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Parametric optimization of rice bran oil extraction using response surface methodology

Syed W. Ahmad[1], Farhan Javed[1], Sajjad Ahmad[*2], Muhammad Akram[3], Abdur Rehman[2]

[1]*University of Engineering & Technology (Lahore) Faisalabad Campus, Department of Chemical & Polymer Engineering*
[2]*University of Engineering & Technology (Lahore) Faisalabad Campus, Department of Basic Sciences and Humanities*
[3]*University of Engineering &Technology (Lahore) Faisalabad Campus, Department of Electrical and Electronics Engineering*
[*]*Corresponding author: e-mail: sajjad.ahmad@uet.edu.pk*

Use of bran oil in various edible and nonedible industries is very common. In this research work, efficient and optimized methodology for the recovery of rice bran oil has been investigated. The present statistical study includes parametric optimization, based on experimental results of rice bran oil extraction. In this study, three solvents, acetone, ethanol and solvent mixture (SM) [acetone: ethanol (1:1 v/v)] were employed in extraction investigations. Response surface methodology (RSM), an optimization technique, was exploited for this purpose. A five level central composite design (CCD) consisting four operating parameter, like temperature, stirring rate, solvent-bran ratio and contact time were examined to optimize rice bran oil extraction. Experimental results showed that oil recovery can be enhanced from 71% to 82% when temperature, solvent-bran ratio, stirring rate and contact time were kept at 55°C, 6:1, 180 rpm and 45 minutes, respectively while fixing the pH of the mixture at 7.1.

Keywords: solvent extraction, rice bran oil, response surface methodology (RSM), central composite design (CCD).

PRACTICAL APPLICATION

A large amount of rice bran is being wasted from rice processing industries. It is worth noting that rice bran contains 13 to 18 percent oil, depending on climate, type of soil and method of cultivation. So, rice bran can be an eminent source of raw material for several edible and non-edible industries in agricultural countries. Therefore, an economic and efficient method for the recovery of rice bran oil should be required to utilize the wastage of rice mills. The current studies composed of parametric optimization, which is required to collect the data for a cost effective process design.

INTRODUCTION

Rice bran is the by-product of rice milling process. It is a natural source of minerals, antioxidants and oil and its composition is similar to that of peanut oil[1]. The constituents of the bran depends on the number of factors like, type of rice, origin, soil composition, climate, rice processing procedures and method of cultivation[2, 3]. In the world, annual rice production is more than 738 million ton. On average, depending upon the rice variety and milling process, rice processing units generate 10–12% rice bran as a byproducts. Rice bran contains approximately 12 to 25% oil that can be a significant source of oil in an agricultural country[4]. Further, rice bran oil can be utilized as raw material in both edible and nonedible industries[5, 6]. Typical edible applications of rice bran oil can be in pharmaceutical and cooking oil industries whereas utilization of rice bran oil in cosmetics, soap and paint industries are among the nonedible applications[7–9].

Both mechanical and physical means can be utilized to extract the oil from rice bran[10, 11]. Solvents like subcritical carbon dioxide and subcritical water have been used for oil extraction from rice bran[1, 11, 12]. Among organic solvents, hexane showed maximum oil recovery from rice bran[13]. However, due to noxious and harmful properties of hexane its use as a solvent is avoided[14]. Ethanol, being its environmentally friendly nature, was used as a solvent for the infusion of edible oil from soybeans[15, 16]. Moreover, extraction parameters, like solvent-bran ratio, temperature, stirring rate and contact time, can change the amount of extracted oil[1, 13, 17]. In our previous work, recovery of bran oil by means of solid-liquid extraction technique has been investigated by employing various combinations of ethanol and acetone. In present study, role of RSM has been exploited to enhance the efficiency in rice bran oil extraction process.

RSM a combination of mathematical and statistical technique, has been used for optimization, development and improvement of several processes[18]. It is an eminent tool for statistical modeling of response variable, i.e. the variable to be optimized. It is worth noting that RSM can effectively be applied for the development of numerical models based on experimental investigations[19]. RSM can be utilized in design optimization and successfully reduce the cost and noise associated with analysis method. Moreover, RSM has frequently been used for the optimization of extraction processes[20]. It is also noted that RSM has superiority over full factorial design due to its short cycle, highly accurate regression equation, simple in calculations and requirement of less number of experimental runs[20, 21]. So, RSM is an effective statistical tool for investigation of complex processes where numerous factors and their interaction affect the desired response[22].

The present investigations comprised of determining the best suited solvent between ethanol, SM and acetone, which was selected on the basis of highest extraction constant. In the study, using best suited solvent, the parametric optimization of rice bran oil extraction process was performed by employing RSM on the basis of CCD. Optimization and experimental design was carried out with the help of Design Expert Software Version 7.0.

MATERIAL AND METHODS

Material

Lab grade acetone and ethanol were obtained from Merck Germany. Raw rice bran, in raw form, was collected from Adam Rice Mills Faisalabad, Pakistan. SM was prepared by mixing ethanol and acetone in equal volume ratio.

Equipment

Solid liquid extraction of rice bran oil was carried in an isothermal batch extraction unit that was fitted with a thermostat, a stirrer and rpm controller assembly. After extraction, the solvent was recovered in a laboratory scale batch distillation unit (see[23] for further details).

Extraction procedure

Thirty experimental runs were designed and performed for the oil extraction from rice bran on the basis of CCD matrix. The detailed experimental procedure was explained in[23]. For optimization, the effect of parameters, like solvent-bran ratio, stirring rate, temperature and contact time at specific level as depicted in Table 3, while pH was kept at 7.1. The percentage recovery of oil from rice bran can be calculated using equation (1).

$$R = \frac{m_e}{m_s} \times 100 \quad (1)$$

where

R= percentage oil recovery,

m_e= mass of oil extracted [g]

m_s=total mass of oil in rice bran sample [g].

Solvent selection

Three solvents were used for solid-liquid extraction of rice bran oil. The suitability of solvent was determined on the basis of extraction constant, which can be calculated by equation (2)[24].

$$\frac{dC}{dt} = kA(C_o - C) \quad (2)$$

On integration

$$\ln\left(1 - \frac{C}{C_o}\right) = -kt \quad (3)$$

where

C = mass of extracted oil [g]

C_o = initial mass of oil [g]

k = extraction constant [min^{-1}]

t = extraction time [min].

For solvent selection, 40 g of stabilized rice bran was treated with 200 ml of pure ethanol, while temperature, stirring rate, solvent-bran ratio and pH were kept at 50°C, 90 rpm, 5.1, and 7.1, respectively (see[23] for further details). The experiments were repeated for acetone and SM (acetone and ethanol (1:1 v/v)). The percentage recovery of oil was calculated after 30 min, 35 min, 40 min, 45 min and 50 min using aforementioned solvent. The data was plotted as shown in Figure 1. It can be deduced that the value of extraction coefficient was the highest for SM, i.e. 0.0047 min^{-1}, see Figure 1. Further, for optimization studies, extraction experiments were performed using SM as a solvent.

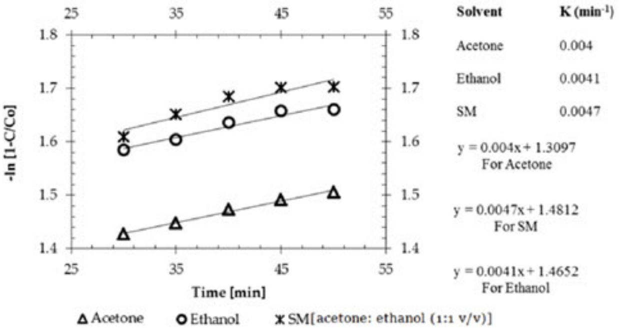

Figure 1. Plot of un-extracted rice bran oil against contact time

Experimental design

CCD of RSM was utilized for parametric optimization of rice bran oil extraction. Following parameters were optimized when pH was kept at 7.1.

A = Temperature [°C]

B = Stirring rate [rpm]

C = Solvent-bran ratio

D = Contact time [min].

The rice bran oil recovery can be represented mathematically as follows, see equation (4):

$$R = f(A, B, C, D) \quad (4)$$

where

R = response or the percentage oil recovery,

f = response function,

A, B, C and D = variables or factors.

A five level CCD consisting of four factors, like temperature, stirring rate, solvent-bran ratio and contact time was developed. The coded and un coded levels of the independent variables have been represented in Table 1 and can be related using equation (5) given below as described by[25].

$$X = \frac{x_i - x_o}{\Delta x} \quad (5)$$

where

X = coded value of independent variable,

x_i = actual value of independent variable,

x_o = midpoint value of actual variable,

Δx = interval of original range.

Thirty experimental runs were resulted from CCD with sixteen factorial points, eight axial and six replicates of central point. The CCD experimental design layout matrix containing actual and predicted response corresponding

Table 1. Coded and un-coded levels of independent variables

Symbol	Variable	Units	Levels				
			–α	–1	0	1	α
A	Temperature	°C	40	45	50	55	60
B	Stirring rate	rpm	90	120	150	180	210
C	Solvent-bran ratio		3	4	5	6	7
D	Contact time	min	30	35	40	45	50

to the experimental runs, which have the values of variables at various levels, have been depicted in Table 2.

It can be observed from Table 2 that the maximum (i.e. 83%) and minimum (i.e. 71.1%) values of oil recovery were obtained at experimental run nos. 16 and 12, respectively. The experimental results were analyzed using RSM to fit a second order polynomial, see equation (6).

$$r = \beta_o \pm \sum_{i=1}^{n} \beta_i X_i \pm \sum_{i=1}^{n} \beta_{ii} X_i^2 \pm \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{ij} X_i X_j \quad (6)$$

where
r = the response,
β_o = constant term,
β_i = regression coefficient of the linear terms,
β_{ii} = regression coefficient of quadratic terms,
β_{ij} = regression coefficient of interaction terms,
X_i, X_j = coded variables,
n = number of independent variables.

RESULTS AND DISCUSSION

Model Fitting & Summary Statistics

As discussed in previous section, for optimization of rice bran oil recovery, four extraction parameters were investigated namely; temperature, stirring rate, solvent-bran ratio and contact time. The statistical summary of rice bran oil extraction utilizing CCD has been shown Table 3. Based on statistics, the highest order polynomial model was selected where model is not alias. Insignificant lack of fit was achieved and by focusing on maximizing the adjusted R-squared and predicted R-squared values, the linear model was suggested for the rice bran oil extraction. The resulted linear model in terms of coded variables is as follows:

$$R = 77.51 + 2.63A + 1.09B + 1.13C + 0.29D \quad (7)$$

While the model in terms of un coded or actual variables is given by the equation (8)

Percentage oil recovery = 37.81600 + 0.52592 Temperature + 0.036458 Stirring rate + 1.12708 Solvent bran ratio + 0.057250 Contact time (8)

Analysis of Variance (ANOVA)

Design Expert Software version 7.0 was used to develop a linear model. Further, summary of analysis of variance (ANOVA) technique was used to evaluate how good the model fits, see Table 4. The regression analysis showed that the coefficient of determination (R-squared) value was 88.26%, which is an indication that the proposed model can explain 88.26% variations. Therefore, it can be concluded that the accuracy and the fitness of linear model was adequate. From Table 3, it may be observed that the model p-value probability F is less than 0.05, which is an indication that the conditions in model are significant. Further, the model F-value of 46.96 implies the model is significant. For the current linear model, the values of probability F are less than 0.05 for factors A, B and C that indicates the model terms are significant. It is also worth noting that factor A, B and C mainly

Table 2. CCD layout

	Coded values				Actual (un-coded) values				Response [Percentage Oil recovery] R [%]	
Run	Temperature [°C] A	Stirring rate [rpm] B	Solvent-bran ratio C	Contact time [min] D	Temperature [°C] A	Stirring rate [rpm] B	Solvent-bran ratio C	Contact time [min] D	Experimental value	Predicted value
1	1	1	-1	-1	55	180	4	35	78.31	79.81
2	0	-2	0	0	50	90	5	40	75.95	75.04
3	-1	-1	-1	1	45	120	4	45	71.31	72.66
4	0	2	0	0	45	120	4	45	80.8	79.98
5	-1	-1	1	-1	45	120	6	35	74.63	74.63
6	0	0	0	2	50	150	5	50	77.93	78.09
7	-1	1	-1	1	45	180	4	45	75.2	75.13
8	-1	1	1	-1	45	180	6	35	76.21	76.81
9	-1	1	1	1	45	180	6	45	77.4	77.39
10	1	-1	1	1	55	120	6	45	79.00	80.47
11	0	0	0	0	50	150	5	40	78.5	77.51
12	-1	-1	-1	-1	45	120	4	35	71.1	72.37
13	1	1	1	-1	55	180	6	35	82.12	82.07
14	-1	1	-1	-1	45	180	4	35	74.63	74.55
15	-2	0	0	0	40	150	5	40	71.33	72.25
16	2	0	0	0	60	150	5	40	83.00	82.77
17	-1	-1	1	1	45	120	6	45	77.56	75.21
18	0	0	-2	0	50	150	3	40	75.80	75.25
19	0	0	0	0	50	150	5	40	77.66	77.51
20	1	1	-1	1	55	180	4	45	79.9	80.39
21	0	0	0	0	50	150	5	40	78.44	77.51
22	1	-1	1	-1	55	120	6	35	80.00	79.89
23	1	1	1	1	55	180	6	45	82.43	82.65
24	0	0	0	0	50	150	5	40	77.51	77.51
25	0	0	0	0	50	150	5	40	76.9	77.51
26	0	0	0	0	50	150	5	40	80.01	77.51
27	0	0	2	0	50	150	7	40	77.9	79.77
28	1	-1	-1	-1	55	120	4	35	77.82	77.63
29	1	-1	-1	1	55	120	4	45	78.23	78.21
30	0	0	0	-2	50	150	5	30	77.6	76.93

Table 3. Satatistical summary of CCD

Sequential Model Sum of Squares [Type I]						
Source	Sum of squares	df	Mean square	F value	P value prob>F	
Mean vs Total	1.802E+005	1	1.802E+005			
Linear vs Mean	227.12	4	56.78	46.96	< 0.0001	Suggested
2FI vs Linear	2.55	6	0.43	0.29	0.9332	
Quadratic vs 2FI	6.04	4	1.51	1.05	0.4163	
Cubic vs Quadratic	14.97	8	1.87	1.97	0.1940	Aliased
Residual	6.66	7	0.95			
Total	1.805E+005	30	6015.76			
Lack of Fit Tests						
Source	Sum of squares	df	Mean square	F value	P value prob>F	
Linear	24.35	20	1.22	1.04	0.5377	Suggested
2FI	21.79	14	1.56	1.32	0.4036	
Quadratic	15.76	10	1.58	1.34	0.3928	
Cubic	0.78	2	0.39	0.33	0.7309	Aliased
Pure Error	5.88	5	1.18			
Model Summary Statistics						
Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	Press	
Linear	1.10	0.8826	0.8638	0.8328	43.03	Suggested
2FI	1.21	0.8925	0.8359	0.6568	88.33	
Quadratic	1.20	0.9159	0.8375	0.6144	99.23	
Cubic	0.98	0.9741	0.8928	0.5280	121.48	Aliased

affect the response. Moreover, lack of fit was insignificant while the predicted R-squared was in reasonable agreement with adjusted R-squared, Table 4.

Model Diagnostic Plots

The diagnostic plots of model are used to evaluate the fitness of model to the analysis of variation (ANOVA) assumptions. The diagnostic graph consists of the normal probability plot of the residuals, which has been depicted in Figure 2. The normal probability plot diagnosis whether the residual data follows a normal distribution, i.e. fits in a straight line. It is worth noting that moderate scatter data may be considered as normal distribution. In contrary, a non-linear scatter of data is an indication of abnormalities in term of error that can be minimized using transformation[22]. It can be deduced from Figure 2 that the current residual data has been closely scattered around the straight line, so the proposed model is considered as normal. The predicted values of response were calculated form linear regression model. The predicted values were plotted against the actual response values, which were obtained from experimentations, see Figure 3. It can be deduced that the diagnostic plot, i.e. Figure 3, represents a linear relationship between predicted and actual values that shows the adequacy of the proposed model.

Response Surface Plots

The 3D response surface plots and the contour plots of the model have been shown in Figure 4 to Figure 7 that indicate the analysis of the interaction effects

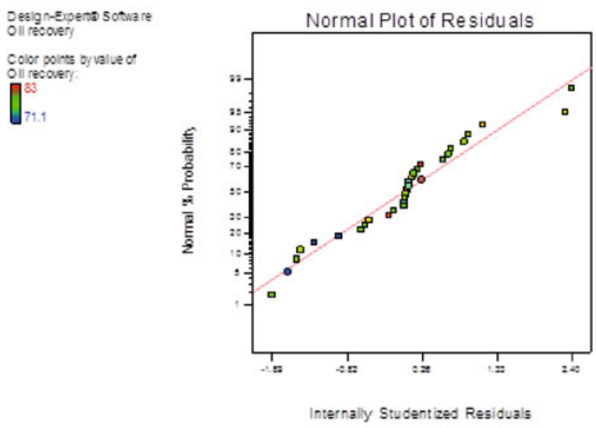

Figure 2. Normal plot of residuals

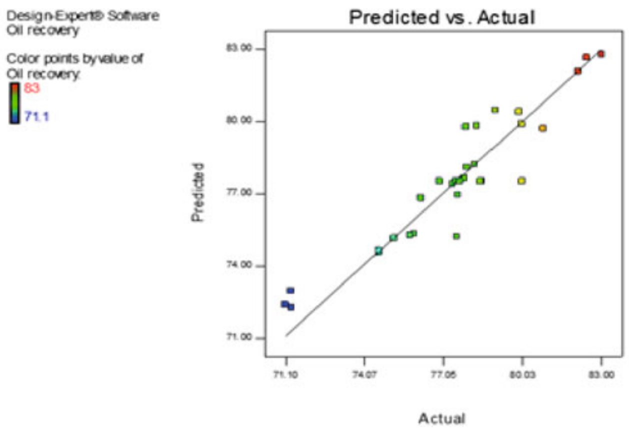

Figure 3. Plot of predicted & actual values of response

Table 4. Analysis of variance (ANOVA) for regression model

Source	Sum of squares	df	Mean square	F value	P value prob>F	
Model	227.12	4	56.78	46.96	< 0.0001	Significant
A-Temperature	165.95	1	165.95	137.27	< 0.0001	
B-Stirring rate	28.71	1	28.71	23.75	< 0.0001	
C-Solvent-bran ratio	30.49	1	30.49	25.22	< 0.0001	
D-Contact time	1.97	1	1.97	1.63	0.2139	
Residual	30.22	25	1.21			
Lack of Fit	24.35	20	1.22	1.04	0.5377	Not significant
Pure Error	5.88	5	1.18			
Cor Total	257.34	29				

of the process variables. The contour plots are the graphical representation of the regression equation for optimization[26]. Further, the contour plots, Figure 4a, and 3D surface plot of response, Figure 4b, indicates the effect of interaction of temperature and stirring rate on percentage recovery of rice bran oil. It can be deduced that the temperature is a highly significant parameter that can affect the percentage oil recovery. As there was an increment in temperature with an increase in stirring rate, the percentage of recovered oil also augmented. The reason may lies in the argument that the solubility of oil in particular solvent increases with temperature elevation, consequently, the value of percentage oil recovery increases, similar experimental observations were reported by[27].

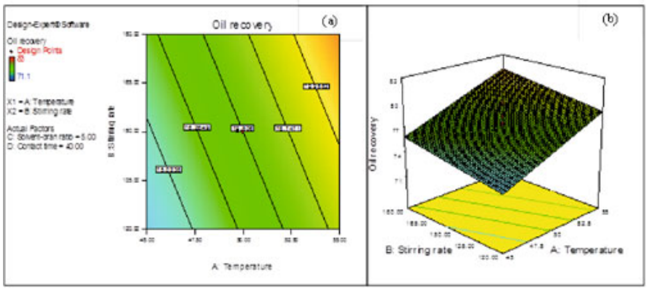

Figure 4. (a) contour plot, (b) 3d surface plot of response with respect to temperature with stirring rate

The contour plot and 3D surface plot of the response has been shown in Fig. 5a and 5b, respectively, which indicate the effect of interaction of solvent-bran ratio and temperature on percentage oil recovery from the solvent extraction process. It can be observed that as solvent-bran ratio increases with an increment in temperature, the percentage recovery of oil from rice bran, the value of response, increases. Further, increment in response value is less sharp as compared to that of due to the temperature increment. So, solvent-bran ratio can affect the amount of extracted oil. Consequently, it can be considered as a significant parameter. This increment in the response value, i.e. percentage recovery of rice bran oil, with the increment of solvent-bran ratio is due to the availability of fresh surface for oil extraction.

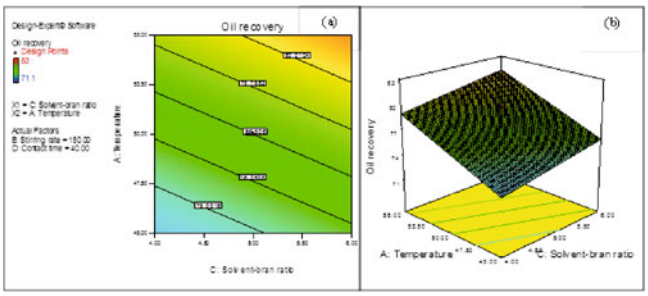

Figure 5. (a) contour plot, (b) 3d surface plot of response with respect to solvent–bran ratio with temperature

The interaction effect of solvent-bran ratio and contact time has been depicted in Figure 6a, (contour plot), and Figure 6b, (3D surface plot of response). It can be deduced that as the contact time increases with the increment in solvent-bran ratio, the change in percentage recovery of oil is not prominent. So, the contact time is

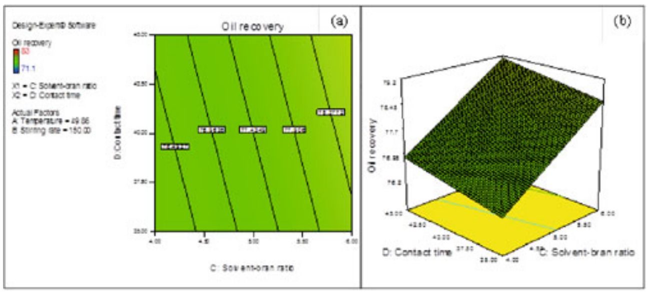

Figure 6. (a) contour plot, (b) 3d surface plot of response with respect to contact time with solvent–bran ratio

an insignificant parameter as compared to solvent-bran ratio. The insignificant effect of contact time in the response, i.e. percentage recovery of rice bran oil, is due to the fact that, initially, driving force (concentration gradient) is high. With the passage of time, concentration gradient decreases. Consequently, contact time become an insignificant parameter.

The interaction effect of stirring rate and contact time has graphically been represented in Figure 7a and 7b. It can be observed that as the stirring rate increase with the increment in contact time, the percentage recovery of rice bran oil also augments. It is an indication that the stirring rate is a significant parameter that can affect the percentage recovery of oil from rice bran as compared to that of contact time. This fact can be supported by the argument that with increasing stirring rate, the diffusion of oil from solid to liquid phase enhances. Consequently, the percentage recovery of oil increases.

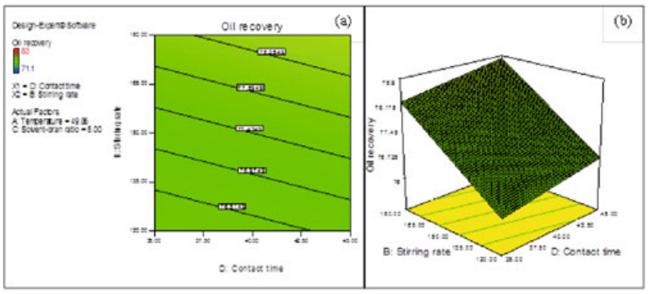

Figure 7. (a) contour plot, (b) 3d surface plot of response with respect to stirring ratio with contact time

Optimization of extraction parameters

With the aid of numerical optimization technique, set of process variables has been found, that gives the maximum value of response function at highest desirability. So the numerical optimization of response function resulted in 29 solutions. The parametric optimization of the extraction of oil from rice bran has been carried out using Design Expert Software version 7.0. In addition, as discussed earlier, the process model has successfully been developed and validated. Further, the objective of optimization is to maximize the response function, i.e. percentage recovery of rice bran oil, within lower and upper limits of 71% and 83%, respectively. The optimum values of temperature, stirring rate, solvent-bran ratio and contact time have been found as 55°C, 180 rpm, 6.1 and 45 minutes, respectively, when predicted oil recovery is 82.6369% at desirability of 0.969. The experimental run was performed at the optimum values set of parameters and was verified.

CONCLUSIONS

The extraction of oil from rice bran was carried out using solid-liquid extraction technique. Three solvents namely, ethanol, acetone and SM, were utilized for extraction study. It was determined that SM was the best solvent on the basis of the highest value of extraction constant, i.e. 0.0047 min^{-1}, as compared to ethanol and acetone. Further, parametric optimization was performed with the best suited solvent. The extraction parameters, like temperature (A), stirring rate (B), solvent-bran ratio (C) and contact time (D) were optimized using RSM under CCD. For optimization, a linear model was developed and validated. Analysis of variance (ANOVA) was performed, which indicates that temperature is the prime parameter that can affect percentage recovery of oil from rice bran, further, solvent-bran ratio and stirring rate were found significant. However, contact time was found insignificant parameter and its effect on percentage recovery of rice bran oil was not prominent. It was found that maximum recovery of rice bran oil, i.e. 82.64% with 0.969 desirability, was determined when temperature, solvent-bran ratio, stirring rate and contact time were 55°C, 6.1, 180 rpm and 45 min, respectively.

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