different fatty acids constituents and their chemical structures were identified as shown in the Table -1.

<table>
<thead>
<tr>
<th>Peak No.</th>
<th>RT (Min.)</th>
<th>Compound Name</th>
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<tbody>
<tr>
<td>1.</td>
<td>17.92</td>
<td>Hexadecenoic acid</td>
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<td>2.</td>
<td>19.44</td>
<td>Butyl-9,12-octadecanoic acid</td>
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<tr>
<td>3.</td>
<td>19.49</td>
<td>Octadecanoic acid</td>
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<tr>
<td>4.</td>
<td>19.72</td>
<td>Octadecyenoic acid</td>
</tr>
<tr>
<td>5.</td>
<td>22.64</td>
<td>Di-n-octyl phthalic acid</td>
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</table>

Table 1: represents the isolated fatty acids from the seeds of Hevea brasiliensis

Figure 1: Typical GC-MS TIC chromatogram for Hevea brasiliensis extracted oil was characterized using GC-MS spectral data and totally fatty acids were identified using NIST GC-MS library.
3.5. Transesterification Process: (Lawrence et al., 2014) have compared that transesterified *Jatropha curcas* and rubber seed oils compared to the petroleum diesel and found that *Jatropha curcas* biodiesel had high Speed of Engine and Power of engine more than transesterified rubber seed oil but that transesterified rubber seed oil contain less emission of carbon monoxide and carbon dioxide than petroleum diesel and transesterified *Jatropha curcas* seed oil. So that transesterified rubber seed oil more eco-friendly biofuels. In the present study, the rubber oil was used for the biodiesel production by transesterification process using 5% H$_2$SO$_4$ as an acid catalyst. The maximum biodiesel yield was achieved more than 87% by using acid catalyst at 100°C for 5 hours. (Sivasubramanian et al., 2013) reported that the biodiesel yield was around 98%, when solid metal oxide catalyst, calcium oxide (CaO) was used for transesterification reaction. This shows that catalyst play an important role for biodiesel production (Figure 7).

**Figure 2.** The EI-MS spectra of Hexadecenoic acid shown in the GC–MS TIC chromatogram spectral data.

**Figure 3.** The EI-MS spectra of shown Butyl-9,12-octadecanoic acid in the GC–MS TIC chromatogram spectral data.

**Figure 4.** The EI-MS spectra of shown Octadecenoic acid in the GC–MS TIC chromatogram spectral data.

**Figure 5.** The EI-MS spectra of shown Octadecynoic acid in the GC–MS TIC chromatogram spectral data.

**Figure 6.** The EI-MS spectra of shown Di-n-octyl phthalic acid in the GC–MS TIC chromatogram spectral data.

**Figure 7.** Biodiesel from the seeds of *Hevea brasiliensis*
Table-2 represents the presence of fatty acid methyl esters in the biodiesel samples from the seeds of *Hevea brasiliensis*

4. Conclusions: In this study rubber seed oil biodiesel have more advantages transesterified rubber seed oil contain less emission of carbon monoxide and carbon dioxide than petroleum diesel and transesterified *Jatropha curcas* seed oil. Soxhlet extraction method using hexane was used for the extraction of lipid from the rubber seeds at 70°C for 48 hours. The maximum oil yield was achieved more than 60%, due to the high temperature and high process duration. The acid transesterification by using 5% H₂SO₄. The maximum yield of biodiesel was achieved more than 87%. The extracted rubber oil was characterized using GC-MS spectral data. Based on the spectral data, totally five fatty acids were identified using NIST GC-MS library. Further, the crude rubber oil was converted into biodiesel using acid-catalyzed trans-esterification reaction. Finally, the produced biodiesel was analyzed for their physicochemical properties using ASTM Standard.

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<th>Compound Name</th>
</tr>
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<tbody>
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<td>1.</td>
<td>17.32</td>
<td>Tetradecanoic Methyl ester</td>
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<td>2.</td>
<td>19.10</td>
<td>9,12 octadecadienoic Methyl ester</td>
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