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*
CO_2 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, CO_2 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 *Heavea* 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

ISSN 2229-5518

solvent and volume of the oil was measured. The percentage of oil was calculated by empricial formula (Warra *et al*., 2011).

$$Oil\ content(\%) = \frac{weight\ of\ the\ oil}{weight\ of\ the\ sample} \times 100$$

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 programmed 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 bench top 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 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 H_2SO_4. 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 transfered into a separating funnel allow it 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%. (Ebewele *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. (Sayyar *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.

Peak No.	RT (Min.)	Compound Name
1.	17.92	Hexadecenoic acid
2.	19.44	Butyl-9,12-octadecanoic acid
3.	19.49	Octadecenoic acid
4.	19.72	Octadecynoic acid
5.	22.64	Di-n-octyl phthalic acid

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

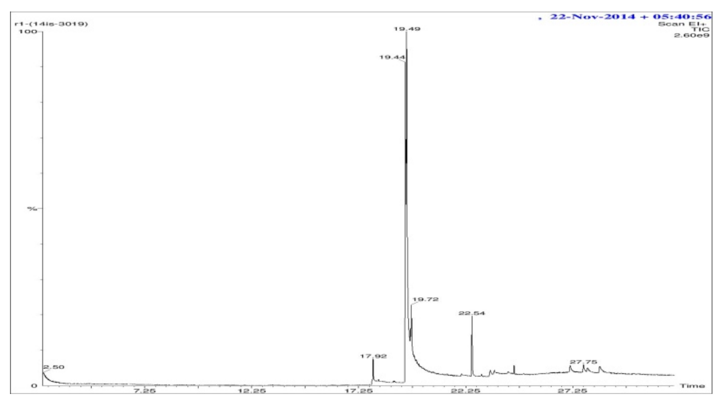

Figure 1. Typical GC–MS TIC chromatogram for *Hevea brasiliensis* extracted oil was characterized using GC-MS spectral data and totally fatty acids were identified using NIST GC-MS library.

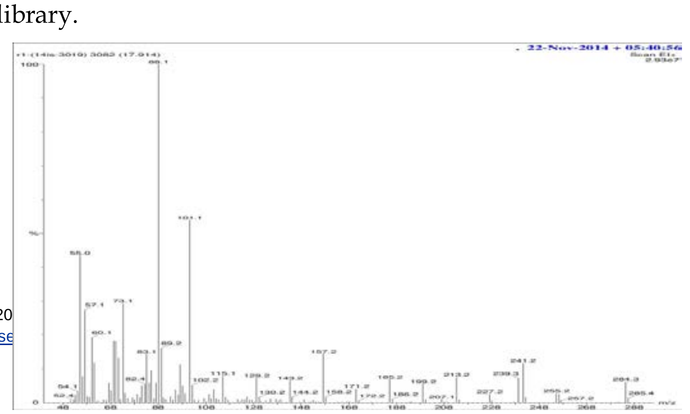

Figure 2. The EI-MS spectra of Hexadecenoic acid shown in the GC–MS TIC chromatogram spectral data

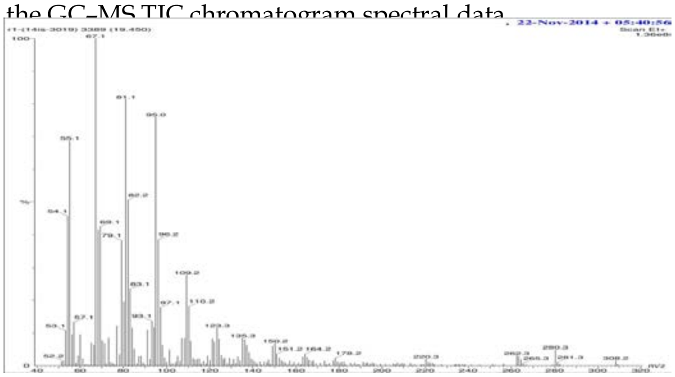

Figure 3. The EI-MS spectra of shown Butyl-9,12-octadecanoic acid in the GC–MS TIC chromatogram spectral data**.**

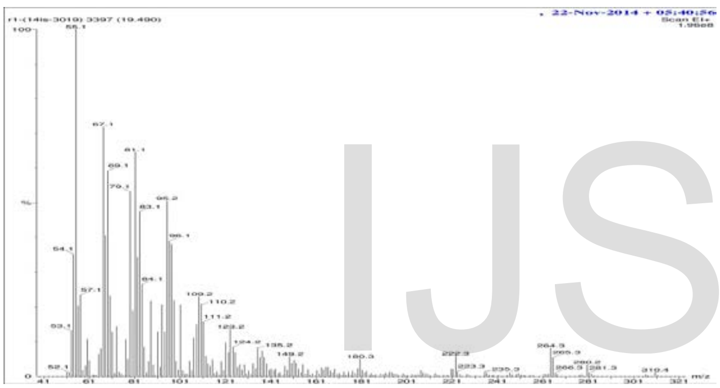

Figure 4. The EI-MS spectra of shown Octadecenoic acid in the GC–MS TIC chromatogram spectral data.

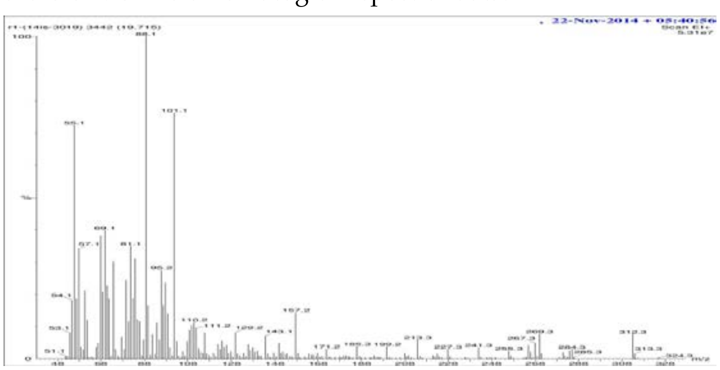

Figure 5. The EI-MS spectra of shown Octadecynoic acid in the GC–MS TIC chromatogram spectral data

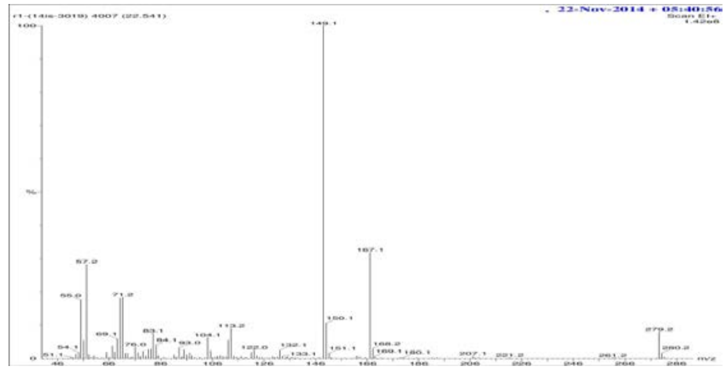

Figure 6. The EI-MS spectra of shown Di-n-octyl phthalic acid in the GC–MS TIC chromatogram spectral data

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_2SO_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 7. Biodiesel from the seeds of *Hevea brasiliensis*

ISSN 2229-5518

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

Peak No.	RT (Min.)	Compound Name
1.	17.32	Tetradecanoic Methyl ester
2.	19.10	9,12 octadecadienoic Methyl ester
3.	19.15	17,Octadecynonic Methyl ester
4.	19.28	Heptadecanoic Methyl ester
5.	20.89	Heptacosanoic Methyl ester

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% H_2SO_4. 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.

References

1. Hill, J., Nelson, E., Tilman, D., Polasky, S., Tiffany, D. (2006). Environmental, economic, and energetic costs and benefits of biodiesel and ethanol biofuels. *Proc. Nati. Acad. Sci. of the U S A.*, 103(30):11206–11210.
2. Chisti, Y. (2007). Biodiesel from microalgae. *Biotechnol. Adv.*, 25:294–306.
3. Vasudevan, P.T., and Birggs, M. (2008). Biodiesel production-current state of the art and challenges. *J. In. Microbiol. Biotechnol.*, 35: 421-430.
4. Sepidar Sayyar., Zurina Zainal Abidin., and Robiah Yunus, (2012). Optimisation of solid liquid extraction of Jatropha oil using petroleum ether. *Asia-Pac. J. Chem. Eng.*, 8: 331–338.
5. Pandian Prabakaran., and David Ravindran, A. (2011). A study on effective lipid extraction methods from certain fresh water microalgae. *Elixir Int. J. Biotech.*, 39: 4589 -4591.
6. Imaekhai, L. (2003). Transesterification of jatropha oil using immobilized *psuodonmans*. *J. Eng. Sci. Design*., 6: 21.
7. Senthil Kumar, S., and Purushothaman, K. (2014). High FFA Rubber Seed Oil as an Alternative Fuel for Diesel Engine – An Overview. *Int. J. Eng. Sci.*, 1(10):16-24.
8. Ramadhas, A.S., Muraleedharan, C., and Jayaraj, S. (2005). Performance and emission evaluation of a diesel engine fueled with methyl esters of rubber seed oil, *Rene.w Ener.*, 30(12):1789-1800.
9. Hairsh, G., and Avianah, K. (2008). Combustion characteristic of Jatropha oil Blends in a transportation engines. *SAE International*, Paper no. 2008-28-0034.
10. Warra, A.A., Wawata, I.G., Gunu, S.Y., and Aujaka, K.M. (2011). Extraction and physicochemical analysis of some selected northern Nigeria industrial oils. *Arch. Appl.Sci. Res.*, 3: 536-541.
11. Ebewele, RO., Iyayi, AF., Hymore, FK. (2010). Considerations of the extraction process and potential technical applications of Nigerian rubber seed oil. *Int. J. Phys. Sci*., 5(6): 826-831.
12. Mohd-Setapar, S.H., Lee Nian-Yian., and Mohd-Sharif, N.S. (2014). Extraction of Rubber (*Hevea brasiliensis*) Seed Oil Using Soxhlet Method. *Mal. J. Fund. Appl. Sci*..,10 (1): 1-6
13. Sala Vila, A., Miles, E.A., and Calder, P.C. (2008). Fatty acid composition abnormalities in atopic disease:evidence explored and role in the disease process examined. *Clin.Exp. Allergy.*, 38:1432–1450.
14. Imaekhai Lawrence., Okhinoghor Jim, I., and Airaodion Justice Uwaifo, (2014). Production of biodiesel from (RSO) rubber seed oil and *Jatropha curcas* seed oil (JCSO). *Standard J.Petro.Gas Exploration.*, 1(1): 1-4.
15. Sivasubramanian, V., Uma Krishnakumar., and Selvaraju, N. (2013). Physico-Chemical Properties of the Biodiesel Extracted From Rubber Seed Oil Using Solid Metal Oxide Catalysts. *Int. J.Eng. Res. App.*, 3(4):2206-2209.