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Development of Mathematical Model for the Prediction of Essential Oil Extraction from *Eucalyptus Citriodora* Leave

***Mu'azu. K[1]., Mohammed-Dabo, I.A[2]. and Waziri, S.M[2].**

[1]Pilot Plants Divison, National Research Institute for Chemical Technology, Zaria, Nigeria
[2]Chemical Engineering Department, Ahmadu Bello University, Zaria, Nigeria

ABSTRACT

The mathematical model for the extraction of essential oil from *eucalyptus citriodora* leave using steam distillation technology was developed by factorial analysis. The factors studied were extraction time (20 – 80min), heating rate (3.30-676kJ/s), volume of water (2-3x$10^{-3}m^3$) and leaves condition (dry -wet). The experimental results demonstrated that extraction time was the major parameter in the extraction process followed by heating rate and volume of water. The oil yield increased as the extraction time, heating rate, and their interaction increases. The model predicted that the highest oil yield was 17.53 x$10^{-6}m^3$(2.66%) at the following conditions: extraction time of 80minutes, heating rate of 6.76kJ/s, 2 x$10^{-3}m^3$ of water using wet leave. However, the predicted oil yields by the developed model were found to be close to that of the experimentally observed ones. The quality of the essential oil produced at the optimum predicted conditions satisfied the relevant quality standard of Association of oil chemist society (AOCS). The developed mathematical model can be employed in simulation of essential oil extraction using steam distillation method.

KEYWORDS*:* eucalyptus citriodora**,** steam distillation, essential oil, oil yield, model equation.

INTRODUCTION

The extraction of essential oil is of great interest in industrial applications as important component in the production of perfume, food, soaps, cosmetics and pharmaceutical.[1]. These oils are odoriferous essence of a number of plants which can be found in its various parts such as flowers, fruits, leaves, roots, seeds and bark. The oils are formed in green (chlorophyll bearing) parts of the plant and with plant maturity are transported to other tissues particularly to the flowering shots [2].

The essential oil obtained from plants is generally volatile in nature, mostly insoluble in water but freely soluble in alcohol ether and vegetable oil as well as mineral oil and may be grouped into five classes according to their chemical structure [3].

Eucalyptus plant is among the main source of essential oil and is mainly grown in tropical and sub-tropical regions primarily as wind breakers due to its resistance to many pest and adaptability to various climatic conditions. These include South Africa, China, Congo Republic, Angola, India and West Africa [2,3]. The plant has up to 700 species and can grow as high as 40 m tall in an altitude of 600m. The principal component of the essential oil extracted from *eucalyptus* leave is cineola which is up to 70 - 80%. However, the yield of essential oil is naturally constrained (usually less than 2%) but with high market value, for instance, essential oil extracted from the leave of *eucalyptus citriodora* and *Helichrysum Italicum* natives of Corista are sold at $0.35 US dollar per gram and $1,305.40 US dollar per gram respectively [4].In case of *eucalyptus citriodora,* oil yield ranges from 0.5 to 2% has been reported in Australia, Brazil, Guatemala and India. However, oil yield as high as 4.80% has been reported from *eucalyptus citriodara* plant growing in Nigeria [4].

Various methods have been employed for the essential oil extraction which includes solvent extraction, water extraction, mechanical expression, extraction under super critical conditions, steam distillation, etc. Among these methods, steam distillation is widely employed because of its simplicity and cost effectiveness [2,5].

Despite the availability of essential oil bearing plants in most part of Africa including Nigeria, the production of essential oil is still at low level partly due to its low yield and partly due to lack of information on best operating conditions for the extraction process using steam distillation method. An attempt had been made to optimize the extraction of essential oil from *eucalyptus citriodora* leave using effect of one- factor- at- a- time (OFAT) as presented in the earlier work [6,7]. However, the relative efficiency of (OFAT) is half of two-factor - at- a- time and the efficiency continue to increase with every factor added [8].

Factorial analysis of Design of Experiment is a useful statistical technique which has been applied in research into complex variable processes. It employs multiple regression and correlation analyses as tools to assess the effects of two or more independent factors on the dependent variables. Its principal advantage over OFAT is reduced number of experimental runs required to generate sufficient information for statistically acceptable results. In addition to that it covers wider area from which to draw inferences about your process and

***Corresponding author:** Mu'azu. K., Pilot Plant Division, National Research Institute for Chemical Technology, P.M.B.1052, Zaria, Nigeria.Tel:+234(0)8027106787.E-mail: kabirumuazu@yahoo.co.uk

also reveals interaction of factors. Factorial analysis has been applied successfully in the optimization and development of mathematical model of several biotechnological and chemical processes [11].

In this study, essential oil was extracted from eucalyptus leave via steam distillation method and developed mathematical model equation to predict best conditions for the extraction process by considering effects of all the main factors at a time and their interactions.

MATERIALS AND METHOD

Materials

Fresh *eucalyptus citriodora* leaves were obtained from Government reserved plantation site in Zaria, Nigeria. The leaves were removed from the stalk and dried for three days at ambient condition (30±5^{0}C).

Apparatus

The extraction was conducted in a 5- Litre capacity distillation flask equipped with thermometer and connected to a coil-type counter-current flow arrangement condenser. The distillation flask was heated with heating mantle such that the extraction process was maintained at 100^{0}C. Cooling water from tap continuously flow through the tube side of the condenser at the rate of 3x10^{-3}m^3 per minute.

Reaction procedures

Initially, the distillation flask was charged with 600g of the eucalyptus leaves and setting conditions of volume of water, heating rate and extraction time as shown in Table 2. As the extraction progressed the steam-oil mixture generated passed through shell side of the condenser and condensed as water-oil mixture which was collected in the conical flask. At every 10 minutes interval, the volumes of condensate (water-oil mixture) were measured and cumulative volume of the oil in each experiment run was recorded as oil yield as shown in Table 2.

Design of experiment

Full factorial design using a two level- four factors (2^4) was adopted in this study requiring a total of 16 experiments [8,9]. The factors which were selected for the study were shown in Table 1 and coded (-1) for low values and (+1) for high values.

Table 1: Statistical design of the extraction process

Factors	Unit	Low level (-1)	High Level (+1)
Extraction time (A)	Min	30	80
Heating rate (B)	kJ/s	3.30	6.76
Volume of water (C)	m^3	2x10^{-3}	3x10^{-3}
Leave condition (D)		Dry	Wet

The effect of each factor and their interactions were calculated using equation (1).

$$Effect = \frac{\sum Y_+}{n_+} - \frac{\sum Y_-}{n_-} \qquad (1)$$

In which Y_+ is the positive responses, Y_- is the negative responses, n_+ and n_- refers to the data points at each level [6].

Table 2: Factorial design of the extraction process showing extraction combinations using coded factors

Std	Exp. run	Extraction time (A)	Heating rate (B)	Volume of water (C)	Leave condition (D)	Oil Yield Experiment x10^{-6}(m^3)	Oil Yield Experiment %	Oil Yield Predicted x10^{-6}(m^3)	Oil Yield Predicted %
1	9	-1.00	-1.00	-1.00	dry	3.20	0.49	3.68	0.56
2	3	1.00	-1.00	-1.00	dry	9.00	1.37	8.95	1.36
3	5	-1.00	1.00	-1.00	dry	12.00	1.82	12.27	1.87
4	7	1.00	1.00	-1.00	dry	17.00	2.58	17.53	2.66
5	2	-1.00	-1.00	1.00	dry	0.20	0.03	0.34	0.05
6	6	1.00	-1.00	1.00	dry	9.90	1.50	10.36	1.57
7	10	-1.00	1.00	1.00	dry	3.80	0.58	4.21	0.64
8	15	1.00	1.00	1.00	dry	14.10	2.14	14.24	2.16
9	14	-1.00	-1.00	-1.00	wet	3.70	0.56	3.68	0.56
10	1	1.00	-1.00	-1.00	wet	9.36	1.42	8.95	1.36
11	16	-1.00	1.00	-1.00	wet	13.00	1.98	12.27	1.87
12	11	1.00	1.00	-1.00	wet	17.60	2.68	17.53	2.66
13	13	-1.00	-1.00	1.00	wet	0.80	0.12	0.34	0.05
14	12	1.00	-1.00	1.00	wet	10.50	1.60	10.36	1.57
15	4	-1.00	1.00	1.00	wet	4.30	0.65	4.21	0.64
16	8	1.00	1.00	1.00	wet	14.70	2.23	14.24	2.16

Mathematical model

Experimental data generated in Table 2 were analyzed via full factorial analysis in order to fit the following polynomial equation generated by Design-Expert 6.0 software (Stat- Ease Inc. USA) [10].
The model for the four independent factors and their interaction is expressed according to equation (2)

$$Y = \beta_o + \beta_1 A + \beta_2 B + \beta_3 C + \beta_4 D + \beta_5 AB + \beta_6 AC + \beta_7 AD + \beta_8 BC + \beta_9 BD + \beta_{10} CD + \beta_{11} ABC + \beta_{12} ABD + \beta_{13} ACD + \beta_{14} BCD + \beta_{15} ABCD \quad (2)$$

Where Y is the predicted response (oil yield), β_o is model constant (intercept) and β_1, β_2, β_3, β_4, β_5, β_6, β_7, β_8, β_9, β_{10}, β_{11}, β_{12}, β_{13}, β_{14} and β_{15} are coefficients associated with variable A, B, C and D and their respective interactions calculated using linear regression method and the model terms were accepted or rejected based on the P-values with 95% confidence level [9,10,11]. Three dimensional plots were obtained based on the effect of the four factors to study their interactions on oil yield at different levels. In order to determine the accuracy of the model developed, experimental data were substituted in the model equation at various conditions of extractions time, heating rate, volume of water and leave condition and the corresponding predicted oil yield was obtained (See Table 2).

Analysis of essential oil

The essential oil obtained at the optimum predicted values was analyzed in terms of some physicochemical properties according to method described by American Oil Chemist Society and presented in Table 6 [3].

RESULTS AND DISCUSSION

Effect of factors on oil yield

In order to develop the mathematical model, 16 experiments at various setting condition were conducted (See Table 2). It is evident from Table 2 that the variation in the yield of oil was as a result of effect of the main factors and their reciprocal interactions. It can be seen in Table 3 that the four factors involved in the extraction process namely; extraction time (A), heating rate (B), volume of water (C) and leave condition (D) have significant effect on the oil yield. It is also evident in Figure 1 that extraction time (A) appears to be the most important factor with main effect of (E_1 = 7.64x10^{-6}) followed by heating rate (B) with (E_2 = 6.23 x10^{-6}) and water (C) with (E_3 = 3.23 x10^{-6}).

Table 3: Complete matrix showing calculated main and interaction effects in standard order

Std	Main Effects				Interaction Effects											
	A	B	C	D	AB	AC	AD	BC	BD	CD	ABC	ABD	ACD	BCD	ABCD	Oil Yield
1	-	-	-	-	+	+	+	+	+	+	-	-	-	-	+	3.20
2	+	-	-	-	-	-	-	-	+	+	+	+	+	-	-	9.00
3	-	+	-	-	-	+	+	-	-	+	+	+	-	+	-	12.00
4	+	+	-	-	+	-	-	+	-	+	-	-	+	+	+	17.00
5	-	-	+	-	+	-	+	-	+	-	+	-	+	+	-	0.20
6	+	-	+	-	-	+	-	+	+	-	-	+	-	+	+	9.90
7	-	+	+	-	-	-	+	+	-	-	-	+	+	-	+	3.80
8	+	+	+	-	+	+	-	-	-	-	+	-	-	-	-	14.10
9	-	-	-	+	+	+	-	-	-	-	-	+	+	+	-	3.70
10	+	-	-	+	-	-	+	+	-	-	+	-	-	+	+	9.36
11	-	+	-	+	-	+	-	+	+	-	+	-	+	-	+	13.00
12	+	+	-	+	+	-	+	-	+	-	+	+	-	-	-	17.60
13	-	-	+	+	+	-	-	+	-	+	+	+	-	-	+	0.80
14	+	-	+	+	-	-	+	-	-	+	-	-	+	-	-	10.50
15	-	+	+	+	-	+	-	-	+	+	-	-	-	+	-	4.30
16	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	14.70
Effect	E_1	E_2	E_3	E_4	E_5	E_6	E_7	E_8	E_9	E_{10}	E_{11}	E_{12}	E_{13}	E_{14}	E_{15}	8.95

Where E_1=7.64x10^{-6}, E_2=6.23 x10^{-6}, E_3=-3.23 x10^{-6}, E_4=0.595 x10^{-6}, E_5=-0.07 x10^{-6}, E_6=2.38 x10^{-6}, E_7=-0.055 x10^{-6}, E_8=-2.355 x10^{-6}, E_9=0.08 x10^{-6} E_{10}=-0.02 x10^{-6}, E_{11}=0.395 x10^{-6}, E_{12}=-0.02 x10^{-6}, E_{13}=0.08 x10^{-6}, E_{14}=-0.105 x10^{-6} and E_{15}=0.045 x10^{-6}

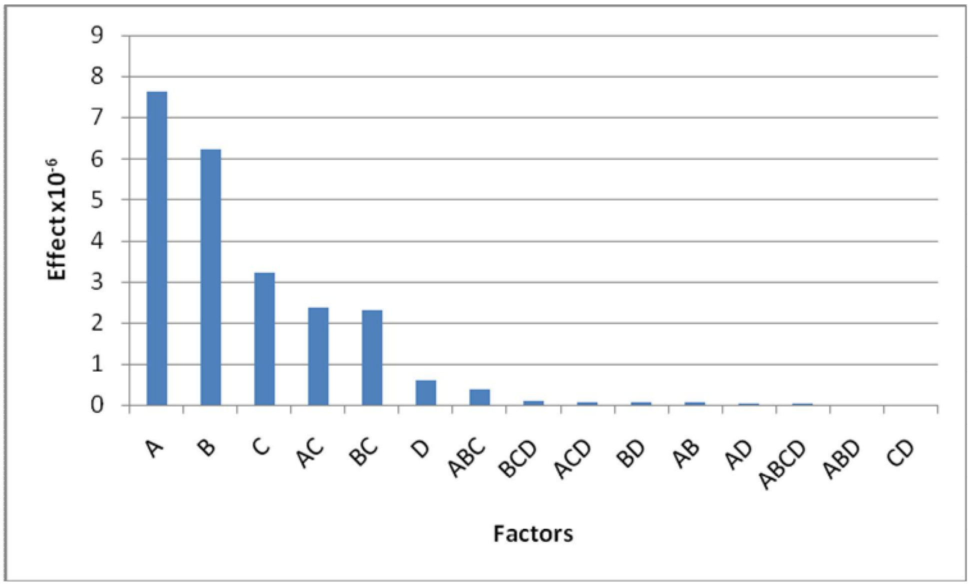

Figure 1: Pareto chart of effects for oil yield

It was observed that at the initial stage of the extraction process, the steam produced travelled through packed bed of the leave and rupture the leave cells before releasing the essential oil. At this period which is usually referred to as induction period or unsteady state condition, the resistance to steam flow across the packed bed of leave and subsequent pressure drop were highly significant and consequently resulted in no oil extraction. The induction period was generally found between 0-21 minutes depending on the operating bulk density and rate of steam supply. As the extraction time (A) was increased, the rate of oil removal also increases due to reduced pressure drop experienced by the steam and the extractable oil had already been transported to the surface of the leave resulting in higher yield of oil. It is interesting to note that most of the oil was extracted between 21-45 minutes of the extraction time [6].

As far as heating rate (B) is concerned, increase in heating rate increases the kinetic energy of water molecule and its vapour pressure. At higher heating rate, the latent heat produced breaks the molecular bonds of the water in shorter time resulting in increase of steam production and consequently increases in oil extraction from the leave. However, at lower volume of water (D), the rate of steam production increases because of the increase in energy absorbed per unit mass of the water molecule [7].

The estimated interaction effect between extraction time and water (AC) and that of heating rate with water (BC) are 2.38 x10^{-6} and - 2.355 x10^{-6} respectively. This means that raising heating rate and volume of water (BC) resulted in decrease in oil yield by 2.355x10^{-6} m^3. On the contrary, increase in the extraction time and water (AC) would results in increased in oil yield by 2.38 x$10^{-6}m^{-3}$ (see Table 3). The effect of leave condition (D) and its interaction with other factors were insignificant (see Figure 1).Therefore, these ten trivial effects (D, AB, AD, BD, CD, ABC, ABD, ACD, BCD) and (ABCD) which are nearest to zero were used in the estimation of error in the analysis of variance and as such did not appear in the model equation [11, 12, 13].

Figure 2 represent the effect of varying extraction time and volume of water an oil yield at a constant heating rate using wet leave. It is evident from Figure 2 that a decreased in the volume of water and simultaneous increase in the extraction time resulted in increase in oil yield and the maximum oil yield obtained was 17.53 x$10^{-6}m^{-3}$ at 2 x$10^{-3}m^{-3}$ of water and extraction time of 80 minutes. When the leave was dried, same value of oil yield of 17.53 x$10^{-6}m^{-3}$ was obtained as shown in Figure 3.

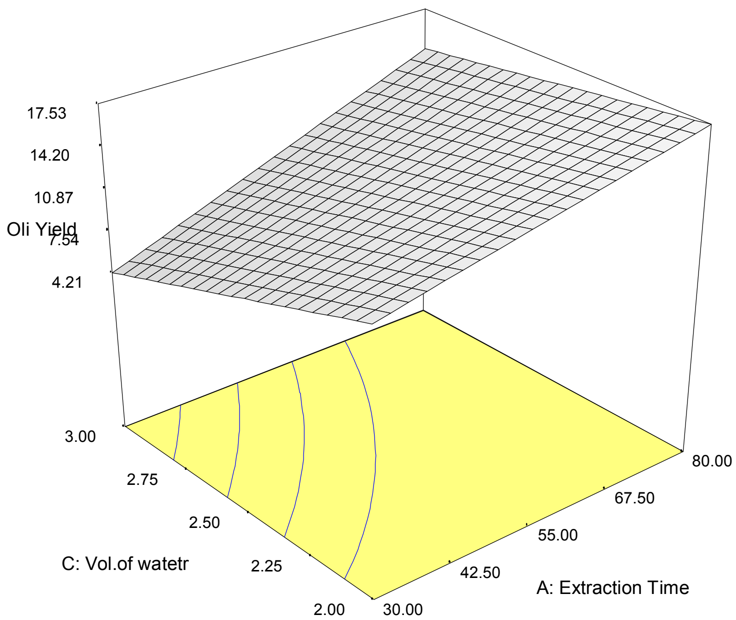

Figure 2: Effect of extraction time and water on oil yield at constant heating rate of 6.76kJ/s using wet leave

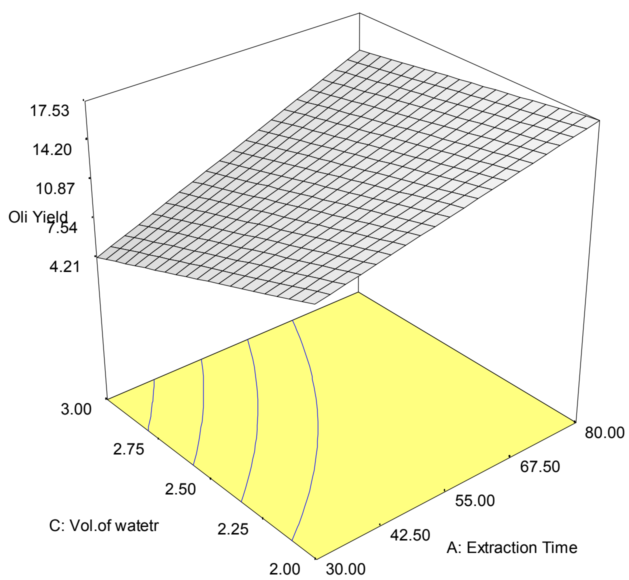

Figure 3: Effect of extraction time and water on oil yield at constant heating rate of 6.76kJ/s using dry leave

This confirmed the fact that, the effect of leave condition is highly insignificant in the oil yields as already shown in Figure 1. However, at low heating rate (3.30kJ/s) and same operating conditions, maximum oil yields of $10.36x10^{-6}$litres was obtained as shown in Figure 5.

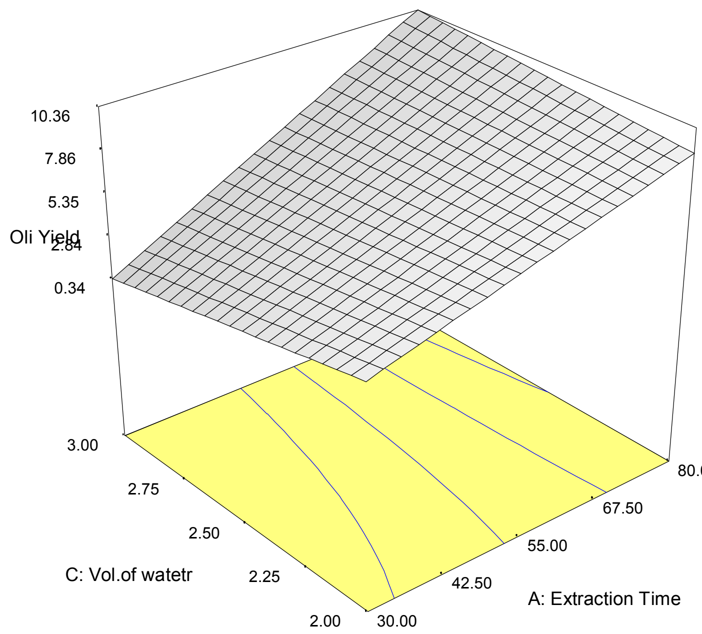

Figure 4: Effect of extraction time and water on oil yield at low heating rate of 3.30kJ/s using wet leave

The interaction effect betweens heating rate and water on oil yield are represented in Figure 5 and 6. It is evident from these two Figures (5 and 6) that at any given condition of the leave (dry or wet), the oil yield increases with increase in heating rate at constant volume of water and maximum oil yield of 17.53 $x10^{-6}m^{-3}$was obtained. At lower heating rate of 3.3kJ/s, maximum oil yield of 10.01 $x10^{-6}m^{-3}$ was obtained under the same operating condition of extraction time. The difference in oil yield in Figure 5 and 6 is as results of the fact that high heating rate increase steam production and consequently increases rate of essential oil removal from the cell pockets of the leave where the essential oil is being held [6,7].

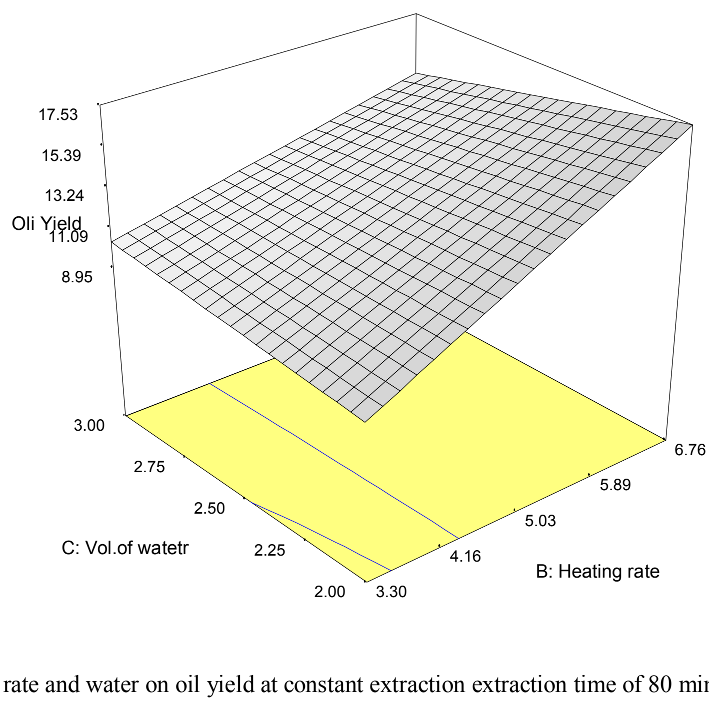

Figure 5: Effect of heating rate and water on oil yield at constant extraction extraction time of 80 minutes using wet leave

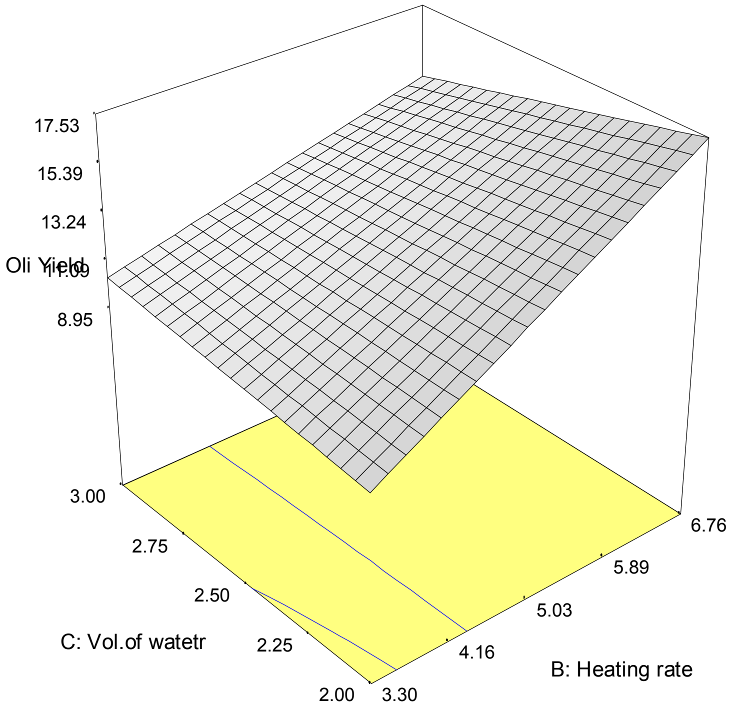

Figure 6: Effect of heating rate and water on oil yield at constant extraction extraction time of 80 minutes using dry leave

Model Equation

In order to verify the conclusion drawn from Table 3 and Figure 1 on the effects of factors and their interactions on oil yield, analysis of variance was employed. The analysis of variance (ANOVA) in Table 4 showed that experiment data are best fitted into a quadratic model at 95% level of confidence [12,13].

Table 4: ANOVA table to identify significance factors influencing oil yield

Source	Sumof squares	DF	Mean square	F-value	Prob >F
Model	$4.78x10^{-10}$	5	$9.559 x10^{-11}$	438.8	<0.0001
A	$2.338 x10^{-10}$	1	$2.338 x10^{-10}$	1073.14	<0.0001
B	$1.553 x10^{-10}$	1	$1.553 x10^{-10}$	712.65	<0.0001
C	$4.409 x10^{-11}$	1	$4.409 x10^{-11}$	202.39	<0.0001
AC	$2.266 x10^{-11}$	1	$2.266 x10^{-11}$	104.01	<0.0001
BC	$2.222 x10^{-11}$	1	$2.222 x10^{-11}$	101.83	<0.0001
Residual	$2.18 x10^{-12}$	10	$2.178 x10^{-13}$		<0.0001
Cor Total	$4.801 x10^{-10}$	15			

The values of $F_{calculated}$ (F = 438.8) of the model equation is greater than $F_{critical}$ at 95% confidence interval ('Prob >F' less than 0.0500), implies that the linear regression model equation is significant. It can also observed in Table 4 that the actual F-values of extraction time (A), heating rate (B) and water (C) and reciprocal interaction of time and water (AC) and that of heating rate and water (BC) exceed the critical F at 95% for these degree of freedom (1,10), implies that they all have significant effect on the oil yield. From this statistical analysis, extraction time, heating rate and water are the most important parameters in the extraction of essential oil [8].

The model equation developed for the extraction of essential oil from *eucalyptus citriodora* leave using steam distillation method in terms of both coded and experimental values are given in equation 3 and 4 respectively.

$$Oil\,Yield = [8.95 + 3.28A + 3.12B - 1.66C + 1.91AC - 1.18BC]x10^{-6} \quad (3)$$

$$Oil\,Yield = [-0.00425 - 0.0000851A + 0.00520B - 1.709C + 0.0952AC - 1.361BC]x10^{-3} \quad (4)$$

Thus, the positive coefficient terms in equation (3) obtained from Table 5 is an indication that the oil yield increases with increase in extraction time, heating rate and interaction between extraction time and water. However, the negative coefficient term indicates increase in oil yield with decrease in volume of water and interaction between heating rate and water [8,13].

Table 5: Regression coefficient and significance of response quadratic model

Factor	Coefficient estimate	DF	Standard error	95%CI low	95%CI high	VIF
Intercept	8.95 $x10^{-6}$	1	1.17 $x10^{-6}$	8.69 $x10^{-6}$	9.21 $x10^{-6}$	-
A-Extraction Time	3.82 $x10^{-6}$	1	1.17 $x10^{-6}$	3.56 $x10^{-6}$	4.08 $x10^{-6}$	1.00
B-Heating rate	3.12 $x10^{-6}$	1	1.17 $x10^{-6}$	2.86 $x10^{-6}$	3.37 $x10^{-6}$	1.00
C-Vol. of water	-1.66 $x10^{-6}$	1	0.12 $x10^{-6}$	-1.92$x10^{-6}$	-1.40 $x10^{-6}$	1.00
AC	1.19 $x10^{-6}$	1	1.17 $x10^{-6}$	9.17 $x10^{-7}$	1.45 $x10^{-6}$	1.00
BC	-1.18 $x10^{-6}$	1	1.17 $x10^{-6}$	-1.44$x10^{-6}$	-0.92 $x10^{-7}$	1.00

Validation of the Model

In order to validate the model developed, experimental data generated in Table 2 (in terms of coded values) were substituted in equation (3) and the predicted oil yield was obtained as shown also in Table 2. The model predicted that the maximum oil yield was 17.532 $x10^{-6}m^{-3}$ at the following conditions, 80 minutes of extraction time, heating rate of 6.76kJ/s, 2 $x10^{-3}m^{-3}$ of water and wet leave. The adequacy of the developed model was also checked by determining the correlation coefficient (R) of the results in Table 2.Since the values of R in the literature lies in the range of -1 to +1 and +1 means perfect relationship [13].Hence, the calculated value of R obtained in this work is 0.995 which means that the developed model has high correlation with the experimental values Comparison of experimental values with the predicted values, both values are in close proximity having standard deviation of 0.47 as shown in Figure 7. These facts suggested reasonably good reliability of the model equation developed to predict oil yield in the extraction process within the selected experimental domains.

Figure 7: Comparison of the actual with predicted oil yield

Quality of Essential Oil

The quality of the essential oil produced at the optimum conditions predicted by the model of 80 minutes of extraction time, heating rate of 6.76kJ/s, 2 $x10^{-3}m^{-3}$ of water and wet leave was analyzed in terms of physicochemical properties as presented in Table 6. The results obtained were in good agreement with the literature values [3].

Table 6: property of essential oil extracted from *eucalyptus citriodora* leave

Property	Experimental value	Literature value[3]	Method
Refractive index,	1.4517	1.4511-1.4570	AOCS Cc 7-25
Specific gravity@30°C	0.912	0.89-0.93	AOCS Cc 10a-25
Ester value, wt %	40.12	12-60	AOCS Ce 1-62
solubility (in 70% ethanol),v/v	1:3	1:3-1:5	AOCS Ce 1-26

CONCLUSION

In this study, essential oil was extracted from *eucalyptus citriodora* leave and the individual and interaction effect of the process parameters viz: extraction time, heating rate, volume of water and leave condition were studied. The conclusion derived from this study is as follows:

1. At higher extraction time, the oil yield increase with higher heating rate and minimum volume of water.
2. The main and interaction effects of essential oil extraction parameters using steam distillation method can be studied emphatically by factorial experimentation technique.
3. The extraction time, heating rate and volume of water has the maximum influence on extraction process.
4. The results obtained from the statistical analysis are in good agreement with the experimental findings for the extraction time, heating rate, water and leave condition. It was found that oil yield increases with increasing time of extraction, heating rate and decreasing volume of water.
5. The developed mathematical model can be used to predict the oil yield in terms of the process parameters investigated from any combinations within the range studied.
6. The developed model can also be employed for simulation of essential oil extraction process with respect to oil yield.

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