IOP Conference Series: Materials Science and Engineering

PAPER • OPEN ACCESS

Optimization of Extraction Process of Jatropha Oil by Using Quenching Agent

IOP Conf. Series: Materials Science and Engineering 1127 (2021) 012003 doi:10.1088/1757-899X/1127/1/012003

Optimization of Extraction Process of Jatropha Oil by Using Quenching Agent

Richa Kothari[*], Shamshad Ahmad[†], M Samykano[#], V.V.Tyagi[‡], A K Pandey[¥], R.Saidur[¥]

*Department of Environmental Sciences, Central University of Jammu, Rahya-Suchani, Samba, Jammu, J&K

[†]Bioenergy and Wastewater Treatment Laboratory, Department of Environmental Sciences, Babasaheb Bhimrao Ambedkar University, Lucknow (U.P.), India.

[#]Faculty of Mechanical & Manufacturing Engineering, Universiti Malaysia Pahang, 26600 Pekan, Pahang, Malaysia

[‡]School of Energy Management, Shri Mata Vaishno Devi University, Kakryal, Katra, Jammu. J&K, India

[¥]Research Centre for Nano-Materials and Energy Technology(RCNMET), School of Science and Technology, Sunway University, No.5, Jalan University Bandar Sunway, Petaling Jaya, 47500 Selangor Darul Ehsan, Malaysia

E-mail: richakothari786@gmail.com

Abstract. Among the various energy plant species, Jatropha curcus is found to be best oil-bearing plant species with a wide range of applications and having enormous economic potentials for its seed, which can be converted into biodiesel via transesterification which is an alternative to petrodiesel. In this present study, the effect of three factors, Time (1-4 hour), temperature (60-120°C) and quenching agents (NaCl, KCl, and ZrCl2) and dose of quenching agent(0.5-2gm) use for the extraction of oil from 10grm of jatropha seed. Using the Box-Benken Design (BBD) approach of Response Surface Methodology (RSM), 17 experimental runs were generated. n-Hexane was used as solvent for each experiment carried out in the 100 ml soxhlet extraction apparatus. Results obtained from the experiments were modeled and analyzed by choosing a quadratic model. From the analysis of modeled found temperature extraction time and dose of quenching agent revealed the good agreement of value (R2=0.98) between observed and predicted value of the experiment and were to be $p>0.05$ that was based on 95% confidence interval. By the use of RSM-BBD an optimizing experiment 60ºC temperature 3h time and 2gm $ZrCl_2$ quenching agent give best extraction of oil yield 41.41%.

1. Introduction

Due to decrease in reserves of fossil fuels and the growing environmental concern have made renewable energy as an attractive alternative energy source for the future in the commercial energy sector. Among the various bioenergy resources from list of renewable energy options, biodiesel is one of the promising alternative sources for diesel engine. It is defined as the mono-alkyl ester of long chain fatty acids derived from vegetable oils or animal fats and alcohol with or without a catalyst [1]. It is renewable, environmental friendly, non-toxic and readily available source. Physico-chemical properties of biodiesel are very close to those of diesel as cited by scientists and researchers [2], and consequently biodiesel or its blends can be used in diesel engines with a few or no modifications. As per the literature review, most commonly used blend is B20 comprises of 1:5 mixture of biodiesel and petroleum diesel, suggested for commercial use. The oil obtained from the substrate cannot be used as fuel directly because of it posses damage to the engine machinery at storage and combustion due to content of free fatty acid, high viscosity, acidity, etc. To convert this oil into fuel at commercial scale chemical modifications are cited by various researchers to improve the qualitative part like direct blending, transesterification, pyrolysis, and emulsification. Among these, transestrification is the key and foremost important step to reduce the cleaner and environmentally safe fuel from vegetable oil [3].

Presently, vegetable oils, animal fats, and microbial oils are dominant sources for biodiesel production. Most of vegetable oils are from edible oils (Soybean, Rapeseed). The prices of these oils are relatively high for fuel-grade production. Whereas, animal fats and microbial oils (yeast, fungi, algal) also have been converted to biodiesel [4] in search of less-expensive fuel production. Use of waste-cooking oil is also cited for biodiesel production by recent researchers in their work [5]. Therefore it is necessary to find a new feedstock for biodiesel production, which is now focused on Jatropha curcus, a non-edible plant of family Euphorbiaceous found to be growing in many parts of the country. It is rugged in nature and can be grown without large monitory requirement. The traditional consists in extracting the oil from the paste using boiling water as a solvent. According to current ratio, the price of Jatropha seen is much lower than the oil price, but as the demand of jatropha oil as biofuel increases, the cost will increases too with consideration of jatropha plantation and the product price [6]. Besides the cost also depends on the quality of the oil extracted from the seed. The increasing demand of jatropha oil also has opened up wide opportunity for global marketing which leads to the requirement of competitive products which comes in advantage in term of quality, cost and production time, hence it is best to identify a best extraction technique, as to extract higher yield of oil with higher quality at lower cost. Other than that, this small investigation with research is also conducted to improve existing extraction process towards more environmental friendly method. Existing extraction method can be improved by optimizing extraction parameters. Response surface methodology (RSM) has been used by Ahmad et al., (2018) [7] and Kirrolia et al. (2014) [8] for optimization of process. RSM is an necessary instrument for designing, formulating, developing, and analyzing the processes and production of any study. This model can be used to analyze combinations of given factor and their responses within the experimental range. In present study, jatropha seed was used extracted oil using hexane as solvent and the objective of this work was to determine the effect of temperature and time on the yield and quality of jatropha oil using RSM. Hence, focus on optimization of oil extraction from jatropha seed can enhance economic status of the seed.

2. Materials and methods

The experiment comprises of selection and collection of plant's seed for the oil after treatment with quenching agents with determination of extraction method.

2.1. Collection of jatropha seed

Jatropha seed collected from the research field station of Department of Environmental sciences, Babasaheb Bhimrao Ambedkar University, Lucknow, U.P. (India). Seed was dry at 50ºC for 24 hours using hot air oven then finally jatropha seed were grinded and sieved with 0.5 and 0.1mm sieve plate.

2.2. Experimental design for optimization of culture conditions and data analysis

Response surface methodology (RSM) is a methodical statistical design tool to discover the collective or multivariate effect of two or more variables within experiment. In the present study, jatropha seed used for extraction of oil using three level, full-factorial Box Benken design (BBD) Design using Expert software (DESv11) (Student version) with three independent variables, the dose of quenching agent (gm), extraction time (h) and extraction temperature (ºC) was applied in this study (Table 1), requiring

17 sets of experiment in triplicate runs comprising 6 replicates of center points, 6 axial (star points) and 5 factorial (cubic points).

Different analysis followed several statistical tests like Fischer's test (F-test), lack of fit, analysis of variance (ANOVA) and other tests were performed identifying the best-fitted model for optimization. In addition to that, the determination coefficient value (R2) of the model articulates the goodness of fit of the suggested model. The effect of the independent factor on dependent variable was analyzed by a Quadratic polynomial equation (Eq.1) expressed as:

$$Y = \beta_0 + \beta_A x_A + \beta_B x_B + \beta_C x_C + \beta_{AB} x_A x_B + \beta_{AC} x x_C + \beta_{AB} x_B x_C + \beta_{AA} x_A^2 + \beta_{BB} x_B^2 + \beta_{CC} x_C^2 \quad (1)$$

Where, Y is the predicted response and$x_A, x_{B,..........,} x_C$ are the variables, which affects the predicted response; $x_A^2, x_B^2 \dots\dots\dots, x_C^2$are the square or quadratic effect of variables; $x_A x_B, x_A x_C \dots\dots, x_B x_C$ are the interaction effect of variables;,β_0 is the intercept;$\beta_A, \beta_B, \beta_C$ is the linear effect $\beta_{AA}, \beta_{BB}, \beta_{CC}$ is the effect of squared; β_{AB},β_{AC}, β_{BC} is the effect of interaction.

After the experimental a point prediction analysis (PPA) done for the optimization according to previous experimental analysis.

Table 1. CODED LIMIT FOR FACTORS USED IN THE EXPERIMENTAL DESIGN

Factor	Unit		Coded level		
			-1	**0**	**+1**
Dose of quenching agent	**gm**	X_1	0.5	1.5	2.5
Time	**Hour**	X_2	1	3.5	6
Temperature	**°C**	X_3	30	45	60

2.3. Quenching agent

In present study, Zirconium chloride (ZrCl2) (Analytical grade) used as a quenching agent for extraction of oil from jatropha seed.

2.4. Extraction of oil from jatropha seed

40 gm of cursed jatropha seed was taken in the cellulose thimble with 150 ml of solvent (Hexane) for extraction by the soxhlet extraction unit fig. (1). The reaction was processed for different time interval and different temperature according to BBD-RSM design. After completing the extraction process the percent yield of jatropha oil calculated by using Eq.(1).

$$\%\, yield\ of\ oil = \frac{(Yi - Yf)x100}{Yi}$$

Yi; Initial weight of seed, Yf; Final weight of oil

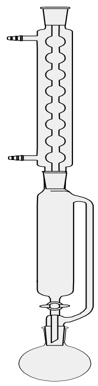

Figure 1. Soxhlet extraction unit

3. Results and Discussions

In order to achieve high yield of extraction from jatropha seed, the factor (dose of quenching agent, time and temperature) taken for extraction and optimizing the maximum % yield. The extraction yield varies between 14.54-46.56% respectively for different combination as shown in Table.2

Statistical and mathematical analysis of Yex% observed by the comparative variation between using predicted and actual observation as shown in Fig.2 and found that predicted and actual value of extraction yield is distributed around the central line and noticed the minimal error. The given regression analysis supporting the results and not any distortion or variation is observed as shown in Table.3 and assured with high degree of accuracy in the experimental data. The best fitted BBD model response for Extraction yield are composed of linear coefficient (X_1, X_2, and X_3) quadratic coefficient (X_1^2, X_2^2 and X_2^3) and Interactive coefficient (X_1X_2, X_1X_3 and X_2X_3) used for the development of predicted responses for the extraction yield by using a quadratic Eq.2.

$$\text{Extraction Yield}=-13.31+28.84*X_1-0.86*X_2+0.64*X_3+0.12*X_1*X_2+0.25*X_1*X_3+0.088*X_2*X_3-0.57*X_1^2-0.34*X_2^2-9.9x10^{-3}*X_3^2 \quad (2)$$

Table 2. MATRIX OF BOX BENKEN DESIGN (BBD) FOR CONDITION WITH OPTIMIZATION FOR EXTRACTION OF JATROPHA OIL PE

Factors				Responses	
S.N	**X_1**	**X_2**	**X_3**	**Y_{Exp}**	**Y_{obs}**
1	0.5	1	45	15.76	15.76
2	1.5	3.5	45	39.76	39.17
3	2.5	3.5	30	23.56	24.89
4	1.5	3.5	45	38.54	39.17
5	1.5	3.5	45	38.54	39.17
6	1.5	3.5	45	39.12	39.17
7	2.5	3.5	60	45.67	45.39
8	1.5	6	30	28.54	27.21
9	2.5	6	45	37.76	37.76
10	0.5	3.5	60	21.67	20.34
11	0.5	6	45	18.54	19.59
12	1.5	1	60	34.45	35.78
13	1.5	3.5	45	39.87	39.17
14	2.5	1	45	33.76	32.71
15	1.5	6	60	46.56	46.84
16	1.5	1	30	29.65	29.37
17	0.5	3.5	30	14.54	14.82

X_1: Quenching agent dose, X_2: Extraction time, X_3: Extraction temperature

Table 3. ANALYSIS OF VARIANCE (ANOVA) FOR EXTRACTION OF JATROPHA OIL

Source	df	Sum^2	Mean Square	F-value	p-value
Model	9	1633.	181.47	113.2	< 0.0001
X_1	1	616.7	616.71	384.8	< 0.0001
X_2	1	39.52	39.52	24.66	0.0016
X_3	1	338.7	338.78	211.4	< 0.0001
X_1X_2	1	0.372	0.3721	0.232	0.6446
X_1X_3	1	56.10	56.10	35.0	0.0006
X_2X_3	1	43.69	43.69	27.2	0.0012
X_1^2	1	470.9	470.91	293.8	< 0.0001
X_2^2	1	19.20	19.20	11.98	0.0105
X_2^3	1	20.95	20.95	13.0	0.0086
Lack fit	3	9.58	3.19	7.8	0.0378
Error	4	1.63	0.4086		
Cor Total	16	1644			
Std. Dev.		1.27			
Mean		32.13			
C.V. %		3.94			
R^2 Adj		0.99			
R^2Pred		0.98			
R^2		0.90			

According to statistical and mathematical analysis the generated response show good agreement with predictive response shown in Table.3 and best-fitted model responses Linear responses (X_1, X_2, and X_3), interactive response (X_1X_3 and X_2X_3) and quadratic responses (X_1^2,X_2^2 and X_2^3). The appropriateness of all the parameter was verified by the analysis of variance (ANOVA) and F test was performed to each model for confirming the significance of the model due to high F value [9].

If the F value less than the 4, the model is not significant but in present study, the value of F value 113.24 which determine model is highly significant.

The Fitting efficiency of the Quadratic model regression equation was used to determine the coefficient (R^2) and an adjustable coefficient (R^2_{adj}). The goodness of fitting for the correctness of quadratic model which is predicted by predicted responses (R_{2Pred}; 0.98) and adjustable responses (R_{2adj}:0.99) indicating the high degree of agreement between experimental and predicted. The model could predict the response to explain 95% of the variability which describe the pure error (Prob>F) to check the significance of regression coefficient. The contribution of individual operating variable toward extraction of oil from jatropha seed maximum 62% contribution by quenching agent and 34% by temperature and 4 % extraction time. The deviation of contribution variable (Quenching agent >temperature>time) for extraction of oil. For the interactive behavior of variables shows the (Quenching Agent: Time> time: Temperature >Quenching Agent: Temperature) responsible for the extraction of oil from jatropha seed. Maximum interactive contribution by Quenching Agent: Time. Kartika et al. [10] only work on the temperature reaction time and methanol to seed ratio (2:1–6:1), was examined to extraction of jatropha oil not involve any type of quenching agent.

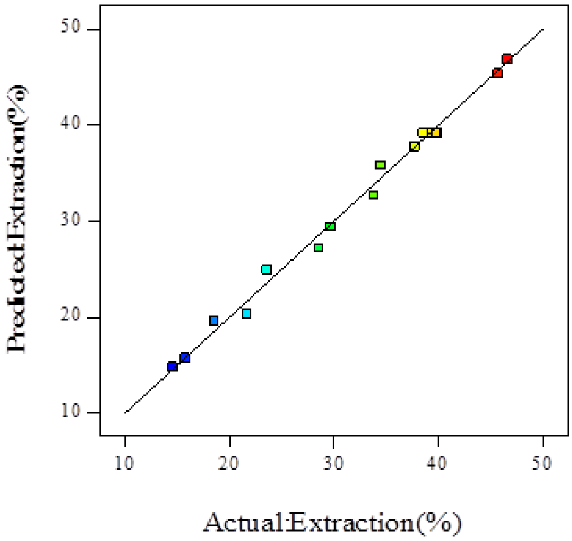

Figure 2. Relation between actual and predicted value of extraction of oil from jatropha seed

The 3D surface plot was drawn to show the interactive effect of variables toward the responses, it also shows the effect of independent variable on the dependent. It was observed from, the graph that interaction between every two variables with maximum prediction of extraction as shown in fig 3a-c.

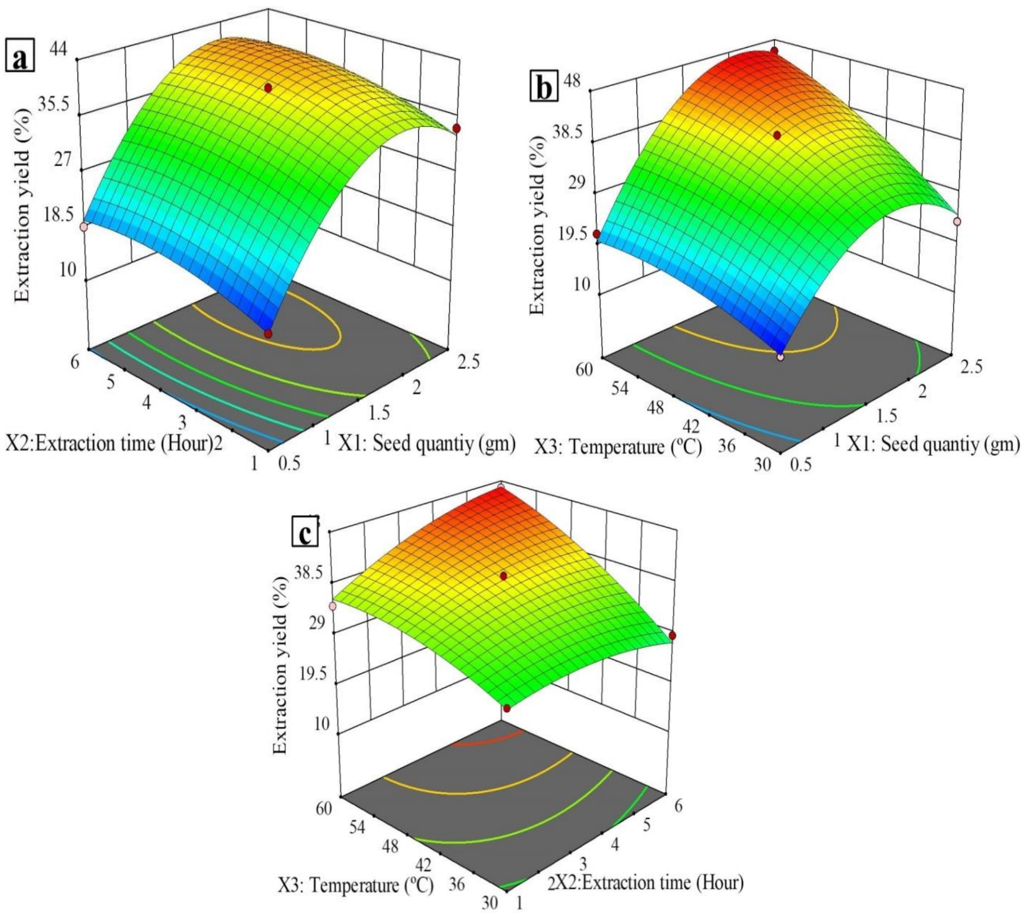

Figure 3. 3D Representation of Biodiesel yield Optimization of variables for extraction jatropha oil

Extraction of oil factor determined by the dose of quenching agent, extraction time and temperature were analyzed for the optimization condition using the point prediction analysis and found different result if variables are changing from lower to higher but for high extraction of oil with using minimal requirement of factor (Quenching agent, Time and Temperature) as shown in fig.3(a-c) provide different results. If minimum quantity of quenching agent (0.5g) used with 1 hour extraction time and 30ºC temperatures give only 14.67% extraction yield but the factor condition is reverse give maximum yield of extraction oil *i.e.* 49%.

4. Conclusions

In this present experimental study, second-order mathematical and statistical model used to predict the variable such as dose of quenching agent , extraction time and temperature for the extraction of oil from jatropha seeds based on box Benken design on response surface methodology (BBD-RSM). Result obtained from the mathematical analysis compared with the experimental study with good agreement of fitness because its estimated value was 0.99. The model developed by BBD-RSM was useful for predicting the optimal extraction condition to achieve maximum extraction. The achievement from the optimizing experiments and found best extraction of oil yield (41.41%) at 60ºC temperature with 3h time and 2gm $ZrCl_2$ quenching agent.

Acknowledgement

The Author and Co-author of this article are very thankful to UGC India for providing financial support and very thankful to head of Department of Environmental Science Babasaheb Bhimrao Ambedkar university Lucknow for providing consistent support for access of laboratory.

References

[1] Kamani MH, Eş I, Lorenzo JM, Remize F, Roselló-Soto E, Barba FJ, et al. Advances in plant materials, food by-products, and algae conversion into biofuels: use of environmentally friendly technologies. *Green Chemistry*. 2019;**21**(12):3213-31.

[2] Anguebes-Franseschi F, Bassam A, Abatal M, May Tzuc O, Aguilar-Ucán C, Wakida-Kusunoki AT, et al. Physical and Chemical Properties of Biodiesel Obtained from Amazon Sailfin Catfish (Pterygoplichthys pardalis) Biomass Oil. *Journal of Chemistry*. 2019;**2019**:7829630.

[3] Kesieme U, Pazouki K, Murphy A, Chrysanthou A. Biofuel as an alternative shipping fuel: technological, environmental and economic assessment. *Sustainable Energy & Fuels*. 2019;**3**(4):899-909.

[4] Li Q, Du W, Liu D. Perspectives of microbial oils for biodiesel production. *Applied Microbiology and Biotechnology*. 2008;**80**(5):749-56.

[5] Ahmad S, Kothari R, Pathak VV, Pandey MK. Fuel Quality Index: A Novel Experimental Evaluation Tool for Biodiesel Prepared from Waste Cooking Oil. *Waste and Biomass Valorization*. 2019;**10**(8):2237-47.

[6] Ramírez V, Martí-Herrero J, Romero M, Rivadeneira D. Energy use of Jatropha oil extraction wastes: Pellets from biochar and Jatropha shell blends. *Journal of Cleaner Production*. 2019;**215**:1095-102.

[7] Ahmad S, Pathak VV, Kothari R, Kumar A, Naidu Krishna SB. Optimization of nutrient stress using C. pyrenoidosa for lipid and biodiesel production in integration with remediation in dairy industry wastewater using response surface methodology. *3 Biotech*. 2018;**8**(8):326.

[8] Kirrolia A, Bishnoi NR, Singh R. Response surface methodology as a decision-making tool for optimization of culture conditions of green microalgae Chlorella spp. for biodiesel production. *Annals of microbiology*. 2014;**64**(3):1133-47.

[9] Ahmad S, Kothari R, Pathania D, Tyagi VV. Optimization of nutrients from wastewater using RSMfor augmentation of Chlorella pyrenoidosa with enhanced lipid productivity, FAME content, and its quality assessment using fuel quality index. *Biomass Conversion and*

Biorefinery. 2020;**10**(2):495-512.
[10] Amalia Kartika I, Yani M, Ariono D, Evon P, Rigal L. Biodiesel production from jatropha seeds: Solvent extraction and in situ transesterification in a single step. *Fuel*. 2013;**106**:111-7.