

BIODIESEL PRODUCTION USING *ERUCA SATIVA* OIL: OPTIMIZATION AND CHARACTERIZATION

MUHAMMAD WASEEM MUMTAZ¹, AHMAD ADNAN¹, ZAHED MAHMOOD²,
HAMID MUKHTAR³, MUHAMMAD DANISH⁴ AND ZAHOOR AHMAD⁵

¹Department of Chemistry, GC University Lahore, Pakistan

²Department of Chemistry & Biochemistry, University of Agriculture Faisalabad, Pakistan

³Institute of Industrial Biotechnology, GC University Lahore, Pakistan

⁴Department of Chemistry, University of Gujrat, Gujrat, Pakistan

⁵Department of Statistics, University of Gujrat, Gujrat, Pakistan

Correspondence: Muhammad.waseem@uog.edu.pk

Abstract

In the present study *Eruca sativa* oil (Taramira oil) was explored for the production of biodiesel by optimizing transesterification process using NaOH, KOH and NaOCH₃ as catalysts. Optimization of different reaction parameters i.e., catalyst concentration, reaction temperature, reaction time and alcohol to oil molar ratio was done using Response Surface Methodology (RSM). The order of catalytic effectiveness regarding production of the biodiesel using three different catalysts was found to be NaOCH₃> KOH > NaOH. The biodiesel was characterized following GC-MS and FTIR analyses. The fuel properties such as cetane number 59.8, density 0.871 g/cm³, specific gravity 0.889, pour point -2.97°C, kinematic viscosity 5.71 mm²/s, flash point 197.5°C carbon residue 0.01% of the biodiesel produced ascertained the suitability of oil as a potential source for biodiesel production.

Introduction

The native energy resources play a pivotal role in the development of a country. Most of the energy utilized in the world comes from petroleum, natural gas, coal and mainly petroleum derived fuels. Because of excessive industrialization there is rapid increase in demand for fossil fuel (Conceicao *et al.*, 2007). On the other hand, the conventional fossil fuel oil resources are depleting gradually which might lead to an alarming situation in future. Furthermore, the continuous use of petroleum fuel is intensifying air pollution releasing air contaminants including NO_x, SO_x, CO and particulate matter (PM) which in turn is magnifying the global warming problem (Shay, 1993; Yii-Der *et al.*, 2008; Klass, 1998). So, there is a growing need to search for alternative renewable, environment-friendly and non-conventional sources of energy (Mushtaq *et al.*, 2009).

Now-a-days biodiesel is getting much importance as an alternative fuel to conventional petro-diesel because of its renewable and environment friendly nature (Knothe *et al.*, 2005; Umer *et al.*, 2009). Biodiesel is usually produced by modifying vegetable oils and fats through transesterification reaction (Schwab *et al.*, 1987; Alencar *et al.*, 1983). The main parameters affecting transesterification are alcohol to oil molar ratio, catalyst concentration, reaction temperature and time, contents of free fatty acids and water in the oils and fats (Freedman *et al.*, 1984; Encinar *et al.*, 2005). Response surface methodology is an emphatic technique, a compilation of statistical and mathematical techniques highly valuable for development, modification and optimization of various processes. Among various feed stocks, *Eruca sativa* (taramira) oil is highly productive feedstock for production of biodiesel. It is a member of the Brassicaceae family, is an annual herb with a sour and unpleasant odor, mostly grown in Middle East, India and Pakistan (Flanders & Abdul Karim, 1985) with 35% oil yield from seeds (Yadava *et al.*, 1998; Mohammad *et al.*, 2009). The oil from *sativa* is not recommended for edible

purposes due to its bad and pungent odor (Mohammad *et al.*, 2009). Therefore, it could be proved as a potential feed stock for production of biodiesel.

To date, there is lack of comprehensive research regarding the optimization of reaction parameters involved in transesterification of *Eruca sativa* oil using RSM in Pakistan. Therefore, we report here optimization of key transesterification reactions for the maximal production of biodiesel using *Eruca sativa* oil as feedstock.

Materials and Methods

Seeds of *Eruca sativa* were procured from the local market of District Gujrat, Pakistan and verified. All the chemicals used were of analytical/research grade. NaOH, KOH, and methanol were purchased from Merck Chemical Company (Darmstadt, Germany) NaOCH₃ from ACROS (New Jersey USA) whereas FAMEs standards were procured from Sigma Chemical Company (St Louis, MO, USA).

Extraction and quality evaluation of *Eruca sativa* oil: Oil was extracted from *Eruca sativa* seeds using Soxhlet extraction method (AOCS, 1997) with *n*-hexane as an extracting solvent (Umer & Anwar, 2008; Umer *et al.*, 2009). Prior to transesterification, the oil extracted was pre-analyzed for its values such as acid, peroxide, iodine and saponification following standard methods (AOCS, 1997). Specific gravity and refractive index were also determined.

Experimental design and protocol: Central Composite Response Surface Design (CCRD) was used to evaluate the effect of reaction parameters including Catalyst concentration (A), Reaction time (B), Reaction temperature (C) and Methanol to oil molar ratio (D) on percentage yield of biodiesel for 30 runs. The summary of CCRD along with the levels of reaction parameters used in present study is given in Table 1.

Table 1. Summary of central composite response surface design applied in the study.

Factor	Name	Units	Low level	High level
A	Catalyst concentration	%	00.25	01.25
B	Reaction time	Minutes	30.00	90.00
C	Reaction temperature	°C	30.00	60.00
D	Alcohol: Oil molar ratio		03.00	09.00

Pre-analyzed *sativa* oil was subjected to base catalyzed transesterification for biodiesel production at specified reaction conditions according to the Central Composite Response Surface Design (CCRD) using three necked flask equipped with a condenser, thermometer, chiller, heating plate and stirrer (Encinar *et al.*, 2002; Rashid & Anwar, 2002). Glycerol was separated and the remaining portion was washed with water to remove catalyst, soap and methanol to recover purified biodiesel and % yield was calculated. All the experiments were performed thrice.

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

Monitoring of transesterification using FTIR spectroscopy: FTIR analysis of *sativa* oil and oil based biodiesel was performed on Interspec 200-X FTIR spectrophotometer equipped with mountable FTIR liquid cells. The spectra were recorded over scanning range of 500 to 5000 cm⁻¹.

GC-MS compositional analysis of *Eruca sativa* oil based biodiesel: Fatty acid methyl esters profile was carried out on (GC-MS), utilizing Agilent- Technologies (Little Falls, CA, USA) 6890N Network Gas Chromatographic system, equipped with an inert XL Mass detector (Agilent-Technologies 5975), auto injector (Agilent-Technologies 7683B series) and capillary column (100 m x 0.25 mm with film thickness 0.20 μm) (Agilent-Technologies RT-2560) 1.0 μL sample was injected in the split mode with a split ratio 1:100. Helium was used as a carrier gas with a flow rate of 1.2 mL/min. The temperature of column oven was programmed from 150 to 250°C @ 4°C /min while initial and final hold up time was 1 and 5 min, respectively, whereas the temperature of the injector and MS transfer line was set at 250 and 260°C, respectively. An electron ionization system (with ionization energy i.e., 70 eV) was used for GC/MS detection while scanning mass ranged from 30–550 m/z (Anwar *et al.*, 2008). The identification of the unknown *sativa* oil fatty acid methyl esters (ESOFAMEs) was performed by comparing their relative retention times with the retention times of authentic fatty acid methyl esters (FAMEs) standards (Sigma Chemical Co., St Louis, MO, USA). For further confirmation of ESOFAMEs profile MS spectra of the sample was compared with those from the NIST mass spectral library of the GC/MS system while quantification was done by Agilent-Technologies data handling software (Chem. Station 6890) and The ESOFAMEs composition was reported as relative percentage of the total peak area(Anwar *et al.*, 2008).

To evaluate the effect of reaction parameters on percentage yield of biodiesel, CCRD experimental results were analyzed using Design Expert-7 and SPSS and suitable mathematical model was suggested and authenticated through necessary diagnostic checks. Optimized reaction parameters and biodiesel yield were estimated from the experimental results and Response Surface Plots were used to ascertain the results. The model can be written as:

Fuel properties of produced biodiesel: The fuel properties of the biodiesel obtained from *sativa* oil were determined using standards methods: cetane number (ASTM D 613), density (ASTM D 5002), kinematic viscosity (ASTM D 445), flash point (ASTM D 93), cloud point (ASTM D 2500), pour point (ASTM D 97) and ash content (ASTM D 874).

Results and Discussion

Quality of extracted *Eruca sativa* oil: For quality evaluation of *Eruca sativa* oil, physico-chemical characterization was carried out before transesterification. The acid value, peroxide value and iodine value of *Eruca sativa* oil was found to be 0.68 mg KOH g⁻¹, 8.0 meq kg⁻¹, and 109.13 g of I₂ per 100 gram, respectively while specific gravity, saponification value, and refractive index were 0.905 , 175.7 mg KOH g⁻¹, and 1.484, respectively.

Biodiesel production: The quality of *sativa* oil along with experimental results of base catalyzed transesterification according to CCRD credited the effectiveness and suitability of the oil methyl ester (ESOMEs). The comparative biodiesel yield (%) at different reaction conditions depicted the comparable catalytic behavior of NaOH, KOH and NaOCH₃ towards biodiesel production (Fig. 1). NaOCH₃ was proven to be the most productive catalyst for transesterification of the oil and executed maximum conversion to biodiesel giving 94.54% yield, whereas % age yield in case of KOH and NaOH catalyzed transesterification was found to be 93.10% and 87.70%, respectively. The high biodiesel yield using NaOCH₃ as catalyst compared to NaOH and KOH can be ascribed to the fact that NaOCH₃ generally catalyzes transesterification of *sativa* oil without water formation when dissolved in methanol in contrast to NaOH and KOH, and water formation during NaOH and KOH catalyzed transesterification results in soap formation thus decreasing the yield of biodiesel (Ellis *et al.*, 2007).

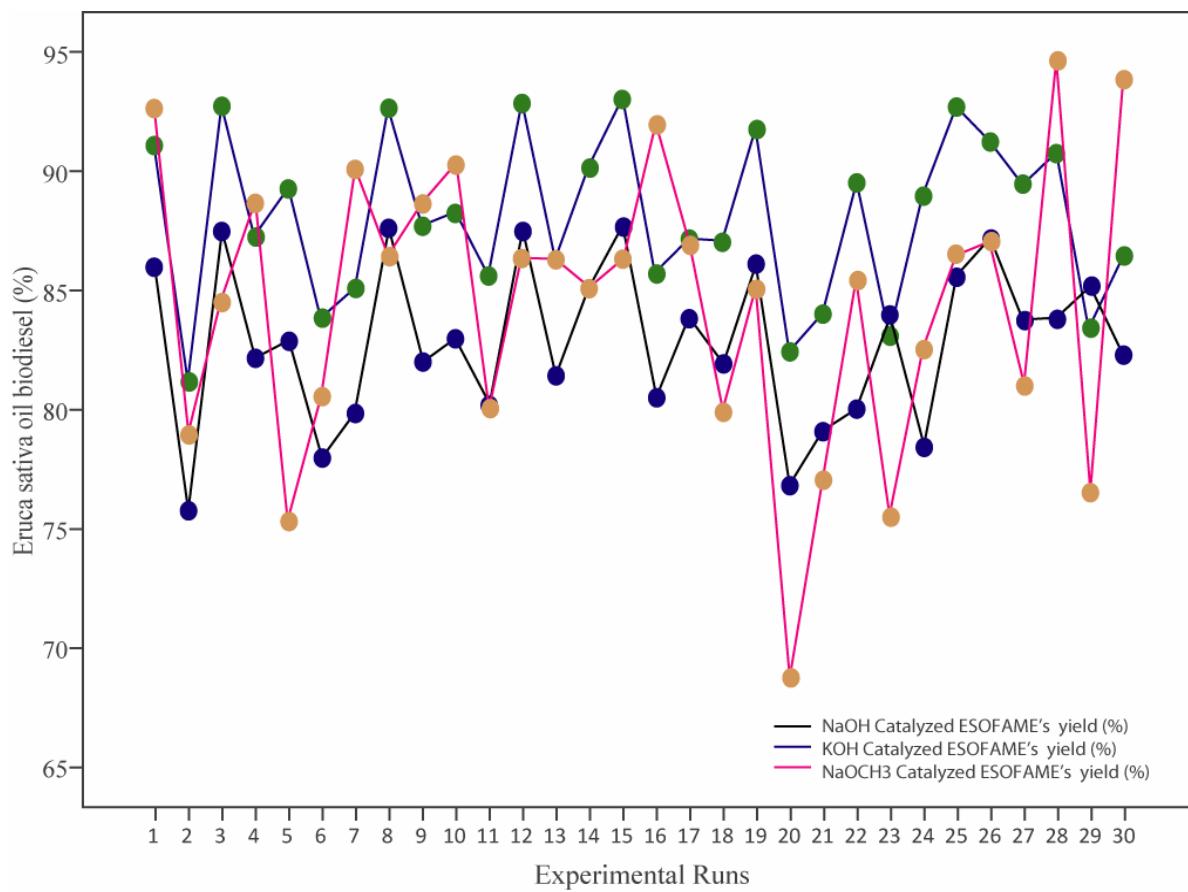


Fig. 1. Comparison of biodiesel yield (%) using NaOH, KOH and NaOCH₃ as catalyst for 30 experimental runs according to CCRD.

Table 2. Summary statistics of selected quadratic model.

Quadratic model	C.V. %	PRESS	R-Squared	Adj R-squared	Pred R-squared	Adeq precision
Model ^a	1.16	66.15	0.9575	0.9178	0.7970	16.818
Model ^b	1.30	103.47	0.9441	0.8920	0.7042	14.286
Model ^c	1.59	140.13	0.9747	0.9512	0.8694	27.836

Model^{a,b&c} represent quadratic models used for optimization of biodiesel yield using NaOH, KOH and NaOCH₃ as catalysts

To assess the contribution of different reaction parameters to biodiesel yield, quadratic models were selected out of linear, 2F1, quadratic and cubic models based upon sequential model testing, lack of fit tests and model summary statistics (Table 2). The fitness of suggested models was verified through diagnostic checks. Residuals were found to follow the normality, and the plots of predicted versus actual biodiesel yields (Fig. 2a, b & c) also ascertained the overall fitness of the suggested quadratic models and showed little difference between the actual and predicted *sativa* oil biodiesel yields.

Analysis of Variance (ANOVA) for quadratic models a, b & c (Table 3) described significance of linear terms i.e., catalyst concentration, reaction temperature and methanol:oil molar ratio for model^a and catalyst concentration and methanol:oil molar ratio for model^b whereas for model^c all the linear terms were found to be significant at 5%, respectively. First order interaction terms i.e., catalyst concentration × reaction time, catalyst concentration × molar ratio and reaction time × methanol:oil molar ratio for model^a while catalyst

concentration × reaction time, reaction time × reaction temperature and reaction temperature × methanol:oil molar ratio for model^b whereas catalyst concentration × reaction temperature for model^c were found to be significant at 5%. All the quadratic terms showed significant contribution for model^{a, b & c} except reaction temperature for model^c.

According to experimental results, NaOH and KOH catalyzed transesterification of the oil resulted in maximum yield of biodiesel (Table 4) when reaction was carried out at 45°C for 60 minutes using 0.75% catalyst concentration and 6:1 methanol:oil molar ratio, whereas maximum biodiesel yield form NaOCH₃ catalyzed transesterification was observed at 52.5°C for 45 minutes using 0.5% catalyst concentration and 4.5:1 methanol:molar ratio. The results are also supported by Response Surface Plots (Figs. 3, 4 & 5) conforming the fact that optimized *Eruca sativa* oil biodiesel was experienced at the defined reaction conditions while beyond these levels a decrease in biodiesel yield was observed.

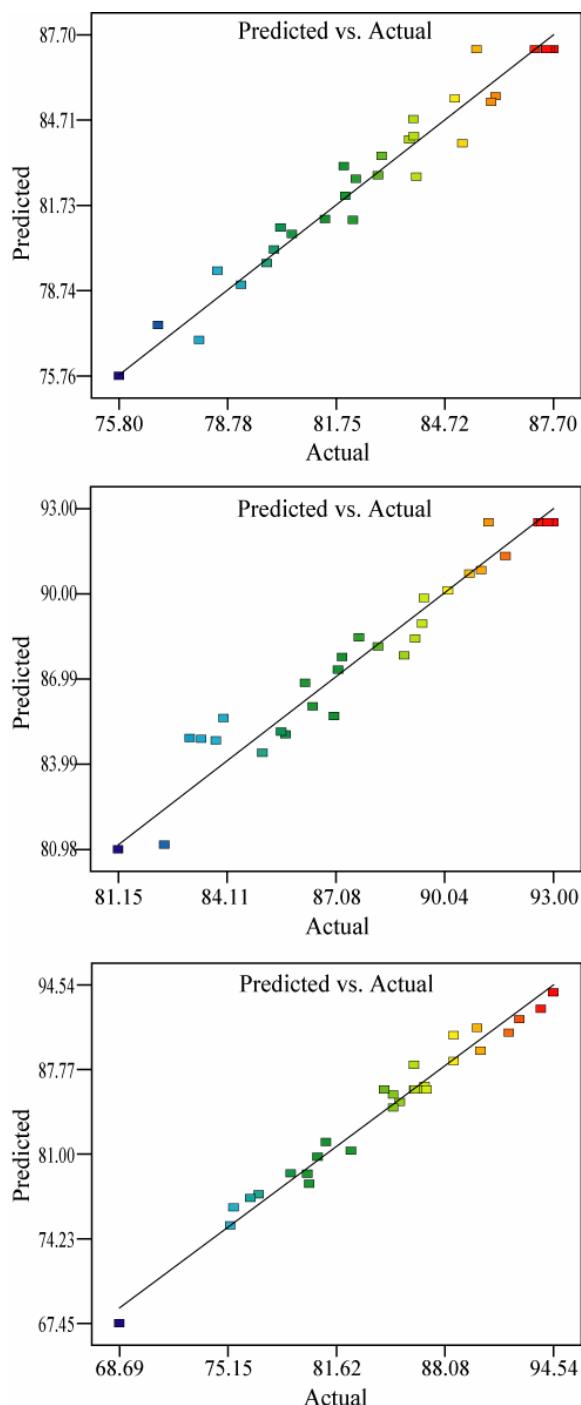


Fig. 2. Plot of predicted Vs actual values when (a) NaOH (b) KOH and (c) NaOCH_3 were used as catalyst to catalyze transesterification of *Eruca sativa* oil

FTIR spectroscopic analysis of *Eruca sativa* oil biodiesel: The FTIR analysis was performed for monitoring transesterification reaction of *Eruca sativa* oil and comparative description was carried out using FTIR spectrum of *Eruca sativa* oil and the *Eruca sativa* oil biodiesel. FTIR spectrums depicted that IR bands in the region $1425\text{-}1447\text{ cm}^{-1}$ for CH_3 asymmetric bending and $1188\text{-}1200$ for O-CH_3 stretching, were absent in *Eruca sativa*

oil IR spectrum while in *Eruca sativa* oil biodiesel spectrum these bands were present (Naresh et al., 2009, Dube, et al., 2004). Further the region $1370\text{-}1400\text{ cm}^{-1}$ for O-CH_2 groups in glycerol (moiety of TG, DG, and MG) and $1075\text{-}1111\text{ cm}^{-1}$ for $\text{O-CH}_2\text{-C}$ asymmetric axial stretching were present in IR spectrum of *Eruca sativa* oil while in biodiesel spectrum these bands were absent (Naresh et al., 2009, Dube et al., 2004, Siatis et al., 2006). Whereas, the region $1700\text{-}1800\text{ cm}^{-1}$ for C=O stretch and $2800\text{-}3000\text{ cm}^{-1}$ for symmetric CH_2 stretching and the asymmetric CH_3 and CH_2 stretching were present in IR spectrum of both *Eruca sativa* oil and biodiesel. Results of our present analysis were in accord to those reported by (Dube et al., 2004, Siatis et. al., 2006, Soares et al., 2008, Oliveira et. al., 2006, Zagonel et. al., 2004 and Younas, 2007).

Compositional analysis of *Eruca sativa* oil biodiesel using GC-MS: The GC-MS analysis of *Eruca sativa* oil methyl esters (ESOMEs) showed that erucic acid is the predominate constituent of ESOMEs (Table 5). The contents of palmitic acid (2.8%) as determined in our analysis was found to be greater to those reported in *Eruca sativa* oil by Sindhu & Kantharaj (1989), while lower (10.2%) investigated by Muhammad et al., (2009). The concentration of stearic acid in *Eruca sativa* methyl esters was found to be 0.90% which was quite comparable to (0.93%) reported by Sindhu & Kantharaj (1989) but somewhat lower (1.60%) than described by Muhammad et al., (2009). Oleic acid content was found to be 16.3% which was lesser 19.88 and 22.8% than reported by Sindhu & Kantharaj (1989) and Muhammad et al., (2009), respectively, while the contents of linoleic acid were found to be (10.3%) which were comparable to (9.23%) described by Sindhu & Kantharaj (1989) whereas, greater than (6.40%) presented by Muhammad et al., (2009). The content of linolenic acid i.e., 12.56% was also comparable to the results of Sindhu & Kantharaj (1989) and Muhammad et al., (2009), whereas, percentage of Cis- 11-ecosanoic acid was found to be (5.15%) which was comparable (6.40%) to those reported in *Eruca sativa* oil methyl ester by Muhammad et al., (2009) but lower (12.61%) with the investigated value of Sindhu & Kantharaj (1989). Finally, erucic acid content i.e., 47.70% was also somewhat greater than the results of Sindhu & Kantharaj (1989) and Muhammad et al., (2009).

Fuel properties of the *Eruca sativa* oil biodiesel: Fuel properties of the *Eruca sativa* oil methyl ester (ESOMEs) were evaluated and results are presented in Table 6. ESOMEs showed a cetane number 59.08 which was found to be compatible with ASTM standard. Density of *Eruca sativa* oil methyl ester (ESOMEs) was found to be 0.871 g/cm^3 at 38°C which was comparable 0.8811 g/cm^3 , 0.875 g/cm^3 , 0.873 g/cm^3 and 0.873 g/cm^3 to *Eruca sativa* oil methyl ester (ESOMEs), rapeseed oil methyl ester (RSOMEs), spent frying oil methyl ester (SFOMEs) and distilled frying oil methyl ester (DFOMEs), respectively (Mohammad et al., 2009, Weber et al., 2001). The Specific gravity at (38°C) of the *Eruca sativa* oil methyl ester (ESOMEs), was 0.889 which was comparable to the reported values (0.883, 0.8796 and 0.853) of palm kernel oil methyl esters (PKOMEs), soy oil methyl esters (SOMEs) and petroleum diesel, respectively (Alamu et al., 2007; Tat et al., 2007).

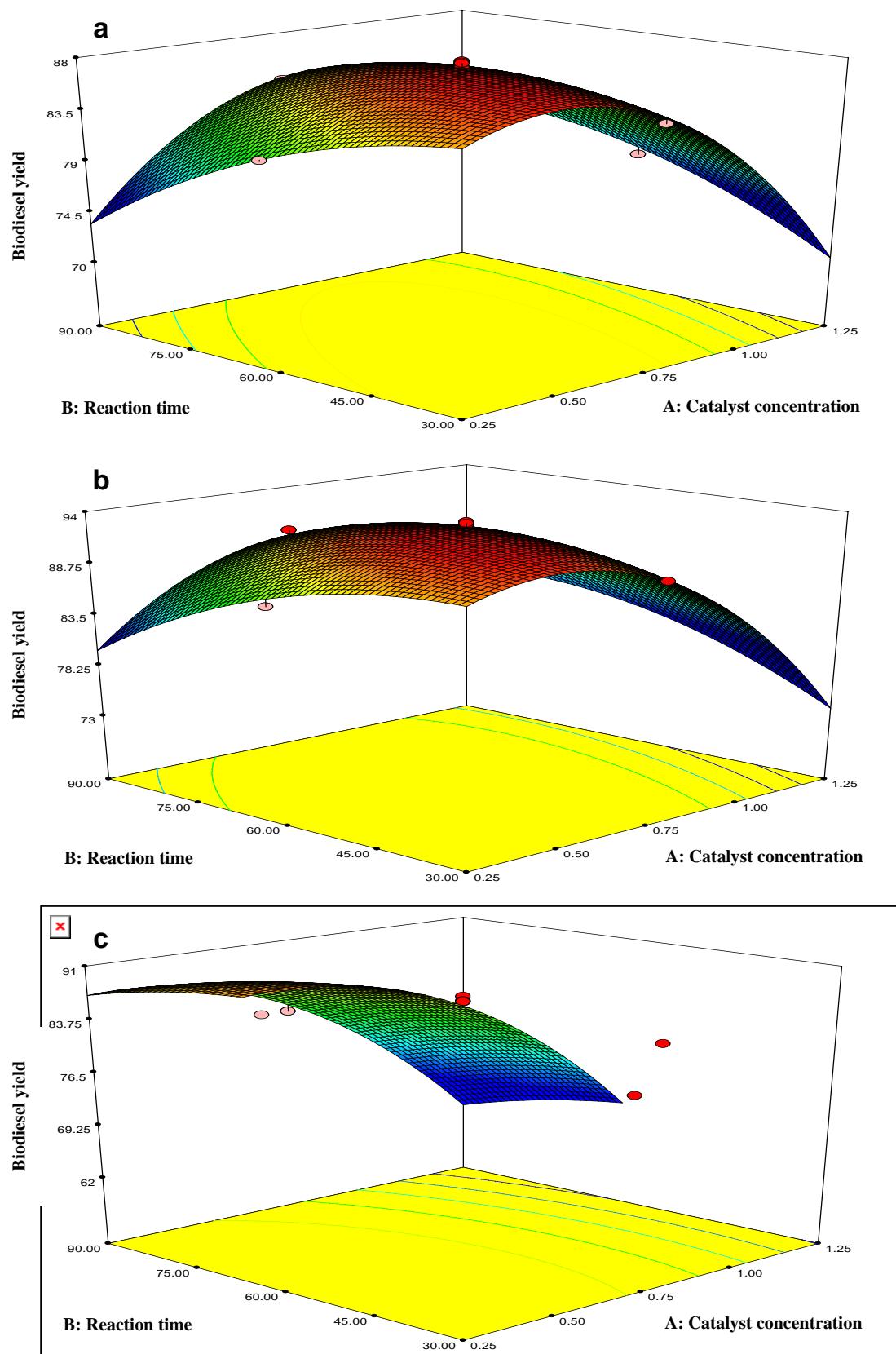


Fig. 3. Response surface plots of ESOFAEs yield predicted from the model showing the effect of (a) NaOH & reaction time (b) KOH & reaction time (c) NaOCH_3 & reaction time on % yield of ESOFAEs.

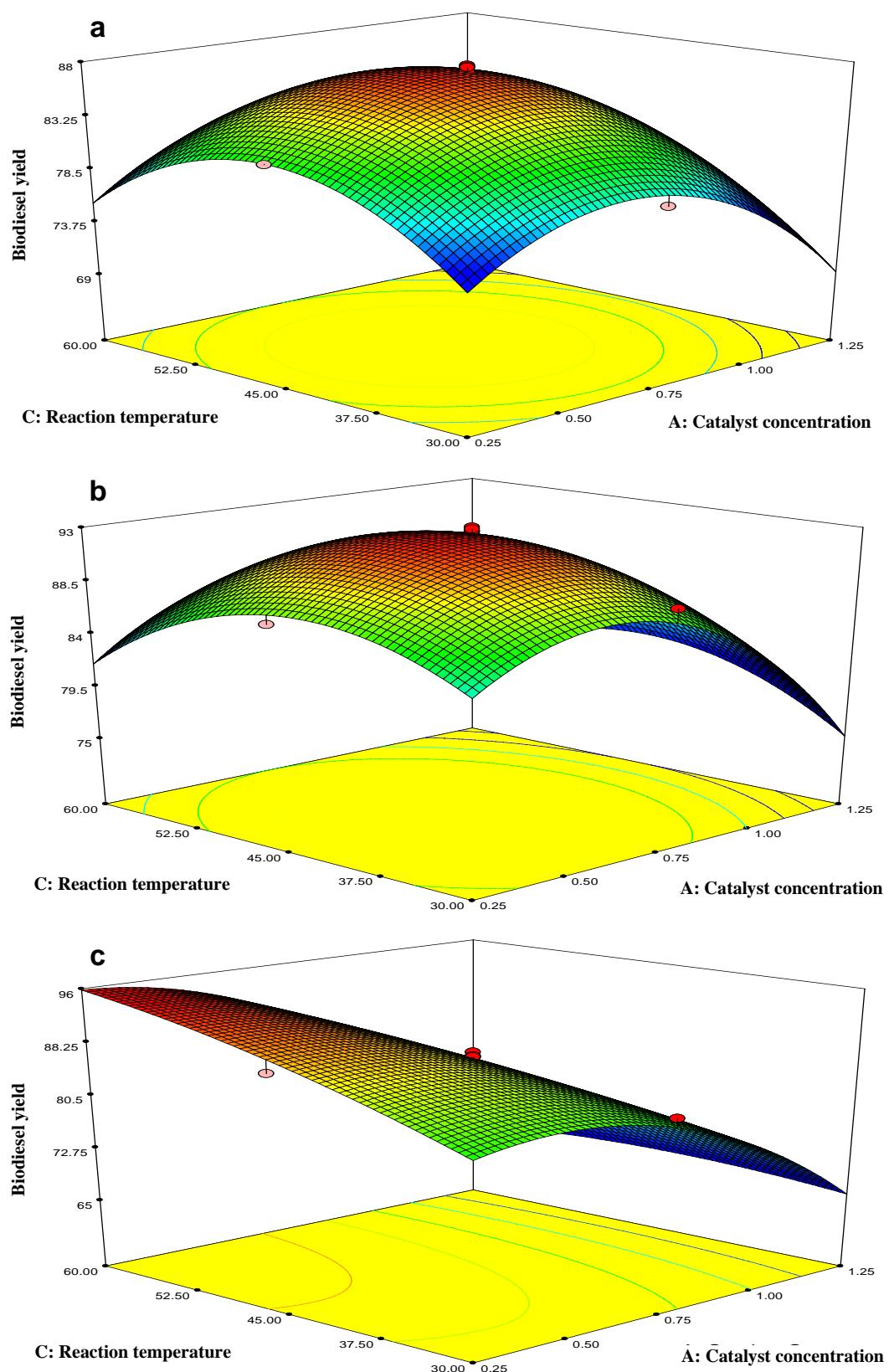


Fig. 4. Response surface plots of ESOFAEs yield predicted from the model showing the effect of (a) NaOH & reaction temperature (b) KOH & reaction temperature (c) NaOCH_3 & reaction temperature on % yield of ESOFAEs.

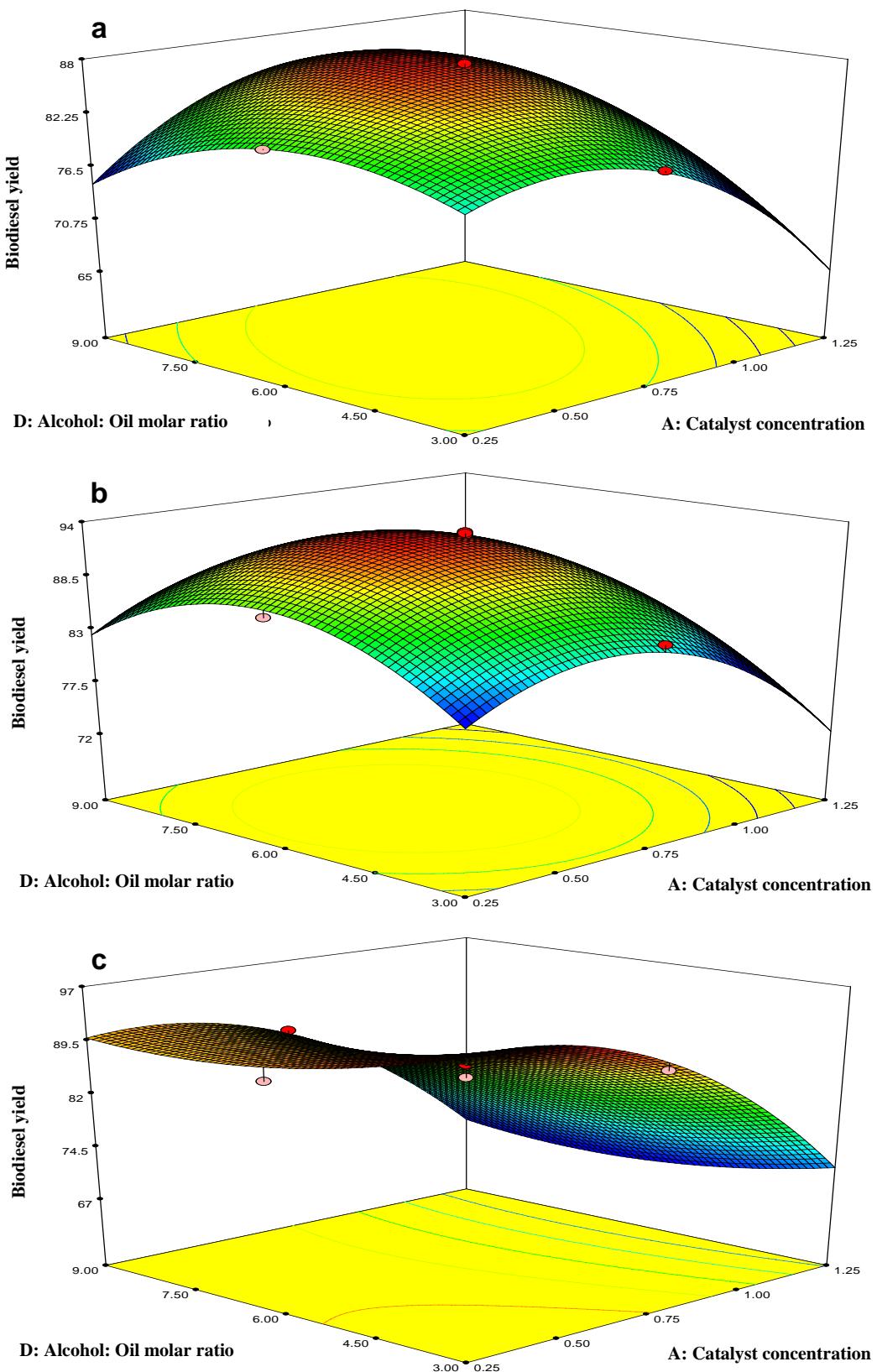


Fig. 5. Response surface plots of ESOFAEs yield predicted from the model showing the effect of (a) NaOH & methanol to oil molar ratio (b) KOH & methanol to oil molar ratio (c) NaOCH₃ & methanol to oil molar ratio on % yield of ESOFAEs.

Table 4. Optimized biodiesel yield and reaction parameters.

Catalyst	Catalyst Concentration (%)	Reaction time (Min.)	Reaction temperature (°C)	Methanol: Oil molar ratio	Biodiesel yield (%)
NaOH	0.75	60.00	45.00	6:1	87.7
KOH	0.75	60.00	45.00	6:1	93.1
NaOCH ₃	0.50	45.00	52.50	4.5:1	94.5

Table 5. Major fatty acid methyl esters of *Eruca sativa* oil biodiesel.

Sr. No.	Fatty acid methyl ester	Retention times	% Age composition	Reference [29]	Reference [15]
1.	Palmitic acid (C16:0)	14.60	2.8 ± 0.04	Trace	10.2
2.	Stearic acid (C18:0)	17.81	0.90 ± 0.01	0.93	1.6
3.	Oleic acid (C18:1)	18.90	16.3 ± 0.15	19.88	22.8
4.	Linoleic acid (C18:2)	20.31	10.3 ± 0.08	9.23	6.4
5.	Linolenic acid (C18:3)	22.08	12.56 ± 0.23	11.70	11.9
6.	Cis-11-Ecosanoic acid	22.19	5.15 ± 0.06	12.61	6.4
7.	Erucic acid (C22:1)	25.93	47.7 ± 1.95	42.4	40.8
8.	Others	-	4.29 ± 0.05	-	-

Table 6. Fuel properties of *Eruca sativa* oil biodiesel.

Fuel property	Value (mean ± SD)
Cetane number	59.08 ± 1.34
Density (g/cm ³)	0.871 ± 0.15
Specific gravity	0.89 ± 0.02
Pour point (°C)	-2.97 ± 0.17
Cloud point (°C)	1.5 ± 0.7
Kinematic viscosity (mm ² /s)	5.71 ± 0.21
Flash point (°C)	197.3 ± 2.1
Carbon residue (%)	0.010 ± 0.002

Pour point is an important parameter for low temperature operation of a fuel (Alamu *et al.*, 2007). The pour point of *Eruca sativa* oil methyl ester (ESOMEs), was found to be -2.97°C which was lower than the pour point of lard methyl esters (LMEs) i.e., 13.7°C, but comparable to those reported (3.0°C) for tallow oil methyl esters (TOMEs) while greater (-15°C) than tallow-oleic estolide 2-ethylhexyl esters, respectively (Victor *et al.*, 2005, Cermak *et al.*, 2007). The observed cloud point of the *Eruca sativa* oil biodiesel was 1.5°C which was lesser (6°C) than palm kernel oil methyl esters (PKOMEs), but greater (-18°C) than petroleum diesel, respectively (Alamu *et al.*, 2007, Tat *et al.*, 2007). Kinematic viscosity of the *Eruca sativa* oil methyl esters was found to be 5.71mm²/s at 40°C which was somewhat lesser than the kinematic viscosity of *Eruca sativa* oil methyl ester (ESOMEs) i.e., 5.9 mm²/s (Mohammad *et al.*, 2009). The observed flash point of the *Eruca sativa* oil methyl ester (ESOMEs) was found to be 197.3°C which was greater than the flash point of the palm kernel oil methyl esters (PKOMEs) i.e., 167°C and the petroleum diesel i.e., 74°C (Alamu *et al.*, 2007). The relatively higher flash point of

the *Eruca sativa* oil methyl ester (ESOMEs) may be due to the presence of High Erucic Acid (HEA) which may be responsible for high flash point of the *Eruca sativa* oil methyl ester (ESOMEs). This indicates that *Eruca sativa* oil biodiesel is more stable at ambient temperature and will pose lesser threat for storage and usage.

Conclusions

Based upon results, it is concluded that *Eruca sativa* oil is highly productive and suitable feed stock for biodiesel production. Results credited the NaOCH₃ catalyzed transesterification of the oil for higher biodiesel yield as compared to KOH and NaOH catalyzed transesterification.

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