SPECIAL ISSUE

Bima Journal of Science and Technology, Vol. 7 (2.1) August, 2023 ISSN: 2536-6041

SYNTHESIS OF BIODIESEL FROM CLOVE OIL VIA TRANSESTERIFICATION

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ABSTRACT

The demand for biodiesel as a sustainable and renewable alternative fuel for diesel engines is steadily increasing due to economic and environmental reasons. Biodiesel can be obtained from vegetable oils (both edible and non- edible) and from animal fat. The synthesis of biodiesel via transesterification of clove biodiesel oil was investigated. Crude clove oil was subjected to purification, then transesterification using methanol and NaOH. FTIR characterization was employed to determine the functional group (presence of biodiesel) of crude, purified and transesterified Clove oil. The FT-IR shows that the ester was produced between $723.10354\,\mathrm{cm}^{-1}$ and $1740.66677\,\mathrm{cm}^{-1}$ peaks in the transesterified sample. This signifies the production of biodiesel.

Keywords: Biodiesel, Clove Oil, Trans-esterification, FTIR

INTRODUCTION

The world energy demand is increasing mainly due to economic growth and population expansion. This high demand together with the negative global environmental impacts of using fossil fuel for energy generations brings a question on dependability of the fossil fuel for sustainable economic growth. Many alternatives have come into being in the recent years and several more are on their way to get established as sustainable fuel substitutes (Ismail *et al.,* 2022).

As a way out, the world is tending to depend more on renewable sources to secure the energy supply for its extensive demands towards the economic growth, improved standard of living and population expansion. Among the promising renewable energy resources for substitution of fossil fuels are biofuels. Biodiesel is one of these biofuels with significant advantages over its counterpart fossil diesel (Marchetti *et al.,* 2007).

The name biodiesel was introduced in the United States in the year 1992 by the National Soy Diesel Development Board which has pioneered the commercialization of biodiesel in the United State (Hariyadi *et al.,* 2020). Pure vegetable oil works well as a fuel for diesel engines itself, as Rudolf Diesel demonstrate in his engine at the 1900s (Indhumathi *et al.,* 2014). Biodiesel can be produced from renewable resources like edible and non-edible vegetables oils,

animal fats, and waste cooking oil. When compared to fossil diesel production, the process technologies usually employed to produce biodiesel are simpler and can easily be implemented in decentralized manner from small to large scale levels. The other major advantages associated with it is its environmental and ecological benefits compared to the fossil diesel. It is biodegradable, non-toxic and free from sulfur and aromatics. Combustion of biodiesel for energy generation releases less greenhouse gas (GHG), less air pollutants, and less particulate matters compared to the conventional fossil diesel, due to the facts that it has relatively high amount of oxygen required for its complete combustion (Marchetti *et al.,* 2007).

Biodiesel is an important new alternative transportation fuel. It can be produced from many vegetable oil or animal fat feed stocks. This Conventional processing involves an alkali-catalyzed process, but this is unsatisfactory for lower cost high free fatty acid feedstock due to soap formation. However, pretreatment processes using strong acid catalysts have been shown to provide good conversion yields and high-quality end products. These techniques have been extended to allow biodiesel production from feedstock like soap stock that are often considered to be waste. (Van Gerpen*,* 2005).

Clove oil is produced from the clove seed obtained from the clove tree. Clove tree, *Syzygium aromaticum* L., originates from the Maluku Islands in Indonesia and has been used for its spice and aromatic properties for millennia. Whilst products from the clove tree were imported regularly to Europe from as early as the 7th century, its story began more recently with the discovery of the Maluku Islands by the Portuguese, and the expedition organised by Magellan. Clove oil (Figure 1) consists of 60-90% eugenoleugenyl, 2-27% eugenyl acetate, 5-12% β-caryophyllene, and minor constituents such as methyl amyl ketone, methyl salicylate, and benzaldehyde. Clove stem oil usually contains 90–95% and clove leaf oil 82–88% eugenol. Stem and leaf oil may have traces of naphthalene (Baker *et al.,* 1813).

Figure 1: Photograph of Clove Oil (Ratri *et al*., 2020)

Transesterification is the process in which fat or oil reacts with an alcohol to form esters and glycerol. A catalyst is used to improve the reaction rate and yield. Because the reaction is reversible, excess alcohol is used to shift the equilibrium to the product side. Transesterification is the general term used to describe the important class of organic reactions where an ester is transformed into another through interchange of the alkoxy moiety. When the original ester is reacted with an alcohol, the transesterification process is called alcoholysis. The alcohol/vegetable oil molar ratio is one of the main factors that influence the transesterification. An excess of the alcohol favors the formation of the products. On the other hand, an excessive amount of alcohol makes the recovery of the glycerol difficult, so that the ideal alcohol/oil ratio has to be established empirically,

considering each individual process (Deng *et al.,* 2010).

MATERIALS AND METHODS

Purification and trans-esterification of crude clove oil was adopted in the production of biodiesel while infrared spectral analysis (FTIR) was done in order to characterize the trans-esterified Clove Biodiesel Oil.

Purification of Clove Oil

The crude clove oil of 200 ml was measured using measuring cylinder which is then prely-heated to 70^{o}C using hot magnet stirrer with thermometer. 1.5ml citric acid was measured and added to the heated oil sample and continuously heated and stirred for 15 minutes at 70^{o}C.The mixture then transferred to the vacuum oven and heated at 80^{o}C for 30 minutes. Then same mixture transferred back to the hot magnetic stirrer and heated at 70^{o}C after which a 2g of silicone reagent added while it's being heated and stirred for 30 minutes. The temperature then increased to 80^{o}C and 4g of activated carbon added to each 100 ml of the oil sample, heated and stirred for 30 minutes. The mixture was then separated using filter paper and separating funnel. Figure 2 and Figure 3 shows the separation of pure Clove oil from the residue.

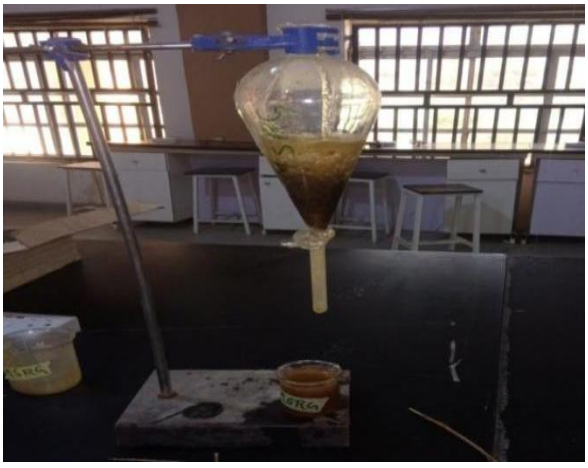

Figure 2: Purified Clove oil under separation

Figure 3: Purified Clove Oil and the Residue after Separation

Trans-esterification of Clove Oil

60ml of the clove oil was measured and heated and stirred until it reaches a temperature of 60-65℃ on a hot magnetic stirrer plate, 0.6g of NaOH measured using the electronic weight machine and allowed to dissolve in 21ml of methanol, the mixture of NaOH and methanol then added to the heated clove oil and allowed it to heat for 60 minutes with the stirrer on the hot magnetic plate. After 60 minute of uniform stirring and heating on the hot magnetic plate maintaining a temperature of 65℃, the mixture of Clove Oil, NaOH and methanol then poured into the separating funnel through a glass funnel. It's allowed to cool for about 40 minute. Afterwards, it is observed to separate into two liquid layers. The upper layer is the biodiesel and the lower layer is triglycrol fatty acid as in figure 4 and figure 5.

Bima Journal of Science and Technology, Vol. 7 (2.1) August, 2023 ISSN: 2536-6041

Figure 4: Transesterified Clove Oil under Separation

Figure 5: CloveBiodiesel Oil and Glycerol after Separation

Characterization of Clove Biodiesel Oil (FTIR)

The Fourier infrared spectral analysis was done using Fourier transform infrared SHIMADZU FTIR-8400S Spectroscopy machine which revealed the functional group of the sample. During the analysis, the sample in a form of thin film was placed between two potassium bromide discs made from single crystals, a drop of the liquid is placed on one of the disc and the other is placed on top which it leads to the spreads of the sample into a thin film.

The source which is located at the FTIR machine generates radiation which passes through the sample and interferometer and finally reaches the detector. The signal is amplified and was converted to digital signal by the amplifier and analog to digital converter respectively. Finally the signal is transferred to a computer in which Fourier transform is carried out.

RESULTS AND DISCUSSION

FT-IR Spectra of the Samples

The band with peaks 650 to 1400 cm^{-1} describes single bond (C-O) bond then 1500 to 1800 cm^{-1} described double bond (C=O) bond while 2700 to 3000 cm^{-1} described single bond (C-H) stretching and finally from 3000 to 3700 cm^{-1} described OH bond. Here C-O and C=O signify the presence of ester or ether group in the sample (Ismail *et al.,* 2022). Figure 6 illustrates the FTIR spectrum plotted for transmittance against the wave number (cm^{-1}) which is the representation of table 1.

Table 1: shows the result of Crude Clove oil FT-IR which revealed the peak number, wave number and the intensity of light absorbed by specific molecules present in the Crude Clove oil.

Table 1: FT-IR of Crude Clove oil

Peak Number	Wavenumber (cm^{-1})	Intensity (W/m^2)
1	726.83088	88.77270
2	894.56108	2 96.30984
3	1002.65388	3 93.76232
4	1177.83876	74.43572
5	1248.65818	88.11837
6	1375.38766	91.19361
7	1461.11643	87.16927
8	1740.66677	60.84853
9	2023.94445	101.15344
10	2426.49693	100.50984
11	2646.40986	100.05484
12	2795.50338	99.21479
13	2858.86812	84.90266
14	2925.96020	74.67377
15	3194.32853	100.18270

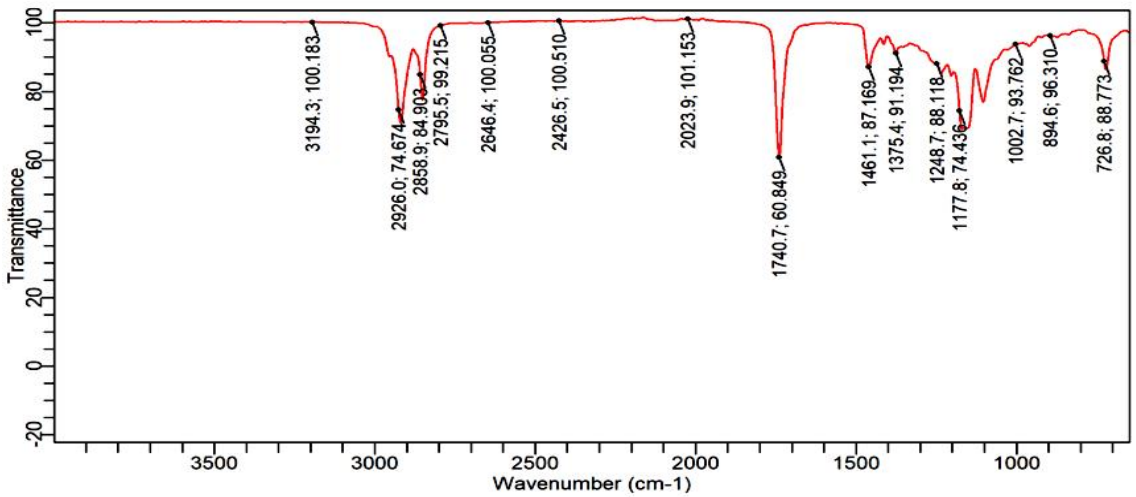

Figure 6: FT-IR Spectra of Crude Clove oil

Based on the amount of light absorbed by specific molecules present in the crude Clove oil, the ester was found to be at 726.83088 cm^{-1} , 894.56108 cm^{-1} , 1375.38766 cm^{-1} , 1461.11643 cm^{-1} and 1740.66677 cm^{-1} peaks this is as a result of the formation of C-O bond (single bond) and C=O bond (double bond) between the carbon and the oxygen present in the molecules of crude clove oil. Similar result was obtained by ((Shammeer and Nishath, 2019; Ismail *et al.,* 2022).

Figure 7 and table 2 indicated that the ester was at 723.10354 cm^{-1} , 961.65316 cm^{-1} , 1107.019344 cm^{-1} , 1155.47473 cm^{-1} , 1233.74882 cm^{-1} , 1379.11500 cm^{-1} and 1740.66677 cm^{-1} peaks. These indicate that there was improvement compare to crude Clove oil due to the fact the number of peaks indicating the present of ester was increased from 5 to 7 peaks. This is as a result of the reduction in the amount of residue present in the crude clove oil. In a research conducted by Shammeer and Nishath, (2019) similar result was obtained.

Table 2 is the result of purified Clove oil FT-IR which shows the peak number, wave number and the intensity of light absorbed by specific molecules present in the purified Clove oil.

Table 2: FT-IR of Purified Clove oil

Peak Number	Wavenumber (cm^{-1})	Intensity (W/m^2)
1	723.10354	82.44724
2	961.65316	90.14304
3	1107.019344	68.70808
4	1155.47473	56.61230
5	1233.74882	77.50253
6	1379.11500	86.12372
7	1461.11643	78.83674
8	1740.66677	43.55456
9	2851.41345	61.44489
10	2922.23286	51.06198

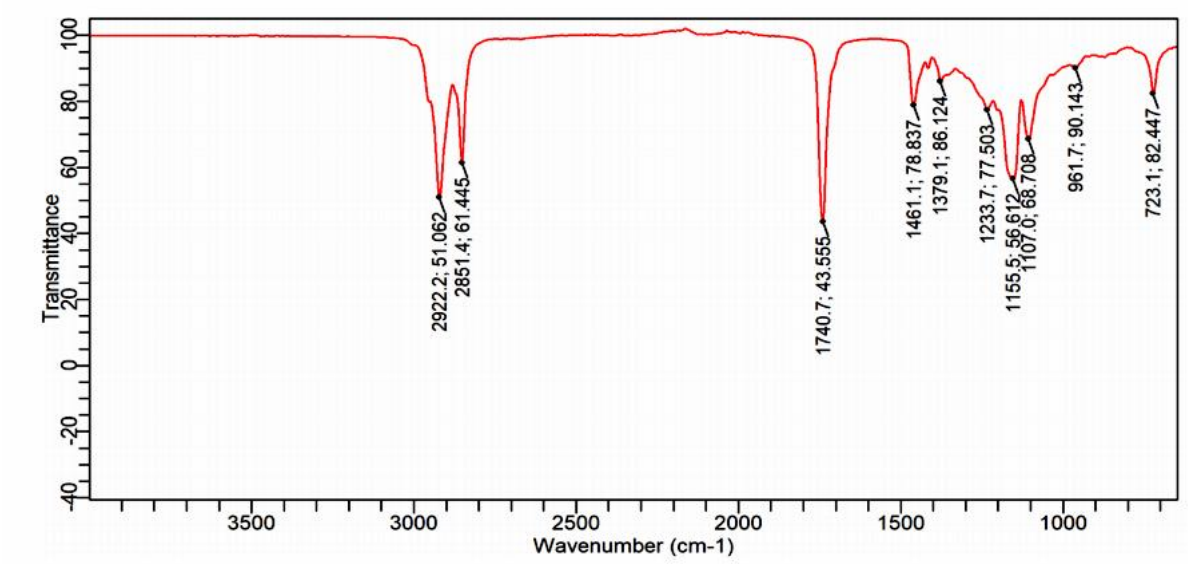

Figure 7:FT-IR Spectra of Purified Clove oil

Table 3 shows the FT-IR of transesterified Clove oil. It shows the peak number, wave number and the intensity of light absorbed by specific molecules present in the transesterified Clove oil.From figure 8 it can be observed that the ester was achieved at 723.10354, 961.65316, 1107.01934 4, 1155.47473, 1233.74882, 1375.38766, 1461.11643 and 1740.66677 peaks. These indicate that there is an improvement compare to purified Clove oil due to the increse in number of peaks signifying C-O and C=O bonds. Despite the increase in the number of peaks, the viscosity of the purified clove oil was equally reduced which is the essence of employing the method of transesterification. The result is similar to the result of Shammeer and Nishath, (2019).

Further, figure 8 illustrates the FTIR spectrum plotted for transmittance against the wave number (cm^{-1}).

Table 3: FT-IR of Transesterified Clove oil

Peak Number	Wavenumber (cm^{-1})	Intensity (W/m^2)
1	723.10354	83.32947
2	961.65316	89.36337
3	1107.01934	69.36702
4	1155.47473	58.50345
5	1233.74882	79.11228
6	1375.38766	86.50037
7	1461.11643	80.20105
8	1740.66677	47.03421
9	2851.41345	64.92558
10	2922.23286	55.51293

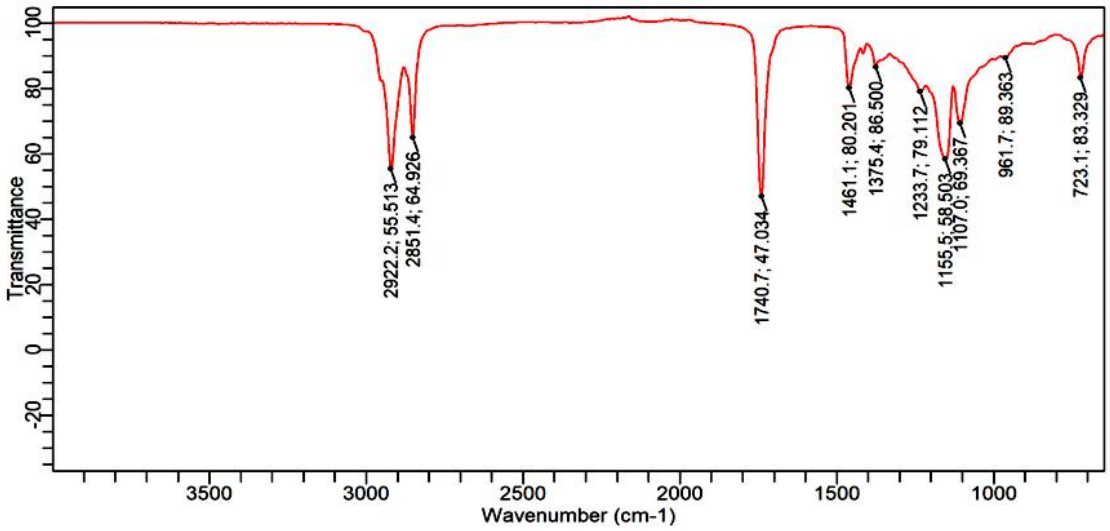

Figure 8: FT-IR Spectra of Transesterified Clove oil

CONCLUSION

Clove biodiesel oil was produced via transesterification process. Crude oil has undergone purification before cunducting the transessterification process using methanol as alcohol and NaOH as catalysts in producing the biodiesel. FTIR characterization has been used to determine the functional groups (presence of biodiesel) of crude, purified and transesterified Clove oil. The FT-IR of crude, purified and transesterified Clove oil confirms the presence of ester between 723.10354cm^{-1} and 1740.66677 cm^{-1} peaks. This signifies the production of biodiesel and affirming the use of Clove oil as a reliable biodiesel feedstock.

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