

Process optimization for biodiesel production from indigenous non-edible *Prunus armeniaca* oil

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Abstract. This work emphasized optimum production of biodiesel using non-edible *Prunus armeniaca* (Bitter Apricot) oil via transesterification collected from the high altitude areas of Himachal Pradesh, India. In this study the author produced biodiesel through the process of transesterification by using an alkali catalyst with alcohol (methanol and ethanol), under the varying molar ratio (1:6, 1:9, 1:12), variable catalyst percentage (1% and 2%) and temperature (70°C, 75°C, 80°C, 85°C). Furthermore, a few strong base catalysts were used that includes sodium hydroxide, potassium hydroxide, sodium metal and freshly prepared sodium methoxide. After screening the catalyst, response surface methodology (RSM) in connection with the central composite design (CCD) was used to statistically evaluate and optimize the biodiesel production operation using NaOH as catalyst. It was found that the production of biodiesel achieved an optimum level biodiesel yield with 97.30% FAME conversion under the following reaction conditions: 1) Methanol/oil molar ratio: 1:6, 2) Reaction time: 3h, 3) Catalyst amount: NaOH 2 wt. %, and 4) Reaction temperature: 85°C. The experimental results showed that the optimum production and conversion of biodiesel through the process of transesterification could be achieved under an optimal set of reaction conditions. The biodiesel obtained showed appropriate fuel properties as specified in ASTM, BIS and En- standards.

Keywords: bitter apricot oil; biodiesel; RSM and CCD; process optimization; fuel properties

1. Introduction

Energy sufficiency is the key to access to modern energy services. With the ever increasing world energy consumption, energy crisis has an acute impact on global social and economic growth. The growth in population, per capita energy use and rapid industrialization has resulted in surge in energy demand of developing nations. Most of the demand in energy is met by the usage of fossil fuels.

The crude oil fuels are important for the industrial growth, transportation, agricultural sector and other human needs. The increased energy requirement has led to overuse of petroleum based

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fossil fuels (Lim and Teong 2009, Demirbas *et al.* 2010) The petroleum based fossil fuels are being used at much higher rate than they are replenished. This has led not only to rapid escalation of crude oil prices, but also to serious health and environmental concerns.

The renewable and new energies are drawing a great deal of attention these days, though the contribution of renewable energy is only 11% of the entire global energy used (Hossain *et al.* 2007) Replacement of fossil fuels with renewable fuels produced within the nation will not merely preserve the foreign exchange of developing nations but will also resolve the ecological problems as well. Biofuels are one of the potential origins of vitality for the hereafter and can constitute the foundation for sustainable growth in terms of socioeconomic and environmental businesses. Biofuels are the liquid fuels that have been derived from waste plant or animal matter. Biofuels include alcohols, vegetable oils, biodiesel etc (Canakci *et al.* 2008, Gerpen 2005). As the vegetable oils have the calorific value close to diesel, there has been attempt by many researchers to utilize vegetable oils in CI engines. However, the vegetable oils have been found to be unsuitable for CI engines due to high viscosity (Knothe 2001, Peterson *et al.* 1983, Baldwin *et al.* 1982). Vegetable oils are several times more viscous as compared to conventional diesel fuel. Higher viscosity of fuel results in poor atomization, incomplete combustion and carbon deposition on the injector and valve seats and engine fouling (Vanderwalt and Hugo 1982, Ryan *et al.* 1984, Fuls *et al.* 1984). Common methods used for decreasing the viscosity of vegetable oils are blending with diesel, emulsification, pyrolysis, cracking and transesterification. Transesterification process has been ordinarily applied to bring down the viscosity of vegetable oil and produce biodiesel (Santori *et al.* 2012, Planning Commission India 2003). The raw material for biodiesel i.e., vegetable oil consists of mainly triglycerides. The triglycerides react with alcohol (methanol or ethanol) in the presence of a catalyst to produce glycerol and fatty acid esters. It is these esters that have come to be known as biodiesel. Temperatures, catalyst type, concentration ratio of alcohol to oil and stirring speed rate were observed to influence the transesterification process to a greater extent (Singh *et al.* 2006, De Oliveria 2005). The transesterification reaction for biodiesel production can be accomplished as one-step (acid/base) or two-step (acid/base) processes depending on the content of FFA. Either two-step process is recommended if a feedstock contains more than 5% of FFA (Rabiah Nizah *et al.* 2014, Ramadhas *et al.* 2004) or elevated reaction conditions at high temperature and pressure are required using specially designed catalyst (Sarma *et al.* 2014, Aslam *et al.* 2014).

There are many non-edible vegetable oils which are unsuitable for human consumption and find little use otherwise. Out of several vegetable oil sources available globally, India is giving prime importance to non-edible oils sources such as *jatropha curcas*, different varieties of *pongamia* species, Mahua, *Mesua ferrea* L, neem etc. in broader perspective to use as substitutes of petroleum diesel (Aslam *et al.* 2014, Chouhan *et al.* 2013) This is attributed to the competition of food vs. fuel for the rising population of the country (Roy *et al.* 2014) In this connection addition of some non-edible vegetable oil in the fuel market may boost the economy. Moreover there is always a liquid fuel crisis situation in high altitude areas both for civil and defense applications in our country.

Bitter Apricot (*Prunus armeniaca*) oil is a non-edible oil which has been used mainly for the medicinal purposes. Literature reveals that *Prunus armeniaca* (*P. armeniaca*) oil contains free fatty acids (FFA) less than 5% (Gumus and Kasifoglu 2010, Ullah *et al.* 2009). Thus, it can be a novel source of non-edible oil obtained from the Himalayan regions of India, and the process of biodiesel production would also be much easier owing to less FFA content. It can be cultivated in cold and arid lands. The marginal lands which were never used for cultivation of any crops, fruits

or vegetable in the hilly terrains can also be opted for the propagation of such species (Gurau *et al.* 2016). The fatty acid profile, physicochemical properties of the oil and process optimization for biodiesel production using Response Surface Methodology (RSM) combined with Central Composite Design (CCD) were used to statistically evaluate and optimize the biodiesel production. The fuel properties were evaluated with respect to appropriate standard methods.

2. Materials and methods

2.1 Materials

Prunus armeniaca (bitter apricot) is an important fruit crop of lower Himalayan regions of India. It wildy grows in mid hills of Himachal Pradesh and Jammu & Kashmir. The kernels of apricot fruits are either sweet or bitter, depending upon the variety. The seed kernels of bitter apricot contains 40-50% of oil, i.e., higher as compared to jatropha kernel's (30-40%) oil content (Chouhan *et al.* 2013). Bitter-kernelled ones thrive better under the warmer climate of mid hills (900-1,500 m), long cool winter (300-900 chilling hours below 7°C), and frost free and warm spring which are favorable for fruiting (accessed on 01-11-2014 at <http://www.fruitipedia.com/Apricot.htm>). The pH of the soil should be 6.0 - 6.8. The psychochemical properties of the apricot oil as compared with *Jatropha curcas* oil are shown in the Table 1 and Table 2. It was earlier reported by Gumus *et al.* 2010 that some composition was found to be similar to *P. armeniaca* oil in Turkey.

Apricot fruits generally appear in first week of May-June. Matured apricot tree can produce 50-

Table 1 Properties of apricot oil and jatropha oil

Property	Apricot oil	Jatropha oil ^a
Acid value (mg of KOH/gm)	6.66	7.4
FFA(in terms of oleic)	3.33	3.7
Iodine value (gm of I ₂ /100 gm)	205	-
Density (kg/m ³)	905	919
Kinematic Viscosity (cSt)	39.5	34.0
Calorific value (MJ/kg)	39.95	36.0

Table 2 Fatty acid compositions of apricot oil and jatropha oil

Free fatty acids	Molecular formula	Composition (%)	
		Apricot	Jatropha ^a
Palmitic acid	C ₁₆ H ₃₂ O ₂	3.305	16.90
Palmetoleic acid	C ₁₆ H ₃₀ O ₂	0.975	-
Stearic acid	C ₁₈ H ₃₆ O ₂	1.625	8.57
Oleic acid	C ₁₈ H ₃₄ O ₂	71.76	38.07
Linoleic acid	C ₁₈ H ₃₂ O ₂	20.19	32.31
Linolenic acid	C ₁₈ H ₃₀ O ₂	1.03	-

^aChouhan *et al.* (2013)

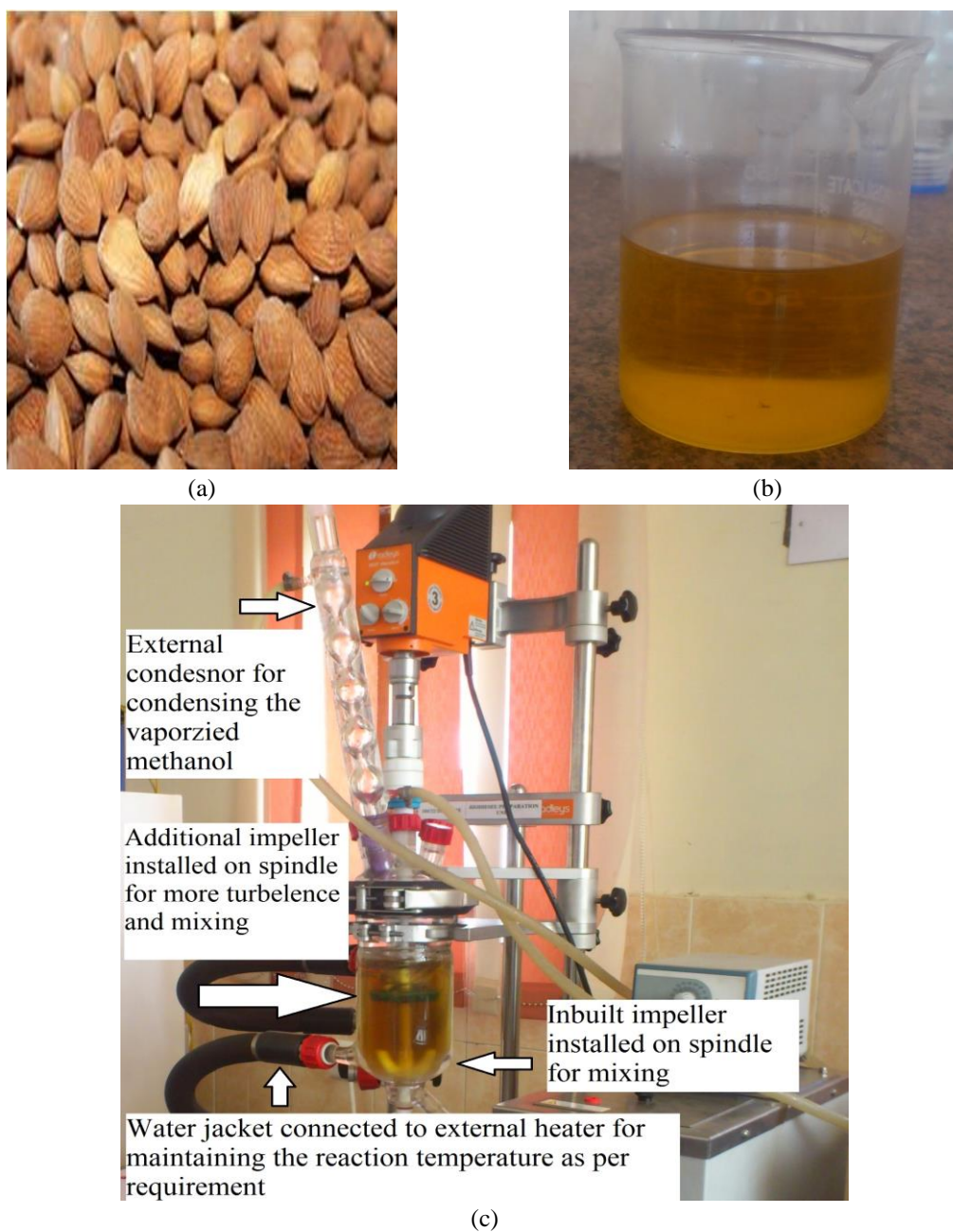


Fig. 1 (a) Apricot kernel seeds, (b) Extracted apricot oil and (c) Modified Radleys reactor for biodiesel production

80 kg/tree of fruits. The tree starts producing fruits after 5 years. About 10 L of the *P. armeniaca* oil and 5 kg of the raw seeds were procured from a local oil expeller Industry in Kullu-Manali, Himachal Pradesh. The psychochemical properties of the *P. armeniaca* oil were determined as per procedure followed by Sarma *et al.* (2005).

For different chemical processes and tests the reagents used were methanol (LOBE CHEMIE; >99.5%), Ethanol (MERCK; >99%), KOH (MERCK; >85%), CHCl_3 (LOBE CHEMIE; >99%), phenolphthalein (HIMEDIA; 98%), NaOH (HIMEDIA; >98), Sodium Metal (MOLY CHEM; >98%), KOI (GR) (MERCK; >99.8), Sodium Sulfate Anhydrous (GR MERCK; >99.5%), Acetic Acid (MERCK; >99%), Starch Powder (HIMEDIA, AR; > 9.995%).

2.2 Production of biodiesel

P. armeniaca oil with low FFA (3.33%) content can be converted into biodiesel using one step transesterification method as reported by Chouhan and Sarma (2013). The experiments for the present work consist of fixed parameters (volume-300 ml, Reaction time-3h, Alcohol-methanol and RPM-500) and variable parameters (catalyst- Na metal, Na methoxide, NaOH & KOH, Temp. 70°, 75°, 80° & 85°C, molar ratio 1:6, 1:9 & 1:12 and catalyst amount 1% and 2%). Initially, calculated amount of *P. armeniaca* oil, methanol and catalyst were poured into a modified Radleys reactor. The reactor consists of water jacket, external heating unit, condenser, and mechanical stirrer. The constant temperature was maintained by an external device and external condenser was also used to control the methanol vaporization. The reaction was carried out with oil, methanol and catalyst. An additional impeller in the mechanical stirrer of Radleys reactor was installed for uniform mixing and turbulence in order to accelerate the transesterification reaction (Fig. 1). The effects of process variables on product yield and ester conversion were evaluated statistically. The biodiesel (Fatty Acid Methyl Ester/FAME) conversion has been estimated using Gas chromatograph (Agilent 7890A) as per EN14214 specification.

2.3 Experimental design

RSM technique can be used for the optimization of biodiesel production as reported by Lee *et al.* (2011). In the present work, optimization of biodiesel processing was done using response surface methodology (RSM) with the help of Minitab 16 (Stat-Ease Inc., Minneapolis, USA). In RSM, Central Composite Design (CCD) technique was employed for studying the response of transesterification reaction variables. Three variables viz. 1) reaction temperature (70°-85°C), 2) catalyst loading (1-2 wt.%) and 3) oil to methanol molar ratio (6-12) were chosen as primary variables and FAME yield were chosen as the response variable in this study. Table 3 provides the actual and the coded values of all the three primary variables. A second order response surface model was fitted using two level three factor, central composite design. For this purpose, 20 experiments were performed according to eight cube points, four centre points in cube, six axial points and two centre points in axial. The complete design matrix obtained using CCD is shown in Table 4.

2.4 Statistical analysis

The RSM technique was employed to analyze the data obtained after CCD experimentation. The second order polynomial equation for the optimized response value is expressed by Eq. (A1). In this equation, Y is the predicted response; β_0 , β_j , β_{ij} and β_{ij} are constant coefficients; x_i and x_j are the coded independent variables or factors; ε is random error.

The coefficient of determination (R^2) was used to determine the quality of fit for the second order polynomial model. Moreover, the Fisher's test (F-test) was implemented for checking the

Table 3 Levels of the transesterification condition variables

Factors	coding	units	Low value -1	High value +1
Temperature	A	°C	70	85
Catalyst loading	B	wt. %	1	2
Oil to methanol (molar ratio)	C	mol/mol	6	12

Table 4 Experimental design matrix and experimental results of the response

A	B	C	FAME	Predicated values	Errors
Reaction temperature	Catalyst loading	Molar ratio	Conversion		
77.50	1.50	9.00	75.39	75.99	0.60
70.00	1.00	12.00	71.00	72.00	0.99
77.50	1.50	9.00	75.00	75.39	0.39
70.00	1.00	6.00	71.05	71.99	0.94
77.50	1.50	9.00	75.39	75.99	0.60
85.00	2.00	6.00	97.43	98.00	0.56
85.00	1.00	12.00	83.37	83.98	0.62
85.00	1.00	6.00	88.00	88.05	0.05
70.00	2.00	6.00	77.50	78.01	0.49
85.00	2.00	12.00	85.12	85.72	0.60
70.00	2.00	12.00	69.82	71.00	1.17
77.50	1.50	9.00	75.39	75.84	0.45
65.25	1.50	9.00	70.00	71.67	1.67
77.50	2.31	9.00	81.58	80.91	0.67
77.50	1.50	9.00	75.39	75.61	0.22
85.00	2.00	9.00	90.00	90.87	0.87
77.50	1.50	4.10	80.98	81.53	0.55
77.50	0.68	9.00	73.89	74.89	1.00
77.50	1.50	9.00	75.39	75.61	0.23
77.50	1.50	13.89	70.00	71.44	1.44

significance of the regression coefficients (Montgomery 2001). Response surfaces and contour plots were obtained from polynomial equation by keeping one independent variable at a constant value while changing the other two variables.

3. Results and discussions

3.1 Screening of the catalyst

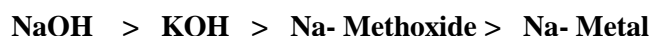
The fatty acid profile as shown in Table II represents the saturated and unsaturated fatty acids present in the bitter apricot oil. More than 90% of unsaturation was present in the oil with 71% of

Table 5 Characteristics of biodiesel based on sodium metal and sodium methoxide, NaOH & KOH

Catalyst	Acid value	FFA	Iodine value	Density (40°C) gm/cm ³	Specific gravity	Kinematic viscosity (cSt)	Flash point (°C)	Carbon residues (%)	FAME conversion (%)
Sodium metal	5.48	2.74	150	0.86403	0.8707	4.8632	125	0.057	74.67
Sodium methoxide	0	0	180	0.86591	0.87267	5.07	115	0.118	66.67
NaOH	0	0	180	0.86278	0.86952	5.23	115	0	80
KOH	2.66	1.33	185	0.86282	0.86956	4.432	115	0	83.33
Feedstock	6.66	3.33	205	0.9005	0.9032	39.09			
ASTM standards	0-0.5	<0.5	120	0.880	0.887	1.9-5	>115	0.01	96.5

oleic acid and 21% Linoleic acid. Initially, the experiments were performed for the production of biodiesel based on four different catalysts namely sodium metal, sodium methoxide, sodium hydroxide and potassium hydroxide. Experimental biodiesel production was carried out from different (1 wt. % each) catalysts (Na-metal, Na-methoxide, NaOH and KOH) at 70°C and a molar ratio (1:12). The characteristics of the biodiesel produced by different catalysts were examined by BIS standards as shown in Table 5. The basic fuel properties viz. density, viscosity, carbon residue and flash point followed by GC analysis are significant for initial evaluation whether the oil is converted to biodiesel or not.

The results showed that there were considerable differences in yield and ester conversion by using four different catalysts. The catalysts used in descending order based upon the yield and ester conversion of oil are as follows:



It was observed that NaOH catalyst based biodiesel gives good results as per requirement of BIS and other available standards. NaOH is an effective catalyst as the properties such as density, viscosity, yield & ester conversion were found to be in range as per standards. The NaOH catalyst not only accelerate the reaction, but also improve ester conversion and yield under applied parameters. The different chromatography peaks confirm the presence of fatty acid methyl esters of C14:0, C16:0, C16:0, C16:1, C18:0, C18:1, C18:2, C18:3, C20:0 and C22:1. However, the rest of the parameters (molar ratio, temp & catalyst loading) were varied for NaOH catalyst. Thus it was decided that sodium hydroxide could be the best option among the four for process optimization using RSM in conjunction with CCD.

3.2 Response surface regression: yield versus A, B, C

It was found that NaOH catalyst based biodiesel gives good FAME yield and characteristics of the biodiesel as per BIS standards, so the effective parameters like temp., molar ratio and catalyst loading were varied for this catalyst. About 20 experiments were performed to optimize the biodiesel production from *P. armeniaca* (bitter apricot) oil.

Table 6 Estimated regression coefficients for FAME yield

Term	Coef	SE Coef	T	P
Constant	75.391	0.4637	162.595	0.0001
A	13.187	0.5859	22.506	0.0018
B	3.347	0.4998	6.698	0.0012
C	-5.046	0.5127	-9.842	0.0002
A ²	9.472	1.0572	8.959	0.0023
B ²	2.851	0.8666	3.289	0.0080
C ²	1.102	0.8479	1.300	0.2230
AB	1.956	1.0351	1.890	0.0881
AC	-3.087	1.0809	-2.856	0.0175
BC	-5.087	1.0809	-4.707	0.0011

Table 7 Analysis of variance for FAME yield

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	1044.37	1044.37	116.041	88.29	0.0001
A	1	665.64	665.70	665.703	506.52	0.0018
B	1	72.22	58.96	58.958	44.86	0.0012
C	1	127.30	127.30	127.297	96.86	0.0002
A ²	1	119.71	105.50	105.500	80.27	0.0023
B ²	1	13.14	14.22	14.221	10.82	0.0080
C ²	1	1.84	2.22	2.221	1.69	0.2230
AB	1	4.69	4.69	4.693	3.57	0.0881
AC	1	10.72	10.72	10.722	8.16	0.0175
BC	1	29.11	29.11	29.114	22.15	0.0011
Residual Error	10	13.14	13.14	1.314		
Lack-of-Fit	5	12.13	12.13	2.425	11.92	0.0081
Pure Error	5	1.02	1.02	0.203		
Total	19	1057.51				

S=1.14642 PRESS=17.547

R-Sq=98.76% R-Sq(pred)=96.11% R-Sq(adj)=97.64%

3.3 Development of regression model

Due to higher order polynomial with significance of additional terms, the quadratic model was found to be the best amongst the other entire fitted model. Table 6 shows the quadratic model as per RSM software. Eq. (A2) represents the model equation based on the coded value of primary variables. The positive sign in front of the terms represents the increase in FAME yield; and negative sign represents the decrease in FAME yield. The model (Eq. (A2)) indicated that positive coefficients are A, B, A², B², C² and AB and negative coefficients are C, AC and BC. The analysis was done using coded units.

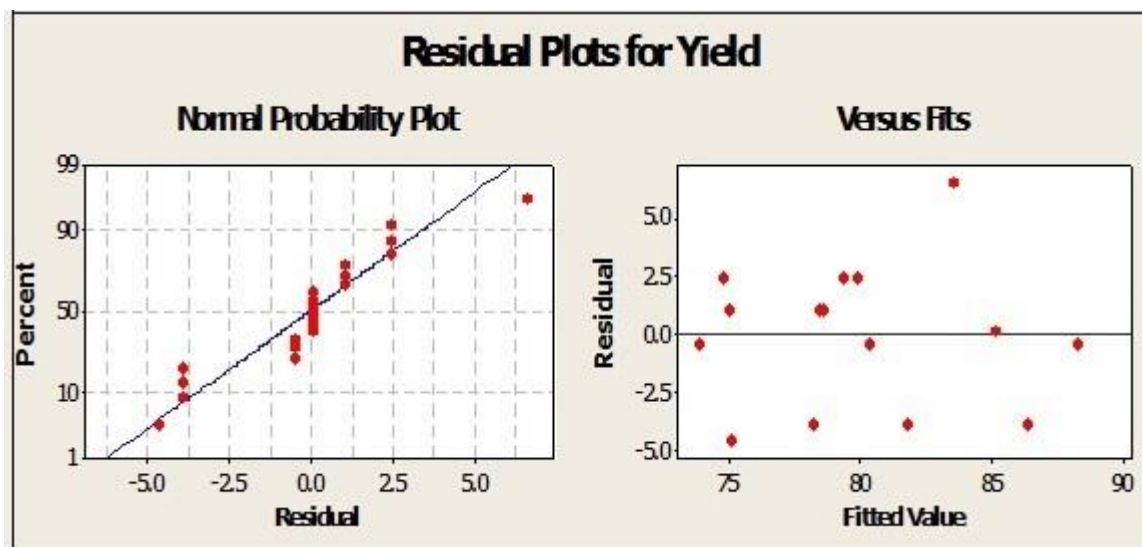


Fig. 2 Residual plots for FAME yield

Table 7 shows the result of statistical analysis of variance (ANOVA). The significance of each regression coefficient was determined using the p-value (probability of error value) as a tool. The regressors or terms incorporated in the Model F-value of 88.29 with p-value 0.0001 imply that the model is significant at 95% confidence level. The p-value less than 0.05 implies that the particular model term was of statistical significance (Chen *et al* 2008).

3.4 Parameter study

Fig. 2 represents the residual plot for yield. Fig. 3 shows the 3D plots for the interaction effect between reaction temperature (A) and reaction catalyst loading (B) and methanol/oil ratio (C) towards FAME conversion. The 3D response surface shown in Fig. 3(a) unveil that increment of reaction temperature from low level (70°C) to high level (85°C) and reduction in methanol/oil ratio from (1:12) to (1:6) leads to the increase of FAME conversion. Hence, it was found that increasing temperature and decreasing molar ratio improves yield. Fig. 3(b) shows the interaction between temperature and catalyst loading which shows that with increasing temperature and catalyst loading, the FAME conversion improves. The FAME conversion remained 75% with the reaction temperature at 70°C. However, the biodiesel yield increase to 97.3% at 85°C. Lee *et al.* 2011 also reported that higher temperature, improved the biodiesel yield. Further, Fig. 3(c) shows that decreasing molar ratio and increasing catalyst loading from 1 to 2 wt. % improved the FAME conversion. It was found that increasing temperature and catalyst loading improves the FAME conversion whereas decreasing molar ratio improves the FAME conversion.

3.5 Optimization of biodiesel (FAME) yield

In this study, the optimization of the biodiesel yield was achieved by varying its operating variables. The variables (reaction temperature, catalyst loading and methanol/oil ratio) were set in a range between low and high levels which coded -1 and +1 to achieve maximum response for the

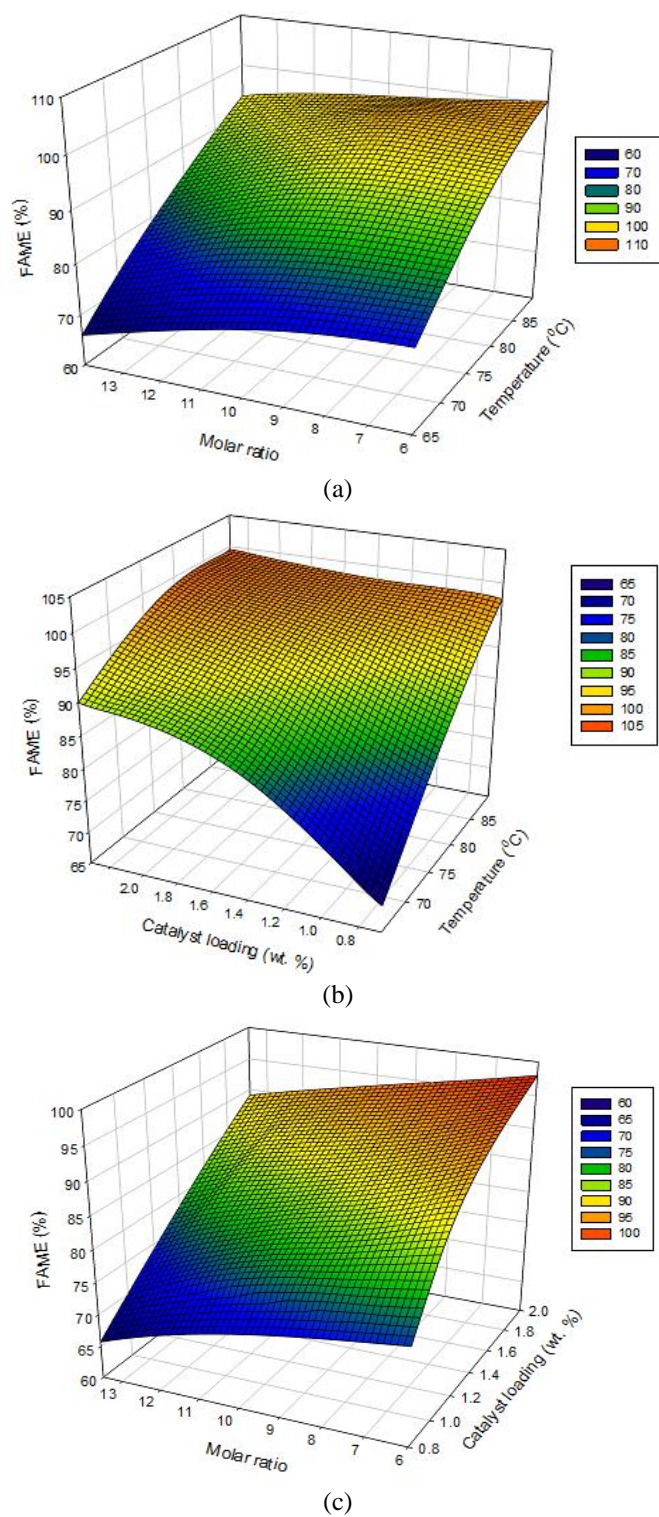


Fig. 3 Comparisons of FAME yield, molar ratio, reaction temperature and catalyst loading

Table 8 Comparison of biodiesel with international standards

Properties	Petro-Diesel	Apricot biodiesel	Jatropha Biodiesel ^a	BIS standards	ASTM Standards D 6751	European Standards EN 14214
Acid value (mg of KOH/gm)	NA	0	0	0-0.5	0-0.5	0-0.5
Iodine value (gm of I ₂ /100 gm)	NA	175	-	120	120	120
Density 40°C (kg/m ³)	850	880	892	860 to 900	880	880
Kinematic Viscosity 40°C (cSt)	2.6-3	4.32	6.80	3.5 to 5	1.9 to 6	3.5 to 5
Flash Point (°C)	65	115	108	>101	>115	>120
Carbon residue (%)	0	0	0.30	.001	.001	.001
FFA (in terms of oleic)	NA	0	0	< 0.5	< 0.5	< 0.5
Colorific Value (%)	42	39.90	37.10	NA	NA	NA
FAME Conversion (%)	NA	97.3	89.43	96.5	96.5	96.5

^aChouhan *et al.* 2013

biodiesel yield as shown in the Table 3. The outcomes with these three variables were generated by the software for the desired reaction based model obtained and the experimental data input criteria which show that biodiesel yield is dependent upon all three parameters i.e. temperature, catalyst loading and molar ratio.

The overall average optimized conditions for biodiesel yield were obtained as follows:

Reaction temperature 85°C, methanol/oil molar ratio 6 and catalyst amount 2 wt. % with biodiesel yield of 97.3%. The predicted biodiesel yield was 98.5%. This means that the experimental value obtained was reasonably close to the predicted value calculated from the model (1.2% of error). It can be concluded that the generated model showed reasonable predictability and sufficient accuracy for the biodiesel yield in the experimental conditions used.

The conditions were optimized using RSM technique in the Minitab software and the mass production of biodiesel were carried out at an optimum set of reaction conditions as evident from the analytical tools, viz. the NaOH catalyzed biodiesel at 85°C, oil alcohol molar ratio (1:6), 2 wt. % catalysts at 500 rpm and at a reaction time of 3h gives 97.3% conversion of oil to methyl ester. The comparison of the basic fuel properties of the *P. armeniaca* biodiesel (obtained at the optimum set of reaction conditions) with petro-diesel, Jatropha methyl ester, BIS, ASTM and EU standards are presented in Table 8. The properties within the limit such as density, viscosity, flash level, carbon residues and % conversion of oil to FAME assured the suitability with respect to all the parameters applicable for its purpose as a substitute of petroleum diesel.

4. Conclusions

P. Armeniaca oil could be potentially important oil for biodiesel production for the high altitude areas where these are grown due the suitability of the oil composition for transesterification at mild condition. This is because the high altitude area faces the fuel crisis frequently due the lack of petroleum supply. It was observed that NaOH catalyzed biodiesel at

85 °C, oil alcohol molar ratio (1:6), 2 wt. % catalysts at 500 rpm and at a reaction time of 3h gives 97.3% conversion of oil to methyl ester. Further, the RSM in conjunction with CCD is the most appropriate tool to select the best possible combination of the process variables during biodiesel production. It has been concluded that NaOH catalyst based biodiesel with increasing temperature and decreasing molar ratio resulted in more efficient conversion.

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Appendix

$$y = \beta_0 + \sum_{j=1}^k \beta_j X_j + \sum_{i < j} \sum \beta_{ij} X_i X_j + \sum_{j=1}^k \beta_{ij} X_j^2 + \varepsilon \quad (\text{A1})$$

$$Y = 75.931 + 13.187 \times A + 3.347 \times B - 5.046 \times C + 9.472 \times A^2 + 2.851 \times B^2 + 1.102 \times C^2 \\ + 1.956 \times A \times B - 3.087 \times A \times C - 5.087 \times B \times C \quad (\text{A2})$$