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PRODUCTION OF BIODIESEL FROM PORK LARD WASTE AND CHARACTERIZATION OF ITS PROPERTIES

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Abstract

The present work explores the potential of pork lard waste as a feedstock for the production of biodiesel. The production involved a unique pathway of reaction using Nitric Acid, an acidic catalyst rather than following the conventional method with a basic/alkali catalyst. The catalyst of choice helped the production to achieve maximum conversion of 92% (9.2g biodiesel/10g fat) by converting the undesired cholesterol in the fat to desired long-chain fatty acids. While achieving a high conversion, the amount of alcohol reagent consumed was recorded to be less than that of the conventional method. Soap, a hindering bi-product formed is also ruled out, unlike in the conventional method. The present work also voids out any hindrance in the yield due to FFA (Free Fatty Acids). The influence of operating conditions such as catalyst loading, alcohol to fat ratio, and reaction time were investigated. The presence of cholesterol in the feedstock and esters in the obtained biodiesel was confirmed through Gas Chromatography analysis. Biodiesel obtained was also tested for the physiochemical properties and was compared to that of the respective standards such as ASTM and IS. The results were found out to be matching to that of the standard range. Thus, from the findings of the present work a conclusion was drawn that the biodiesel produced from pork lard waste could be a promising supplementary fuel to the commercial diesel. Moreover, considering the amounts of reagents used, the explored method is more economically feasible compared to the conventional method where a basic catalyst is utilized. The finding from the current work also offers a new methodology to work with high 'cholesterol-containing' fats to produce biodiesel.

Keywords: Biodiesel, Esterification, Pork lard, ASTM

1. Introduction

Because of rapid industrialization and urbanization, the energy requirement has increased enormously around the world [1]. The transportation industry which contributes to the world's main major economy is also dependent on the energy from petroleum sources [2]. Therefore, there is a continuous exploitation of fossil fuels like coal, oil, and natural gas which will be exhausted in the near future [3]. Although these non-renewable resources are fulfilling the present energy requirements, globally as the world is facing the issue of depletion of these fossil fuels [1-2]. The use of these fossil fuels results in the emission of carbon dioxide into the atmosphere [4]. This has led to an increase in the global temperature and in turn affects the animal and human health [5]. Therefore, there is a need to search for an alternative source of fuel other than the non-renewable resource to meet the energy needs, while overcoming the drawbacks of the non-renewable sources [6]. The various sources such as tidal, solar, nuclear, geothermal, and biomass are considered to be renewable and energy can be produced [7, 8]. The energy source is to be chosen carefully so that it is environment friendly and efficient at the same time. Recent studies have shown that biodiesel obtained from various biomasses is an efficient and effective energy source to replace fossil fuels [5, 9]. Biodiesel is environmentally friendly when compared to fossil fuels [10]. It can be produced from a variety of sources such as edible oils, non-edible oils, animal fats, and other wastes such as waste grease oil [11]. The biodiesel produced from edible oils has high yield because edible oils contain fatty acids less than 1.5% [12].

Considering the issue of food security, there is a growing concern for selecting the feedstock such as edible oils over non-edible oils [13]. Recently, a lot of efforts are being taken for the exploration of biodiesel from animal wastes, refinery waste oil and grease oil, etc. [14]. More sources to produce biodiesel will improve the standard of agriculture and also the economy [15]. Research can be carried out with various feedstocks other than edible products for biodiesel production. This will address the problem of food security around the world [16]. By establishing potential plants as a feedstock for biodiesel, will improve the employment sector especially in the rural areas, and promotes social integration [17]. The government has already taken initiatives to blend biodiesel with commercial diesel for transportation purposes. In this way, the government has supported biodiesel production [18]. This also encourages new researches to work with the development of biodiesel synthesis.

Recent studies have shown that waste animal fats are also being used for producing biodiesel which reduces the overall cost of the process [19]. The production of biodiesel is simple, technically viable, economical and environmentally benign process [20]. When a biodiesel is produced, it should emit less pollutants, have a higher flashpoint and cetane number, less of sulphur compounds, non-toxic and can be blended with commercial diesel to any proportions [6, 21]. Different techniques such as ultrasonic method, microwave technique, pyrolysis, membrane process, reactive distillation and transesterification using the catalyst from various sources can be used for the production of biodiesel [22]. Among these, transesterification using different types of catalysts is considered to be effective because it is technically feasible and economically viable. Various triglycerides present in the feed oil reacts with an alcohol and forms fatty acid esters of another functional group (biodiesel) along with glycerol [23] and this process is called transesterification. However, fatty acids can be directly converted to esters using suitable catalysts and is called

esterification, which is followed in this work. The biodiesel is obtained will be at the top as a lighter layer and the glycerol produced at the bottom as a denser layer. Glycerol is used for preparing cosmetic products [24].

The existing methods of producing biodiesel only follows the mechanism of transesterification of fats. Transesterification consumes a lot of reactant alcohol, increasing the cost of production [25, 26]. Transesterification of fats occurs only when the feedstock fat/oil contains enough triglycerides [27]. Additionally, if the amount of fatty acids present is more, it will form soap as a side product which is not desired during the production of biodiesel [12]. This happens because fatty acids react with basic catalysts to form soap, also fat/oils containing lesser amounts of triglycerides and more of fatty acids or cholesterol will not follow transesterification. Hence, a new methodology has to be introduced to address this issue and the current work proves to be efficient in addressing this issue.

Different types of animal wastes are used for the production of biodiesel. The prominent among them are fish processing waste [28, 29], poultry waste [30, 31], swine waste [32], animal fat waste [33, 34] and pork lard [35]. The above research works showed that a high yield of biodiesel was obtained and the properties of the biodiesel were promising and comparable with the petro-diesel fuel. In countries like Korea and China, a major portion of the animal waste from slaughterhouses includes pork waste and it's reported that a large number of pigs are dying every year due to fever [36, 37]. Therefore, careful handling of this waste and dead animal could be a supplement to the current energy requirements. Moreover, the studies have shown that pork lard consumption could risk in cardiovascular diseases due to the very high composition of cholesterol [38]. Hence, pork lard could be considered as a potential feedstock for the synthesis of biodiesel.

The current study attempts to develop a new methodology to produce biodiesel from pork lard, using ethanol as alcohol and Nitric acid as the catalyst. The catalyst of choice was carefully chosen so to convert cholesterol to long-chain fatty acids meanwhile catalysing to produce biodiesel esters. The methodology developed implicitly takes care of the two steps mentioned while consuming very less amount of alcohol and catalyst.

2. Materials and Methods

Pork lard waste was collected from a pig slaughterhouse in Hebri, Udupi, India. The semi soft white solid fat was taken from the loin and intestinal area. The amount of waste fat ranges anywhere between 500g to 2kg per pig as the size of the slaughtered pig varies. However, an average pig can be 60-70kgs and the waste lard extracted from it averaged at 1kg. The solid fat was dry heated in an open pan and the molten fat/liquid fat is collected and is used for the reaction. The solid remains of the fat are discarded. The molten pork lard was pale yellow in color, with a foul smell and having a density of 0.790 g/cc. Nitric acid (Fisher chemicals) mol wt 63.012g/mol (assay 70%), was used as the catalyst, and Ethanol (CS Analytical) was used as the reagent for the reaction.

2.1. Esterification

The reaction was carried out in a 500 ml three necked round bottomed flask placed on the heating mantle (REMI company). Initially, 10 g of the liquid pork lard was heated up to 50- 60^{o}C in a three necked round bottomed flask with constant stirring.

One of the necks was attached with a thermometer to monitor the temperature and another neck was connected with a reflux condenser. The representation of the experimental setup is shown in Fig. 1.

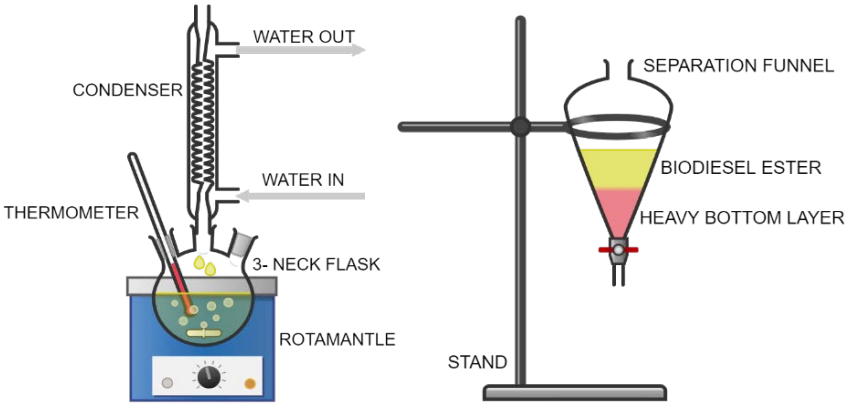

Fig 1. Schematic representation of the experimental setup.

Initially, 5 g of ethanol and 1 g of nitric acid as catalyst was heated up to 50ºC in a beaker. Ethanol and nitric acid reacts to form ethyl nitrate and the ethyl nitrate which is at 50ºC is added through one of the necks to the round bottomed flask containing oil. Then the oil and ethyl nitrate are heated in the flask with constant stirring at 120 rpm for 8 hours. After 8 hours the stirring was stopped and the solution is allowed to cool down to 30-40ºC. Later it is transferred to a decanter and kept overnight for the separation of two layers. After the separation, two separate layers are formed. The top light layer being biodiesel, golden yellow in color and the bottom heavy layer was glycerol, reddish in colour as shown in Fig. 1. The experiments were conducted by varying the amounts of alcohol, catalyst and reaction time.

2.2. Characterization of biodiesel

The conversion of oil to fatty acid ethyl esters was examined by gas chromatography. A gas chromatograph-mass spectroscopy instrument Agilent GC model 7890A and MS model 5975C MSD, equipped with a column DB 5 MS of dimensions (30 m L x 0.25 mm ID x 0.25 um film thickness) was used for the analysis. The mass spectrometer was operated in the electron impact ionization mode at 70 eV in the scan range of 30–700 m/z. Helium was used as the carrier gas at a flow rate of 1 mL/min. The sample was diluted with hexane and 1µL of the sample is injected into the instrument at an inlet temperature of 260ºC. The column initial temperature was at 40 ºC and was raised to 290ºC at 6ºC/min with a total run time of 47 min. The temperature of the transfer line and the ion source was set at a value of 300ºC and 230ºC respectively. Peaks from the analysis were identified by comparing with standards and mass spectra from the NIST libraries 2011. The biodiesel after the reaction process was separated from the glycerol which is present as high density bottom layer. The yield of biodiesel obtained after the reaction was calculated as [39]:

$$\%\ yield = \frac{weight\ of\ biodiesel\ obtained}{weight\ of\ feed\ oil\ taken} \times 100 \quad (1)$$

The physiochemical properties of the pork lard biodiesel were calculated according to standard testing procedures prescribed under ASTM (American Standard for Testing

and Materials), EN (European standard), and IS(Indian Standard). Density, Viscosity, Flash point, Sulphated ash, Water by distillation, and Copper corrosion were measured following the IS 1448 standard procedures. Properties such as Sulfur content, and Carbon residue were calculated using procedures under ASTM D5453, and ISO 10370 (International Organization for standards).

3. Results and Discussion

3.1. Influence of operating parameters

The influence of operating parameters such as catalyst loading, alcohol to oil ratio, temperature, and time of the reaction was analysed and shown in Figs. 2-4. For the experiment, preliminary studies were conducted to establish the range of parameters to be discussed. Suggestions from standard literature following conventional method also aides the study to establish this range of parameters [40, 41].

3.1.1. Effect of catalyst loading

The effect of catalyst loading on the biodiesel yield was studied by varying the amount of catalyst from 0.5 to 2.5 g of nitric acid by maintaining the parameters constant such as alcohol to oil ratio 7:10, temperature 60°C and reaction time 8 hours. It was observed from the results that by increasing the amount of catalyst from 0.5 to 1.5 g, there was a gradual increase in the yield of biodiesel. The maximum biodiesel yield was observed to be 90.9%. There is a possible interaction between the catalyst and the feed to give a high yield [42]. The catalyst, Nitric acid being a good oxidizing agent oxidizes the cholesterol present in the feed to long chain fatty acids. Furthermore, it also converts all the fatty acids to biodiesel without forming any side products such as soap. Soap is formed from FFAs when an alkali catalyst is used. The choice of catalyst as nitric acid rules out this soap formation and converts these FFAs to ester, making the percentage yield even higher. This finding can be seen in the GC results in Tables 1 and 2 as well. Hence, providing maximum conversion.

Beyond 2g of catalyst, there was a drop in the yield as the reaction mixture degrades because the catalyst used is very corrosive and highly oxidizing in nature. At this higher concentration of catalyst, it was observed that the feed oil degraded itself to a viscous mass, reddish in appearance. Due to higher mass transfer resistance, the diffusion of reactants into the reaction system is limited and mixing of reactants becomes poor [43, 44].

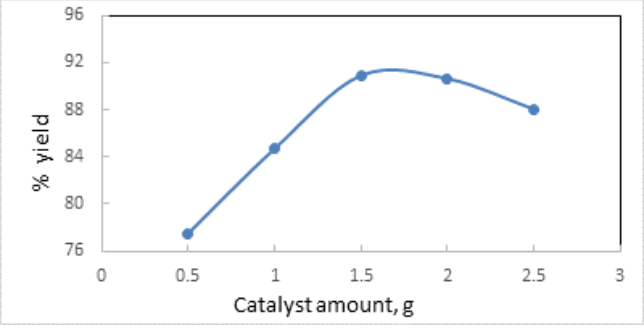

Fig. 2. The effect of catalyst loading on the yield of biodiesel. (alcohol to oil ratio 7:10; temperature 60°C; reaction time 8 hours).

3.1.2. Effect of alcohol to oil ratio

The influence of alcohol to oil was varied in the range of 3:10 to 15:10 by keeping the temperature 60ºC, catalyst concentration 1.5g and reaction time 8 hours. The results showed that by increasing the ethanol to oil concentration, the biodiesel yield increased to the maximum. This is because of the reason that by increasing the alcohol ratio the reaction shifts towards the product side and the yield increases [39, 45, 46]. The yield value is obtained up to 91.5% at 7:10 alcohol to oil ratio. But after increasing the ratio above 7:10, the percent biodiesel yield decreased. This is because of the reason that a formation of glycerol layer takes place, this layer interferes with the alcohol content and separation of this layer is difficult [47, 48]. Also, the glycerol is soluble with the ethanol and shifts the reaction towards the reactant side [42].

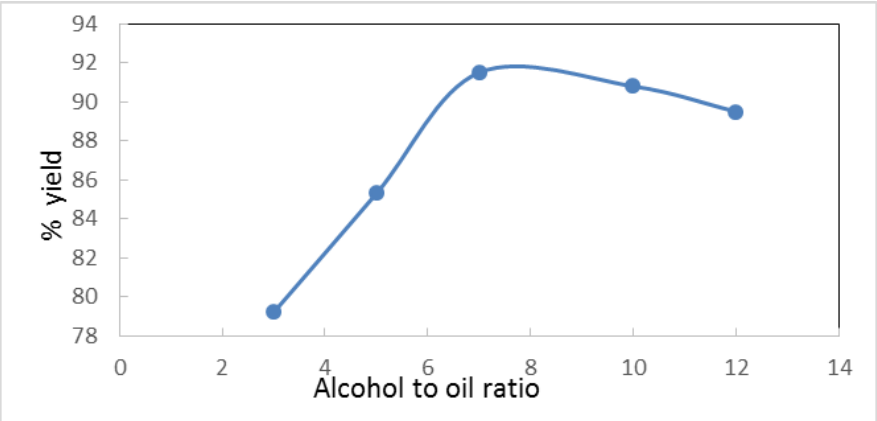

Fig. 3. Effect of alcohol to oil ratio on biodiesel production (temperature 60ºC; catalyst concentration 1.5g; reaction time 8 hours).

3.1.3. Effect of reaction time

The variation of biodiesel yield with respect to time was studied by varying the time from 4 to 12 hours while the other parameters alcohol to oil ratio 7:10, reaction temperature 60ºC and catalyst content as 1.5 g constant.

The biodiesel yield increased up to 8 hours and a yield of 92% was reached. Because the longer reaction time provided enhanced contact between the reactant molecules and the reaction progressed [49]. Afterward the yield of biodiesel remains constant due to the equilibrium which has been reached in the reaction system.

The maximum conversion was noted at the 8th hour of reaction. Once the maximum conversion has reached, the reaction system tends to shift its direction towards the reactants to maintain an equilibrium.

To attain this, few of the esters formed would dissociate to form its parent fatty acid through hydrolysis with side products such as glycerol. However, this decrease in the yield was not more than 1%, and this equilibrium was constant after the eighth hour.

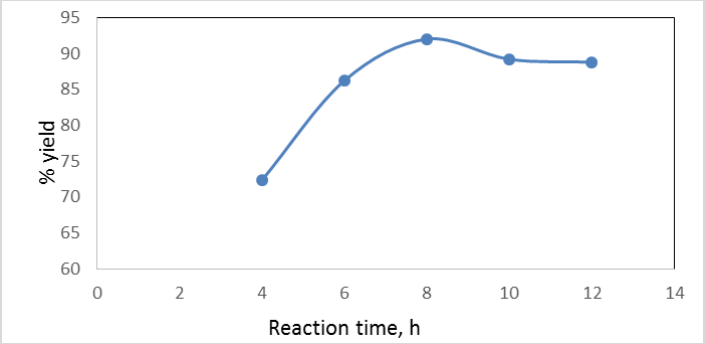

Fig. 4. Influence of reaction time on the reaction (alcohol to oil ratio 7:10; reaction temperature 60°C; catalyst amount 1.5 g).

3.2. Characterization of biodiesel produced

3.2.1. Estimation of various properties of biodiesel

The properties of the produced biodiesel were evaluated as per standard testing procedures and are shown in Table 1. Different properties such as density, viscosity, flash point, sulphur content, carbon residue, water content, acid number, cloud point, pour point and presence of sodium, potassium were determined.

Cloud point and Pour point are properties used to assess the low temperature performance of the fuel especially during winter [50-52]. Flash point is important from the safety perspective of the fuel with respect to its storage and transportation. The fuel with a higher flash point has better storage capacity and the risk of fire hazards are minimum [53-55]. T

he acid value determines the corrosiveness of the fuel as the engine and storage tanks are corroded faster containing fuels with high acid values [50]. Viscosity determines combustion performance and operational difficulties [51, 55, 56]. The sulphur and moisture contents are to be maintained as low as possible [52, 54]. All the measured physiochemical properties were under the range specified by the respective Standard procedures except for carbon residue.

This higher value of carbon residue is because of the reason that the biodiesel esters formed were long chain esters compounds with 14-20 carbon atoms, and this could be seen in the GC results.

The values obtained from biodiesel from pork lard feedstock when compared with the ASTM specifications are within the range and have met the ASTM standards.

3.2.2. Confirmation for the conversion of oil to biodiesel by gas chromatography

The chemical composition of pork lard oil prior to the reaction was found using GCMS and the data are shown in Table 2. It can be observed from the results that various chemical components were present in the feed oil and were determined at different peaks. The average molecular weight of the components was calculated as 201.47. The major components in the pork lard were cholesterol (50%), E,E-

1,9,17-Docasatriene (7.498%), 2,4-Decadienal (8.366%), 2-Decenal, (E) (6.686%) and 2- Hydroperoxide, 1-methylhexyl (5.704%).

Table 1. Various properties of biodiesel produced from pork lard as feedstock.

Sl. No.	Parameter	Units	Method	Result	Standard value
1	Density at 15.0°C	g/mL	IS 1448-P16	0.8994	0.870
2	Kinematic viscosity at -40°C	cSt	IS 1448	12.44	8-12
3	Flash point	°C	IS 1448 – P21	40.0	5 to 65
4	Sulphur content	ppm (m/m)	ASTM D5453-2016	15	15
5	Carbon residue	% (m/m)	ISO 10370	1.30	<0.1
6	Sulphated ash	%	IS 1448 [P:4]:2014	0.004	Max 0.01
7	Water by distillation	%	IS 1448 [P:40]:2014	1.10	1.10
8	Copper corrosion (3h at 50°C)		IS 1448 P-15	1a	Not worse than class 1.
9	Acid number (Inflection end point)	mg KOH/g	ASTM D664 (Method A)-2017a	14.2	5 to 20
10	Cloud point	°C	ASTM D2500-2016	16	<49
11	Pour point	°C	ASTM D97-2017	12	>9
12	Elements by ICP Sodium Potassium	mg/kg mg/kg mg/kg	ASTM D7111-2016	<1 <1	1

Since the feed oil contains a maximum percentage of cholesterol i.e. 50%, it was not ideal for the reaction because of the absence of fatty acids which are essential for ester formation. Therefore, it was treated with nitric acid, a strong oxidizing agent that would breakdown the complex cholesterol structure into long chain fatty acids. The treated feed was subjected to GCMS analysis. The chemical composition of the feed oil after treatment with nitric acid is represented in Table 3. It was observed that the treated feed did not contain any cholesterol and instead contained long chain fatty acids. The peeks obtained at 19.898, 21.633 and 21.799 are long chain fatty acids were contributing a significant sum of 80.502% of feed oil content. This implies that the cholesterol was converted to fatty acids and these fatty acids were responsible for the formation of various esters.

Gas Chromatography (GC) is an efficient method used for confirming the purity of biodiesel in terms of fatty acid esters formed. The technique will quantify how

much oil is getting converted into fatty acid esters [57]. The composition of the final biodiesel product was analysed by GC-MS technique.

The peaks obtained for different fatty acid esters, molecular formula, molecular weight, % content and the retention time are summarized in Table 4. The results shows that various fatty acid esters were obtained at different retention times. The major of these components were (E)-9-Octadecenoic acid ethyl ester (38.572%), Ethyl palmitate Hexadecanoic acid, ethyl ester (21.706%), (Z)-9-Octadecenoic acid ethyl ester (20.927%), Ethyl stearate Octadecanoic acid, ethyl ester (10.424%) and Ethyl (9E)-9-hexadecenoate 9-Hexadecenoic acid, ethyl ester (5.044%). It was found that mainly the conversion of fatty acid n-Hexadecanoic acid, 9-Octadecenoic acid and Octadecanoic acid into fatty acid esters occurred during the formation of biodiesel. It was found that compounds with carbon atoms in the range of C18- C20 were maximum than the compounds with carbon atoms in the range of C16-C17. Esterification reaction is a reaction where esters of corresponding long chain fatty acids are formed. Hence, if the starting fatty acid is 'x', then ethyl ester of 'x' will be the final product. Therefore if the feed contains Octadecanoic acid (C18) **,** Octadecenoic acid ethyl ester (C18+ C2(ethyl group has 2 carbons) = C20) will be the final product. Since most of the feed compounds were in the range of C16-C18, the final ester will have a maximum combination of C18-C20. The compounds with carbon atoms in the range of C18-C20 had an average molecular weight 201.47g/mol. Similar kinds of results were obtained with pork lard as feed oil for biodiesel production [35].

Table 2. Feed oil composition obtained using gas chromatography.

Sl. No.	Feed oil composition	Molecular formula	Molecular weight	Content (%)	Retention time, min
1	Hydroperoxide, 1-methylhexyl	$C_7H_{16}O_2$	132.2	5.704	4.166
2	2-Heptenal, (E)-	$C_7H_{12}O$	112.16	4.510	7.618
3	Nonanal	$C_9H_{18}O$	142.2	2.250	11.408
4	2-Decenal, (E)-	$C_{10}H_{18}O$	154.24	6.686	15.304
5	2,4-Decadienal	$C_{10}H_{16}O$	152.23	8.366	16.629
6	Heptadecane, 2,6,10,14-tetramethyl-	$C_{21}H_{44}$	296.57	1.394	20.581
7	E-14-Hexadecenal	$C_{16}H_{30}O$	238.40	1.565	24.102
8	3-Trifluoroacetoxydodecane	$C_{14}H_{25}F_3O_2$	282.34	2.122	31.856
9	Butanamide	C_4H_9NO	87.12	2.302	32.460
10	Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl)ethyl ester	$C_{19}H_{38}O_4$	330.50	5.577	34.227
11	unknown			1.973	36.608
12	E,E-1,9,17-Docasatriene	$C_{22}H_{40}$	304.6	7.498	36.690
13	RT Cholesterol	$C_{27}H_{46}O$	386.65	50.053	44.521

Table 3. The composition of nitric acid treated feed oil using gas chromatography.

Sl. No.	Feed oil composition	Molecular formula	Molecular weight	Content (%)	Retention time, min
1	Pyridine	C_5H_5N	79.10	6.03	4.268
2	n-Hexadecanoic acid	$C_{16}H_{32}O_2$	256.4	17.28	19.898
3	2-octyl-Cyclohexane	$C_{14}H_{28}$	196.3	3.39	21.575
4	9-Octadecenoic acid, (E)-	$C_{18}H_{34}O_2$	282.4	46.65	21.63
5	Octadecanoic acid	$C_{18}H_{36}O_2$	284.4	16.58	21.77
6	Allyl octadecyl ester	$C_{23}H_{42}$	382.5	5.93	22.34
7	9-Octadecenal, (Z)-	$C_{18}H_{34}O$	266.4	4.15	23.89

Table 4. Fatty acid ester composition using gas chromatography analysis.

Sl. No.	Fatty acid ester composition	Molecular formula	Molecular weight	Fatty acid ester (%)	Retention time, min
1	Dodecanoic acid, ethyl ester	$C_{14}H_{28}O_2$	228.37	0.732	22.477
2	Tetradecanoic acid, ethyl ester	$C_{16}H_{32}O_2$	256.42	1.823	26.231
3	7-Hexadecenoic acid, methyl ester, (Z)-	$C_{17}H_{32}O_2$	268.43	0.772	29.287
4	Ethyl (9E)-9-hexadecenoate 9-Hexadecenoic acid, ethyl ester	$C_{18}H_{34}O_2$	282.4	5.044	29.377
5	Ethyl palmitate Hexadecanoic acid ethyl ester	$C_{18}H_{36}O_2$	284.47	21.706	29.693
6	(Z)-9-Octadecenoic acid ethyl ester	$C_{20}H_{38}O_2$	310.5	20.927	32.410
7	(E)-9-Octadecenoic acid ethyl ester	$C_{20}H_{38}O_2$	310.5	38.572	32.549
8	Ethyl stearate Octadecanoic acid ethyl ester	$C_{20}H_{40}O_2$	312.5	10.424	32.794

4. Conclusions

From the obtained results it can be concluded that pork lard waste is a promising feedstock for the production of biodiesel. The present work suggests new improvisations to the existing method of producing biodiesel by proving that an acidic catalyst can also be used instead of an alkali/Basic.

The catalyst of choice was apt as it oxidised cholesterol in the fat to fatty acids, and avoided the formation of undesired side products such as soap, unlike the conventional methods. The properties of the pork lard measured indicates the suitability of feedstock for biodiesel. The esterification reaction showed that the highest yield obtained was 92%.

The optimum conditions for the biodiesel production were found to be catalyst content as 1.5 g, alcohol to oil ratio 7:10, reaction time 8 hours and reaction temperature 60^{o}C. The fuel properties measured such as density, viscosity, flash point, sulphur content, carbon residue, water content, acid number, cloud point and pour point were in accordance with the respective standards.

The confirmation of different fatty acid ethyl esters were confirmed by gas chromatography. It was analysed that Ethyl palmitate hexadecanoic acid ethyl ester, (Z)-9-Octadecenoic acid ethyl ester, (E)-9-Octadecenoic acid ethyl ester and Ethyl stearate octadecanoic acid ethyl ester were present in major portion in the biodiesel.

Finally, it can be concluded pork lard waste is a potential feedstock for biodiesel production. The production process does not produce any harmful fumes and the side products from the reaction are disposable and non-hazardous. Therefore this process addresses two problems such as using low cost waste for biodiesel and also producing biodiesel which is non-toxic, less hazardous and environment friendly.

Conflict of interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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