Conventional and Non-Conventional Transesterification for Biodiesel Production Using Waste Cooking Oil-Derived Soybean Oil

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Abstract

This study concern the production of biodiesel from waste cooking oil utilizing both conventional and non-conventional heating techniques employing a homogeneous base catalyst (NaOH). The research objective was to establish a process that is ecologically sustainable, highly efficient, and economically viable. The investigation effectively showcased the feasibility of biodiesel synthesis using both heating methodologies, resulting in commendable yields. The reaction time, molar ratio, and biodiesel quality of this process were compared with conventional transesterification. The assessment of biodiesel yields encompassed techniques such as Thin-Layer Chromatography (TLC), Fourier Transform Infrared Spectroscopy (FTIR), Proton Nuclear Magnetic Resonance (1H NMR), and American Society for Testing and Materials (ASTM) analysis. The optimization of different parameters influencing biodiesel synthesis under microwaves irradiation was done. Notably, the transesterification reaction of soybean oil into biodiesel under heating and ultra-sons sonication irradiation exhibited the highest conversion rate to methyl esters, yielding: 99%. As results ultrasonic technique a fast time reaction and smaller amount of methanol obtained a high conversion of methyl ester. Moreover, the properties of the produced biodiesel aligned with ASTM standard specifications.

Keywords: Biodiesel, tranesterification, microwaves irradiation, waste cooking oil, ultrasonic, NMR Qantification.

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1. Introduction

In recent decades, the quest for sustainable solutions to the challenges posed by fossil fuels has propelled the interest in biodiesel and other renewable fuels to the forefront. These alternatives offer promising resolutions to issues such as depleting oil reserves, the environmental fallout of carbon dioxide and greenhouse gas emissions, and the geopolitical vulnerabilities arising from fuel import dependencies (Zapata et al., 2012; Saka and Kusdiana, 2001). Biodiesel, notably distinguished by its renewable origin, non-toxic composition, biodegradability, and absence of aromatic and sulfuric compound, presents a compelling alternative to conventional fuels (Grande et al., 2021). Its combustion efficiency is heightened by a greater oxygen content compared to petroleum diesel, leading to a noteworthy reduction of up to 22% in carbon dioxide and particulate matter (PM) emissions (Osorio-González et al., 2020). Presently, the landscape of biodiesel production is predominantly shaped by edible vegetable oils, contributing to approximately 95% of the global output (Gui et al., 2008; Leung et al., 2010). Biodiesel produced from waste cooking oil (WCO) has the potential to significantly reduce the overall cost of biodiesel production. This is because the feedstock costs typically account for approximately 70-95% of the total expenses in biodiesel production (Chhetri et al., 2008). This biodiesel synthesis involves the transformation of triglycerides into fatty acid methyl esters through a reaction with methanol catalyzed by specific agents (Grande et al., 2021; Wong et al., 2021; Tariq et al., 2012; Wijaya et al., 2022). Primary feedstocks for biodiesel generation encompass first-generation biofuels, including palm, soybean, rapeseed, and sunflower oils (Paneque et al.,. 2011) . However, these sources are encumbered by inherent limitations due to associated negative externalities (Osorio-González et al., 2020a). The capacity of vegetable oils and animal fats to supplant liquid fossil fuels, while noteworthy, remains insufficient to meet current demand. Consequently, biodiesel derived from these materials could potentially address up to a quarter of the total diesel consumption (Quesada-Medina and Olivares-Carrillo, 2011). Intriguingly, exploration of alternative non-edible oil sources such as castor oil, jatropha oil, algae oil, karanja (Pongamiapinnata), tobacco (Nicotianatabacum), rubber plant (Heveabrasiliensis), and recycled cooking oil is gaining momentum, commanding extensive research efforts (Atabani et al., 2012; Lam et al., 2010; Hailegiorgis et al., 2013a). However, the efficacy of the transesterification reaction, despite catalyst utilization, is hampered by the limited solubility of oil in methanol. Ingenious strategies, including ultra-sonication, co-solvent utilization, phase transfer mechanisms, and microwave irradiation, have emerged to enhance mass transfer between reactants, thus expediting transesterification rates (Hailegiorgis et al., 2011b, 2012c, 2011d). The integration of microwave energy as a catalyst for organic reactions captured attention in the mid-1980s (Larhed et al., 2002). Subsequent studies underscore its potential for rapid, safe, and high-yield reactions, attributed to the selective absorption of microwave energy by polar molecules. This energy mode enables consistent, rapid, and widespread internal heating, setting it apart from conventional methods (Nagariya et al., 2010). Central to microwave energy heating is the orchestrated oscillation of polar molecules or ions under the influence of an oscillating electromagnetic field. This motion encounters resistance forces, generating randomized particle movement and subsequent heat release (Yadav et al., 2004).

Sono chemistry is a method that harnesses ultrasonic waves for chemical reactions. Ultrasonic technology is known for its effectiveness in enhancing mass transfer in liquid-liquid heterogeneous systems (Ji et al., 2006). By improving liquid-liquid mass transfer, it facilitates the thorough mixing of oils and methanol. Under ultrasonic irradiation, transesterification can occur at lower temperatures, requiring smaller amounts of catalyst and methanol. For instance, Georg ogianni et al. Conducted transesterification using waste oils with both alkaline and heterogeneous catalysts, employing low-frequency ultrasonication at 24 kHz in combination with mechanical stirring at 600 rpm. Their results highlighted numerous advantages of ultrasonic irradiation, such as achieving high yields of methyl esters and significant time savings (Georgogianni et al., 2007a).

Colucci et al. demonstrated the feasibility of producing biodiesel from soybean oil using ultrasonic mixing, reporting conversion rates exceeding 99% to fatty acid methyl esters (FAME) when applying ultrasonic energy (via a probe) for more than 15 minutes at 40°C.

The objective of this paper is to explore a new process through a parameter study of transesterification using non-conventional methods such as microwaves irradiation and the ultrasonic technique to convert waste cooking oil into biodiesel and compare it with the traditional heating methods. Therefore, this approach represents an efficient and cost-effective technique for biodiesel production. Furthermore, methods heating effects on methyl esters formation during transesterification were analyzed and the content of these esters were quantified by 1H NMR

2. Materials and methods

2.1 Materials

Waste cooking oil (WCO) collected from chip cracker potatoes factory in Algeria.Food remaining in the WCO used in thisstudywasremoved by filtration. The acid value of the samplesis 71.62±0.036 mgKOH/g.For the transesterification reaction, the chemical reagent analytical grade methanol (Sigma Aldrich, 99.8% (v/v) purity) and NaOH in pellets (Sigma Aldrich, purity > 99% (w/w)) were used. During biodiesel washing procedures a solution of hydrochloric acid (HCl) 37 % (Sigma-Aldrich), was used. The reagents for the determination of quality parameters were of analytical grade and in agreement with each standard specification (further referred).

2.2. Methods

2.2.1. Conventional Transesterification of Waste Cooking Oil (Biodiesel 1)

In a 100 mL reaction flask equipped with a reflux condenser (Figure 1), we introduce a 12.5 g of waste cooking oil-derived Soybean Oil in continuous stirring, a solution of 0.125 g of NaOH in 2.83 g of methanol (99.8%) was added at room temperature, maintaining an alcohol-to-soybean oil molar ratio of 6:1. The reaction mixture was then heated to 60°C to initiate the transesterification process, allowing it to proceed for 2 hours. After completion, the mixture separated into distinct glycerin and methyl ester (biodiesel) layers. The biodiesel phase underwent a thorough washing process involving HCl and distilled water, followed by decanting and subsequent heating to eliminate residual water and methanol (Tapanes et al., 2008); Georgogianni et al., 2008b). The progression of the reaction was monitored through thin-layer chromatography (TLC). Upon completion, the reaction was halted, and any excess solvent was eliminated using a rotary evaporator. The resulting products were separated via centrifugation at 3600 rpm. The upper layer (biodiesel) was then purified through a sequence of hot distilled water washes and desiccation with anhydrous sodium sulfate. The yield of biodiesel was quantified using the following equation (1):

$$\text{Yield (\%)} = (\text{weight of methyl ester (g)} / \text{weight of waste cooking oil (g)}) \times 100\% \qquad (1)$$

Characterization of the liquid products involved the use of thin layer chromatography (TLC), Fourier transform infrared spectrometry (FTIR), and proton nuclear magnetic resonance (1H NMR) techniques. Furthermore, the quality assessment of the produced biodiesel adhered to the American Standard for Testing Materials (ASTM) standards for biodiesel.

2.2.2 Microwave-Assisted Transesterification (Biodiesel 2)

In a 100 mL reaction flask equipped with a septum port, we introduce a 12.5 g of waste cooking oil-derived Soybean oil in continuous stirring, a solution of 0.125 g of NaOH in 2.83 g of methanol (99.8%) was added at room temperature, maintaining an alcohol-to-soybean oil molar ratio of 6:1. The mixture underwent irradiation using a common modified domestic household microwave (Figure 2) oven (Samsung model), operating at 2.45 MHz with programmable power ranging from 100 to 800 W.

2.2.3. Ultrasonic assisted transesterification(Biodiesel 3)

In a 100 mL reaction flask equipped with a septum port, we introduce a 12.5 g of waste cooking oil-derived soybean oil in continuous stirring, a solution of 0.125 g of NaOH in 2.83 g of methanol (99.8%) was added at room temperature, maintaining an alcohol-to-soybean oil molar ratio of 6:1. The mixture underwent irradiation using an Elma type S30H ultrasonic bath equiped with anrefregerant (Figure 3), the reaction carried out at a frequency of 80 KHz for 10 minutes.

2.3. Analytical methods

2.3.1.TLC chromatography

To monitor the reaction's progress, thin-layer chromatography (TLC) was conducted using a mobile phase composed of n-Hexane and acetic acid (9:1 v/v) on silica gel G-coated glass plates (Merck). The revelation was carried out using a UV lamp at a wavelength of 254 nm. Following completion of the reaction, the crude mixture was allowed to cool, and catalyst removal was achieved through filtration. Subsequently, the mixture was transferred to a separatory funnel, enabling the separation of glycerol via gravity over a 12-hour period. Distillation was employed to remove alcohol, followed by washing the ester phase with water (2 × 50 mL) to eliminate any remaining catalyst, glycerol, and alcohol.

2.3.2. ATR- FTIR

Attenuated Total Reflection Infrared Spectroscopy (ATR-IR) is commonly employed for the swift analysis of quality control. ATR-IR measurements were conducted using a Perkin Elmer Spectrum 100 instrument, with data processed through Spectrum Express software. The resultant vibrational spectrum reveals distinct and highly focused "fingerprints" of functional groups within the IR region (4000-650 cm-1). Specifically, the formation of the FAME (Fatty Acid Methyl Ester) group was identified within the spectral range of 1300 to 1060 cm^{-1}.

2.3.3.^{1}H NMR measurements

The 1H NMR analyses were conducted using a Bruker AMX 300 instrument, maintaining a temperature of 300 K. A 5 mm probe head and CDCl3 were employed as the solvent. In each analysis, 40 mg of the sample was dissolved in CDCl3, which also served as the internal reference, featuring a shift value for residual protons at 7.32 ppm.

Before initiating the analysis, the obtained spectra underwent treatment for phase and baseline corrections through the NMRNotebook program. Spectral integration was manually performed, with each spectrum integrated ten times, and the results were subsequently averaged. This averaging process helped mitigate uncertainties, ensuring that the final results remained within a margin of less than 5% originating from manual adjustments.

3. Results and discussion

Using basic conditions for the methanolysis of soybean oil the best results were achieved under conventional conditions with 0.125 g of NaOH stirred at 60 °C for 2 h. When this mixture was irradiated with microwaves (150 W) for 3 min we observed complete conversion of the oil to the respective Methyl esters (figure 4).

Aiming to reduce still more the reaction time, the mixture was irradiated (100 W) with microwaves (Kingston, 1988). When the same protocol was performed at higher MW power (200 W), we observed incomplete consume of the oil after 3 min, and the reaction mixture turned black.

However, the reaction was incomplete under conventional condition as well as under microwave irradiation. To verify the intervention of microwave specific effects it is necessary to perform a study with controlled temperature of the system (Perreux and Loupy, 2001a). Microwave specific effects can be expected when the polarity of the system is altered during the progress of the reaction (Perreux et al., 2003b). Ultrasonic experiments were conducted using identical quantities of reactants, catalyst, and a methanol-to-triglyceride (TG) molar ratio as employed in the conventional heating with mechanical stirring transesterification process. The notably high conversion achieved under ultrasonic conditions can be attributed to the rapid mixing and enhanced mass transfer between methanol and TG. Additionally, the formation of a microemulsion resulting from the ultrasonic cavitation phenomenon (Colucci et al., 2005), as depicted in Figure 4, played a crucial role in this efficiency.

Specifically, it was observed that conventional heating afford an impressive conversion, whereas the ultrasonic method achieved a significantly lower conversion of 76 % over a duration of 10 minutes. These results clearly indicate that reactions carried out in the conventional mechanical stirring occur at a much faster rate compared to those under the non-conventional ultrasonic tubular reactor process. The histogram below shows that the yield of the reaction for obtaining biodiesel through conventional heating and microwave irradiation is similar, around 87% and 89% respectively, whereas the yield decreases to 76% in the case of the ultrasonic reaction.

3.1. Optimization of parameters influencing production of biodiesel 2 under microwaves irradiation

To investigate the influence of key parameters on biodiesel yield when utilizing microwave irradiation, specifically:

- The impact of microwave radiation power

- The influence of catalyst quantity

- The effect of irradiation time

- The influence of alcohol quantity

A series of experiments were conducted, and the results are presented in Figure 5-8. Notably, the optimal conditions for achieving biodiesel 2 yield of 83% are as follows: 1% NaOH catalyst, a 6:1 ratio of MeOH to oil, a reaction time of 3 minutes and a microwave radiation power of 150 watts.

3.2. Biodiesel characterization using Thin-Layer Chromatography (TLC)

Thin-layer chromatography (TLC) confirms the formation of methyl ester, the desired biodiesel, as evidenced by the distinct spot observed on the plate (Figure 6). It is noteworthy that biodiesel 1, 2, and 3 exhibit identical Rf values. Furthermore, TLC also detects the presence of monoglycerides, diglycerides, and residual vegetable oil, revealing their formation through distinct spots on the plate. The eluent (mobile phase) employed in this study consists of n-Hexane and glacial acetic acid in a ratio of 9:1.

3.3. ATR-FTIR Characterization of biodiesel

Figure 10-14 displays the FTIR spectrum of soybean soil, waste cooking oil and biodiesel derived from waste cooking oil respectively. The FTIR analysis revealed distinct peaks at 2922 cm^{-1} and 2853 cm^{-1}, signifying the presence of alkane C–H stretching absorption. Additionally, a prominent peak at 1743 cm^{-1} indicated the absorption of the carbonyl group (C=O), found both in the carbonyl group itself and the carboxylic ester. A smaller peak at 1623 cm^{-1} was confirmed as the absorption peak of the C=C double bond. The peaks at 1458 cm-1 and 1165 cm^{-1} were attributed to $–CH_3$ and C=O absorptions, respectively (Attia et al., 2020).We also observe a band at 3007 cm^{-1} characteristic of the functional group =C-H with a cis geometry, which characterizes unsaturated fatty acids in the oil. Upon closer examination, the starting material exhibited an absorption peak at 1160 cm^{-1}, suggesting the presence of the $C–CH_2–O$ group in the waste cooking oil spectra. However, in the FAME (Fatty Acid Methyl Ester) products of biodiesel 1, 2, and 3, this absorption diminished, replaced by new peaks at 1435 cm^{-1} and 1196 cm^{-1}, indicative of the $–CH_3$ and $O-CH_3$ groups formation. Of particular significance is the emergence of the $O–CH_3$ peak at 1196 cm^{-1}, indicating the formation of methyl groups during the transesterification of waste cooking oil. The presence of methyl functional groups also serves as an indicator of the successful transesterification process and the production of FAME, affirming the effectiveness of the reaction.

3.4.^{1}H-NMR Study

Figure 15 shows 1H NMR spectra with characteristic signals and chemical shifts (ppm) of waste cooking oil derived soybean oil, which consists of linoleic acid (Tapanes et al., 2008). The transformation of waste cooking oil into biodiesel (Figure 12-14) was determined by the decrease or disappearance of the glyceride’s protons signal at δ= 4.15 to 4.30 ppm and the appearance of new signals at δ= 3.495 and 3.688 ppm. The new peak was the peak of the CH_3 protons (Tapanes et al., 2008). Based on the differences of ^1H NMR spectra between waste cooking oil and the produced biodiesel, it can be concluded that the soybean oil in this study have been converted into biodiesel.

The ^1H NMR spectra of crude waste cooking oil (doublet of doublets), pure methyl ester (singulet) and a waste cooking oil/methyl ester mixture from 4.15 to 4.30 ppm. (Figure 19)

In the quantification of the methyl esters using 1H NMR spectroscopy, there is a superimposition of glycerilmethylenichydrogens of the crude oil and the $-OCH_2$ from methyl ester in biodiesel.

Quantification of biodiesel using NMR can be done using several equations that have been reported in the literature; one of them will be described and used in the present work for the quantification of methyl esters. The notation used to describe the methods corresponds to that reported by da Fonseca et al. (Fonseca et al., 2008) in their study of transesterification of soybean oil with ethanol, was applied to the present research (Eq. 2)).

$$(\%)C_{ME} = \frac{\frac{4I_E}{3}}{\frac{4I_E}{3}+\frac{I_{TAG}}{2}}.100 \qquad (2)$$

The notation used in their paper was adapted to the one followed in this research:

%CMEcorrespond to the pourcentage of methyl esters conversion, ITAG corresponds to integration of glycerilmethylenichydrogens of TAG molecule at 4.33–4.45 ppm in soybean oil. Also, the term IE which corresponds to integration of signal at 3.66 ppm, this is exclusive to methoxyhydrogens (see Fig. 20-22).

The conversion to fatty ester was determined by 1H NMR analysis (Table-1) based on the signal at the methoxy (3.66 ppm; s, OCH3) for the methyl esters, in comparison with the signal of the a-methylene hydrogens (4.15–4.30 ppm) present in soybean oil (Goodrum and Geller, 2005; Ramezani et al., 2010).

As shown in the table, it is evident that both conventional heating and ultrasonic irradiation methods yielded highly significant conversions of soybean oil, exceeding 99% for the desired methyl esters.

3.5. ASTM analysis of Biodiesel

Biodiesel, produced through the transesterification of vegetable oils, can be evaluated using ASTM tests to ensure its quality and compliance with industry standards. The parameters assessed include specific density (specific gravity), kinematic viscosity, water content, lowtemperature fuel properties (cloud point and pour point), flash point, and residual sulfur (Patil, K.R, 2023).

Table 2 provides a comparison between the analysis results of biodiesel derived from waste cooking oil and the established ASTM specifications for biodiesel. The standards for biodiesel in accordance with The EN 14214 is an international standard that describes the minimum requirements for Biodiesel that has been produced from rapeseed, soya, jatropha or other virgin biomass, UCO (used-cooking oil), MSW (municipal solid waste) or from any viable fuel stock, serve as the reference.

The analytical findings indicate that some parameters meet the biodiesel criteria effectively. However, there is a need for improvement in certain parameters to align with the standards. Notably, high kinematic viscosity is a drawback when using soybean oil as a biodiesel feedstock. This is attributed to the predominance of linoleic methyl compound. Considering the comprehensive evaluation of parameters based on EN 14214 standards, the biodiesel produced is more suitable as an additive to fossil fuels rather than being used as a standalone fuel.

4. Conclusions

Biodiesel production derived from waste cooking oil proves viable through both conventional and non-conventional methods. However, the conventional approach yields significantly higher conversions compared to the non-conventional counterpart. The encouraging outcomes of the conventional method are notable, particularly given the utilization of lower-quality oils. These findings represent pioneering non-conventional methods to transesterification results for soybean oil. Nonetheless, further endeavors are warranted to enhance conversion rates. Notably, NMR spectroscopy emerges as a valuable technique for quantifying the conversion of triglycerides (TAG) into desired methyl esters.

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References

[1] Atabani, A. E., Silitonga, A. S., Badruddin, I. A., Mahlia, T. M. I., Masjuki, H. H., &Mekhilef, S. (2012). A comprehensive review on biodiesel as an alternative energy resource and its characteristics. Renewable and Sustainable Energy Reviews, 16, 2070-2093.

[2] Attia, A. M. A., El-Seesy, M. N., & Nada, S. A. (2020).The effect of castor oil methyl ester blending ratio on the environmental and the combustion characteristics of diesel engine under standard testing conditions.Sustainable Energy and Technology Assessment, 42, 100843. https://doi.org/10.1016/j.seta.2020.100843.

[3] Chhetri, A. B., Watts, K. C., & Islam, M. R. (2008). Waste cooking oil as an alternate feedstock for biodiesel production. Energies, 1, 3-18.

[4] Colucci, J. A., Borrero, E. E., &Alape, F. (2005).Biodiesel from an alkaline transesterification reaction of soybean oil using ultrasonic mixing. Journal of the American Oil Chemists' Society, 82, 525-530.

[5] da Fonseca, M. C., Biaggio, F. C., &GarcíaCortéz. (2008). XII Encontro Latino Americano de IniciaçãoCientífica e VIII Encontro Latino Americano de Pós-Graduação-Universidade do Vale do Paraí.

[6] Georgogianni, K. G., Kontominas, M. G., Pomonis, P. J., Avlonitis, D., &Gergis, V. (2008b).Conventional and in situ transesterification of sunflower seed oil for the production of biodiesel.Fuel Processing Technology, 89, 503-509.

[7] Georgogianni, K. G., Kontominas, M. G., Tegou, E., Avlonitis, D., &Gergis, V. (2007a). Biodiesel production: reaction and process parameters of alkali-catalyzedtransesterification of waste frying oils. Energy & Fuels, 21, 3023-3027.

[8] Goodrum, J., & Geller, D. (2005). Influence of fatty acid methyl esters from hydroxylated vegetable oils on diesel fuel lubricity. Bioresource Technology, 96, 851-855.

[9] Grande, L., Pedroarena, I., Korili, S. A., & Gil, A. (2021). Hydrothermal liquefaction of biomass as one of the most promising alternatives for the synthesis of advanced liquid biofuels: A review. Materials, 2021, 1-19. https://doi.org/10.3390/ma14185286

[10] Gui, M., Lee, K., & Bhatia, S. (2008). Feasibility of edible oil vs. non-edible oil vs. waste edible oil as biodiesel feedstock. Energy, 33(11), 1646-1653.

[11] Hailegiorgis, S. M., Mahadzir, S., &Subbarao, D. (2011b). Enhanced in situ ethanolysis of Jatrophacurcas L. in the presence of phase transfer catalyst. Renewable Energy, 36, 2502-2507.

[12] Hailegiorgis, S. M., Mahadzir, S., &Subbarao, D. (2011d). Reactive extraction of Jatrophacurcas L. assisted by phase transfer catalyst for the production of biodiesel. In National Post Graduate Conference (NPC2011) (pp. 245). DOI: 10.1109/NatPC.2011.6136271

[13] Hailegiorgis, S. M., Mahadzir, S., &Subbarao, D. (2012c). In situ transesterification of non-edible oil in the presence of cetyltrimethylammonium bromide. International Journal of Global Environmental Issues, 12, 161-170.

[14] Hailegiorgis, S. M., Mahadzir, S., &Subbarao, D. (2013a). Parametric Study and Optimization of In Situ Transesterification of Jatrophacurcas L. Assisted by Benzyltrimethylammonium Hydroxide as a Phase Transfer Catalyst via Response Surface Methodology. Biomass and Bioenergy, 49, 63-73.

[15] Ji, J., Wang, J., Li, Y., Yu, Y., &Xu, Z. (2006).Preparation of biodiesel with the help of ultrasonic and hydrodynamic cavitation.Ultrasonics, 44, 411-414.

[16] Kingston, H. M. (1988). The oven powers were determined as described by. In Jassie, L. B. (Ed.), Introduction to microwave sample preparation – theory and practice. Washington, DC: American Chemical Society.

[17] Lam, M. K., Lee, K. T., & Mohamed, A. R. (2010). Homogeneous, heterogeneous and enzymatic catalysis for transesterification of high free fatty acid oil (waste cooking oil) to biodiesel: a review. Biotechnology Advances, 28(4), 500-518.

[18] Larhed, M., Moberg, C., &Hallberg, A. (2002).Microwave-Accelerated Homogeneous Catalysis in Organic Chemistry. Accounts of Chemical Research, 35(9), 717-727.

[19] Leung, D. Y. C., Wu, X., & Leung, M. K. H. (2010).A review on biodiesel production using catalyzedtransesterification.Applied Energy, 87(4), 1083-1095.

[20] Nagariya, A. K., Meena, A. K., Kiran, A., Yadav, A. K., Niranjan, U. S., Pathak, A. K., ... Rao, M. M. (2010). Microwave-assisted organic reaction as a new tool in organic synthesis. Journal of Pharmacy Research, 3, 575-580.

[21] Osorio-González, C. S., Gómez-Falcón, N., Sandoval-Salas, F., Saini, R., Brar, S. K., &Ramírez, A. A. (2020). Production of biodiesel from castor oil: A review. Energies, 13, 1-22. https://doi.org/10.3390/en13102467

[22] Paneque, M., Román-Figueroa, C., Vásquez-Panizza, R., Arriaza, J. M., Morales, D., &Zulantay, M. (2011).Bioenergía en Chile. Santiago, Chile: FAO y Universidad de Chile.

[23] Patil, K.R., (2023). Experimental investigation into the combustion, performance, and emissioncharacteristics of oxygenated DEE and ethanolblendingwith KOME biodiesel fuelled CI engine. Journal of RenewableEnergy and Environment (JREE), Vol. 10, No. 2, 1-8. (https://doi.org/10.30501/jree.2022.328607.1330).

[24] Perreux, L., &Loupy, A. (2001a). A tentative rationalization of microwave effects in organic synthesis according to the reaction medium, and mechanistic considerations. Tetrahedron, 57, 9199–223.

[25] Perreux, L., Loupy, A., &Delmotte, M. (2003b). Microwave effects in solvent-free esters aminolysis. Tetrahedron, 59, 2185–2189.

[26] Quesada-Medina, J., & Olivares-Carrillo, P. (2011).Evidence of thermal decomposition of fatty acid methyl esters during the synthesis of biodiesel with supercritical methanol. Journal of Supercritical Fluids, 56, 56-63.

[27] Ramezani, K., Rowshanzamir, S., &Eikani, M. H. (2010). Castor oil transesterification reaction: a kinetic study and optimization of parameters. Energy, 35, 4142-4148.

[28] Saka, S., &Kusdiana, D. (2001). Biodiesel fuel from rapeseed oil as prepared in supercritical methanol. Fuel, 80(2), 225-231.

[29] Tapanes N, O., Aranda D, G., Carneiro J, M., &Antunes O, C. (2008).Transesterification of Jatrophacurcas oil glycerides: theoretical and experimental studies of biodiesel reaction. Fuel, 87, 2286-2295.

[30] Tariq, M., Ali, S., & Khalid, N. (2012). Activity of homogeneous and heterogeneous catalysts, spectroscopic and chromatographic characterization of biodiesel: A technical review. Renewable and Sustainable Energy Reviews, 16, 6303-6316. https://doi.org/10.1016/j.rser.2012.07.005

[31] Wijaya, K., Saputri, W. D., Aziz, I. T. A., Wangsa, Heraldy, E., Hakim, L., et al. (2022). Mesoporous silica preparation using sodium bicarbonate as a template and application of the silica for hydrocracking of used cooking oil into biofuel. *Silicon, 14(4), 1583-1591.

[32] Wong, K. Y., Ng, J. H., Chong, C. T., Lam, S. S., & Chong, W. T. (2021). Pareto-hierarchical clustering framework for biodiesel transesterification.Sustainable Energy and Technology Assessment, 45, 101160. https://doi.org/10.1016/j.seta.2021.101160

[33] Yadav, G. D., &Bisht, P. M. (2004). Novelties of microwave-assisted liquid-liquid phase transfer catalysis in enhancement of rates and selectivities in alkylation of phenols under mild conditions. Catalysis Communications, 5, 259-263.

[34] Zapata, N., Vargas, M., Reyes, J. F., & Belmar, G. (2012). Quality of biodiesel and press cake obtained from Euphorbia lathyris, Brassica napus and Ricinuscommunis. Industrial Crops and Products, 38(1), 1-5.

[35] Zhang, Y., Dubé, M., McLean, D., &Kates, M. (2003). Biodiesel production from waste cooking oil: 2. Economic assessment and sensitivity analysis. Bioresource Technology, 90, 229-240. https://doi.org/10.1016/S0960-8524(03)00150-0.

List of Table

Table1 conversion to fatty methyl esters of waste cooking oil determined by ^{1}H NMR analysis

Biodiesel	Biodiesel 1(Conventional heating)	Biodiesel 2 (under microwaves)	Biodiesel 3 (under sonication)
$\%C_{ME}$	99.92	95.85	99.07

Table2 ASTM Analysis results of biodiesel.

Parameters	Biodiesel*	Waste cooking oil derived Soybean Oil biodiesel 2 (non Conventional heating under microwaves irradiation)
Specific density at 15 °C, g/cm^3	0.800-0.900	0.886
Kinematic viscosity 40 °C, mm^2/s	3.5-5.0	4.18
Flash point °C	>101	151
Cetane number	51	47
Sulfur content, mg/Kg	10	0.067
Water content, mg/Kg	500	87

*Standards of biodiesel for the EN 14214-standard.

List of Figure

Fig.1. Experimental set-up of conventional heating

Fig.2. Experimental set-up of microwaves irradiation in domestic modified microwave for production of biodiesel

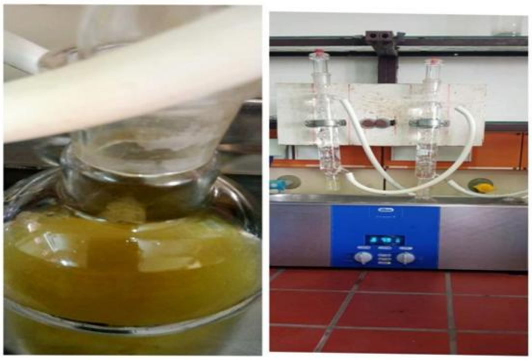

Fig.3. Experimental set-up of ultrasonic bath for biodiesel 3 production

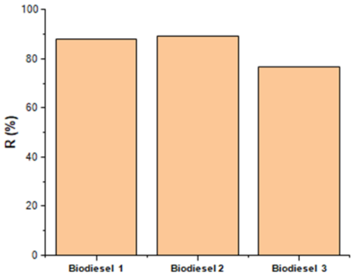

Fig.4. Yield Histogram for synthesis of Biodiesel 1, 2, and 3.

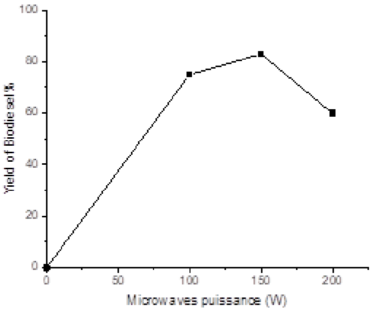

Fig.5. Yield of biodiesel2 vs. Microwaves puissance. Reaction

Temperature 60 °C, reaction time 3mn, amount of NaOH 1 wt%, methanol/iol ration 6:1.

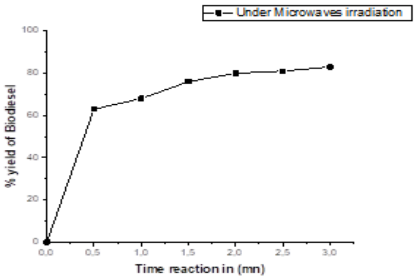

Fig.6. Yield of biodiesel2 vs. Time reaction. Reaction

Temperature 60 °C, puissance of iradation 150 W, amount of NaOH 1 wt%, methanol/iol ration 6:1.

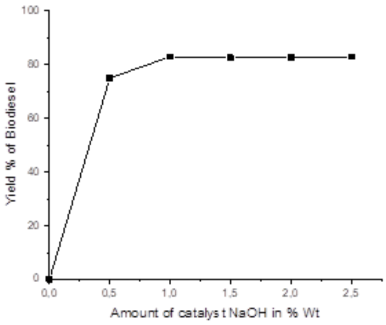

Fig.7. Yield of biodiesel2 vs. %Wt of NaOH. Reaction

Temperature 60 °C, reaction time 3mn, puissance 150 W, methanol/oil ration 6:1.

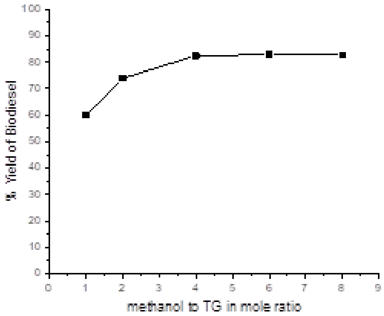

Fig.8. Yield of biodiesel2 vs. methanol to TG in mole ratio. Reaction temperature 60 °C, reaction time 3mn, amount of NaOH 1 wt%, Puissance 150 W

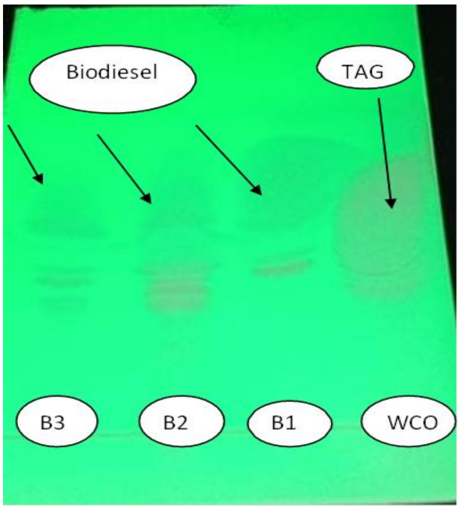

Fig.9. TLC plate of synthtetized biodiesel

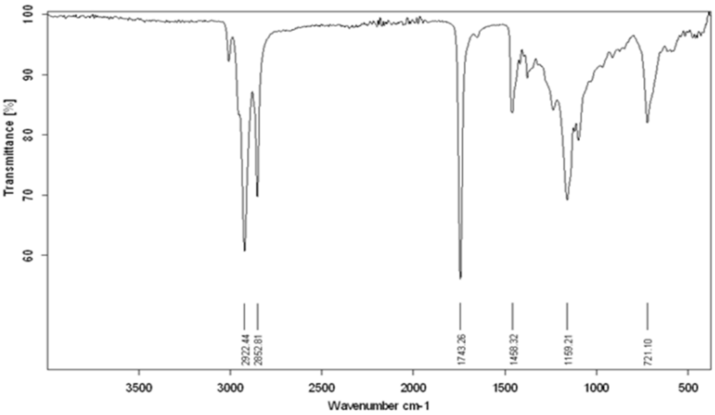

Fig. 10. FTIR spectra of Soybean oil

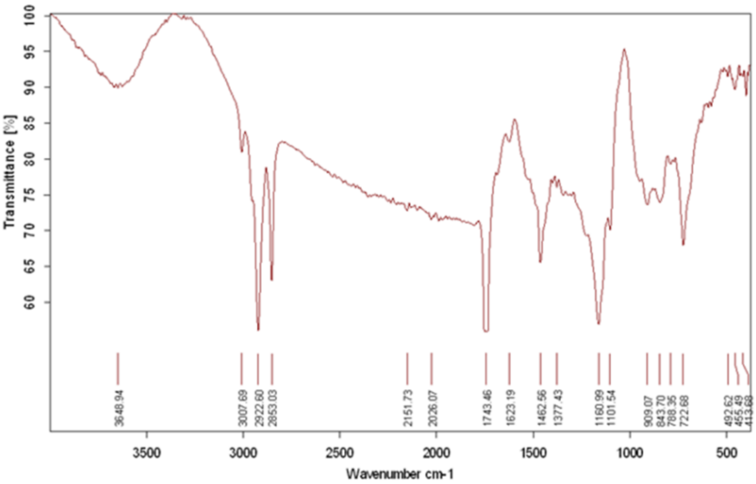

Fig.11. FTIR spectra of Waste Cooking Oil-derived Soybean Oil

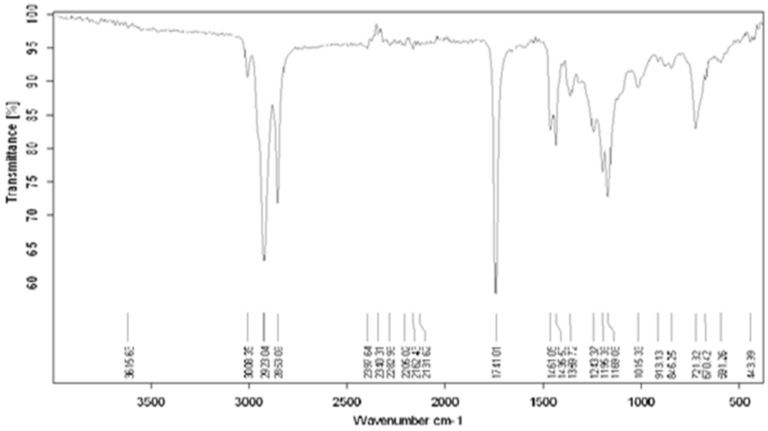

Fig.12. FTIR spectra of biodiesel1.

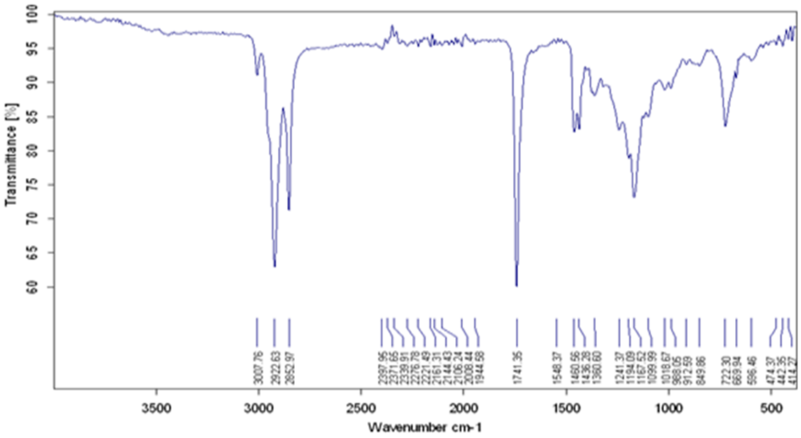

Fig.13. FTIR spectra of biodiesel2.

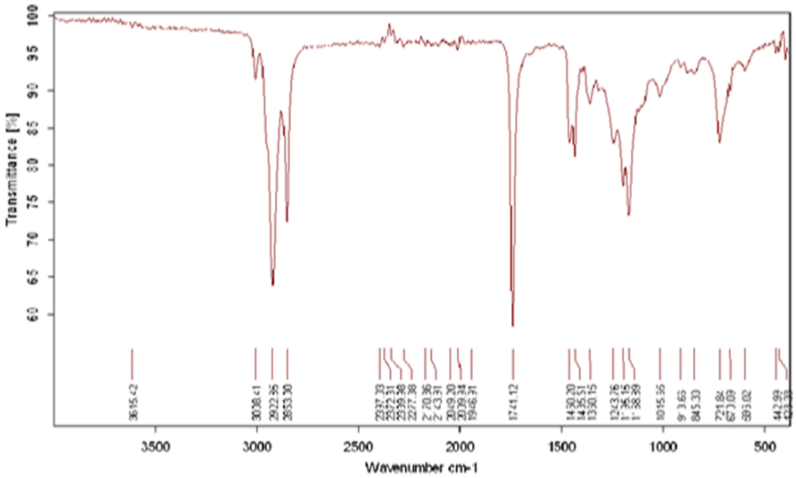

Fig.14. FTIR spectra of biodiesel3

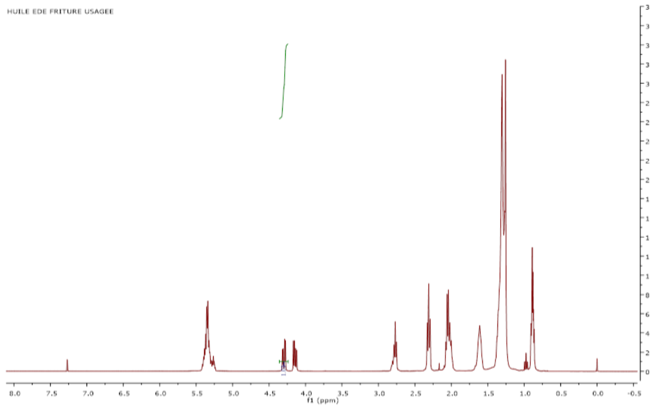

Fig.15. Full 1H NMR spectra ofwaste cooking oil soybean derived

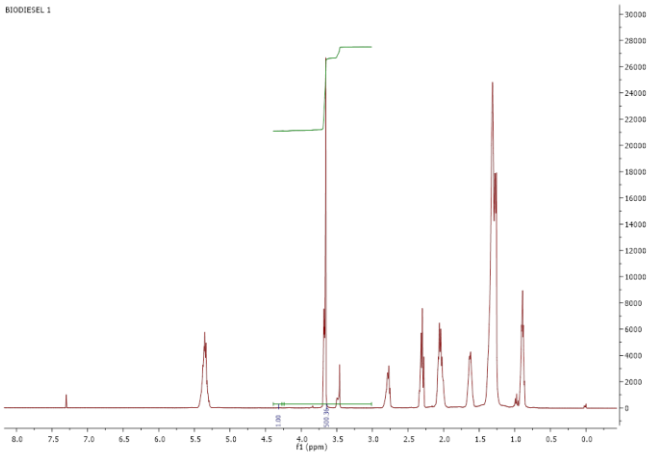

Fig.16. Full 1H NMR spectra of biodiesel 1 produced by conventional transesterification of Soybean Oil.

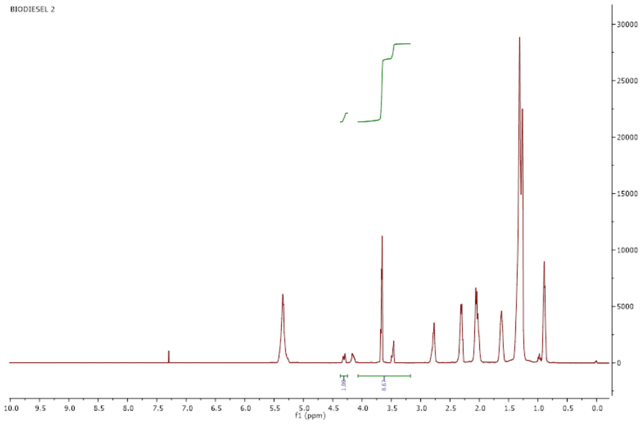

Fig. 17. Full 1H NMR spectra of biodiesel 2 produced by non-conventional microwaves transesterification of Soybean Oil.

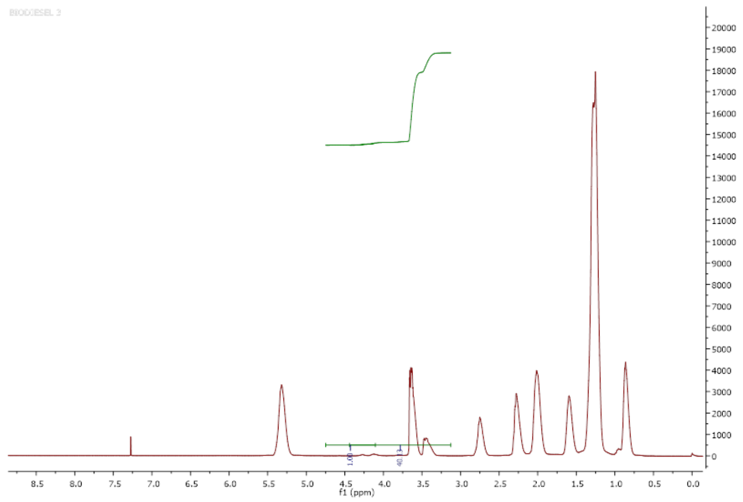

Fig.18. Full 1H NMR spectra of biodiesel 3 produced by non-conventional transesterification (Ulra-sons) of Soybean Oil.

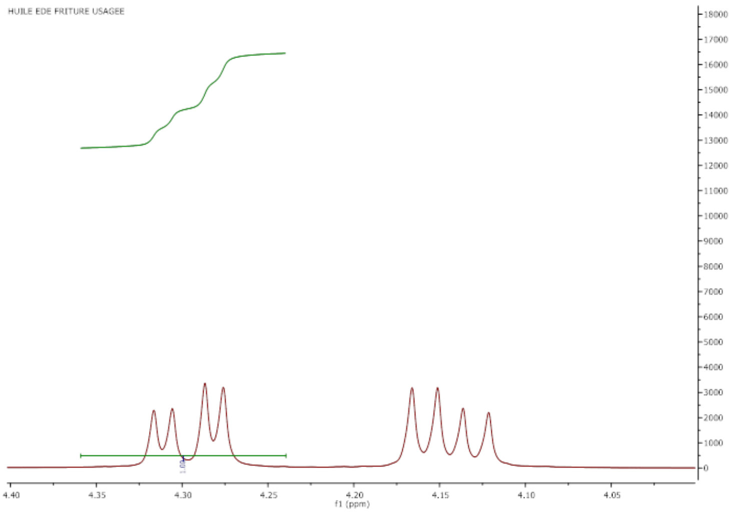

Fig.19.^{1}H NMR spectra of waste cooking oil derived soybean (doublet of doublets) from 4.05–4.40 ppm.

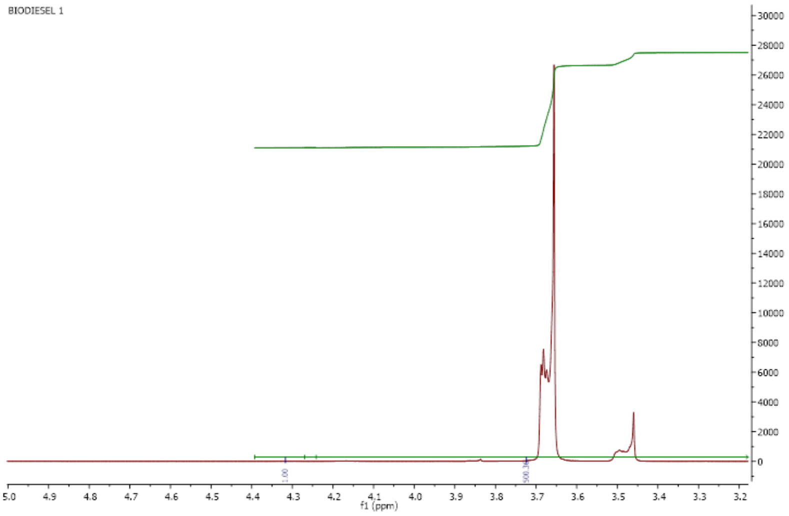

Fig.20.^{1}H NMR spectra of pure methyl ester (singluet) from and 4.15–4.50 ppm.

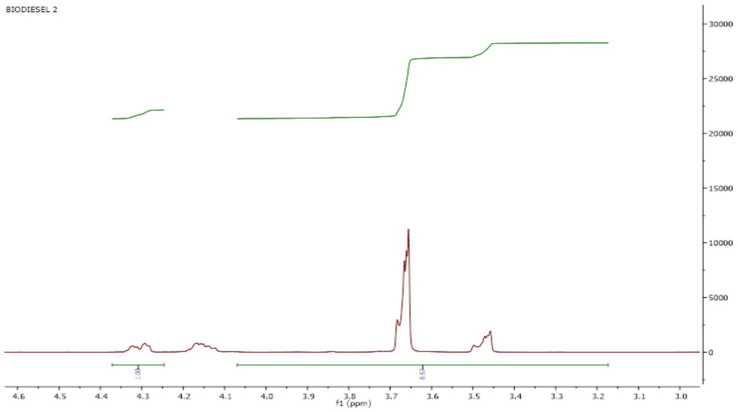

Fig. 21.^{1}H NMR spectra of soybean oil/methyl ester (biodiesel 2) mixture from 3.40–4.50 ppm.

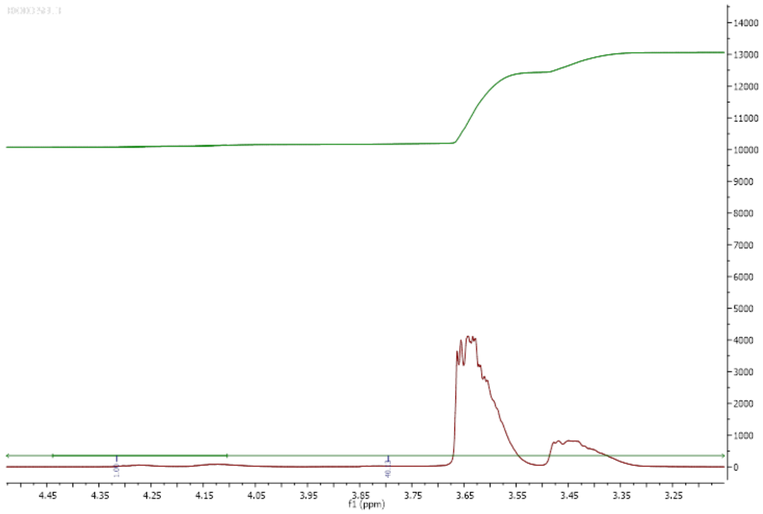

Fig.22.^{1}H NMR spectra of soybean oil/methyl ester (biodiesel 3) mixt