Optimization of oil extraction from Scleropyrum pentandrum (Dennst.) mabb seeds by surface response approach

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Abstract

Scleropyrum pentandrum (Dennst.) Mabb tree grown in India local fields facilitates diet, and medicinal benefits for humans as well as animals. This work is through the interest given to extract the oil from Scleropyrum pentandrum (Dennst.), the feedstock grown in those trees. Solvent extraction method being employed as it demonstrates to be efficient and straightforward for researchers to have the oil obtained from vegetative feedstock. Response surface methodology (RSM) approach in Box-Behnken model being effective which is used to enhance the oil yield contemplating the factors solvent/seed ratio 1, 1.25 and 1.5ml/gm. The extraction temperature 60 ºC to 70ºC, at interval of 5°C. the extraction performed with I hour interval starting from 3hours to 5hours range. The research revealed optimum yield of 61.64% at temperature level of 66.4646 °C, 4.4141-hour extraction time at solvent/seed ratio of 1.3182 ml/g through RSM tool.

Keywords: *Scleropyrum pentandrum (Dennst.) Mabb, Solvent extraction, Surface response methodology, Box-Behnken design, Response optimiser.*

1. Introduction

Demand of fossil fuel and meeting the challenges along with the pollution control concern has led the researchers to work on the area of alternative fuels. The vegetative stock, mainly nonedible feedstock is vital to be considered as the source to obtain the bio diesel. The reliability of humans and the animals' constraints the use of edible feedstock as the demand must be met. Scleropyrum pentandrum (Dennst.) is one such vegetative feedstock grown plentiful in the coastal areas of Dakshina Kannada which can be opted to see the presence of oil and use it as alternative fuel for CI engines. Adding to the awareness on the difficulties faced to meet fossil fuel needs in transport and other sectors indeed it is right to mention that impact of fossil fuel emissions on the environment. The discharge of toxic gasses on combustion has been severely destroying the eco structure and the life of humans. High levels of Particulate materials and NOx contribute towards the respiratory disorders and cancer on the skin [1]. Apart from the 50% of the fossil fuel is essential to meet up the needs of transport sector followed by stationary engines in industry and agriculture [2]. Main kind of bio-oils are bio ethanol derivative also labelled first generation got from starch, sugar etc. and the second generation attained through feedstock [3]. It will be highly appreciable in the case where bio diesel gained make up the engine run devoid of physical revisions. Good lubricity and being nontoxic in nature and reasonable Cetane number promotes the safe use of bio diesel. Authors have inferred on the modest levels of sulphur presence in bio diesel [4]–[6]. Simultaneously quite researchers have revealed the adverse concerns on use bio diesel like oxidization, handling difficulties, fuel flow at low temperatures. Synthetic antioxidants and other additives are included to address the above-mentioned hurdles. Merging of feedstock oil prior to transesterification have improved the properties in comparison to standards. The Fatty acid levels are mentioned the cause of cold flow issues. Cold temperature movement of fuel issues impact the filter and the combustion characteristics [7].

The present investigation focused more on the mining of oil from the available feedstock. The studies [8], [9] shows that the oil content of 55-60% for the Seeds of S. pentandrum. The obtained oil can be further used as a biodiesel for diesel engines after separating the triglycerides. Regarding the production or preparation of the oil, there are traditional as well as scientific methods like boiling, cold press method, chemical extraction, supercritical fluid extraction process and enzymatic extraction etc. The nature of the seed or behaviour of feedstock are the factors to choose the best suited method to produce oil. Comparing to the above said methods, supercritical fluid extraction looks to be costlier and boiling method is cheaper for the oil extraction. The solvent extraction method is most popularly utilised strategy which is financially savvy as well as faster compared to the normal boiling process [10]. Many researchers [11]–[13] have dealt with number of methodologies for numerous available feedstock's', but the conditions and influence of different parameter plays a vital role in extraction of oil yield. Therefore, researchers investigate the steps to get maximum yield by using different statistical tools and implement the best suited condition to get the optimised value and confirmed with the experimentations. RSM has been depicted as a viable statistical approach which tells the impact of individual factors and their interaction on responses. Researchers are using this tool to catch on the effective parameters and their levels which are significant towards the oil yield. This is considered essential to effectively use the natural resources available. Hence in the current study during the oil yield from Scleropyrum pentandrum (Dennst.) Mabb RSM is employed to understand the significance of major 3 factors.

Response surface methodology (RSM) here being used for augmentation, incorporates the Factorial Design and Regression Evaluation [14]. Mathematical model developed to assess and evaluate key variable and influence. The pictorial representation in form of 3-D plot which tells the trend for influence of two different parameters over the output result. The chemical extraction using solvent is proficient technique for removing most of oil accessible in the oil seed.

2. Materials and Methods:

Figure 1. (A) Raw yield in bunches

Figure 1. (B) Outer sugary pulpy rind

From March to July, S. pentandrum plants produce fruit; infrequently, some trees also produce fruit in the winter, but considerably lower than in summer. Figure 1(a) shows how seeds are formed in bunches, with each bunch containing roughly 15–25 seeds. Due to its outer sugary pulpy peel and hard shell covering the inner kernel, ripe orange fruits fall from the trees and are devoured by cattle (Figure 1b). Every matured tree can produce 8 – 10kg of seeds in two seasons a year.

Ripe harvested seeds initially being dried completely which needs 12days to make the moisture dry. This is facilitated by the aid of solar drier to get unison quality. Amongst the observation made it in noticed high loss in weight during the drying process. Using a mechanical type of crusher, the dried seeds are pulvarised to fine grit. It took almost a month duration to prepare the powder which aids in extraction of oil.

The following steps are involved to obtain the final product that is bio-oil

- Collection of seeds
- Deshelling

- Oven frying or drying
- Blender
- Extraction
- Distillation
- Filtration
- Final product

2.1 Chemicals Used

Oil extraction was carried out using the solvent n-hexane, and the oil-dissolving solvent for the acid-base titration was isopropyl alcohol.

2.2 Extraction of oil from Scleropyrum pentandrum (Dennst.) Mabb seeds make use of solvent extraction technique

Soxhlet apparatus of 1000cc volume with n hexane along with the pulverised seed used to root out the bio-oil. 1000gms of seed powder per sample was necessary at every trail with the extraction duration kept 3 hours to 5 hours. This facilitated in maximum oil extraction. The heater was set to the selected values of temperature as shown in table1. Box-Behnken design array obtained using Minitab software, the oil extraction procedure was carried out. Once the extraction is done, the retraction of oil with n-hexane is done by distillation process. For other parameters, the experiment was repeated, and the yield was computed. Equation 1 was used to calculate the percentage oil yield for the extraction [15].

$$\text{Percentage oil yield} = \frac{Mass\ of\ oil\ extracted}{Mass\ of\ the\ seed\ powder} \times 100 \quad (1)$$

Table 1. Levels and range in form of code for Box-Behnken design (BBD)

Factors	Unit	Coded symbol	Levels & range		
			-1	0	1
Solvent / seed ratio	ml/g	A	1	1.25	1.5
Extraction temperature	^{0}C	B	60	65	70
Extraction time	h	C	3	4	5

2.3 Experimental Design

The combination of parameters for experimentation is done as per the generated array as per Box-Behnken experiment array of Minitab statistical software. The Box-Behnken array of DOE is intimated to generate the sequence of experiments with three factors and three levels [16]. The generated array has 15 number of trials with different parametric variable combinations at different levels. The detailed factors and its level are clearly shown in Table 2. The experiment has been conducted for the obtained array in a sequence.

3 Results and Discussion:

3.1 RSM-based optimization:

RSM is one of the statistical tool to optimize the chemical methods and outcomes [17], [18] which eases the implementation of RSM technique during extraction process to delve into the most favourable process considerations for the extraction of Scleropyrum pentandrum (Dennst.) Mabb oil from the pulverised and dried raw material by aid of solvent extraction process. Box-Behnken design is being employed through the aid of Minitab software in 3 level variables for 3 factors. The created trials for the BBD has been experimentally performed through this research consist of 15 runs.

The oil yield obtained solvent extraction by Soxhlet apparatus is tabulated for 3 factors and 3 level experimental combination is shown in ***Table 1***. The regression equation is developed as par with the oil yield values for 15 experimental runs suggested for BBD. The predicted values are obtained using regression equation for each trial runs. It is found that minimal error when comparing the oil yield and predicted values for entire table. Equation 2 represents regression model developed based on the regression analysis for 15 runs.

$$\%\ oil\ yield = 59.667 + (2.4 * A) + (3.763 * B) + (5.312 * C) - (5.896 * A * A) -$$

$$(9.021 * B * B) - (7.921 * C * C) + (0.9 * A * B) + (1.15 * A * C) + (3.175 * B * C) \quad (2)$$

Table 2. Box-Behnken design experimental table

Run	Solvent/seed ratio (w/w of oil)	Extraction temperature (^{0}C)	Extraction Time (hr)	Oil yield (%)	Predicted value	Residual
1	-1	-1	0	40.1	39.487	0.613
2	1	-1	0	42.2	42.487	-0.287
3	-1	1	0	45.5	45.213	0.287
4	1	1	0	51.2	51.813	-0.613
5	-1	0	-1	38.5	39.288	-0.788
6	1	0	-1	41.9	41.788	0.112
7	-1	0	1	47.5	47.612	-0.112
8	1	0	1	55.5	54.712	0.788
9	0	-1	-1	37	36.825	0.175
10	0	1	-1	38.5	38.001	0.499
11	0	-1	1	40.6	41.099	-0.499
12	0	1	1	54.8	54.975	-0.175
13	0	0	0	58.8	59.667	-0.867
14	0	0	0	59.3	59.667	-0.367
15	0	0	0	60.9	59.667	1.233

Table 3. Variance Analysis

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	1010.39	112.265	109.01	0.000
Linear	3	385.11	128.371	124.65	0.000
Factor A	1	46.08	46.08	44.75	0.001
Factor B	1	113.25	113.251	109.97	0.000
Factor C	1	225.78	225.781	219.24	0.000
Square	3	576.42	192.141	186.57	0.000
Factor A * Factor A	1	128.35	128.348	124.63	0.000
Factor B * Factor B	1	300.46	300.463	291.76	0.000
Factor C * Factor C	1	231.65	231.654	224.94	0.000
2-Way Interaction	3	48.85	16.284	15.81	0.006

Factor A * Factor B	1	3.24	3.24	3.15	0.136
Factor A * Factor C	1	5.29	5.29	5.14	0.073
Factor B * Factor C	1	40.32	40.322	39.15	0.002
Error	5	5.15	1.03		
Lack-of-Fit	3	2.74	0.914	0.76	0.611
Pure Error	2	2.41	1.203		
Total	14	1015.5			

Table 4. Model response values

S	R-sq	R-sq(adj)	R-sq(pred)
1.01481	99.49%	98.58%	95.15%

Results obtained through ANOVA analysis through RSM displays, the fact that the model's F-value being 109 indicates which is exceptional. Value of P lower than 0.05 delegate model positions or values being considerable. The obtained table shows that Factor B, Factor C, Factor B * Factor B, Factor C * Factor C are major significant factors. The effects of Factor A* Factor B and Factor A* Factor C are negligible. The Table 4 illustrates how the R-Squared and Adjacent R-Squared value model work together in practise.

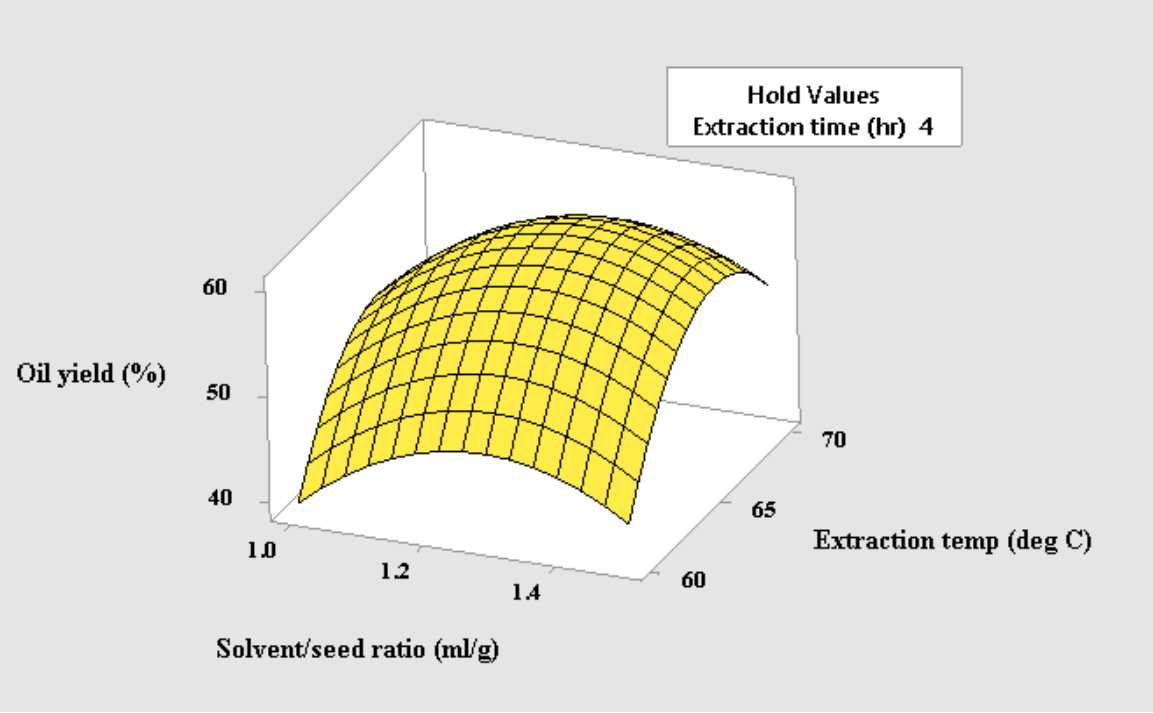

Figure 2. Effect of Extraction temperature and Solvent/seed ratio on oil yield – Surface plot

Locking the time to 4 hours, it is clear from figure 2 that a solvent to seed ratio of 1.2 to 1.35 (ml/g) contributes to a higher yield when supported by a temperature range of 65 °C to 69 °C. This data is discussed fixing the duration of extraction being 4 hours. Likewise, in accordance with variation in the extraction duration in the steps of 1 hour that is 3, 4 & 5h steps as represented in figure 3 the ideal time range can be noted as in range of 4 to 4.5h duration to obtain the maximum yield. As the duration

approaches to 5 hours the oil yield drops. Again, locking the temperature of 65°C as shown in figure 4, in accordance with variation of extraction duration and solvent to seed ratio it is depicted solvent to seed ratio 1.3 ml/g and 4.5h extraction duration results in maximum oil yield of 61%. Hence from figure 5 showing the optimal levels of factors to contribute the maximum oil yield and based on the above discussion, it is concluded that solvent seed ratio of 1.312 ml/g at temperature 66.4°C fixing the extraction duration of 4.4 hours will yield in 61% of bio-oil under solvent extraction method for the feedstock selected in this research study.

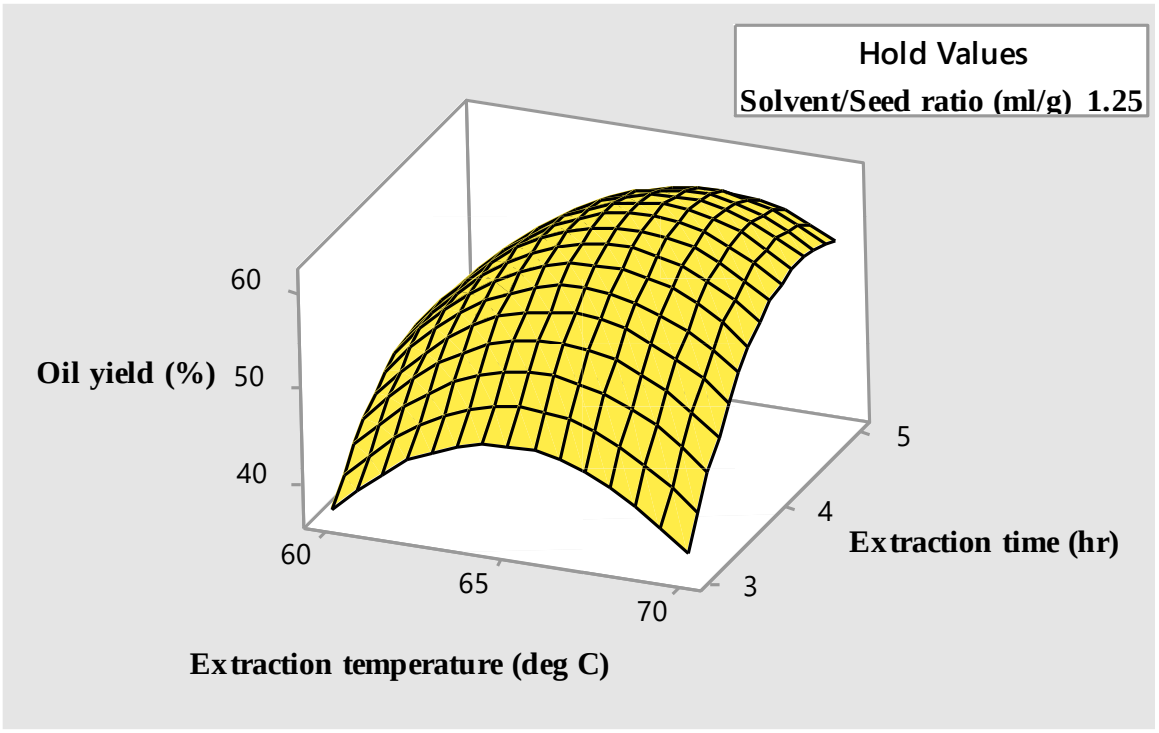

Figure 3. Effect of Extraction temperature and Extraction time on oil yield – Surface plot

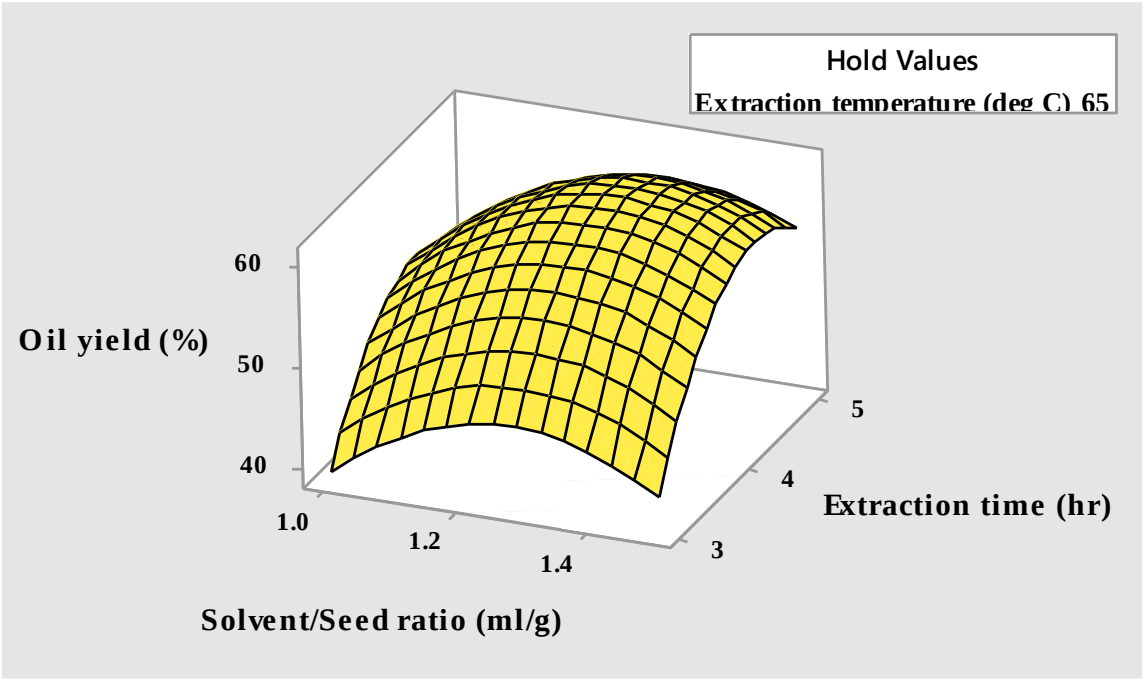

Figure 4. Effect of Solvent/seed ratio and Extraction time on oil yield – Surface plot

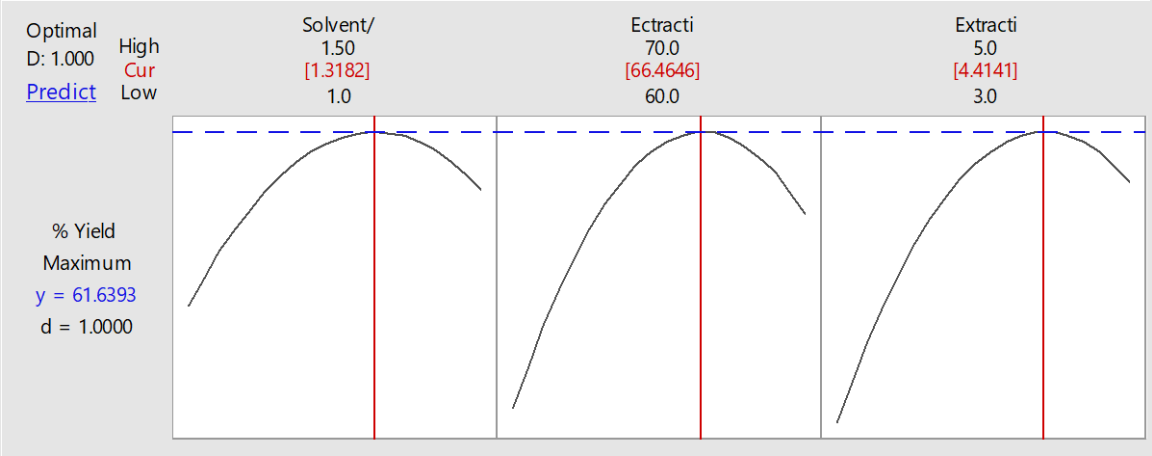

Figure 5. Optimal level of factors contributing to oil yield

4. Conclusion

Scleropyrum pentandrum (Dennst.) Mabb seed oil proved to be influencing the researchers to work on the yield of bio-oil which facilitates its use as bio diesel for various applications. The present work concentrated on the extraction of oil only with selection of right parameters and its levels. The oil yield is comparatively competent in quality as well as richness in oil content. The n-hexane Soxhlet apparatus is used for the extraction and 3 factor with 3 level BBD. Through this study it is evident that very good or promising yield of oil obtained through solvent extraction. The yield obtained is 61.6393% at temperature of 66.4646 °C, oil removal duration of 4.4141 hour fixing the solvent to seed proportion of 1.3182 ml/g. It is observed that the length of the process and the use of the proper temperature have a big impact on the amount of oil produced.

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