Tropical Plant Research
An International Journal
© Society for Tropical Plant Research

ISSN (E): 2349 – 1183
ISSN (P): 2349 – 9265
4(1): 31–36, 2017
DOI: 10.22271/tpr.2017.v4.i1.005

Research article

Optimization of extraction parameters for dye from *Pterocarpus santalinus* L.f. (red sanders) wood using response surface methodology

Surendra S. Bisht[1]*, Pradeep S. Hiremath[2], P. L. Muralidhara[2], Mamata Ravindra[1] and V. S. Shetteppanavar[1]

[1]Institute of Wood Science and Technology, Bengaluru, Karnataka, India
[2]Rashtreeya Vidyalaya College of Engineering, Bengaluru, Karnataka, India

***Corresponding Author:** ssbchem@gmail.com [Accepted: 27 January 2017]

Abstract: The present study deals with the extraction of dye from Red sanders (*Pterocarpus santalinus*) wood by chloroform and ethanol solvents using response surface methodology (RSM) technique. Through RSM using three-level three-factor Box-Behnken Design, the optimal conditions such as extraction temperature, time, and feed ratio were identified to be affecting extraction efficiency. The best predicted yield of dye with chloroform solvent extraction was 17.9% at 35°C, 105 minutes, feed ratio 200 mL.g^{-1} while experimental yield was 17%. Similarly for ethanol solvent extraction 24.36% was the predicted yield at 65°C, 105 minutes and feed ratio of 200 mL.g^{-1} while experimental yield was found to be 23%.
Keywords: *Pterocarpus santalinus* - Response surface methodology - Dye - Extraction.

[Cite as: Bisht SS, Hiremath PS, Muralidhara PL, Ravindra M & Shetteppanavar VS (2017) Optimization of extraction parameters for dye from *Pterocarpus santalinus* L.f. (red sanders) wood using response surface methodology. *Tropical Plant Research* 4(1): 31–36]

INTRODUCTION

Natural dyes such as indigo, chlorophyll, heme and red sanders are considered as sustainable and eco-friendly (Strych & Trauner 2013). Red sanders (*Pterocarpus santalinus*) wood is renowned for its characteristic timber of exquisite colour, beauty and yielding a red natural dye which belong to the molecular class of condensed bioflavonoid (Arnone *et al.* 1975, 1977a,b). It also plays an important role in Ayurveda medicine for treating digestive tract problems and coughs. Extraction of dye is influenced by various process parameters such as solvent composition, pH, temperature, extraction time and solid to liquid ratio (Borges *et al.* 2011, Wijngaard & Brunton 2010). Therefore it is very much important to optimize the extraction parameters, so that high yield of dye can be achieved.

Response Surface Methodology (RSM) provides superb statistical tools for design and analysis of experiments aimed at process optimization. Design of Experiments (DOE) deals many RSM designs with options depend on the number of design variables or factors, which can range from one to ten offering Box-Behnken designs (BBD) for three to seven factors require only three levels, coded as -1, 0, and +1 creating designs with desirable statistical properties (Bafna 2012). In view of these points the present study deals with the evaluation of RSM technique for the extraction of dye from Red sanders wood.

MATERIALS AND METHODS

Red sanders wood was powdered by mechanical pulveriser. Chemically inert borosilicate glassware was used during the experiments. Laboratory grade chemicals were purchased from HiMediaLaboratories Pvt. Ltd., while distilled water was used during all set of experiments. Instrument Buchi Rota vapour was used for vaporisation of excess amount of solvents from samples. A Box-Behnken model of three factors was adopted in this study for modelling using MiniTab17.

Received: 21 October 2016 Published online: 31 January 2017
https://doi.org/10.22271/tpr.2017.v4.i1.005

Optimization of extraction parameters using Response Surface Methodology (RSM)

10g of the wood powder was mixed with various volumes of chloroform and ethanol solvents to give a solid to liquid ratio ranging from 1:50 to 1:200 ($g.mL^{-1}$).The flask containing sample powder along with solvent was incubated in thermostatic water bath at various temperatures (35–65 °C) and various time intervals (30–200 min). Observe the change in colour within solvent and filtering it into filtrate. Extract containing coloured filtrate is subjected to evaporation in rota vapour at 50°C and dye extract remained in the round bottom flask was weighed. Design summary of extraction is given in table 1 and 2.

Table 1. Design summary of RSM for chloroform extraction method.

Factors	Units	Low	High
Temperature	°C	35	55
Time	min	30	180
Feed Ratio	$mL.g^{-1}$	50	200

Table 2. Design summary of RSM for ethanol extraction method.

Factors	Units	Low	High
Temperature	°C	35	65
Time	min	30	180
Feed Ratio	$mL.g^{-1}$	50	200

Statistical screening and optimization by design of experiments

A Box-Behnken model of three factors was adopted in this study for modelling using MiniTab17. This method is preferred as design model, since relatively few combinations of the variables are adequate to estimate potentially complex response function. In total 15 experiments are needed in each sample of individual solvent to calculate its 10 coefficients of the second order polynomial equation which was, fitted on the experimental data (Table 3 & 4).

Percentage recovery of dye was taken as response of the system while the three process parameters *i.e.*, temperature, extraction time and solid to liquid ratio were taken as input independent variables with respect to solvent. The system was stated by the following equation:

$$Y=X_0+X_1A+X_2B+X_3C+X_{11}A^2+X_{22}B^2+X_{33}C^2+X_{12}AB+X_{13}AC+X_{23}BC$$

Where, $X_0$is the intercept; X_1, X_2, and X_3 are linear coefficients; X_{11}, X_{22}, and X_{33} are squared coefficients; X_{12}, X_{13}, and X_{23} are interaction coefficients and the experimental variables are temperature (A), Extraction time (B) and Feed ratio (C), Dof- Degrees of freedom. The model adequacies were checked in terms of the values of $R^2$and analysis of variance (ANOVA) was employed to determine the significance of the models.

RESULTS AND DISCUSSIONS

Box-Behnken analysis

BBD was used for three process variables *i.e.*, extraction temperature, time, and feed ratio at three levels. The design points fall within a safe operational limit, within the nominal high and low levels. Design arrangements and responses of experimental and predicted values of chloroform and ethanol solvents were shown in table 3 and 4 respectively.

Table 3. BBD responses for extraction by chloroform.

S.N.	Experiment number	Time (Minutes)	Temperature (°C)	Feed ratio ($mL.g^{-1}$)	Experimental Yield (%)	Predicted Yield (%)
1	1	105	45	125	12	12.34
2	2	30	45	200	13	14.35
3	3	105	45	125	11	12.34
4	4	180	45	50	16	14.65
5	5	30	45	50	05	4.13
6	6	105	35	50	06	7.27
7	7	180	35	125	15	15.14
8	8	105	45	125	14	12.34
9	9	105	35	200	17	17.99
10	10	105	55	200	16	14.73
11	11	30	35	125	08	9.04
12	12	105	55	50	11	12.02
13	13	30	55	125	09	8.86
14	14	180	55	125	15	15.39
15	15	180	45	200	17	17.87

Table 4. BBD responses for extraction by ethanol.

S.N.	Experiment number	Time (Minutes)	Temperature (°C)	Feed ratio ($mL.g^{-1}$)	Experimental Yield (%)	Predicted Yield (%)
1	1	105	50	125	22	20.09
2	2	30	65	125	18	16.83
3	3	180	35	125	13	14.33
4	4	30	35	125	09	9.83
5	5	105	50	125	21	20.09
6	6	105	65	50	19	19.81
7	7	180	50	50	14	14.07
8	8	30	50	50	15	15.56
9	9	30	50	200	10	10.11
10	10	180	65	125	21	20.35
11	11	180	50	200	20	19.62
12	12	105	35	200	14	13.36
13	13	105	35	50	19	17.80
14	14	105	50	125	22	20.09
15	15	105	65	200	23	24.36

Statistical analysis

Multiple regression analysis of the data yielded, the following equation for the recovery of natural dye using chloroform and ethanol recovery of batch solvent extraction method in terms of coded factors:

For chloroform

$$\mathbf{Y_1 = -224 + 1.161*A + 5.56*B + 1.606*C - 0.00074*A^2 - 0.0167*B^2 + 0.00148*C^2 - 0.0033\ A*B - 0.00311A*C - 0.0267B*C}$$

For ethanol

$$\mathbf{Y_2 = 89 + 1.40*A + 1.6*B - 1.12*C - 0.00778*A^2 - 0.017*B^2 - 0.00156*C^2 - 0.0022\ A*B + 0.00489\ A*C + 0.0200\ B*C}$$

Where, Y_1 and Y_2 are chloroform and ethanol responses variables respectively. The student t-distribution and the corresponding p-values along with the f-values of chloroform and ethanol responses are listed in table 5 and 6 respectively.

Table 5. ANOVA for response surface quadratic model of chloroform recovery.

Source	DoF	Squares of sum	Squares of mean	F-value	P-value
Model	9	22231.7	2470.19	8.01	0.017
$\mathbf{X_1}$	1	9800	9800	31.78	0.002
$\mathbf{X_2}$	1	112.5	112.5	0.36	0.572
$\mathbf{X_3}$	1	9112.5	9112.5	29.55	0.003
$\mathbf{X_1^2}$	1	64.1	64.1	0.21	0.668
$\mathbf{X_2^2}$	1	10.3	10.26	0.03	0.862
$\mathbf{X_3^2}$	1	256.4	256.41	0.83	0.404
$\mathbf{X_1*X_2}$	1	25	25	0.08	0.787
$\mathbf{X_1*X_3}$	1	1225	1225	3.97	0.103
$\mathbf{X_2*X_3}$	1	1600	1600	5.19	0.072
Error	5	1541.7	308.33		
Lack-of-fit	3	1075	358.33	1.54	0.418
Pure Error	2	466.7	233.33		
Total	14	23773.3			

In ANOVA for response surface quadratic model of chloroform recovery of batch solvent extraction method, the Model F-value of 8.01 implies the model is significant and there is only a 1.7% chance that a "Model F-

Value" this large could occur due to noise. Values of "Prob < F" >0.05 indicate model terms are significant. In this case feed ratio is significant model terms. Values greater than 0.1 indicate the model terms are not significant. The "Lack of Fit F-value" of 1.54 implies it is not significant relative to the pure error. The values for the coefficient of determination R^2=0.9352 and Adjusted R^2 =0.8184 represents the proportion of variation in the yield or response in the model.

Table 6. ANOVA for response surface quadratic model of ethanol recovery.

Source	DoF	Squares of sum	Squares of mean	F-value	P-value
Model	9	27566.7	3063	15.84	0.004
X_1	1	3200	3200	16.55	0.01
X_2	1	8450	8450	43.71	0.001
X_3	1	7950	7950	22.64	0.017
X_1^2	1	10016	10016	51.81	0.001
X_2^2	1	539.1	539.1	2.79	0.156
X_3^2	1	1077.6	1077.6	5.57	0.065
X_1*X_2	1	25	25	0.13	0.734
X_1*X_3	1	3025	3025	15.65	0.011
X_2*X_3	1	2025	2025	10.47	0.023
Error	5	966.7	193.3		
Lack-of-fit	3	900	300	9	0.102
Pure Error	2	66.7	33.3		
Total	14	28533.3			

In ANOVA for response surface quadratic model of ethanol recovery of batch solvent extraction method, the model F-value of 1.18 implies the model is significant. There is only a 4.5% chance that a "model F-value" this large could occur due to noise. Values of "Prob<F" >0.05 indicate model terms are significant. In this case extraction temperature, time, feed ratio, squared temperature coefficient and temperature-solid to liquid interaction coefficients are significant model terms. The "Lack of Fit F-value" of 9.0 implies it is not significant relative to the pure error. The values for the coefficient of determination R^2=0.9661 and adjusted R^2=0.9051 represents the proportion of variation in the yield or response in the model.

Plot response surface design

To visualize the relationship between response and experimental levels of the independent variables for the natural dye extraction, three dimensional surface and contour plots were constructed according to the quadratic polynomial model equation. The variation of chloroform and ethanol recovery with extraction time and temperature are graphically presented below in figures 1 to 4. As the extraction time of both solvents increases and temperature were decreased, the natural dye recovery increased significantly with curvature contour lines.

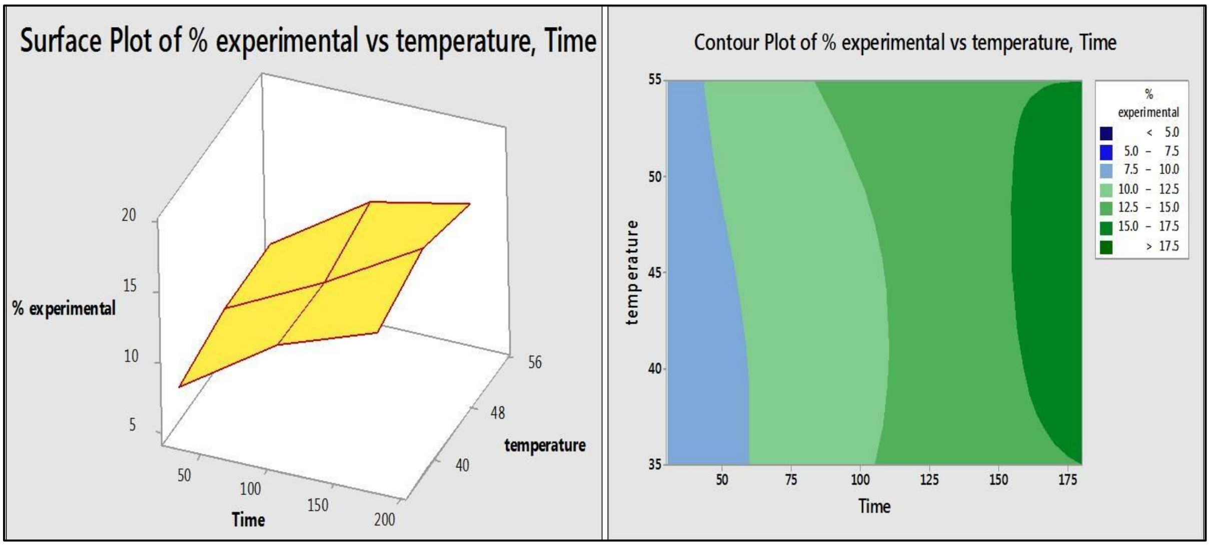

Figure 1. Chloroform recovery surface and contour plots of experimental *vs.* time and temperature.

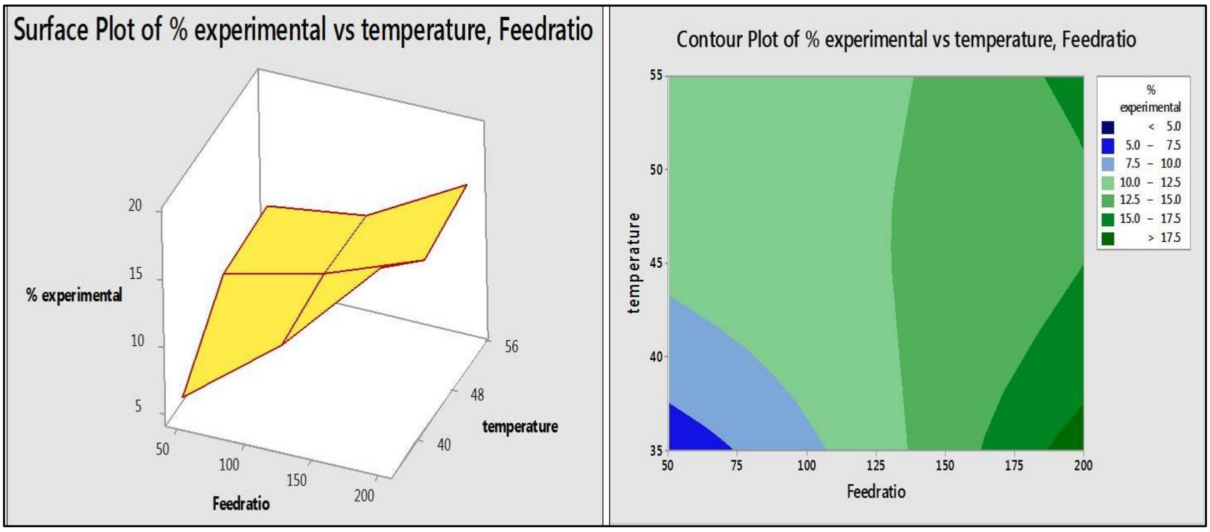

Figure 2. Chloroform recovery surface and contour plots of experimental *vs.* feed ratio and temperature.

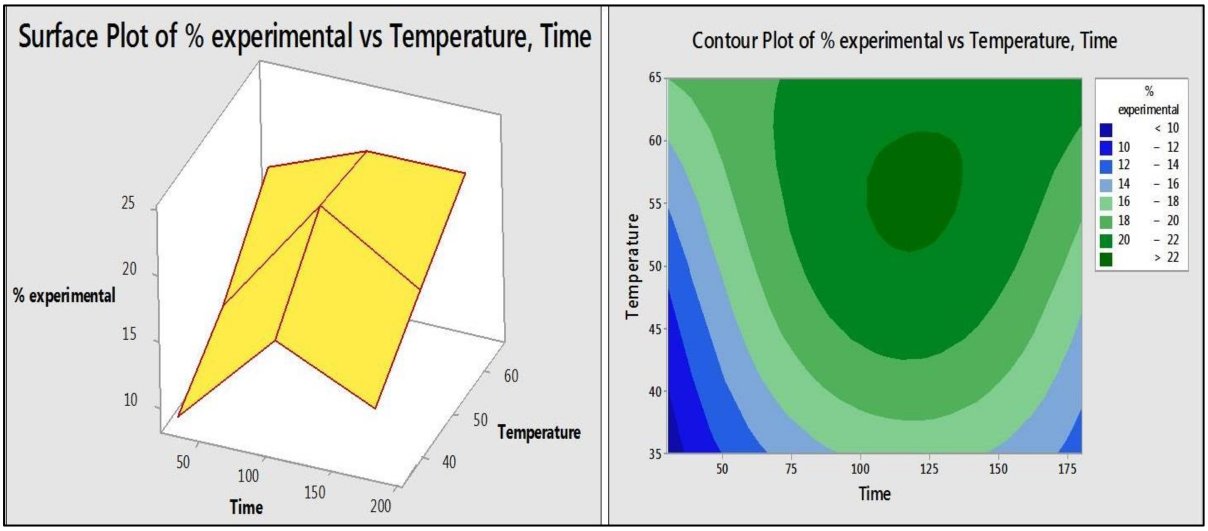

Figure 3. Ethanol recovery surface and contour plots of experimental *vs.* time and temperature.

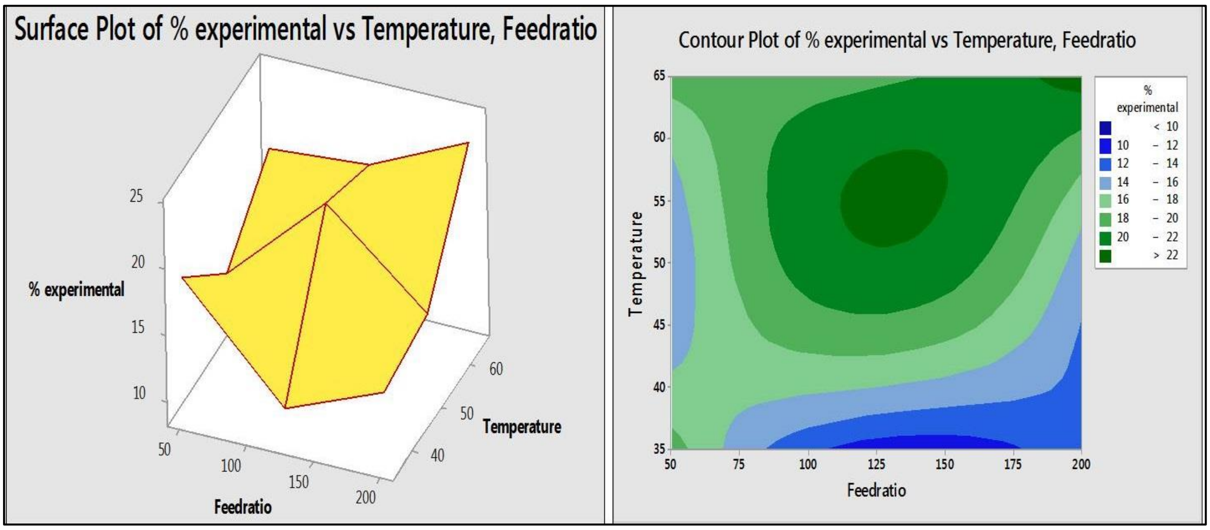

Figure 4. Ethanol recovery surface and contour plots of experimental *vs.* feed ratio and temperature.

CONCLUSION

Response surface methodology was used to optimize the batch solvent extraction of natural dye from *Pterocarpus santalinus* wood. The optimum process parameters and the multiple regression analysis for predicting responses were obtained by applying Box-Behnken design. Under optimum condition ethanol extraction method showed the highest natural dye yield of 23% at 65°C, 105 minutes and feed ratio of 200 $mL.g^{-1}$ compared to chloroform extraction method, which had a maximum yield of 17% at 35°C, 105 minutes and feed ratio of 200 $mL.g^{-1}$. In conclusion we found RSM using Box-Behnken design as a useful tool for optimization of parameter for high yield dye from red sanders wood.

ACKNOWLEDGEMENTS

Director, Institute of Wood Science and Technology, Bangalore is acknowledged for providing the infrastructure during this work. Pradeep is thankful to Dr. M. A. Lourdu Antony Raj, Professor and Head, Department of Chemical Engineering, R. V. College of Engineering, Bengaluru for providing all possible help and advice during the project.

REFERENCES

Strych S & Trauner D (2013) Biomimetic Synthesis of Santalin A,B and Santarubin A,B, the Major Colorants of Red Sandalwood. *Angewandte Chemie International Edition* 52: 9509–9512.

Arnone A, Camarda L, Merlin L & Nasini G (1975) Structures of the red sandalwood pigments santalins A and B. *Journal of the Chemical Society, Perkin Transactions* 1: 186–194.

Arnone A, Camarda L, Merlin L & Nasini G (1977a) 13 C nuclear magnetic resonance spectral analysis of santalin and santarubin permethyl ethers. *Journal of the Chemical Society, Perkin Transactions* 1: 2118–2122.

Arnone A, Camarda L, Merlini L, Nasini G, Taylor DAH (1977b) Colouring matters of the West African red woods Pterocarpus osun and P. soyauxii. Structures of santarubins A and B. *Journal of the Chemical Society, Perkin Transactions* 1: 2116–2118.

Borges GDSC, Vieira FGK, Copetti C, Gonzaga LV & Fett R (2011) Optimization of the extraction of flavanols and anthocyanins from the fruit pulp of Euterpe edulis using the response surface methodology. *Food Research International* 44(3): 708–715.

Wijngaard HH & Brunton N (2010) The optimisation of solid–liquid extraction of antioxidants from apple pomace by response surface methodology. *Journal of Food Engineering* 96(1): 134–140.

Bafna PG (2012) Optimization of Process Parameters for Extraction of Kokum (*Garcinia Indica*) Fruit Pulp using Response Surface Methodology (RSM). *International Journal of Scientific & Engineering Research* 3(8): 1293–1299.