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High quality biodiesel production from pork lard by high solvent additive

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ABSTRACT: Large scale biodiesel production by using low cost and abundant feedstock as waste animal fat is becoming more important due to petroleum reserves crisis and adverse environmental problems. This paper has experimentally developed a new method for lard synthesis by using suitable conditions of production with high solvent blending. A polynomial equation was obtained for lard biodiesel yields as a function of synthesis parameters. The validity of the predictive model was confirmed by validation experiments. The suitable combination for high quality biodiesel production from lard was less than 2.0 wt.% catalyst with 10:1 methanol/lard molar ratio and 65.0 wt.% solvent additive. The significant point of this method was to use high solvent blending ratios for lard synthesis to overcome poor solubility problem between highly concentrated free fatty acid pork lard and catalyst. Using this method, gave high yields of biodiesel in short reaction time. The evaluation of the synthesis was made using gas chromatography. The final products fulfilled all of the requirements of ASTM D6751-09 and EN 14214 standards. Overall, this process can be useful for larger scale industrial process with low energy consumption.

KEYWORDS: renewable energy, animal fat, methyl ester, synthesis, statistical analysis

INTRODUCTION

Alternative fuels must be environmentally acceptable and economically competitive. Research on biodiesel (BD) derived from vegetable oils and animal fat is being conducted to alternate this kind of fuels with petroleum based diesel fuel. The waste frying oils are known to be scarce but waste animal fats are more abundant and inexpensive. Animal fat BD has higher cetane number, and it more prone to oxidation than diesel fuel. BD from animal fat contains no aromatic moieties and almost no sulphur. It contains a high oxygen concentration by weight, is biodegradable, and has high lubricant ability. But the use of animal fat BD fuels has not been developed in depth in South Korea.

Several research studies have been carried out on the BD production from pork lard. Temperature and catalyst amount are the most important parameters determining the BD quality[1–7]. Immobilized lipase catalysed BD production from lard gives a BD yield of 87.4 wt.% with 8 wt.% solvent (*n*-hexane) blending[2]. Also BD production yields from animal fat varied from 81.7–88.0 wt.% without solvent blending[3]. BD production from chicken and mutton fats using an acid catalyst resulted in higher yield in comparison to base catalysis. As long time reaction requires high energy consumption, the final product price always increase[4].

Many studies report statistical analysis to optimize the parameters influencing the synthesis process in various vegetable oils[8–15]. But only a few studies to optimize lard synthesis process have been carried out[16,17]. Berrios et al[16] found a regression equation describing the relation between the fatty acid methyl ester (FAME) concentration of BD and the operational variables (catalyst concentration and agitation speed). They reported that for lard synthesis, the most important variable is the catalyst concentration. Huang et al[17] compared two types of catalyst, methanol/oil molar ratio, and reaction time effects to lard methyl ester extraction and reported that the under optimal conditions, BD yield reached to 97.2 wt.% with methanol/oil molar ratio of 5.12 and reaction time of 20 h.

The main objective of this study was to experimentally investigate high quality BD production process from pork lard synthesis by using optimization methods and improve the lard BD properties in a larger scale production with low energy consumption. To achieve this objective, the present work is focused on the analysis of the high blending ratios of solvent during lard synthesis process to reduce chemical reaction time and increase BD yield.

MATERIALS AND METHODS

Materials

Commercial purified pork-lard (hereinafter referred to as "lard") was provided by Samyang (South Korea) from a local market and used without any further purification. Methanol 99.5 wt.%, KOH powder 95 wt.%, and *n*-hexane as a solvent used for the synthesis process were purchased from Samchun pure chemical Co., Ltd. (South Korea).

Biodiesel synthesis using lard as raw material

The experiments were conducted in a laboratory-scale setup, which consisted of a 2000 ml flask and the reaction mixture agitated by a magnetic stirrer at 600 rpm. A sample of 300 ml of lard was placed in a flat-bottom flask. The lard was heated to 55 °C slowly until melted and blended with 0–240 ml (0–80 wt.%) solvent. The reaction temperature for lard BD production varied from 50–60 °C. In another beaker, methanol (considering a methanol/lard molar ratio of: 6:1, 10:1, 14:1, and 18:1) was mixed with 0.48–3.05 wt.% catalyst. This mixture was then added to the melted lard and stirred rigorously for 120 min. The mixture was then transferred to a separator funnel and glycerol was allowed to separate. After draining off the glycerol, BD was washed to remove excess of methanol. Finally, the lard BD was distilled to remove the residual water and solvent.

Analytical methods

Gas chromatography (GC) was used to determine the composition of esters in BD and to characterize pure BD. The lard BD was diluted 1:20 in GC-grade pure acetone in order to achieve the vaporization of the sample at the injector. Reference materials and samples were analysed using an Agilent 6890 GC, 5973 MSD. The GC was equipped with a capillary column DB-5MS (30 m/0.25 μm). Helium was used as the carrier gas at a constant flow rate of 1.2 ml/min. The injector temperature was kept at 280 °C with an electro ionization of 70 eV. The analysis of BD for each reaction mixture was carried out by dissolving 50 μl of diluted sample (BD sample in *n*-hexane) into 50 μl of internal standard solution (concentration = 2 g/l) and 1 μl of this mixture was injected using a cold on column injection part. To determine the FAMEs present in lard BD, a comparable standard FAME mixture from Supelco-37/Component FAME Mix was used. Unidentified esters of lard BD go uncounted and compounds present in minor amounts (less than 0.1% peak area) were not considered further. Korean Institute of Petroleum Management analysed for lard BD properties and all test methods are described in Table 1.

Table 1 Test methods for BD fuel properties determination.

Test items	Unit	Test method
Ester content	wt.%	KS M 2413:2004
Pour point	°C	ASTM D 6749:2007
Flash point (PM)	°C	KS M ISO 2719:2003
Viscosity (40 °C)	mm^2/s	KS M 2014:2004
Carbon residue	wt.%	KS M ISO 10370:2006
Sulphur content	mg/kg	KS M 2027:2005
Sulphated ash	wt.%	KS M ISO 6245:2003
Cold filter plug point	°C	KS M 2411:2006
Density (15 °C)	kg/m^3	KS M ISO 12185:2003
Total glycerin	wt.%	KS M 2412:2004
Methanol content	wt.%	EN 14110:2003
Calorific value	MJ/kg	KS M 2057:2006

Statistical analysis

A factorial design of experiments was applied to determine the influence of the operational conditions of the synthesis process. The DESIGN EXPERT (Version 8.0.4, StatEase, Inc., USA) software was used to perform the regression and graphical analysis of the data obtained. The experimental design applied to this study was a full 2^3 factorial design consisting of 18 experiments. Four central points were added to evaluate the experimental error and six additional star points are encoded as $+\alpha$ and $-\alpha$ (Table 2). Experiments were run at random to minimize errors due to possible systematic trends in the variables. The polynomial model for the yield of lard BD was regressed with respect to the reaction conditions as follows:

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i x_i + \sum_{i=1}^{3} \beta_{ii} x_i^2 + \sum_{i=1}^{3} \sum_{j=i+1}^{3} \beta_{ij} x_i x_j + \varepsilon, \quad (1)$$

where Y is the predicted response variable; β_0, β_i, β_{ii}, and β_{ij} are the intercept, linear, quadratic, and interaction constant regression coefficients of the model, respectively; x_i, x_j ($i = 1 \ldots 3$; $j = 1 \ldots 3$; $i \neq j$)

Table 2 Experimental matrix and lard BD yield results.

EN	Run order	SO (ml)	ME	CA (wt.%)	x_1	x_2	x_3	Y (wt.%)
1	13	200	14	2.5	+1	+1	+1	92.3
2	11	200	6	2.5	+1	+1	−1	98.4
3	5	0	14	1.5	−1	−1	+1	68.5
4	10	0	6	2.5	−1	+1	−1	68.6
5	1	200	14	1.5	+1	−1	+1	88.7
6	2	0	14	2.5	−1	+1	+1	78.1
7	17	200	6	1.5	+1	−1	−1	87.2
8	14	0	6	1.5	−1	−1	−1	64.3
9	8	100	10	2	0	0	0	82.1
10	3	100	10	2	0	0	0	82.9
11	4	100	10	2	0	0	0	82.2
12	15	100	10	2	0	0	0	81.2
13	9	240	10	2	$+\alpha$	0	0	98.2
14	18	0	10	2	$-\alpha$	0	0	74.2
15	12	100	16.8	2	0	$+\alpha$	0	83.2
16	7	100	4.8	2	0	$-\alpha$	0	77.8
17	16	100	10	3.05	0	0	$+\alpha$	87.9
18	6	100	10	0.48	0	0	$-\alpha$	71.7

are independent variables (the reaction conditions) in the form of coded values; ε is the standard error. Response surface 3D plots were developed using the polynomial equation obtained from the regression analysis, holding one of the independent variables at constant values corresponding to the stationary point.

RESULTS AND DISCUSSION

The standard experimental matrix for the factorial design and the results of lard BD yields (Y, wt.%) are shown in Table 2. Here, EN: Experiment number, SO and x_1: Solvent amount, ME and x_2: Methanol/lard molar ratio, CA and x_3: Catalyst concentration. Lard BD yield is defined as the weight percentage of the transesterified final product relative to the weight of lard at the experiment start.

Statistical analysis of the regression model was performed to evaluate the analysis of the variance. ANOVA for the response surface linear model is shown in Table 3. The p-value of the model was smaller than 0.05, which indicated that the model was suitable for use in this experiment. The most statistically significant factor from ANOVA analysis was the solvent amount because the p-value for x_1 factor was $p < 0.0001$. Methanol/lard molar ratio was also an important factor ($p = 0.0114 < 0.05$). Other factors of the model had no statistically significant effects. In this study, the value of the determination coefficient ($R^2 = 0.9425$) indicates that the sample variation of 94.2% for lard BD yields is attributed to the independent variables and 5.8% of the total variations are not explained by the model. A lower value of the coefficient of variation (CV = 3.94%) indicates a better precision and reliability of the experiments carried out. In the regression analysis, the following expression was obtained:

Table 3 ANOVA for the polynomial model.

Source	SS	DF	MS	F-value	p-value	Sig.
Model	1524.99	8	190.62	18.45	0.0001	Yes
x_1	1267.13	3	422.38	40.88	< 0.0001	Yes
x_2	254.37	4	63.59	6.15	0.0114	Yes
x_3	0.10	1	0.10	9.8×10^{-3}	0.9233	
Residual error	92.99	9	10.33			
Lack of fit	92.34	6	15.39	71.03	0.0025	Yes
Pure error	0.65	3	0.22			
Total	1617.98	17				

$R^2 = 0.9425$, CV = 3.94%.

$$Y = 82.9 + 12.34\,x_1 + 1.17\,x_2 + 11.08\,x_3 - 4.12\,x_1x_2 + 0.35\,x_1x_3 - 1.11\,x_2x_3 + 0.5\,x_1^2 - 2.46\,x_2^2 - 4.37\,x_3^2 \quad (2)$$

The linear effect of x_1, interaction effect of x_1x_2, and quadric effect of x_3^2 were the general determining factors of lard BD yield as they had the larger coefficients. The linear effect of x_3 and the interaction effect of x_2x_3 were secondary factors of the response. Other terms of the model had no significant effects on lard BD yield. In this statistical analysis solvent amount has the highest coefficient value among the three independent variables, implying that the yield of lard BD is very influenced by this parameter ($\beta_1 = 12.34 > \beta_2, \beta_3$ and $\beta_{11} = 0.5 > \beta_{22}, \beta_{33}$). But in the other studies[8–15], the most important parameter in the conversion to BD was catalyst concentration.

Fig. 1 shows a plot of experimental values versus predicted for lard BD yields. It is clearly observed that the actual data are in great agreement with the predicted data by the model which proves the reliability of the model.

Fig. 2 shows the response surface 3D plots indicating effects of interaction between parameters on the yield of lard BD. Fig. 2a presented the lard BD yields response surface corresponded solvent amount and methanol/lard molar ratios. Solvent blending with lard is an important parameter as it influences the reaction rate of synthesis significantly and subsequently the yield of lard BD. In the case of using high methanol/lard molar ratio from 10:1 without solvent, lard BD yields were about constantly but increase as solvent concentration lard BD yields decrease. Fig. 2b shows the 3D plot of catalyst concentration and solvent amount on the yield of lard BD. Lard BD yield was low at high catalyst and low solvent concentration

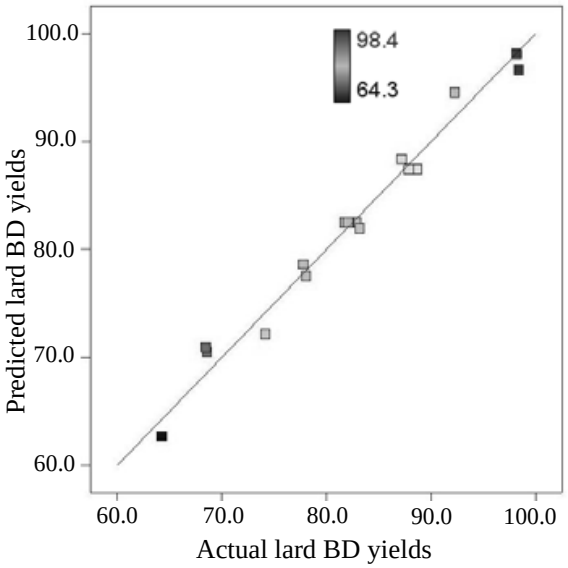

Fig. 1 Experimental versus predicted values.

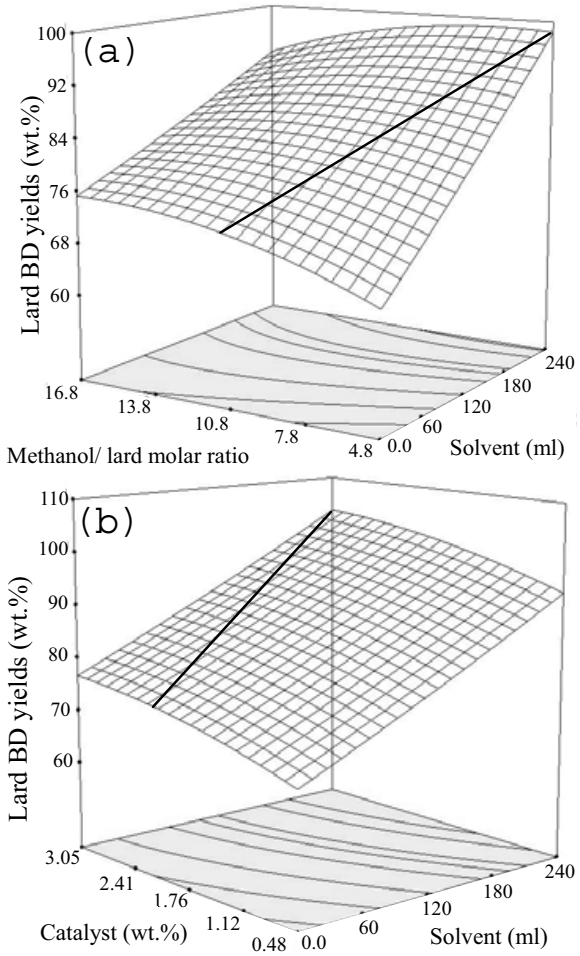

Fig. 2 Response surface plot of lard BD yields. Conditions: (a) catalyst concentration of 2.0 wt.%; (b) methanol/lard molar ratio of 10:1.

due to the poor solubility between lard and catalyst. Although enhanced mutual solubility between lard and catalyst increased as solvent concentration increased, it is observed that the lard BD yield increased steadily when the concentration of catalyst increased until 2.0 wt.%; thereafter was almost constant. Hence the best yield of lard BD could be obtained at 2.0 wt.% of catalyst concentration.

From Fig. 1, it can be seen that the predicted maximum value of lard BD yield was 98.0 wt.%, and the actual experimental value was 98.2 wt.%. Therefore, the most suitable conditions for the process were obtained by actual experiment number-13 (Table 2): methanol/lard molar ratio of 10:1, 240 ml solvent (80.0 wt.% to lard) and catalyst concentration of 2.0 wt %. To evaluate the validity of the model, additional experiments were performed.

One of the variables affecting the BD yields is the reaction temperature. In order to optimize the reaction temperature, samples were prepared by varying the reaction temperatures (50 °C, 55 °C, and 60 °C). Usually the transesterification reaction temperature should be below the boiling point of alcohol in order to prevent the alcohol evaporation. The optimal reaction temperature was found to be 60 °C (Fig. 3). Samples were taken after every 15 min and lard BD yield was measured after purification procedure (see Materials and Methods). Reaction speed was high as the reaction starts and then it decreases after 20 min. It is indicating that, the chemical reaction speed increase rapidly after addition of the methanol and KOH mixture. It can be seen that lard synthesis process optimal reaction time was 90 min even at various temperatures (Fig. 3). The lard BD yield was approximately constant around this time periods. Therefore, the reaction time and temperature in the next all verification experiments were set at 90 min and 60 °C, respectively.

The solvent effect on the lard BD yield with various methanol/lard molar ratios is illustrated in Fig. 4. Lard BD yield increase significantly as solvent concentration increase until 65.0 wt.%, but further increase of solvent concentration tends to slightly decrease lard BD yield due to retarding of the catalyst activity by use of too much solvent. The optimal solvent concentration obtained by statistical analysis was 80.0 wt.%. But, as shown Fig. 3, the optimal solvent amount can be selected as 65.0 wt.%. Solvent

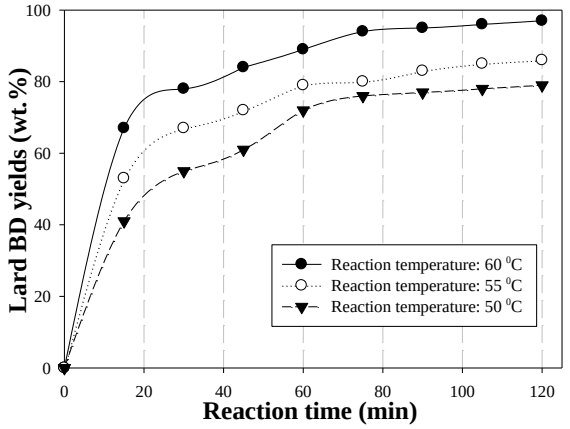

Fig. 3 Lard BD yields versus reaction time and temperature (Conditions: solvent concentration of 65.0 wt.%; methanol/lard molar ratio of 10:1; catalyst concentration of 2.0 wt.%).

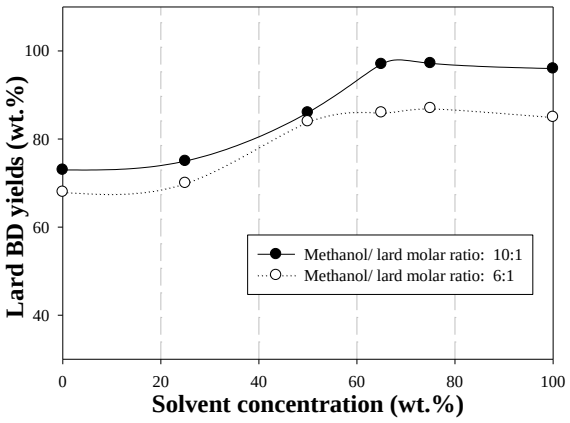

Fig. 4 Effect of solvent on the yield of lard BD (catalyst concentration of 2.0 wt.%).

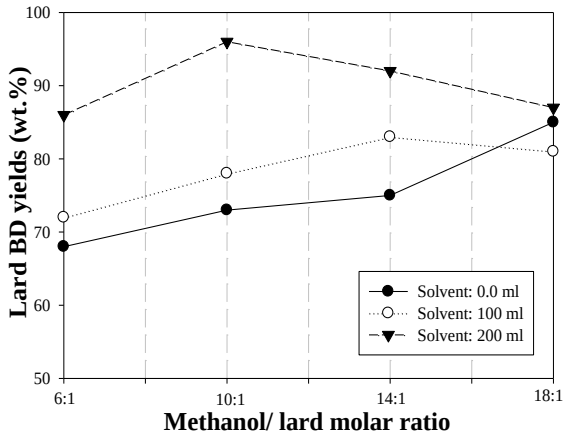

Fig. 5 Effects of methanol and solvent on the yield of lard BD (catalyst concentration of 2.0 wt.%).

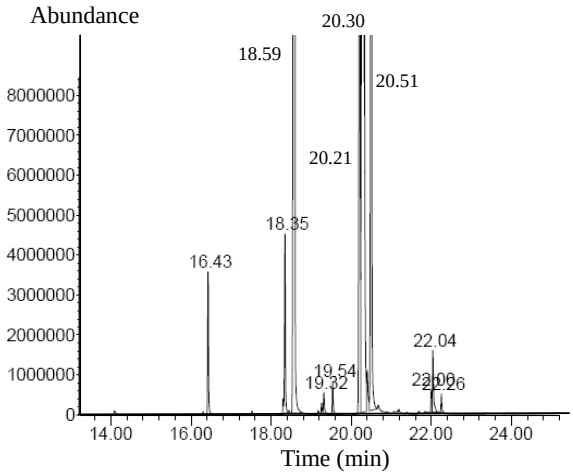

Fig. 6 Chromatogram (GC/MSD) of lard BD.

makes the lard and chemical reactants enable to be miscible. Therefore, this homogeneous single phase reaction leads to fast and complete reaction when compared to conventional reaction.

Fig. 5 illustrates lard BD yields with condition of various methanol/lard molar ratios from 6:1–18:1. The molar ratio of methanol to lard is an important factor that affects the conversion to BD. Stoichiometrically, 3 moles of methanol are required for each mole of triglyceride, but in practice a higher molar ratio is employed in order to drive the reaction towards completion. In the case of lard BD synthesis without solvent, lard BD yield was increased continuously with an increase in the methanol/lard molar ratio. However, the conversion rates were lower than those of lard BD synthesis with solvent. The solvent blending increases the rate of reaction by making the lard soluble in methanol. The highest lard BD yield of 98.0 wt.% was achieved when the molar ratio was 10:1 with high solvent additive of 200 ml.

Fig. 6 shows a chromatogram from the chromatographic analysis with the identification of the peaks of lard BD samples. The standard ten FAMEs which match both the BD and the standard were taken into account. Table 4 gives the composition of lard BD obtained from the suitable production conditions. The methyl ester profile of the mixture showed higher content of palmitic and lower contents of oleic acid and linoleic compared to referenced vegetable BD composition. The increased rate of palmitic indicates that the applicable conversion procedure of lard fat

Table 4 FAMEs and chemical composition of lard BD.

FAMEs	Study result [wt.%]	Ref. 18 [wt.%]	C [wt.%]	H [wt.%]	O [wt.%]
Myristic	1.78	-	1.31	0.21	0.24
Palmitic	29.36	11.0	22.0	3.69	3.66
Palmitoleic	2.39	-	1.8	0.28	0.3
Heptadecanoic	0.45	-	0.33	0.05	0.05
Stearic	12.5	4.0	9.22	1.74	1.53
Oleic	37.85	22.0	28.9	4.58	4.28
Linoleic	13.79	53.0	10.6	1.58	1.57
Eicosanoic	1.05	1.0	0.81	0.12	0.11
Total			75.1	12.3	11.7

occurred during the synthesis in this experiment. The lard BD obtained in the experiment mainly contained seven FAMEs.

The main difference between animal fat and vegetable oil BD is their FAMEs composition. The soybean [18] oil BD has high content of unsaturated fatty acids, mainly $C_{18:1}$ (22.0 wt.%) and $C_{18:2}$ (53.0 wt.%) fatty acids, while lard BD contains large amounts of $C_{16:0}$ (29.36 wt.%), $C_{18:1}$ (37.85 wt.%), and $C_{18:2}$ (13.79 wt.%) fatty acids (Table 4). The analytical method used heptadecanoic acid as an internal standard; however, such methyl ester might exist in animal fats composition which if even in small amounts, would affect the results leading to lower purities. The lard BD analysis showed that the methyl ester profile included 0.45 wt.% of heptadecanoic, which is lower than the standard limit.

All properties of lard BD analysed by Korean Institute of Petroleum Management and predicted cetane number [19] were determined for BD synthesized at suitable condition (Table 5). The properties were compared with ASTM D6751-09 and EN 14214 standards and the final products fulfilled all of the requirements of these standards. The lard BD's most important parameters like viscosity and density met the specifications required by ASTM and EN 14214.

Lard BD is non-flammable and, in contrast to commercial diesel, is non-explosive, with a higher flash point of 130 °C and it makes the storage safer. Lard BD has higher cetane number than diesel fuel and this can help the engine start quicker and run more quietly. The presence of monounsaturated compounds gave the lard BD a higher cetane number. Animal fats are highly saturated, which means that the fat solidifies at a relatively high temperature. Therefore, BD made from animal fat has a high pour point. But result of this study show that lard BD pour point and cold filter plug points meets the limits by ASTM standard. The primary reason for these good agreements is probably that high blending rate of solvent enhanced complete conversion of lard's long chain molecules.

Table 5 Properties of lard BD compared to other standards.

	Result Lard BD	Diesel	ASTM D6751-09	EN 14214
Density (kg/m^3)	837.1	820–860	-	860–900
Viscosity (mm^2/s)	2.491	1.3–5.5	1.9–6.0	3.5–5.0
Cetane number	57.8	30–40	$>$ 47	$>$ 51
Flash point (°C)	130	38–55	$>$ 130	$>$ 120
Pour point (°C)	4.0	$-$15 to 3	$-$15 to 10	-
Cold filter plug point (°C)	1.0	-	$-$3 to 12	-
Calorific value (MJ/kg)	38.8	42.7	-	-
Carbon residue (wt.%)	0.03	-	$<$ 0.05	$<$ 0.3
Sulphur content (mg/kg)	1.00	10	$<$ 0.05	$<$ 10
Sulphated ash (wt.%)	0.002	0.02	$<$ 0.02	$<$ 0.02
Total glycerin (wt.%)	0.038	-	$<$ 0.24	$<$ 0.25
Methanol content (wt.%)	0.09		-	$<$ 0.2

The calorific value of lard BD is about 38.8 MJ/kg, being 9.13% lower than the one of commercial diesel.

CONCLUSIONS

In conclusion, the present study experimentally investigated high quality BD production from lard by using optimization methods and the final product properties fulfilled all of the requirements of ASTM and EN standards. Verification experiments confirmed the validity of the RSM model. Thus the suitable conditions for lard BD production were found to be as follows: solvent amount of 65 wt.%, methanol/lard molar ratio of 10:1 and catalyst concentration as 2.0 wt.%. Experimental study by using RSM analysis was successfully carried out with the maximum lard BD yield of 98.2 wt.%. The most important factor is the solvent amount, which improves mutual solubility between lard and chemical components. Higher cetane number and oxygen concentration are major advantages of lard BD than that of diesel fuel.

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