Research Article

Evaluation of Process Parameters on Rice Bran Oil Yield in Lab Scale Automated Solvent Extractor Using Central Composite Design

Ruby Pandey[1,*] and S L Shrivastava[2]

[1]Centurion University of Technology and Management, Odisha, India
[2]Agricultural and Food Engineering, Indian Institute of Technology Kharagpur, Kharagpur, India

Abstract

Automated solvent extractor is a modified design of soxhlet for solvent extraction of oil where extraction and miscella collection takes place in the same chamber unlike soxhlet. Fresh solvent continuously percolates through the solid matrix thus increasing the rate of extraction. The present study has been carried out to evaluate the effect of process parameters for rice bran oil extraction in automated solvent extractor using hexane as the solvent. Effect of three different parameters *viz.* solvent to bran ratio, extraction time, and heater temperature on the rice bran oil yield and free fatty acid (FFA) content has been evaluated. Response surface methodology with rotatable central composite design has been used to optimize the process parameters at five different levels: solvent to bran ratio (1.2, 1.5, 2, 2.5 and 2.8 g solvent/g bran), extraction time (2, 5, 10, 15 and 18 minutes), and heater temperature (60°C, 90°C, 135°C, 180° and 210°C). The extraction efficiency was determined on the basis of percentage oil yield. All the parameters significantly influenced the oil yield (p value < 0.001). No significant effect of any of the parameters was found on the FFA content of oil. Well-fitting model was successfully established for oil yield from rice bran (Adjusted R^2 = 0.97) through multiple regression with backward elimination.

Keywords: Rice Bran Oil, Optimization, Solvent Extraction, Central Composite Design, Automated Solvent Extractor

***Correspondence**
Author: Ruby Pandey
Email: ruby.pandey@cutm.ac.in

Introduction

Rice bran oil (RBO) is one of the most nutritious oil among all the edible oils due to its balanced fatty acid profile and presence of bioactive and antioxidants compounds such as γ-oryzanol, tocopherol, tocotrienol and phytosterols [1]. In addition, it also contains a good amount of wax esters that are reported to be the source of policosanols [2].

Rice bran is a cuticle between rice husk and grain. Depending upon the variety of rice and degree of milling, it contains around 12-25 % oil [3] which is generally extracted using solvents, commonly hexane [4]. It is an underused co-product of rice milling [5]. Its value is partially captured through extraction and refining of RBO. Still, there exists wide gap between the production and utilization of rice bran. The annual RBO production potential in world is around 3.5 MT out of which only 1.5 MT is produced presently [6]. Abundant paddy production and bran being cheap source of oil (12-25% oil) are the main reasons behind utilisation of rice bran for oil extraction. Also, de-oiled bran being a good source of protein and fiber has a good commercial value. As per the current situation, RBO is extracted using food grade hexane by keeping bran in contact with excess amount of hexane for long time (2-2.5 hours)[7].

The paddy processing capacity of most of the rice mills in developing nations are 2-4 tonnes/hour. Considering on an average 5% degree of polishing and 16 hours of processing per day, the bran produced in these mills ranges from 1.6-3.2 tonnes/day. However, currently available RBO extraction plants have the bran processing capacity between 5-500 tonnes/day. This means that for full utilization of the plant capacity, bran needs to be collected and stored for 2-3 days. This requires storage space and bran stabilization which adds to the cost of production. Also, rise in FFA during storage causes heavy loss in refining. Rice bran crude oil with less than 5% FFA is desirable for refining [8]. If FFA is more than 5%, the oil is used for soap manufacture or other industrial purposes. On top of that, it is uneconomical and unaffordable for the small rice millers to install large scale solvent extraction unit for rice bran oil production. Hence, bran from these mills is wasted. It is to note that the transportation of bran from mills to the extraction unit causes rapid rise in its FFA content which renders it unfit for extraction of edible grade oil.

Solvent extraction of solid samples commonly known as solid-liquid extraction or leaching is one of the oldest methods of oil extraction. Among all the techniques, soxhlet is the mostly used since long time [9]. In this, sample is placed in thimble holder which is gradually filled with condensed fresh solvent from distillation flask. Once liquid reaches the overflow level, miscella from the thimble holder is siphoned back to distillation flask through siphon arm. This is called one cycle and like this around 72 cycles are run to achieve complete extraction. This kind of operation makes soxhlet hybrid continuous-discontinuous technique [10]. Along with some attractive advantages of soxhlet extraction such as complete extraction of the analyte and low cost of the basic equipment, it suffers from some serious drawbacks such as long extraction time and thermal decomposition of thermo labile species. Automated solvent extractor is an improved design of soxhlet which provides substantial savings in extraction time and solvent (B811 Extractor, BUCHI). In this apparatus, both extraction and boiling takes place simultaneously in the same chamber thus eliminating the concept of cycles (as in Soxhlet) where 7-10 minutes is consumed per cycle. Therefore, if the design of automated solvent extractor is up-scaled, it would be possible to process bran from small rice mills in place thus adding value to the bran that otherwise is wasted. Investment of setting up this kind of extraction equipment will also be many times lesser than the continuous counter current extraction unit whose processing capacity is in the order of many tonnes.

There is no report on the optimized process parameters for RBO extraction in automated solvent extractor. Identifying this gap, this work has been done to determine minimum solvent to bran (STB) ratio, extraction time (ET) and heater temperature (HT) for RBO extraction in automated solvent extractor to get maximum RBO yield. Model equation has been determined that can be used to predict the oil yield by varying the process parameters. Further optimization will be required for pilot scale or large scale units but results of this study would be useful in giving initial estimates.

EXPERIMENTAL

Rice Bran Preparation

Paddy cv. Lal Swarna was procured from local farmer in Kharagpur, West Bengal. The bran was collected by milling in bench top modern rice mill (Satake), milled at 4% degree of polish. The collected bran was sieved through 30 mesh sieve and was stored (moisture content 10±1%) in zipper pouches at -20°C to prevent mould growth and FFA rise. The bran samples were used for oil extraction on the same day.

Solvent Extraction

Rice bran oil was extracted with hexane in automated solvent extraction apparatus (SOCS PLUS, Pelican), the schematic diagram of which is shown in **Figure 1**. Samples of 15 g of rice bran were weighed in cellulose thimbles and were inserted into pre-weighed glass vessels. Solvent was then added into the thimble seeping into the extraction vessel and was kept immediately for extraction on pre-heated heater at set temperature. The temperature of the cooling water being circulated in condenser was maintained at 25±2°C. The image of the setup used for extraction is given in Figure 1. Thimbles were removed from the extraction vessel after stipulated time and the solvent was recovered. The concentrated miscella was then kept in vacuum oven for 1 hour to remove residual hexane. The extraction vessel was kept in desiccator for cooling and weighed to calculate the oil yield using following formula:

$$\%\,\text{Oil yield} = \frac{(W_f - W_i)}{W_s} \times 100 \quad \text{eq.(1)}$$

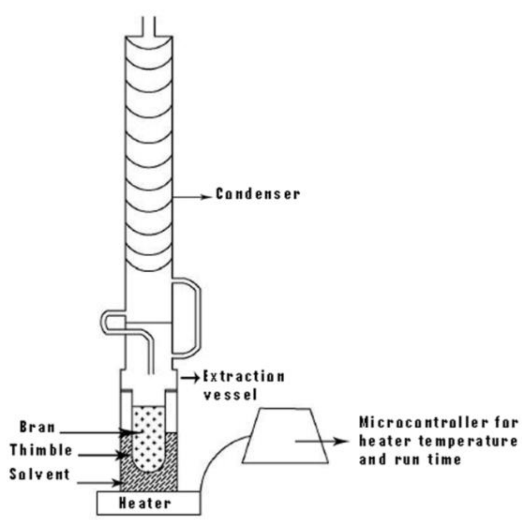

Figure 1 Schematic diagram of automated solvent extractor [6]

Where, W_i = Initial weight of extraction vessel (g); W_f = Final weight of extraction vessel (g); W_s = Weight of rice bran (g). The total amount of oil in bran samples was determined by taking 5 grams of sample and extracting with 200 ml of hexane for 8 hours in soxhlet apparatus. This was done to check lot to lot variation in oil content of bran. The total oil content in bran samples was found to be 21±0.6%.

Free Fatty Acid (FFA) Determination

Rice bran and its oil quality is generally determined by its FFA content. It is expressed as oleic acid percentage (by weight) of the oil. The typical percentage of FFA on crude RBO lies in the range of 2 – 6 % [11]. Free fatty acid was determined by AOCS Method Ca 5a-40 using 0.1 N NaOH and was expressed as free oleic acid percentage. All the experiments were done in triplicate.

Experimental Design

Response Surface Methodology (RSM) with central composite design (CCD) was used to optimize the parameters of oil extraction from rice bran obtained from *cv.* Lal Swarna. Three independent parameters *viz.* solvent to bran ratio, extraction time, and heater temperature, each at five different levels were selected to evaluate their effect on responses i.e. oil yield and FFA. The parameters and their levels were selected based on preliminary trials in lab. The experiments were designed and analysed with the help of Design Expert version 7.0 (Stat-Ease Inc., Minneapolis) software. In this study, a total of 20 experiments were carried out in triplicate and were repeated twice. The experimental design consisted of eight factorial points (2^3), six star points to form a rotatable CCD and six replicates of the centre point. Optimization was done using alpha value of ±1.68 for three factors. The experimental runs were executed in random order to minimize the variability in responses due to extraneous factors. The ranges of selected levels of three different variables are listed in **Table 1**.

Table 1 Range of selected parameters for RSM

Independent Variables	**Levels**				
	-α	**-1**	**0**	**1**	**+α**
Solvent to Bran ratio [r] (w/w)	1.2	1.5	2	2.5	2.8
Extraction time [t] (min)	2	5	10	15	18
Heater temperature [T] (^{0}C)	60	90	135	180	210

Results and Discussion

RBO optimum extraction conditions

The highest oil yield was observed for the process parameters in the range 2.2-2.5 (r), 10-15 minutes (t) and 135-180°C (T) giving more than 95% of the total oil yield. No correlation was found between FFA % and model parameters. The FFA content of oil samples was found to be in the range of 1.5-5% with a mean value of 3±0.7%. It was observed that the experimental conditions giving low oil yield resulted in higher FFA content. It is possibly due to higher rate of extraction of FFA at initial time of extraction [12]. **Table 2** lists the experimental conditions and their respective observed and predicted responses.

Model Fitting

A third-order polynomial equation is proposed for predicting the oil yield from rice bran in lab-scale automated solvent extractor. It is a function of three variables as follows:

$$y = 18.56 + 5.28r + 3.41t + 2.91T + 1.16tT - 2.60r^2 - 2.30t^2 - 1.94T^2 - 1.39rtT - 1.04r^2t - 1.31rt^2$$

The model was validated by random selection of parameter levels within the selected range. Five experiments were performed for validation, the observed and predicted oil yields of whom are listed in **Table 3**.

The response surface model obtained from the experimental design was evaluated using ANOVA and analysis of residuals. The results of statistical analyses including f-value and p-value are listed in **Table 4**.

The model was found to conform to the data at 99% confidence level. Accuracy of the model was evaluated by the value of adjusted R^2. It indicates how well the data fits to the statistical model. Greater is the value of adjusted R^2, lesser is the chance of variability in outcome due to variables not selected. The adjusted R^2 value of the proposed model was 0.9684, implying that 97% of the variability in oil yield is due to selected parameters (r, t, and T) and there is good fit between the observed and predicted values as shown in **Figure 2**. The analysis of variance (ANOVA) results (Table 4) also indicate that the proposed model is statistically good with a significance level of P < 0.0001 and insignificant lack of fit (P > 0.05).

Table 2 Observed and predicted responses for different experimental runs

Runs	Solvent to bran ratio [r] (w/w)	Extraction time [t] (min)	Heater temperature [T] (^{0}C)	Observed		Predicted
				%Oil yield[a] (w/w)	%FFA[b] (w/w)	%Oil Yield[a] (w/w)
1	1.5	5	90	5.73	2.79	5.02
2	2.5	5	90	10.37	2.8	10.18
3	1.5	15	90	5.21	4.02	4.66
4	2.5	15	90	15.7	3.01	15.38
5	1.5	5	180	6.00	3.92	5.74
6	2.5	5	180	16.93	2.50	16.46
7	1.5	15	180	8.67	2.73	15.58
8	2.5	15	180	21.34	2.98	20.74
9	1.2	10	135	1.76	3.78	2.33
10	2.8	10	135	19.52	3.38	20.09
11	2	2	135	5.79	3.12	6.32
12	2	18	135	20.24	3.24	17.79
13	2	10	60	7.51	4.70	8.18
14	2	10	210	17.54	1.83	17.97
15	2	10	135	18.63	2.22	18.56
16	2	10	135	18.31	2.97	18.56
17	2	10	135	19.01	2.7	18.56
18	2	10	135	18.37	2.43	18.56
19	2	10	135	18.95	2.22	18.56
20	2	10	135	18.22	2.43	18.56

[a]Mean value with standard deviation between replicates at ±0.5%
[b]Mean value with standard deviation between replicates at ±0.2%

Table 3 Model validation data

Input Parameters (r, t, T)	Predicted oil yield (%)	Experimental oil yield (%)
2.0, 12, 166	20.96 ± 0.23	20.66 ± 0.34
2.2, 10, 135	20.26 ± 0.12	19.87 ± 0.08
2.5, 5, 90	10.18 ± 0.40	10.43 ± 0.34
1.5, 15, 150	13.65 ± 0.28	13.29 ± 0.44

Table 4 ANOVA results for oil yield

Source	Sum of Squares	Degree of freedom	Mean square	F value	P value
Model	1299.99	10	130.00	102.23	<0.0001
A – Solvent to bran ratio	315.24	1	315.24	247.91	<0.0001
B – Extraction time	131.22	1	131.22	103.19	<0.0001
C – Heater temperature	231.94	1	231.94	182.40	<0.0001
BC	21.58	1	21.58	16.97	0.0004
A^2	152.98	1	152.98	120.30	<0.0001
B^2	118.80	1	118.80	93.42	<0.0001
C^2	84.71	1	84.71	66.62	<0.0001
ABC	30.80	1	30.80	24.22	<0.0001
A^2B	7.21	1	7.21	5.67	0.0259
AB^2	11.39	1	11.39	8.96	0.0065
Residual	29.25	23	1.27		
Lack of fit	6.90	4	1.73	1.47	0.2512
Pure error	22.34	19	1.18		

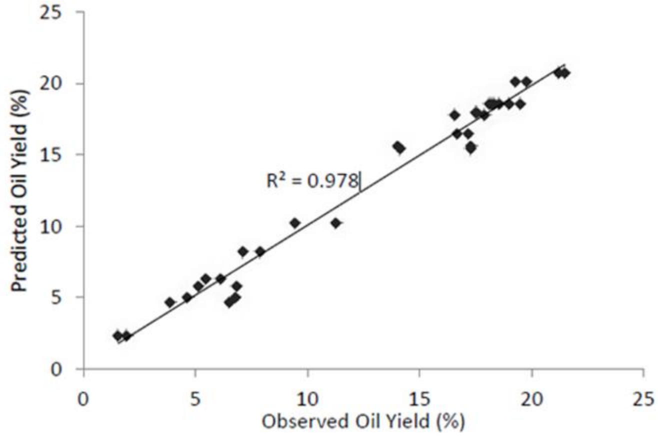

Figure 2 Predicted vs observed oil yield

Variables affecting RBO yield

There are many factors which could affect the RBO yield extracted by the method of solvent extraction. These include solvent to bran ratio, extraction time, pelletization, moisture content of the bran, solvent temperature, heater temperature etc. Since, this optimization study has been carried out keeping in mind the development of industrial scale mini solvent extraction unit; only few parameters which will be important in designing have been selected. The extraction will be carried out at atmospheric pressure; therefore solvent temperature has not been selected as the independent variable. In a mini scale extraction, the problem of channelling in bran could be avoided; therefore pelletization will not be required. It has been found that all the three selected variables (r, t, and T) significantly affect the RBO yield.

Effect of solvent to bran ratio

The trend in oil yield with varying solvent to bran (STB) ratio and extraction time (t) has been shown in **Figure 3**. It can be interpreted that there is steady increase in oil yield with increase in STB ratio. Similar effect of STB ratio has been reported in the findings of [13] who have compared RBO extraction yield in two different solvents viz. hexane and isopropanol. Among all the factors, the individual effect of STB ratio is maximum on the oil yield (F value = 247.91), as evident from the ANOVA results (Table 4). Keeping STB ratio between 2 to 2.5 gives the maximum oil yield (>20%). At lower solvent to bran ratio (1.5), even if heater temperature and extraction time is kept maximum, the oil yield doesn't go beyond 14% (Figure 3a). This is due to lesser solvent available to extract given amount of oil. Around 40-60% of solvent gets trapped inside the bran and some amount is trapped in cellulose thimble. Therefore, at 1.5 STB ratio, very less solvent is left to extract oil and recirculate after condensation. Increasing the STB ratio from 1.5 to 1.8 leads to rapid rise in oil yield from 14% to 18.5%. The oil yield becomes constant (20.5±0.1%) after STB ratio of 2.2 (**Figure 4** (b) and (c)). However, maximum oil can be extracted in lesser time as the STB ratio increases from 2.2 to 2.5 (Figure 4 (c)). There is no significant change in the required heater temeprature to get maximum oil yield at higher STB ratio (2.2 to 2.5) as shown in Figure 4 (b) and (c). The quadratic terms of the STB ratio showed significant interaction with the extraction time and vice versa. These observations reveal that overall there is a positive effect of STB ratio on oil yield and its interaction with extraction time significantly influence the oil yield.

Effect of extraction time

Increase in oil yield was observed with increase in extraction time from 5 to 12 minutes after which it became almost constant (**Figure 5**). This is possibly due to diffusion process coming into picture after 12 minutes which is extremely slow process compared to washing. Since, it is desirable to get maximum oil yield in lesser time, it is suggested that keeping the extraction time between 10-12 minutes is sufficient to get maximum oil yield (~20%) from rice bran. The extraction time showed significant interaction with STB ratio and heater temperature. It decreases with increase in both. Increasing heater temperature beyond 150°C doesn't cause much reduction in extraction time to get maximum oil yield (**Figure 6**). High STB ratio (1.8 - 2.5) and heater temperature between 160-170°C causes significant reduction in extraction time (Figure 4 (b) and (c)). Overall, there exists positive effect of extraction time on RBO yield and its interaction with heater temperature and STB ratio significantly influences the oil yield.

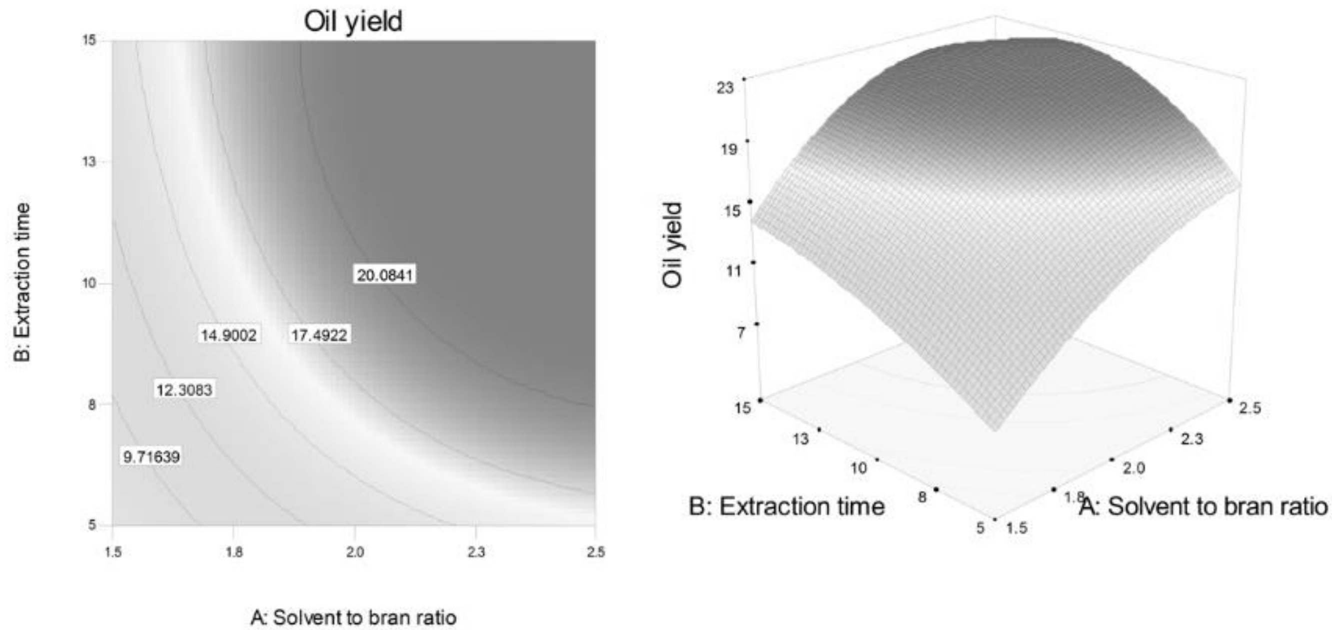

Figure 3 Contour plot and response surface of the oil yield percentage vs. 'r' and 't' at 'T' = 154°C

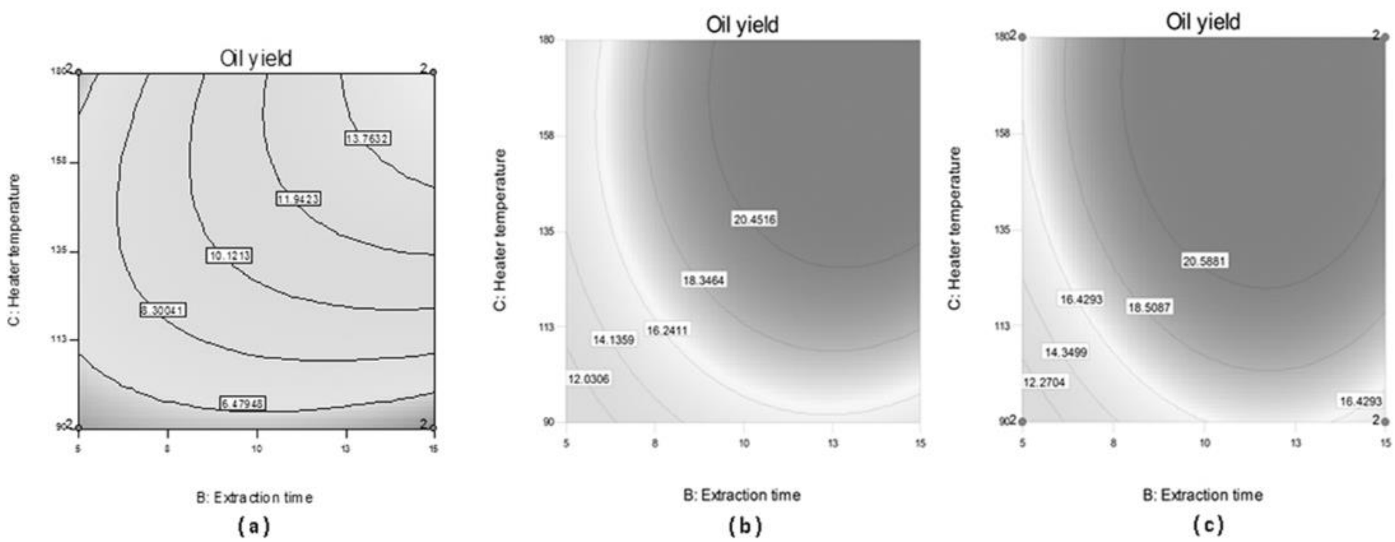

Figure 4 Contour plot of oil yield vs. 'T' and 't' at 'r' = (a) 1.5 (b) 2.2 and (c) 2.5

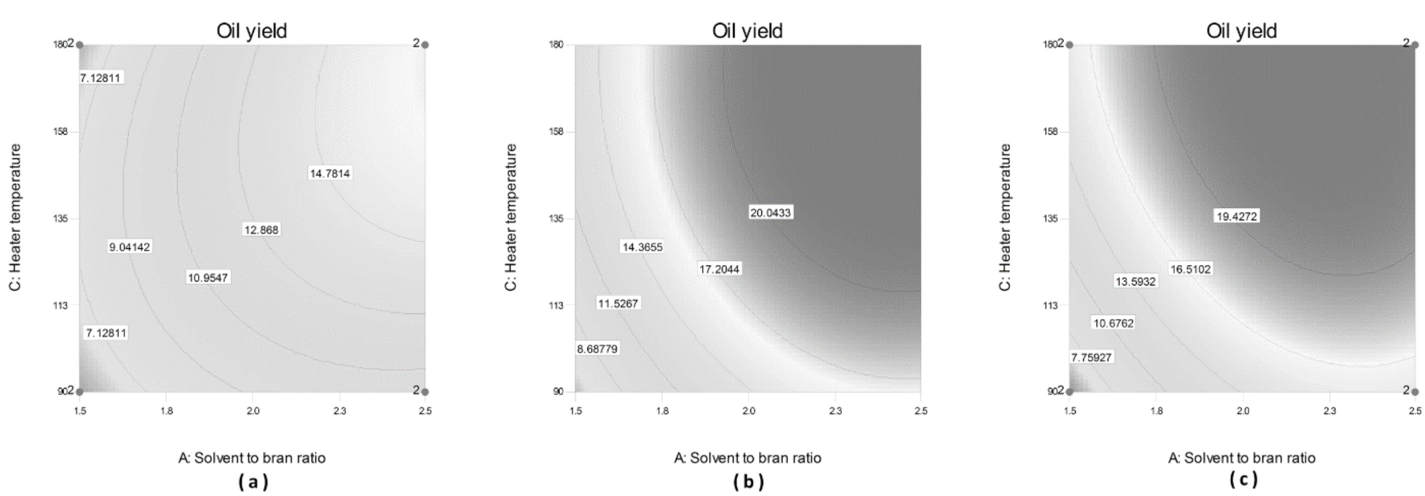

Figure 5 Contour plot of oil yield vs. 'T' and 'r' at 't' = (a) 5 mins; (b) 12 mins and (c) 15 mins

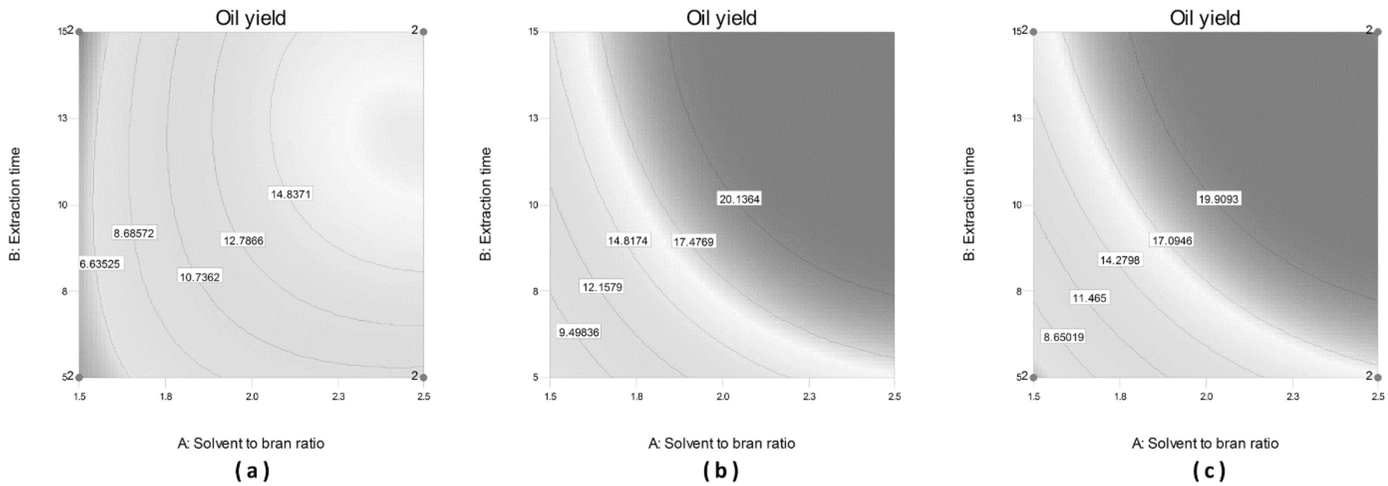

Figure 6 Contour plot of oil yield vs.'t' and 'r' at 'T' = (a) 90°C (b) 164°C and (c) 180°C

Effect of heater temperature

The individual effect of heater temperature on RBO yield is positive i.e. increasing the heater temperature increases the oil yield, maximum yield observed between 160-170°C (Figure 6). Boiling point of hexane lies between 67-69°C at atmospheric pressure. At higher heater temperature the rate of vaporization of hexane increases. This causes more amount of pure solvent condensing in the extraction vessel in a given time. However, if heater temperature is very high (170-210°C), the rate of vaporization of hexane is so fast that it remains for lesser time in liquid state to extract oil from bran. Therefore, a slight reduction in oil yield was observed when heater temperature was raised beyond 170°C (Figure 5c). From ANOVA results, it has been found that there is no significant two factor interaction (rT) between STB ratio and heater temperature (p value = 0.527>0.05). However, in contour plot (Figure 6a and 6b) it has been observed that increasing the heater temperature significantly reduces both the STB ratio and extraction time required to get maximum oil yield. This is possibly due to significant three factor interaction (rtT) between all the three factors or due to indirect effect of interaction between STB ratio and extraction time (rt).

Conclusion

In automated solvent extraction, the rate of oil extraction increases significantly as compared to soxhlet extractor. It depends on the solvent to bran ratio, extraction time and rate of fresh solvent percolating through the solid matrix. Therefore, maintaining higher heater temperature causes rapid rate of solvent evaporation and hence condensation. It requires reduced solvent and extraction time. In this study, higher heater temperature has shown positive effect on rice bran oil yield, extraction time and solvent to bran ratio. Effect of individual parameters and their interaction have been studied so as to utilise the results in up scaling this type of solvent extraction unit. This will cause significant reduction in processing time and solvent consumption. In lab scale study, it has been found that maintaining heater temperature of 160 - 170°C and solvent to bran ratio of 2.2 (g/g), around 20% rice bran oil yield can be obtained in 12 minutes.

Acknowledgement

The author is grateful to the Indian Institute of Technology Kharagpur for providing lab facilities for carrying out this work.

References

[1] F. Cicero, A. Gaddi. Rice bran oil and gamma-oryzanol in the treatment of hyperlipoproteinaemias and other conditions. Phytotherapy Research, 15: 277–289.
[2] G. Cravotto, A. Binello, G. Merizzi, M. Avogadro. Improving solvent-free extraction of policosanol from rice bran by high-intensity ultrasound treatment. European Journal of Lipid Science and Technology, 2004, 106: 147–151.
[3] B.M.W.P.K. Amarasinghe, N.C. Gangodavilage, T. Rao. Data for Process Equipment Design. Trans IChemE, 2004, 82:54–59.

[4] S. Zullaikah, C. Lai, S. R. Vali, Y. Ju. A two-step acid-catalyzed process for the production of biodiesel from rice bran oil. Bioresource Technology, 96: 1889–1896.
[5] F. Shahidi. Rice Bran Oil, Bailey's Industrial Oil and Fat Products, John Wiley & Sons, Inc., Canada, 2005, p266.
[6] N. Kasabe. Asian nations join hands to promote rice bran oil. Retrieved from http://www.financialexpress.com/article/markets/commodities/asian-nations-join-hands-to-promote-rice-bran-oil/117063/, 2015.
[7] R. Pandey, S. L. Shrivastava. Comparative evaluation of rice bran oil obtained with two-step microwave assisted extraction and conventional solvent extraction. Journal of Food Engineering, 2018, 218: 106–114.
[8] R. V. Enochian, R. M. Saunders, W. G. Schultz, E. C. Beagle, P. R. Crowley. Stabilization of rice bran with extrusion cookers and recovery of edible oil: A preliminary analysis of operation and financial feasibility. Marketing Research Report, ARS, USDA, 1980, 1120.
[9] M. D. Luque de Castro, F. Priego-Capote. Soxhlet extraction: Past and present panacea. Journal of Chromatography A, 2010, 1217:2383–2389.
[10] M. Luque de Castro, L. García-Ayuso. Soxhlet extraction of solid materials: an outdated technique with a promising innovative future. Analytica Chimica Acta, 1998, 369:1–10.
[11] M. Ghosh. Review on Recent Trends in Rice Bran Oil Processing. J Amer Oil Chem Soc, 2007, 84:315–324.
[12] T. A. Toda, M. M. Sawada, C. E. C. Rodrigues. Kinetics of soybean oil extraction using ethanol as solvent: Experimental data and modeling. Food and Bioproducts Processing, 2016, 98: 1–10.
[13] W. Hu, J. H. Wells, T. Shin, J. S. Godber. Comparison of isopropanol and hexane for extraction of vitamin E and oryzanols from stabilized rice bran. J Amer Oil Chem Soc, 1996, 73:1653–1656.

flavor.

Publication History

Received	08.07.2021
Revised	08.09.2021
Accepted	09.09.2021
Online	30.09.2021