

Modelling of the Effect of Pretreatment Methods, and Time on the Yield of Eucalyptus Essential Oil Using Response Surface Methodology

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ABSTRACT:

The wide application of essential oils as a result of their medicinal properties has prompted continuous research towards determining factors affecting their yield. One of such essential oils with medicinal properties is *Eucalyptus camaldulensis*. In line with this trend is the aim of this study centred on modelling the effect of pretreatment methods applied to plant leaves and the extraction time on the yield of essential oils from *Eucalyptus camaldulensis*. Different polynomial regression models were developed showing the relationship between the pretreatment methods, time and the yield of oil using response surface methodology in Design Expert Version 11. The different multiple regression models were fitted to experiment data and the best model was further validated using analysis of variance (ANOVA), and analysis of residuals. Further optimization of the extraction of the yield was carried out using the desirability function in Design Expert Version 11. Results of the study showed that at optimum values of extraction time of 145 min, a yield of 1.48% was obtained with chilled pretreated leaves. The model that best explains the data was a linear model and the model showed a good fit with the experimental data, with a coefficient of multiple determination (R^2) value of 0.9938. The study would pave the way for further scale-up in the recovery of essential oils from Nigerian-based *Eucalyptus camaldulensis* specie.

Keywords: *Eucalyptus camaldulensis*, modelling, optimization, response surface methodology.

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INTRODUCTION

The therapeutic application of essential oils from different plants due to their medicinal, and aromatic properties has driven its wide industrial use in the cosmetic and pharmaceutical industries. Essential oils are a component of skin care products, fragrances, and creams [1]. Consequently, essential oils are a source of exports for many countries. However, one of the main challenges of wide-scale commercialization of the conventional methods of essential oil recovery from plants is the low yield and high energy cost. Consequently, there is a need for studies on increasing the yield of essential oils from plants. In line with this, the study aims to determine the relationship between different factors and the yield of essential oils and to optimize the yield.



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According to the World Health Organization, about 21000 plants have therapeutic properties [2]. Essential oils from the Eucalyptus plant, a family of Myrtaceae, have been applied in dentistry due to their antiseptic property. However, the yield of essential oils is a function of different plants [1]. An example of a plant with a different species that is rich in essential oils is Eucalyptus. There are more than 750 species in the Eucalyptus genus out of which more than 300 contain volatile oils and less than 20 species contain about 70% 1,8 cineole (eucalyptol), a main therapeutic component of Eucalyptus plant. They have been used commercially in the cosmetic and pharmaceutical companies. Accordingly, essential oils have been extracted from *Eucalyptus tereticornis* using steam distillation, [3] solvent extraction [4]; *Eucalyptus globulus* using solvent extraction [5], *Eucalyptus urograndis* using hydrodistillation [6]; *Eucalyptus nitens* using solvent extraction [7], *Eucalyptus camaldulensis* using steam distillation [8], with these species exhibiting different yields.

Consequently, to study the relationship between different extraction factors and the yield of essential oils from Eucalyptus, many empirical models have been proposed. Galadima, et al. [3] modelled and optimized the yield of essential oils from *Eucalyptus tereticornis* as a function of the solute: solvent, time and steam rate. An optimum yield of 2 % was achieved at 105 min and a steam extraction rate of 0.03 kg/h. Ammer, et al. [4] modelled the anti-bacterial components of essential oils in *Eucalyptus tereticornis* as a function of pH and temperature. A pH of 7 and temperature of 40 °C achieved optimum anti-bacterial components of 3, 11, and 15 µL of Limonene, 1, 8-Cineole, and Terpinen-4-ol respectively. Gullón, et al. [5] optimized the yield, total phenolic and flavonoid content, and anti-oxidant components of *Eucalyptus globulus* as a function of the temperature, time and solvent-to-water ratio. Optimum conditions were 32.7% yield, 92.9 mg GAE/g dried leaf total phenolic content and 53.7 mg RE/g dried leaf total flavonoid content respectively. The antioxidant levels were 205.4, 363.4 and 185.2 mg TE/g dried leaf for the different analyses. Palma, et al. [9] also optimized the bio-active molecules in *Eucalyptus globulus* as a function of ultrasound power, pH, temperature, time, and ethanol to water percentage. The results showed that temperature was the most important factor affecting these molecules. However, modelling and optimization studies centred on optimizing the yield of essential oils of *Eucalyptus camaldulensis* as a function of the pretreatment type are lacking.

Response Surface Methodology is a statistical and mathematical technique used to optimize processes by analyzing the relationships between multiple input variables and their impact on one or more response variables. Response surface methodology builds predictive models to describe how changes in input factors influence the outcome by systematically designing experiments. Further optimal conditions are identified through response surface analysis, graphical visualizations, and mathematical techniques. It is widely applied in industries such as manufacturing, pharmaceuticals, food processing, and agriculture. The application of response surface methodology in the design of experiments minimizes experimental effort while providing valuable insights into complex systems, making it a crucial tool for process optimization. Hence, this work will specifically model the relationship between the pretreatment type, extraction time and the yield of essential oils from *Eucalyptus camaldulensis* using response surface methodology. The specific objectives are:

- To carry out a Design of Experiment using I-optimal custom design to assess the relationship between the yield of essential oils as the response and pretreatment type, and other extraction parameters as independent variables.
- To develop polynomial models explaining the relationship between the response and independent variables.
- To determine conditions for optimum yield of essential oils.

MATERIALS AND METHODS

Materials

Nigerian *Eucalyptus camaldulensis* specie was obtained from the Lagos State University, Epe, Nigeria. The water used for steam generation was obtained from the laboratory.



Extraction studies

Extraction of essential oils from the *Eucalyptus camaldulensis* was carried out using a steam distillation set-up (Figure 1). The leaves of obtained *Eucalyptus camaldulensis* were washed and air dried until a constant weight was obtained. Another set of leaves was washed and kept in the refrigerator in sealed nylons at -4 °C until when it was ready to be used while the third set of wet leaves was used directly for extraction studies. The leaves were charged in a distillation flask and steam vapour generated at 110 °C in a steam flask was allowed to flow over the leaves in the distillation flask. Vapourised essential oils evaporated and cooled through a connected condenser. The mixture of essential oils and water was collected in a separating funnel. Water as the denser oil was decanted from the mixture after settling and the essential oil was recovered.



Figure 1. Steam Distillation Apparatus Used for Extraction Studies

Design of Experiment

The different experimental runs were determined using the I-optimal custom Design of Experiment to accommodate for one numeric factor (extraction time) and one categoric factor (different pretreatment types). I-optimal custom Design of Experiment was developed using 2-levels for the extraction time, and three levels for the pretreatment types. Additional centre points were added to check for curvature in the model equation. Design Expert Version 11 was used to carry out the Design of Experiment and was used for further optimization studies. The extraction time at different levels was converted to coded variables and the relationship between the actual variables and the coded variables is given by Equation 1. The different levels of variables used, the coded values (x_i) and the actual values (X_i) of the variables are presented in Table 1.

$$x_i = \frac{X_i - (X_{max} + X_{min})/2}{(X_{max} - X_{min})/2} \quad (1)$$

Where X_{max} , X_{min} are the maximum and minimum values of the actual variable.

Table 1. Different Levels of Variables were Used for the Design of Experiment

Factors	Unit	Type	Actual levels		Coded values	
			Low	High	Low	High
Extraction time	min	Numeric	30	240	-1	1
Pretreatment		Categoric	Wet, chilled, dried		{0 1}, {-1 1}, {1 0}	



Modelling

For a two-variable system, the relationship between the response value y and regressor variables x could be expressed by a multiple linear regression model in Equation 2.

$$y = \alpha_0 + \alpha_1 x_1 + \alpha_2 x_2 + \dots + \alpha_k x_k + \varepsilon \quad (2)$$

Where y is the yield of essential oils and α_1 to α_k are the coefficients of the regressor variables x_1 to x_k .

For more complex relationships including interactions between 2 variables, an interaction was introduced into equation 2 to give equation 3.

$$y = \alpha_0 + \alpha_1 x_1 + \alpha_2 x_2 + \alpha_{12} x_1 x_2 + \varepsilon \quad (3)$$

For higher-order models in x , the relationship between the response y and variables x in Equation 2 can be written as Equation 4.

$$y = \alpha_0 + \alpha_1 x_1 + \alpha_2 x_2 + \alpha_{11} x_1^2 + \alpha_{22} x_2^2 + \alpha_{12} x_1 x_2 + \varepsilon \quad (4)$$

The coefficients in the regression models were determined by fitting to experimental data using the method of least squares. Design Expert Version 11 was used for the model fitting and screening. The models were screened by determining how best the model describes the experimental data.

RESULTS AND DISCUSSION

Model Relationship between Pretreatment, Extraction Time and the Yield of Essential Oil

The relationship between the process variables and the yield is presented in Table 2 and the best equations that describe the data for *Eucalyptus camaldulensis* are given by equations 5-7. Comparing different models using Design Expert Version 11, the relationship between the variables and the yield is that of a first-order.

For *Eucalyptus camaldulensis* specie

The equation for yield of oil from dried pretreated leaves is:

$$yield = 0.75 + 0.00197 * Time \quad (5)$$

The equation for yield of oil from wet leaves is:

$$yield = 1.197 + 0.00197 * Time \quad (6)$$

The equation for yield of oil from chilled pretreated leaves is:

$$yield = 0.75 + 0.00197 * Time \quad (7)$$



From the interaction between all variables and the yield (Figure 2), the chilled leaves yielded more oil with increasing extraction time than other pretreatment types.

Table 2. Relationship between Process Variables and the Experimental Yield of Oil

Run	Actual factor 1	Actual factor 2	Coded factor 1	Coded factor 2	Response
	Time	Pretreatment	Time	Pretreatment	% Yield
1`	182.89	wet	0.456	{0 1}	1.4
2	182.89	wet	0.456	{0 1}	1.4
3	87.75	dried	-0.450	{1 0}	0.89
4	30	wet	-1.000	{0 1}	1.13
5	240	dried	1.000	{1 0}	1.24
6	135	chilled	0.000	{-1 1}	1.475
7	87.75	dried	-0.450	{1 0}	0.93
8	135	chilled	0.000	{-1 1}	1.45

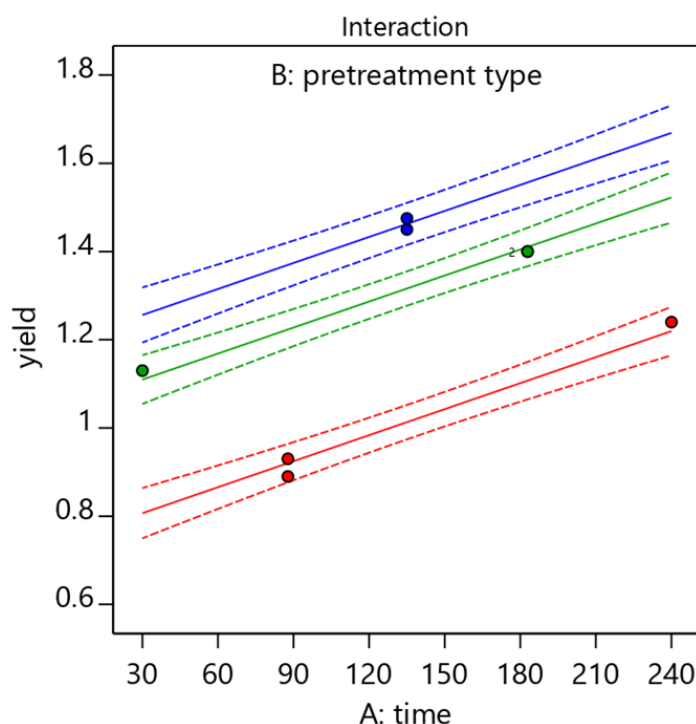


Figure 2. Interaction between the Pretreatment Types (drying-red, wet-green, chilled-blue) and the Extraction Time as It Affects the Yield of Oil

Model Validation

The models were validated by carrying out statistical analysis. ANOVA was carried out to test for the significance of regression at a significance level of 0.05 (Table 3). The results showed that F_0 value for the model $> F_{0.05,2,5} = 3.37$ implies the model is significant. Also, a P-value of $0.0001 < 0.05$ implies that the model terms are significant. Results of R^2 in Table 4 show a reasonable agreement between the predicted R^2 and adjusted R^2 value. A value close to 1 shows that the model best explains the data.

Further residual analysis tests were also carried out to check the accuracy of the model. Results showed the model points and experimental data fit on a straight line (Figure 3).



Table 3. ANOVA Results for Model Validation

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.3789	3	0.1263	213.72	< 0.0001	significant
A-time	0.1199	1	0.1199	203.00	0.0001	
B-pretreatment type	0.2705	2	0.1352	228.87	< 0.0001	
Residual	0.0024	4	0.0006			
Lack of Fit	0.0013	1	0.0013	3.37	0.1636	not significant
Pure Error	0.0011	3	0.0004			
Cor Total	0.3812	7				

Table 4. Values of Coefficient of Determination (R^2)

Std. Dev.	0.0243	R^2	0.9938
Mean	1.24	Adjusted R^2	0.9892
C.V. %	1.96	Predicted R^2	0.9677
		Adeq Precision	31.5482

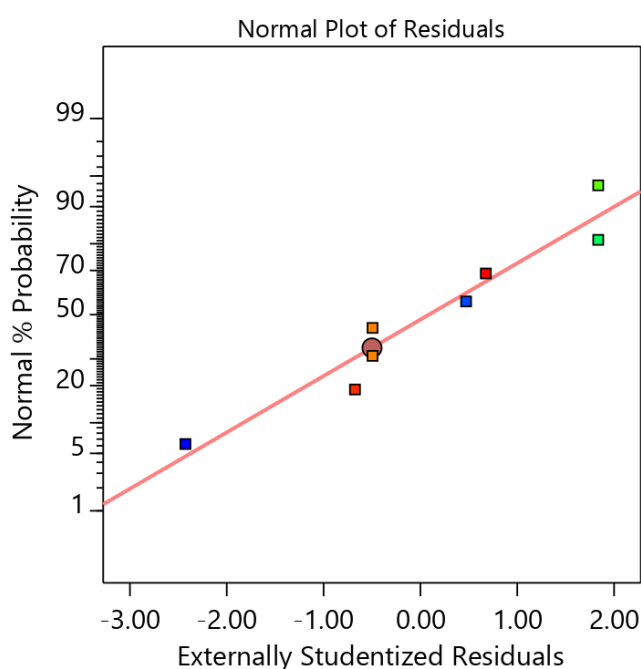


Figure 3. Normal Probability Plot for the Model

Optimization

Optimization was carried out numerically using the desirability approach in Design Expert Version 11. This involves maximizing the yield at specific desirability from 0-1 for the variables. The closer the value of the desirability to 1, the more the level of importance. Since the response is just the yield, a weight of 1 was assigned to the yield. The boundary conditions used were time (30-240 min) and pretreatment type (drying, chilled and wet). At time 145 min and for the chilled pretreatment type, the optimum yield obtained was 1.48% (Figure. 4). The optimum results obtained were different from that obtained by Ayub et al. [10] for the extraction of essential oils from *Eucalyptus globulus* using superheated steam. The extraction time was lower (40 min) but the extraction temperature was higher as a result of the use of superheated steam. The results obtained in this work are justified by the fact that at a lower steam temperature of about 110 °C, it would take a longer extraction time to achieve the required yield of oil.



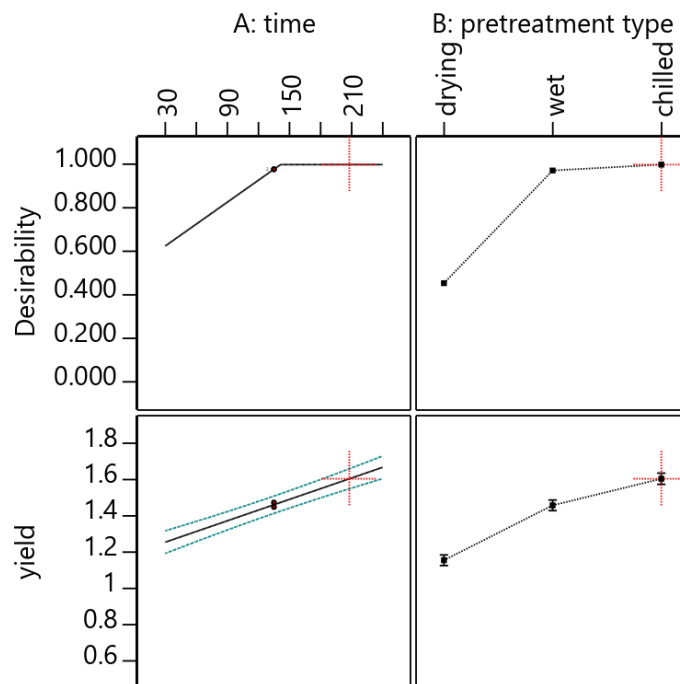


Figure 4. Desirability Graphs for Optimization of the Yield of Oil

CONCLUSION

The relationship between the pretreatment type and the extraction time has been modelled and optimized. The relationship between the different pretreatment types, extraction time and the yield of essential oils is that of a first order. The model showed a reasonable agreement with the experimental data with an R^2 value of 0.9938. At a time of 145 min and chilling of the leaves at -4°C , a maximum yield of 1.48% was obtained. The modelled results can help to predict the recovery of essential oils from wet, chilled or dried *Eucalyptus* oil. This would pave the way for further scale-up in the recovery of essential oils from Nigerian-based *Eucalyptus camaldulensis* specie.

CONFLICT OF INTERESTS

No conflict of interest.

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