Jatropha curcas L.: A Non-food Oil Source for Optimized Biodiesel Production

^{1,2}Tahir Mehmood*, ¹Adeela Naseem, ¹Farooq Anwar, ³Mudassir Iqbal and ¹Muhammad Ashraf Shaheen ¹Department of Chemistry, University of Sargodha, 40100 Sargodha, Pakistan.

²Institute of Biochemistry and Biotechnology, University of Veterinary and Animal Sciences,

54000, Lahore, Pakistan.

³Department of Chemistry, School of Natural Sciences, National University of Sciences and Technology

(NUST), H-12, Islamabad, 44000-Pakistan.

tahiruosbiochem@yahoo.com*

(Received on 6th February 2018, accepted in revised form 7th November 2018)

Summary: Response Surface Methodology (RSM) was applied based on central composite rotatable design (CCRD) to optimize transesterification reaction parameters for obtaining optimal biodiesel yield from *Jatropha curcas* oil. Transesterification variables such as: catalyst concentration (CC) (0.16-2%), reaction temperature (RT) (40-65°C), molar ratio of oil and methanol (0.95-11.5), and reaction time (30-140 min) were optimized via RSM involving 2⁴ full factorial CCRD design. The molar ratio of methanol to oil and RT were the most significant (p< 0.5) factors affecting the yield of *Jatropha curcas* oil methyl esters (JOMEs). A linear relationship was recorded between the observed and predicted values (R² = 0.766). Using multiple regression analysis, a quadratic polynomial equation was constructed to predict JOMEs yield. The quadratic term of molar ratio as howed a significant impact on the JOMEs yield. The interaction terms of molar ratio and CC with reaction time exhibited positive impact on ester yield (p< 0.05). The optimum reaction conditions including CH₃OH to oil ratio of 6:1, 1.0 % CC, 60 °C RT and 60 min reaction time offered the highest yield of JOMEs (99.90%). JOMEs were analytically characterized using GLC and FTIR. The fuel properties of produced JOMEs were in accordance to ASTM D6751 and EN 14214 standards.

Key Words: Jatropha oil, Methanolysis, Optimization GLC, FTIR, Fuel Properties.

Introduction

The worldwide bouncy panorama based on fossil fuels is characterized by continuous headway of energy demand, causing scarceness of resources, nationwide sanctuary issues and the typical weather allegations due to the persistent combustion of the vestige fuels [1]. With the advent of life, man is always in search to hunt the new ways of development to cope with ever-growing demand of energy, as energy is a vital driver for civilization. Eighty-five percent of the essential energy demands i.e. industries, agriculture and other human needs are fulfilled via fossil fuels, out of which seventy-five percent is utilized for the transportation. The nonrenewable energy resources are progressively decreasing from the nature [2] so exploration of alternative and renewable energy resources has to play a vital role to meet the challenges of future developments and save environment as well. The scope of bio-products is becoming the latest trend to meet the energy demands which are crafting a great deal of monetary, assertive and biological concerns.

Predominantly, biodiesel is sustainable and recyclable source of energy. Hence it is conquering an ample thoughtfulness to be the most suitable substitute of petro-diesel [3]. Biodiesel can be produced by using edible and non-edible oils, animal fats, dissipated cooking oils, wood and wood waste, algae and other microorganisms etc. [4, 5]. Transesterification processes is employed for converting triglycerides into fatty acid methyl esters (FAMEs)/biodiesel with short chains alcohol (methanol and ethanol) in the presence of acidic or basic catalysts [6]. Glycerol is obtained as a by-product in this reaction.

In order to cope with the challenges of fuel versus fuel dilemma and transesterification process cost-effectiveness, currently there is greater focus on production of biodiesel using some non-edible oils, animal fats, waste frying oils, acid oils, wood and wood waste, algae and other microorganisms etc. technologically [12]. The most advanced transesterification reactions use a base catalyzed system nowadays. Stoichiometrically, the chemical equation needs one mole of triglyceride and three moles of alcohol to form three moles of fatty acid alkyl esters (biodiesel) and one mole of glycerol (as by-product) in the presence of a strong base [13]. The by-product (glycerol) can be utilized in the pharmaceutical industry along with many other applications. Moreover, as the reaction is reversible so an excess of alcohol is used to shift the reaction equilibrium to the product's side. The most important factors affecting transesterification are molar ratio of oil to alcohol, concentration of catalyst and its type, reaction time, reaction temperature, mixing intensities, contents of free fatty acids (FFAs), moisture and impurities in the feedstock oils and fats [14]. Base-catalyzed transesterification is much faster than acid-catalyzed transesterification and is most often used commercially. Generally, alkaline catalysts (NaOH) are used for oils with low FFA content to catalyze the transesterification reaction, nevertheless, in most of the feedstocks, there is need to optimize process variables for gaining best yields of biodiesel[14,15].

Response surface methodology (RSM) is a valuable statistical tool that can be employed to optimize the process variables in the production of biodiesel utilizing various edible and non-edible triglycerides via transesterification process [15, 16]. The RSM technique has been employed to deliver a rapid mean to comprehend important parameters of the under study process, thereby saving the time and preserving noteworthy facts and fig of each constituent. It takes account of factorial design and regression analysis that assist in calculating important parameters and make a plot of these simultaneously beside each other to estimate the collaboration and their effects on per capita instantaneously [17].

The seeds of *Jatropha curcas* L. contain viscid oil, not so suitable for cooking due to presence of lethal fatty acids. On the other hand, it is reported that this oil has long being intricate in medicine and as an insecticide [18], grain protectant [19], a molluscicides [20] and in cosmetics [21]. The oil from *J. curcas* seeds is helpful with rashes and parasitic skin diseases [22]. When the oil is mixed with benzyl benzoate, it becomes effective against scabies and dermatitis [23]. The oil from the seeds can also be applied to soothe rheumatic pain and it may cause premature abortions [24].

In Pakistan, J. curcas has been introduced as a non-conventional oilseed crop on experimental basis. The yield of J. curcas oil is relatively better as compared to other conventional vegetable oils. Due to non-edible nature, alternatively, this potential oil can be used for the production of biodiesel, however no systematic RSM based optimized transesterification has yet been reported in this regard. This study was aimed to optimize the transesterification reaction parameters/variables by using RSM and to develop a comprehensive protocol for obtaining best biodiesel yield from indigenous J. curcas seed oil. ASTM and European biodiesel standard were used as a reference for evaluation of the quality and fuel properties of the biodiesel produced.

Experimental

Collection of Sample

The seeds of Jatropha *(Jatropha curcas)* were procured from National Agriculture Research Center, Islamabad (NARC).

Pretreatment of Jatropha seeds

Firstly, seeds were deshelled and made clean of dirt and any other impurities. The dried seeds were then crushed into small particles using grinder. The ground material was packed in polythene bags.

Oil extraction

100 g of ground seeds were filled a thimble made from filter paper and placed in a Soxhlet apparatus fitted with a 500 mL round bottom flask. The extraction was carried with n-hexane on water bath for 6 hours. The excess solvent was evaporated from the extracted oil under vacuum in a rotary evaporator (Heidolph HB digital, LABOROTA, 4001-efficient). The percentage oil yield was estimated by using the following formula:

% oil yield = Grams of oil recovered oil / Grams of sample taken $\times 100$

Preliminary Assessment for Jatropha Oil

The physicochemical properties such as specific gravity, refractive index, free fatty acids, acid value, iodine value, peroxide value, and saponification number of Jatropha oil were made following standard methods of AOCS (AOCS, 1997).

Experimental Design of Transesterification using Response Surface Methodology (RSM)

Optimization of process factors/ variables such as molar ratio (A), catalyst concentration (B), time (C) and temperature (D), for transesterification of Jatropha methyl esters was made via response surface methodology using CCRD. The levels of the process factors are: A (0.95:1-11.5:1), B (0.16-2%), C (30-140min.) and D (40-65°C). A five-level, fourfactorial CCRD was employed with total 30 runs. Second order multiple regression model was employed to optimize the biodiesel yield on the basis of data obtained from CCRD. Overall thirty experimental runs were made $(2^k + 2k + 6)$, in which 'k' represents the number of autonomous variables. Sixteen factorial and eight axial experimental runs were improved with 6 rotations at central axis to estimate the pure inaccuracy.

Experimental Procedure for Transesterification

Optimization of Reaction Parameters

$$Y = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ij} X_i^2 + \sum_{i>i}^{k} \sum_{j=1}^{k} b_{ij} X_i X_j$$

Various reaction parameters such as molar ratio of oil to methanol, catalyst concentration, reaction time and temperature were optimized to get highest yield and good quality biodiesel with the help of central composite response surface design (CCRD). A quadratic polynomial (second order) was used to predict the response as a function of independent variables and their interactions and a second-order polynomial equation [25] was used as shown below in the equation:

Pretreatment of Jatropha Oil

The crude jatropha oil was first treated with 1% sulfuric acid to minimize the FFAs up to 1% prior to methanolysis. Acid-catalyzed esterification of crude oil involved the transformation of FFAs into esters in the existence of acid catalyst i.e. sulfuric acid along with methyl alcohol. FFAs of the reacting oil were checked at regular intervals of 25-30 minutes, when the FFAs reduced up to 1% the reaction was stopped. Excess of methanol was evaporated in rotary evaporator. Esterified oil, with FFAs less than 1%, was further subjected to base catalyzed transesterification reaction.

Transesterification Reaction

To carry out the base catalyzed transesterification reaction a 2 liter three necked round bottom laboratory scale biodiesel reactor equipped with a thermometer, magnetic stirrer, a condenser, and fitted on a hot plate set on 600 rpm was used. 100 g of pretreated jatropha oil was fed

into the three necked flask. Calculated amounts of NaOH and CH₃OH were taken in a beaker and shaken well to dissolve the contents. Then this solution was introduced into oil sample and stirred well. The resulting mixture was agitated at 600 rpm as set for the design. On the completion of reaction, the contents were permitted to cool down at ordinary temperature. The reaction scheme for the synthesis of biodiesel is shown below (Scheme 1)

Separation and Purification of Biodiesel

The cooled mixture was poured into a separating funnel and left for 8-10 hours for separation of biodiesel layer from by product glycerol. The separated layers consisted of upper phase biodiesel (jatropha methyl ester) and lower phase of glycerol, some methanol and unused catalyst, in some cases soap formation also noted. Then separation of the two phases was done, and the upper layer of jatropha methyl ester was purified by successive washes with distilled water. Then anhydrous sodium sulfate was used to remove any moisture present in the methyl ester followed by filtration. At last purified jatropha methyl ester (biodiesel) was obtained. The percentage yield of biodiesel produced was calculated by following formula:

Percentage yield of fatty acid methyl esters = grams of methyl ester produced/ grams of oil used in reaction $\times 100$

FT-IR Monitoring of Jatropha Methyl Esters (JMEs)

The purified JMEs were analyzed by Fourier Transform Infra-Red (FT-IR) with a DLATGS (Deuterated Triglycin Sulfate Doped with L-Alanine) detector was used and measurements were performed in dry atmosphere 278 (18 \pm 0.5 °C). The empty accessories were used to obtain the background spectrum. The approximate total time required for spectral collection was 5 min. All spectra were recorded with the range of 4000–600 cm⁻¹. Analysis was carried out in triplicates.



Scheme-1: Synthesis of biodiesel.

Fuel characteristics of Jatropha biodiesel

The fuel properties of the biodiesel produced including specific gravity (kg/L), cetane number, oxidative stability (h), kinematic viscosity (mm²/s, 40°C), lubricity (HFRR, μ m), cloud point (°C), pour point (°C), flash point (°F), cold filter plugging point (°C), copper strip corrosion (100°C, 3h), sulfur content (ppm), distillation temperature (°C), bottom sediments & water (B.S&W) (% vol.), free glycerin (%), acid value (mg KOH/g) and ash content (%) were analyzed according to ASTM methods. Triplicate measurements of each parameter were made which were then presented as mean ± SD.

Statistical Analysis

Analysis of Variance (ANOVA) was done with the help of Minitab 6 software for response surface, contour and diagnostic checks. The mathematical model for experimental design was chosen based on different tests such as ANOVA and high value of \mathbb{R}^2 . The model was only thought to be acceptable if ANOVA reached the maximum statistical significance, with F-values within 95% of level of confidence and *p*-values <0.05. Validity and significance of different parameters was evaluated using F-test and t-test. Validity of model and best possible value of the variables was checked through diagnostics plots like normal probability plot of Table-2: Experimental data for second-order CCRD. residuals, predicted versus actual residuals, residuals versus variables plot, contour and 3D-surface plots.

Results and Discussion

The physicochemical analysis of jatropha oil includes the investigation of the following properties (Table-1).

Table-1: Physicochemical properties of *Jatropha curcas* seed oil.

Parameters	Values
Yield of Triglyceride (%)	46.00 ± 1.40
Density (g/cm ³)	0.92 ± 0.103
Specific Gravity (g/cm ³)	0.91 ± 0.02
Refractive Index (25 °C)	$\textbf{1.477} \pm \textbf{0.020}$
Free Fatty Acid (%)	11.38 ± 0.09
Acid Value (mg KOH/g)	22.76 ± 0.18
Iodine Value (g I ₂ /100g)	177.63 ± 0.41
Peroxide Value (m mol/kg)	11.50 ± 0.08
Saponification Value (mg KOH/g)	195.41 ± 2.86
Values are mean + SD	

Process optimization using central composite rotatable design for manufacturing the biodiesel from Jatropha curcas seed triglyceride

Optimization of process factors was followed with the aid of CCRD. A five-level, four-factorial CCRD was employed with total 30 runs. Experimental design of four independent parameters at five levels, methanol/oil molar ratio, temperature, catalyst concentration and time of reaction is given in Table-2.

Sr.no	Molar ratio	Catalyst Conc. %	Time (min)	Temp(°C)	Experimental yield %	Predicted yield%	Residual values
1.	3.00	0.50	60.00	40	57.08	60.79	-3.71
2.	3.00	0.50	120.00	50	89.98	90.63	-0.65
3.	3.00	1.50	60.00	65	57.43	52.90	4.53
4.	3.00	1.50	120.00	60	90.14	91.42	-1.28
5.	9.00	0.50	60.00	55	94.31	92.73	1.58
6.	9.00	0.50	120.00	50	78.13	82.36	-4.23
7.	9.00	1.50	60.00	50	93.71	92.76	0.95
8.	9.00	1.50	120.00	55	95.08	91.07	4.01
9.	6.00	1.00	30.00	50	71.60	73.45	-1.85
10.	6.00	1.00	140.00	60	98.55	97.13	1.42
11.	6.00	0.16	90.00	55	89.17	84.86	4.31
12.	6.00	1.50	90.00	60	80.83	85.56	-4.73
13.	0.95	1.00	90.00	60	65.61	64.81	0.80
14.	11.05	1.00	90.00	55	90.14	91.37	-1.23
15.	6.00	1.00	90.00	55	96.50	97.87	-1.37
16.	6.00	1.00	90.00	50	95.42	96.87	-1.45
17.	6.00	1.00	90.00	55	97.00	97.87	-0.87
18.	6.00	1.00	90.00	65	98.89	99.07	-0.18
19.	6.00	1.00	60.00	60	99.10	99.87	0.03
20.	6.00	1.00	90.00	55	98.95	99.01	-0.06
21.	11.05	1.50	60.00	50	75.24	76.99	-1.75
22.	6.00	0.5	60.00	60	87.1	86.76	0.34
23.	9.00	1.00	120.00	50	93.4	92.65	0.75
24.	6.00	1.50	60.00	50	85.2	84.18	1.02
25.	9.00	1.00	90.00	55	87.9	89.69	-1.79
26.	11.05	0.50	60.00	50	74.1	76.00	-1.9
27.	6.00	1.50	140.00	50	77.3	75.94	1.36
28.	9.00	1.00	120.00	65	93.7	92.65	1.05
29.	6.00	0.50	60.00	50	79.8	80.76	-0.96
30.	9.00	2.00	90.00	40	73.2	73.37	-0.17

follows:

 $Y = -384 + 13.1 \text{ A} + 140 \text{ B} + 1.56 \text{ C} + 9.64 \text{ D} - 0.96 \text{ AB} - 0.0703 \text{ AC} + 0.101 \text{ AD} - 0.182 \text{ BC} - 1.71 \text{ BD} - 0.0015 \text{ CD} - 0.775 \text{ A}^2 - 12.8 \text{ B}^2 - 0.00375 \text{ C}^2 - 0.0688 \text{ D}^2$

where, $\mathbf{Y} =$ dependent or response variable (biodiesel yield)

A, B, C, D = independent variables (molar ratio, catalyst concentration, time and temperature of reaction, respectively).

Table-3 depicts the statement for ANOVA to fit quadratic model by least square method. The F-value = 3.52 with *p*-value = 0.011 shows the significance of the built-in second order quadratic model. The evaluation of dependability of built-in model is carried out by determining the adjusted coefficients (\mathbb{R}^2). The value of adjusted coefficient is noted to be 0.548 which depicts that % of unpredictability (55%) is due to experimental variables while only 45% due to other uncontrolled reasons. The value of $\mathbb{R}^2 = 0.766$ agrees that built-in model may be applied for estimation with realistic precision.

Table-3: ANOVA by least square method for second order quadratic model.

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F-values	P-values
Regression	14	3286.24	234.73	3.52	0.011
Residual Error	15	1001.54	66.77		
Total	29	4287.78			

Table-4 presents the mathematical worth of the process factors of the recommended model for response variable (biodiesel yield %) as evaluated using T Statistics along with their corresponding pvalues. The p-values less than 0.05 of variables depict the significance of variable terms related to it. To augment the importance, cross matching is done of all variables and low probability displays fine communication strength of cross matched variables. The p-values of AC depict a noteworthy role in recommended model. Values lower than 0.05 are highly significant. In this study AC and A² are significant terms.

Table-4: Parameters estimated by Least Square Fit and p-values for their significance.

Source	Estimated Co-efficient	Standard Error	T Statistic	P-value
Intercept	-384.2	288.8	-1.33	0.203
Α	13.07	13.11	1.00	0.335
В	139.54	92.67	1.51	0.153
С	1.5642	0.8692	1.80	0.092
D	9.638	8.512	1.13	0.275
AB	-0.963	2.256	-0.43	0.676
AC	-0.07035	0.02886	-2.44	0.028
AD	0.1007	0.2002	0.50	0.622
BC	-0.1817	0.2165	-0.84	0.415
BD	-1.710	1.266	-1.35	0.197
CD	-0.00151	0.01358	-0.11	0.913
A^2	-0.7754	0.2675	-2.90	0.011
B ²	-12.788	9.841	-1.30	0.213
C^2	-0.003748	0.002192	-1.71	0.108
\mathbf{D}^2	-0.06885	0.06470	-1.06	0.304

Significant (p-value<0.05)

Normal probability plot is designed to verify the ordinary percentage probability of the residuals. Normality plot is illustrated in Fig. 1. As the plot is displaying that there is a straight line and there exist no suspicion in the normal probability of the residuals or responses found. And also the analysis of variance is giving a powerful and beneficial explanation of the probability of residuals. The residuals are required to lie in the range of probability for the authenticity of ANOVA results.



Fig. 1: Plot for normal versus residuals values.

A plot of predicted yield versus experimental yield is designed to display the responses actually found and those expected from the model. This plot is shown in Fig. 2. It is obvious from the plot that both the parameters are parallel to each other, contained by the sequences of working conditions with a high value of $R^2 = 0.766$. It is evident from the fig. that most of the responses lay between 85-99 % other lie in the perimeter of 60-80 %.





Fig. 2: Plot for predicted versus Experimental Yield.

The residual plots of residuals versus process factors are plotted to analyze the influence of individual factors. These plots are presented in Fig. 3 (A-D) In Fig. 3 (A) relationship between molar ratio and residuals, is exposed in which large amount of residuals is observed at 9 but highest yield is observed at 6. Fig. (B) describes that maximum yield of biodiesel is attained at 1.0% catalyst concentration, although a large amount of residuals is present at 0.5% catalyst concentration however the low percentage yield is observed. Fig. 3 (C) also shows an arbitrary effect of time on residuals, huge number of residuals is observed at 60 minutes but maximum yield of the biodiesel is observed both at 60 and 90 minutes. Temperature has influence on the residuals as is obvious from the Fig. 3 (D) that highest residuals are found at 55°C, 50°C and 90°C. On the other hand, 60°C is evident to give maximum biodiesel yield.

To present the regression equation for adjusting the process variables graphically, contour and 3-dimentional surface plots are designed. These plots are designed to display an association and influence of two independent factors on each other. They also help in evaluating their ideal levels maintaining the other factors at constant levels. Fig. 4 shows contour plots. Fig. 4 (A) displays a direct relation between these temperature and catalyst concentration as the temperature proliferates, biodiesel yield also rises with increasing catalyst concentration but to a definite limit after which further rise pull down the yield of biodiesel. Fig. 4 (B) also portrays the direct relation of catalyst concentration and molar ratio on the biodiesel yield. Fig. 4 (C) revealed the insignificant relation of catalyst concentration with time. Fig. 4 (D), Fig. 4 (E) and Fig. 4 (F) designates no interaction between temperature and molar ratio, temperature and time, and molar ratio and time correspondingly.









Fig. 4: Contour plots for the renovation of Jatropha triglyceride into biodiesel.





Fig. 5: Surface plots for plot for the renovation of Jatropha triglyceride to methyl ester.

Similar interactions are displayed in Fig. 5 (A-F) shows specific three dimensional-surface plots between different variables. The above discussion proves greater impact of molar ratio on yield of biodiesel as well as process variables which is also apparent through the ANOVA that depicts molar ratio is more significant.

All the interpretations made above accomplish that most optimal factors to get highest methyl ester yield (99.90%) are 6.00 MeOH to triglyceride molar ratio, 1.00% catalyst concentration and 60°C reaction temperature for 60 minutes.

FT-IR analysis of Jatropha curcas methyl esters (JMEs)

Analysis of JMEs was done by FT-IR (Fig. 6). The IR band positioned at 1452 cm^{-1} indicates the section for CH₃ irregular bending and region of 2675.27-3001.24 cm⁻¹ indicates for O-CH₃ stretching. The region 1753.29 cm⁻¹ displays the presence for C=O stretch that validates it is an ester [26] and

2800-2900cm⁻¹ region for indication of symmetric CH₂ stretching and the irregular CH₃ and CH₂ stretching. The presence C-H specifies predominance of properties such as pour and cloud points that affect the enactment of biodiesel during cold weather in engine on ignition. However, the occurrence of ample groups with carbon to carbon double bonds (C = C, = C-C) which can cause the fuel to remain in liquid form but during long storage there is chance of possible oxidation. The obtained results show concurrency with Rashid *et al.* and Oliveira *et al.* [27, 28].

Quantitative analysis of Jatropha oil methyl ester content (JMEs)

JMEs were characterized by GLC. Identification of JMEs was accomplished by comparing the retention times of the unknown with those of pure FAMEs standards. Quantitative analysis was made by comparing the peak area of the separated compounds with those of pure standards.



Fig. 6: FT-IR spectrum of jatropha methyl esters.



Fig. 7: GLC profile of Jatropha curcas methyl esters.

Table-5 shows that J. *curcas* methyl esters comprises following fatty acids: caproic (2.5%), myristic (9.9%), palmitic (18.5%), palmitoleic (7.8%), steric (9.0%), Oleic (18.8%), elaidic (1.4%), linoleic (20.2%), linolenic (2.1%), and behenic (2.4%) acids.

Table-5: Fatty acid profile of Jatropha curcas oil.

Sr. no	Fatty acid	Formula	Composition (%)
1.	Caproic acid	C5H11COOH	2.5
2.	Myristic acid	$C_{14}H_{28}O_2$	9.9
3.	Palmitic acid	C16H32O2	18.5
4.	Palmitoleic acid	C16H30O2	7.8
5.	Steric acid	C18H36O2	9.0
6.	Oleic acid	C18H34O2	18.8
7.	Elaidic acid	C18H34O2	1.4
8.	Linoleic acid	$C_{18}H_{32}O_2$	20.2
9.	Linolenic acid	$C_{18}H_{30}O_2$	2.1
10	Behenic acid	C21H43COOH	2.4

Fuel characteristics of Jatropha biodiesel

Table-6 describes the performance characteristics of optimized Jatropha methyl esters as fuel compared with ASTM D6751 and EN14214 specifications. The fuel performance and quality of biodiesel greatly depends on these properties.

Specific gravity of the biodiesel produced is 0.862 kg/L; this is well within the range of the standard. Flash point (FP 169°C) of the biodiesel is within the recommended range of American biodiesel standards but it is also greater than petro-diesel [29, 30]. Distillation temperatures are also in congruency with standard limits of ASTM [26]. The thickness of biodiesel manufactured in the current study is low as compared to the parent triglyceride. In the current investigation, Jatropha triglyceride methyl esters

have kinematic viscosity (4.5mm²/s) that agrees with the American and EN biodiesel specification limits.

The copper strip corrosion of the under study biodiesel is found to be within the limits of specifications of standard methods i.e. 1a. Sulphur content was 0.05 ppm in the investigated biodiesel. No specific limits are given by ASTM or EN standards.

Table-6: Fuel properties of biodiesel obtained from non-edible *Jatropha curcas* seed oil according to ASTM D6751 and EN 14214

Properties	Unit	Jatropha Biodiesel	ASTM D6751	EN 14214
Specific Gravity	kg/L	0.865 ±0.002	0.95 max	
Flash point (PMCC)	°F	169 ± 0.581	90 min	120 min
Distillation IBP	°C	290 ± 1.500		
Т 90	°C	360 ± 2.583		
Kinematic Viscosity	@ 40 (°C)	$\textbf{4.50} \pm \textbf{0.490}$	1.9-6.0	3.5-5.0
Copper Strip Corrosion	3h@100(°C)	1 a	No. 3 max	No. 1 min
Sulphur Content	ppm	$\textbf{0.05} \pm \textbf{0.250}$	0.0.5 max	
Bottom Sediments &water (B.S&W)	% vol.	Trace	<0.03	<0.05
Cetane No.		51±0.788	47	51
Cloud point	°C	4 ± 0.985		
Cold filter plugging point	°C	9.10± 3.55		
Pour point	°C	-1 ± 0.88		
Acid value	(mg KOH/g)	0.37±0.11	0.5max	0.5max
Oxidative stability	h	3.15	3min	6min
Lubricity (HFRR)	μm	$145{\pm}0.563$		
Ash content	%mass	0.017		
Free Glycerin	%mass	0.015	0.02	0.02

Values are mean ± SD (-----) not specific value

 H_2O is in the trace amount in the investigated biodiesel. Occurrence of H_2O in biodiesel can cause corrosion and bacterial growth which generally indicates inappropriate handling. Water and sediment can lessen plug fuel filter lifespan, which can cause engine fuel starvation [30]. Cetane number significantly affects the specificities of engine like exhaust expulsion and ignition parameters [30]. A fuel with higher cetane number has lower NOx liberation just like biodiesel which has huge amount of saturated fatty acids. In this investigation cetane number is noted to be 51.00 \pm 0.78 which lies within ASTM and EN range.

In a recent study [30] the cold flow properties like cloud point CP, pour point PP and cold filter plugging point CFPP are found to be 4 $^{\circ}$ C, -1 $^{\circ}$ C and 9.10 $^{\circ}$ C, respectively. Oxidative stability is related with the storage stability of biodiesel attributing itself towards a technological issue for the use of biodiesel. Polyunsaturated chains of fatty acid are the main cause of this oxidative stability. The oxidative stability of under study jatropha biodiesel is 3.15 h. Lubricity is the property which lowers the friction between two moving objects. There is no specific value so far set for lubricity parameter by international standards; the investigated biodiesel has lubricity 145 μ m. Other fuel properties like acid value (0.37), ash content (0.017) and free glycerin (0.015) were noted to be in concordance with standard ASTM and EN limits.

Conclusions

This research work concluded that Jatropha oil can be used as raw material to produce biodiesel. It is proved from the experimental results that alkali based transesterification is a favorable method for the production of biodiesel on commercial scale. Glycerin produced as a byproduct can be sold to the pharmaceutical companies, since it is used in manufacturing of valuables such as cosmetics and toothpaste. Effects of different parameters on the biodiesel yield were examined. The optimized parameters were found as MeOH to triglyceride molar ratio of 6:1, 1.0 % catalyst concentration, 60 °C temperature, and 60 minutes' reaction time. Under optimized set of transesterification conditions an optimal biodiesel yield of 99.90% was achieved. The fuel properties of biodiesel produced were consistent with the ASTM specifications which specified that Jatropha triglyceride can be used as a potential feedstock to produce ecofriendly biodiesel with potential to replace petro-diesel for combustion in compression-ignition engines.

Acknowledgments

The authors would like to acknowledge Pakistan Arab Refinery Company (PARCO) for analyzing different fuel properties of JOMEs.

References

- 1. D. Agarwal and A. K. Agarwal, Performance and emissions characteristics of Jatropha oil (preheated and blends) in a direct injection compression ignition engine, *Appl. Therm. Engineering.*, **27**, 2314 (2007).
- 2. Anonymous, International Energy Agency. World Energy Outlook. (2002).
- 3. S. J. Deshmukh and L. B. Bhuyar, Transesterified Hingan (Balanites) oil as a fuel for compression ignition engines, *Biomass Bioenergy*, **33**, 108 (2009).
- A. Demirbas, Progress and recent trends in biodiesel fuels, *Energy Convers. Manag.*, 50(1), 14 (2009).
- 5. M. Guru, B. D. Artukoglu, A. Keskin and A.

Koca, Biodiesel production from waste animal fat and improvement of its characteristics by synthesized nickel and magnesium additive. *Energy Convers. Manag*, **50**, 498 (2009).

- 6. A. Demirbas, Diesel fuel from vegetable oil via transesterification and soap pyrolysis, *Energy. Sources*, **24**, 835 (2002).
- 7. T. T. Kywe and M. M. Oo, Production of biodiesel from Jatropha oil (*Jatropha curcas*) in pilot plant, Proceedings of *World Acad Sci Eng Technol.*, **38**, 481(2009).
- 8. D. Y. C. Leung, X. Wu and M. K. H. Leung, A review on biodiesel production using catalyzed transesterification, *Appl. Energ.*, **87**(4), 1083 (2010).
- 9. J. Tickell, *From the fryer to the fuel tank*, 2nd ed. Sarasota, FL: GreenTeach Publishing, (1999).
- 10. E. Alptelkin and M. Canakci, Optimization of pretreatment reaction for methyl ester production from chicken fat, *Fuel.*, **89**, 4035 (2010).
- Y. Zhang, M. A. Dube, D. D. McLean and M. Kates, Biodiesel production from waste cooing oil, *Bioresour. Technol.*, 89, 1-16 (2003).
- A. K. Azad and S. M. A. Uddin, Performance study of a diesel engine by first generation biofuel blends with fossil fuel: An experimental study, *J. Renew. Sustain. Energy.*, 5, (2013).
- 13. Y. Chisti, Biodiesel from microalgae, *Biotechnology Advances.*, **25**, p. 294 (2007).
- 14. S. P. Singh and D. Singh, Biodiesel production through the use of different sources and characterization of oils and their esters as the substitute of diesel: a review, *Renew Sust Energ Rev.*, **14**(1), 200 (2010).
- G. F. Silva, F. L. Camargo and A.L.O. Ferreira, Application of response surface methodology for optimization of biodiesel production by transesterification of soybean oil with ethanol, *Fuel. Process. Technol.*, **92**, p. 407 (2011).
- 16. X. Duan, J. Peng, C. Srinivasakannan, L. Zhang, H. Xia, K. Yang and Z. Zhang, Process optimization for the preparation of activated carbon from Jatropha hull using response surface methodology, *Energy Sources, Part A: Recovery, Utilization and Environmental Effects.*, 33, 2005 (2011).
- A. A. Refaat, N. K. Attia, H. A. Sibak, S. T. Sheltawy and G. I. Diwani, Production optimization and quality assessment of biodiesel from waste vegetable oil, *Int. J. Environ. Sci.*

Technol., 5(1), p. 75 (2008).

- B. A. Boateng and F. Kusi, Toxicity of Jatropha seed oil to *Callosobruchus maculatus* (Coleoptera: Bruchidae) and its Parasitoid, Dinarmus basalis (Hymenoptera: Pteromalidae), *J. Appl. Sci. Res.*, 4, 945 (2008).
- K.O. Adebowale and C.O. Adedire, Chemical composition and insecticidal properties of the underutilized Jatropha curcas seed oil, *Afr. J. Biotechnol.*, 5, 901 (2006).
- M. Rug and A. Ruppel, Toxic activities of the plant *Jatropha curcas* against intermediate snail hosts and larvae of schistosomes, *Trop. Med. Int. Health.*, 5, 423 (2000).
- A. A. Warra, Cosmetic potentials of physic nut (*Jatropha curcas* Linn.) seed oil: A review Am. J. Sci. Ind. Res., 3, 358 (2012).
- 22. R. I. Okoli, O. Aigbe, J. O. O. Obodo and J. K. Mensah, Medicinal herbs used for managing some common ailments among Esan people of. Edo State, Nigeria, *Pak. J. Nutr.*, 6(5), 470 (2007).
- 23. K. Prminik, Properties and use of *Jatropha curcas* oil and diesel fuel blends in compression ignition engine, *Renew. Energ.*, **28**, 239 (2003).
- 24. B. S. Nayak and K. N. Patel, Pharmacognostic studies of the *Jatropha curcas* leaves, *Int. J. Pharmtech Res.*, **2**(1), 140 (2010).
- 25. D. C. Montgomery, *Design and analysis of experiments*, 8th Edn, John Wiley and Sons, New York, (2001).
- K. Pranab and Barua, Biodiesel from Seeds of Jatropha Found in Assam, India I. J. E. I. C, 2(1), p. 53 (2011).
- U. Rashid, F. Anwar and G. Knothe, Evaluation of Biodiesel obtained from cottonseed oil. Fuel Process *Technol.*, **90**, 1157(2009).
- J. S. Oliveira, R. Montalvao, L. Daher, P. A. Z. Suarez and J. C. Rubim, Determination of methyl ester contents in biodiesel blends by FTIR-ATR and FTNIR spectroscopies, *Talanta.*, 69(5), 1278 (2006).
- 29. A. M. Syam, R. Yunus, T. M. I. Ghazi and T. C. S. Yaw, Synthesis of Jatropha curcas-based methyl ester and ethyl ester as biodiesel feedstocks, *Pertanika J. Sci. & Technol.*, **20**, 165(2012).
- O. A. Ogunwole, Production of biodiesel from Jatropha oil (Curcas Oil), *Res. J. Chem. Sci.*, 2(11), 30 (2012).