

Biodiesel Production from Industrial Waste Assisted by Ultrasonication

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Abstract

The food industries vegetation and non-vegetarian generates a large amount of waste material that is responsible for the environmental damage. Biodiesel production from waste cooking oil, industrial fat and animal fat provides an alternative energy means of producing liquid fuels from biomass for various uses. Biodiesel production from non-edible feed stock has good alternative option to produce environment friendly fuel economically. Animal fat have

recently increased in popularity as alternatives to vegetable oils in the production of biodiesel. Biodiesel production from waste biomass is not easy because high free fatty acids (FFA) in waste oils are a serious bottleneck for the process of transesterification, so we need to neutralize the feed stock or use the acidic catalyst (H_2SO_4 and H_3PO_4). Acid catalyzed reaction (H_2SO_4) were found to be the most efficient against the feed stock containing 88-90% free fatty acid content at $70^\circ C$ with the alcohol to oil molar ratio 1:5 at 5% catalyst concentration. Transesterification with alkaline homogenous catalysis (KOH and NaOH) is still preferred in industries producing biodiesel against low FFA containing (0.5mgKOH/gm sample) feed stock. Recent developments in heterogeneous catalysts that can be easily recovered, regenerated and reused are promising for future industrial use. Ultrasounds energy helps to improve the liquid-liquid interfacial area through emulsification and reduce the reaction time 5-6 times as compare to the conventional process. Biodiesel is an excellent renewable source of energy because it is biodegradable, non-toxic, environment friendly and has a good combustion emission property and can be blended up to 20% with fossil diesel for its use in many countries. Biodiesel properties were tested according to the American (ASTM D6571) fuel standards.

Keywords: Biodiesel, Industrial Waste, Ultrasonication, Animal fat, FFA.

Introduction

Fossil fuels increase greenhouse gas emissions and cause global warming, the use of alternative resources like bio-fuels are more pronounced every day. The majority of the energy sources are supplied through petrochemical, coal and natural gases. Petrochemical energy is very important in the

world, especially diesel, because this is the main fuel used for industrial and agricultural transportation. Pumps, heavy trucks, city transport buses, locomotives, electric generators, etc. the huge energy demand in the industrialized world and the population problems caused due to the widespread use of fossil fuels, depletion of the world petroleum reserves and increasing environmental concerns has stimulate the search for renewable fuels and made it increasingly necessary to develop alternative sources of energy to resolve these question [1, 2]. Alternative diesel fuels must be acceptable, economically competitive, environmental friendly and easily available. From this view of point, the use of vegetable oil as alternative fuels for diesel engines is a promise in many countries [3]. Animal byproduct production, as part of the meat and poultry processing chain, is huge. For instance, it represents nearly 17 million tons per year only in the European Union [4]. Most of the waste results from over 328 million pigs, sheep, beef, goats and dairy cattle and 6 billion chickens, turkeys and other poultry that are slaughtered every year in Europe [5]. After rendering, materials classified as edible which amount up to 12 million tons, are processed in a variety of food and feed related sectors [6]. The remaining byproducts that are considered inedible have other applications for disposal such as bio-fuels and biodiesel for energy generation [7,8]. Biodiesel also contributes to sustainability by reducing the carbon footprint due to lower CO₂ emission compared to fossil diesel fuel [9].

US production of biodiesel was more than 5.6 million tons in 2019 and came from 91 plants with a capacity of 8.3 million tons per year [10, 11]. Biodiesel is usually blended up to 20% with fossil diesel fuels in most countries due to its complete miscibility and the unnecessary need for engine

modification at such percentage. For instance, a 20% blend is used in the United States while at least a 10% blend is used in China [12]. Blends receive the name B5, B10 or B20 when the biodiesel volume content is 5%, 10% and 20%, respectively. Today, more than 78% of diesel vehicles coming off production lines are approved for up to B20 use [10]. For biodiesel to be blended with normal fossil diesel, it must comply in Europe with EN14214 from the European Committee of Standardization (ISO) and in the US with ASTM D6751 from the American Society for Testing and Materials [8]. Biodiesel can be used in existing diesel engines without the need for substantial modification. Biodiesel has higher oxygen content than conventional diesel and the carbon to hydrogen ratio is also lower. This explains the major advantages of biodiesel such as lower emission of particulate matter, but also a lesser content of sulfur, hydrocarbon and carbon monoxide [13,14]. The major challenge nowadays is the production of environmentally and economically viable biodiesel [12] and the use of animal fat waste could contribute towards achieving this goal. This review is highlighting the latest advances in the available processes for biodiesel production from animal fat waste. To elaborate on the present review, the literature search was performed in Web of Science (WoS) database from 1 January 2010 to 28 February 2020. The terms "biodiesel production," "animal fat" and "transesterification" were used for the survey of published papers.

The most common way to produce biodiesel is by transesterification reactions [15, 16]. In this reaction triglycerides, the main components of vegetable oils, react with alcohol to produce fatty acid mono-alkyl esters and glycerol. The stoichiometry of methanolysis, reaction required

3 mol of methanol and 1 mol of triglyceride to give 3 mol of fatty acid methyl ester and 1 mol of glycerol. However excess alcohol is used to increase the yield of the alkyl esters and allow phase separation from glycerol formed [17, 18]. Several aspects, including the type of temperature, purity of the reactants (mainly water content) and free fatty acid content, have an influence of the transesterification rates. *Jatropha curcas* oil (triglyceride) reacts with alcohol in the presence of a homogenous base catalyst producing biodiesel and glycerol using ultrasonic irradiation. The conventional catalysts used are acid and alkali catalysts depending upon the nature of the oil used for biodiesel production. Choice of the acid and alkali catalysts depends on the free fatty acids (FFAs) content in the oil. FFA should not exceed 3% in oil for transesterification to occur by alkali catalyst [19, 20].

The alkali catalysts are the most commonly used in the biodiesel industry, it processes faster and the reaction conditions are moderated. These catalysts include sodium hydroxide, potassium hydroxide and sodium methoxide. However, sodium methoxide is more expensive than the hydroxides and also more difficult to manipulate since it is a very hygroscopic. Potassium hydroxide has the advantage that it can be neutralized with phosphoric acid after the reaction, resulting in potassium phosphate, which may be used as fertilizer. The utilization of potassium or sodium hydroxide in vegetable oil methanolysis produces soaps by neutralizing the free fatty acid in the oil or by triglyceride saponification. Owing to their polarity, the soaps dissolve into the glycerol phase during the separation stage after the reaction. In this sense, both soap formations decrease the biodiesel yield obtained after the separation and purification stages. In addition, the dissolved soaps increase the methyl

ester solubility in the glycerol, an extra cause of yield loss [21].

The present research work, the transesterification of *Jatropha curcas* oil/ animal fat with methanol, ethanol and various mixtures of methanol/ethanol in the presence of base catalyst, KOH producing biodiesel and glycerol using ultrasonic irradiation [22] was investigated. The applications of ultrasound are those which produce changes through wave propagation [23]. Ultrasound energy may activate various mechanisms to promote the effects but the mechanisms involved are not always known. So far, most investigators agree that there are three phenomena attributed to the effect of ultrasonic irradiation. First, a rapid movement of fluids caused by a variation of sonic pressure causes solvent compression and rarefaction. The second one, by far the most important, is cavitation. Third, there is microstreaming in which a large amount of vibrational energy is confined in small volumes with little heating. Most investigators accept that the formation and collapse of micro bubbles are responsible for most of the significant chemical effects observed [24]. Ultrasounds help improve the liquid-liquid interfacial area through emulsification, which is important for viscous films containing gas-filled bubbles and cavitation bubbles [25]. Gas-filled bubbles within the films, oscillating because of ultrasound and mobilized by acoustic streaming, entrain sonic of the film. Simultaneously, cavitation bubbles spray solvent on the film that covers the pulsing gas bubble. The pulsing action of the gas bubble is therefore disrupted and the liquid is scattered on its surface, leading to highly dispersed emulsions. Very fine ultrasonic emulsions, which are much smaller in size and more stable than those obtained conventionally, greatly improve the interfacial area available

for reaction, increase the effective local concentration of reactive species, and enhance the mass transfer in interfacial region [26]. Therefore it leads to a remarkable increase in reaction rate under phase-transfer conditions. Furthermore, cavitations during sonication produces extreme local conditions and a micro environment with high temperature and high pressure, which may also create active intermediates that, permit the reaction to proceed instantaneously [27].

On the basis of the above concept, transesterification of *Jatropha curcas* oil was performed using low frequency ultrasound which gives excellent biodiesel yield with base catalyst in the presence of methanol, ethanol and various mixtures of methanol/ethanol in a much shorter time than with mechanical stirring. The formation of ethyl esters is environmentally acceptable because unlike methanol, ethanol is produced from renewable resources. Also, ethanol has better solvent properties than methanol for solubility of oil. However, formation of emulsion after the transesterification reaction makes ethanolysis more complicated and impractical [28]. In case of methanolysis the reaction mixture remains in two phases due to the low solubility of oil in methanol. Hence, the mass transfer limitations make the rate of the reaction slower. It was reported that the rate of formation of methyl esters from soybean oil is 15 times slower than the butyl esters [29].

Catalyst for biodiesel production

Catalysts are employed to activate the transesterification process. The synthesized catalyst comprises homogeneous and heterogeneous, heterogeneous nanocatalysts and supercritical fluids (SCFs). These catalysts are effective in stimulating the process to completeness,

although the reaction demands high energy and a cumbersome purification process to obtain the purified end product [30, 31]. The different catalytic processes of transesterification used to produce biodiesel. Enzymatic transesterification has an environmental advantage over the other methods. Immobilized lipase can be used to catalyze transesterification of fat and oil using ultrasonic irradiation for biodiesel Synthesis.

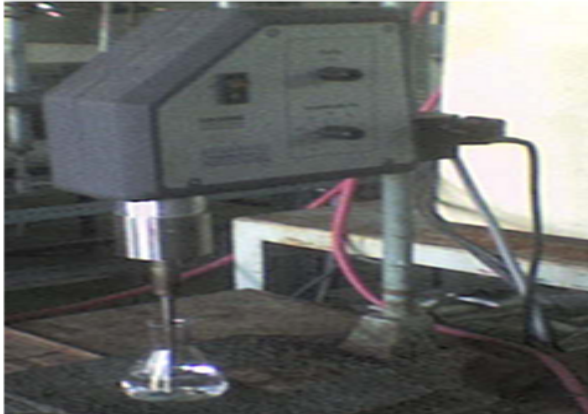
Methods

Reagents and Materials

West animal and vegetable industrial fat and oils are collected from various industries. Anhydrous methanol (purity 99.8%) and KOH (purity 85.1%) were purchased from M/S Ranbaxy. Ethanol (purity 99.8%) was purchased from Sigma-Aldrich. All reagents were analytical grade and standard of fatty acid methyl esters from Fluka.

Apparatus

An ultrasonic procedure UP 200S from Hielscher ultrasonic GmbH was used to perform the transesterification reaction. The ultrasonic processes operate at 200W and 24 kHz frequency. The amplitude and cycle for the reaction were adjustable from 20 to 100% and from 0.1 to 1 cycle per tone, respectively. The titanium sonotrode S₇ with a diameter of 7 mm and length of 100 mm, for samples from 20ml up to 500ml was used to transmit the ultrasound into the liquid. All experiments were performed in an Erlenmeyer type flask, having 50 ml total volume.



Ester preparation By methanolysis

A typical procedure of the production of biodiesel (methyl ester) from industrial waste (animal and fat) involves the base catalyzed transesterification with methanol to give methyl esters. Glycerin is a byproduct in the transesterification reaction. In the case of methanolysis the reaction mixture remains in two phases due to the low solubility of oil in methanol.

KOH was dissolved into methanol and the mixture was transferred to the Erlenmeyer type flask to be subjected to the ultrasound waves. A S7 type sonotrode was submerged up to 25 mm into the solution. The ultrasonic wave cycle and its amplitude as well as the time of the reaction were adjusted by the controller. Amplitude of 60% was set and cycle of 0.3 was set. Under these parameters reactions were carried out for different time durations depending upon the category and concentration of catalyst. After completion of the reaction, the unreacted methanol was evaporated in a rotavapor flask and recovered products were transferred to a separating funnel for phase separation by which the ester mixture was ultimately

isolated from glycerol. The traces of homogenous catalyst in the ester layer were washed with warm water and heterogeneous catalyst removed by filtration and the biodiesel dried over the anhydrous sodium sulphate or under vacuum. At last the conversion was checked by the HPLC method.

Ultrasonic assisted transesterification carried out in the presence of methanol/ ethanol and catalyst (homogeneous/ heterogeneous), keeping the molar ratio of oil/ fat to alcohol 1:5, catalyst concentration 0.75 wt% (homogenous) and 5% (heterogeneous) of oil, ultrasonic amplitude 50% and pulse 0.3 cycle, 6 min and 20 min reaction time homogeneous and heterogeneous, respectively, under atmospheric condition. Ultrasonic mixing has increased the rate of transesterification reaction as compared to mechanical mixing.

By ethanolysis

The preparation of biodiesel (ethyl ester) from industrial west fat (animal and plant) used to catalytic transesterification reaction with ethanol assisted by ultrasonication to give ethyl ester. This process is called ethanolysis. Glycerin is the fatal product in the transesterification reaction and all conditions are the same as the methyl ester formation.

HPLC analysis

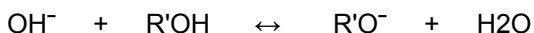
HPLC was used to analyze the purity, conversion and FAME composition of the biodiesel esters sample. The Reverse phase high performance liquid chromatography (RP-HPLC) separates different components according to their polarity. The chromatographic apparatus consisted of a model waters 600 pump with waters 600 controller, waters 2996 photodiode array detector, a nova-pack®, 3.9 X 150 mm column with guard column of dimension 3.9 X 20 mm, both

packed with C18 particle with diameter 4 μm . (all from waters, Milford MA, USA).

HPLC condition: RP-HPLC method flow rate of 1ml/min, an injection volume of 5 μl , a column temperature of 45oC, the UV detection at 215 nm and a 40 min gradient mobile phase 15% H₂O + 85% CH₃OH in 10 min, 100% CH₃OH in 0 min, 60% CH₃OH + 15% hexane + 25% propan-2-ol in 30 min and for the last 10 min system back to initial state 15% H₂O + 85% CH₃OH were used for the separation and determination of the compound produced during the methanolysis of industrial fat in all the experiments.

Mechanism of alkaline catalyzed transesterification reaction

In case of the alkaline transesterification reaction, triglyceride molecules react with the lower alcohol, the active catalyst species is alkoxide anion. In case of methanol, CH₃O⁻ methoxide and in case of ethanol, C₂H₅O⁻, ethoxide anions are formed. This is preliminary step in alkaline catalyzed transesterification reaction



The alkoxide anions formed in the preliminary step attacks the carbonyl atom of the triglyceride molecule to form tetrahedral intermediate in the first step of the reaction. This is a rate determining step of transesterification reaction. Therefore, the rate of the alkaline catalyzed transesterification reaction is determined by the reactivity of the alkoxide anion. Reactivity of methoxide is more than the ethoxide anion. The reason for this is, as the length of the carbon chain increases, nucleophilicity of the alkoxide anion decreases leading to a decrease in the reactivity of alkoxide anion. This causes the slower reaction rate of ethanol than methanol and ultimately less amount of ethyl esters were formed compared to methyl

when mixture of methanol/ethanol was used for transesterification of oil/ fat [32]. Even through the formation of ethyl esters was slow, the overall rate of reaction was fast.

Characterization

Physical and Chemical Characteristics

Kinematic Viscosity, Acid number and oxidation stability. All the physicochemical properties of the biodiesel are given in Table. The kinematic viscosity of biodiesel at 40 °C was determined following ASTM D 445 [33] using a Rheotek AKV 800 automated kinematic viscometer (Poulten Selfe and Lee Ltd., Essex, England). The acid number of biodiesel was determined according to ASTM D 664 [34] using a Brinkman/Metrohm 809 Titrand (Westbury, NY). The oxidation stability of biodiesel was determined as the induction period (IP) according to EN 14112 [35] using a Metrohm 743 Rancimat instrument (Herisau, Switzerland).

Cold flow properties

The cloud point (CP), pour point (PP) and cloud filter plugging point (CFPP) measurements were done as per ASTM standards, D 2500-05 [36], D 97-96a [37] and D 6371-05 [38], respectively. A Lawler model DR-34H automated cold properties analyzer (Lawler Manufacturing Corporation, Edison, NJ) was used to measure the cold flow properties.

Table 1. Physico chemical characterization of biodiesel

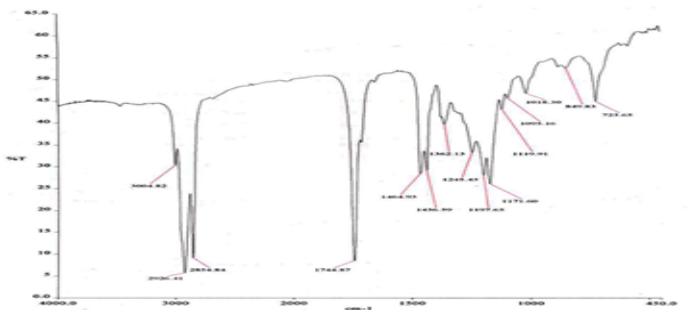
Property	ASTM method	ASTM Specification	BIS Specification
Viscosity 40 °C (mm ² /s)	D 445	1.9-6.0	2.5-6.0

Acid number (mgKOH/g)	D 664	0.5	<0.5
Free glycerin (mass %)	D 6584	0.020	----
Total glycerin (mass %)	D 6584	0.24	----
Oxidation stability (IP, h)	EN 14112	3 minimum	----
Cloud point	D 2500-05	-3 to +12	----
Pour point	D 97-96a	-15 to +10	----
Cetane number	D 613	>47	>51
Cold filter plugging point	D 6371	-4 to -9	----
Density at 15 °C (kg/ml)	D 976	0.575 to 0.900	0.860-0.900
Flash point (°C)	D 93	>100	>120
Carbon residue (wt%)	D 4530	<0.02	<0.02
Copper strip corrosion (3 h at 100 °C)	D 6751	<No. 1.0	1.0

Water content (ppm)	D 95	<500	<500
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FTIR analysis

The sample was analyzed using a FTIR spectrophotometer (Perkin Elmer) in the range 4000-200 cm^{-1} . The IR spectra of biodiesel produced from industrial waste shows in figure. The ester carbonyl group stretching vibration ($\text{C}=\text{O}$) at 1745 cm^{-1} , esteric ($-\text{CO}-\text{OR}$) vibration at 1437-1465 cm^{-1} , and the presence of the $(\text{CH}_2)_n$ group vibration band is seen at 725 cm^{-1} . The presence of a broad band at the 2855-3004 cm^{-1} confirms the low moisture and free fatty acid content in the sample [39]. The IR spectra of esterified and non esterified oil show the pronounced function group, which indicate the presence of alkanes and lesser extent of aromatic and poly aromatic groups. The IR spectra of the oil after the transesterification also show that they contain significant amount of esters. The higher percentage of esters, alkanes and absence of phosphorous and sulfur makes it environment-friendly fuel.



FTIR spectra of Biodiesel

¹H-NMR Spectroscopy.

Proton NMR is used as a complementary spectroscopic technique to quantify the conversion of

vegetable oil into FAMES through the transesterification reaction without the need of the derivatization of test sample. The assignment of chemical shifts of protons for glycerides and for methyl esters are summarized in the scheme below [38, 39]. Integrated intensities of the doublet of methoxy group in methyl esters at 3.7 ppm and a triplet of -methylene groups in all fatty acid derivatives at 2.3 ppm are required for the quantitation of the conversion of vegetable oil into FAMES. Allylic protons also give a multiplet at 2.1 ppm. The terminal methyl group might also be considered, but it appears as a broad superposition of the triplet between 1.1 and 0.8 ppm. Therefore, it is essential to properly resolve proton NMR spectra of biodiesel for an accurate measurement of the intensities of biodiesel peaks [40, 41]. A 200 MHz or higher field NMR spectrometer is preferred for this purpose. A typical example of the use of ^1H NMR spectroscopy in the analysis of oils.

Conclusion

Various groups around the world are pursuing the development of advanced technologies for commercially viable production of biodiesel from a large number of renewable resources and waste materials to supplement and subsequently replace middle distillate fuels. Due to the large diversity of feeds from which biodiesel is being/will be produced, it is not feasible to harmonize its specifications at Global level. Ultrasonication is a fast, easy and environment friendly process for the transesterification of industrial waste using both acid and base catalyst in homogenous and heterogenous phase. It is expedient therefore to establish standards and test methods unique to the biodiesel produced from non-edible resources (waste animal fat and vegetable oil) in India.

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