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OPTIMIZATION OF LEMON GRASS OIL EXTRACTION PROCESS USING FACTORIAL DESIGN TECHNIQUE

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ABSTRACT

The need for quality lemon grass oil for medicinal and other important purposes globally cannot be overemphasized. In this regard, there is the need to critically examine the combination of parameters (such as temperature, pressure, energy flow rate among others) that determine the quality and quantity of extracted lemon grass oil. However, the determination and combination of the critical extraction parameters have always been the major challenge in ensuring the quality and quantity of lemon grass oil. This paper, therefore, aims at determining the best combination of these critical extraction parameters that can give the best quality and quantity of the lemon grass oil. The objective of the research is to optimize the extraction of lemon grass oil processes using factorial design technique to develop and establish an empirical model to relate the extraction parameters. The empirical models were established using only response variables that had an effect on the critical parameters. These models can be used to monitor, forecast and control the operation parameters or processes of the extraction. It was found that, the mass of the lemon grass, the flow of the LPG and the interaction of the mass of grass and flow of LPG have significant effect on the quantity of LPG used. The operating pressure and the interaction of the mass of grass and flow of LPG were the only response variables that have significant effect on the quality of the oil obtained.

Keywords: *Parameters, Temperature, Pressure, Effects, Model*

INTRODUCTION

Lemon grass (*Cymbopogon citratus)* is an odorous tropical grass which yields oil that smells of lemon, used in cooking, perfumery and medicine (Concise Oxford Dictionary, 2000).The name Cymbopogon is derived from the Greek words ‘kymbe’ (boat) and ‘pogon’ (beard), referring to the flower spike arrangement (Shah et al., 2011). Cymbopogon citratus has been used by the Brazilian Quilombolas tribe to decrease blood pressure and to calm individual’s anxiolytic (Rodriques and Carlini, 2004). Cymbopogon citratus has been traditionally used to treat gastro-intestinal discomforts (Devi et al. 2011). Other uses include: medicinal, perfumery industry, vitamin A manufacturing, pharmaceuticals, as food crop. For example, it is used in herbal tea because of its sharp lemon flavour, as perfume in soaps, and as medicine to treat various health disorders, such as acne, athlete’s foot, turgidity, muscle aches and scabies (Rozzi,et al, 2002). Optimization of the lemon grass extraction process offers an alternative with the most cost effective or highest achievable performance under given constraints, by maximizing desired factors and minimizing undesired ones. Uncertainty of the extraction process offers the opportunity to optimize the process by varying key factors in order to get the best combination of factors using factorial technique that can give best quality and quantity of the oil.

Over the years, a lot of extraction methods have been developed and used globally for the extraction of essential oils. For the purpose of this paper, the direct steam distillation method (DSDM) (Kumar, 2010) was adopted because it enables a compound or mixture of compounds to be distilled and subsequently recovered at a temperature substantially below that of the boiling points of the individual constituents (Denny, 2001).

A factorial design technique is often used by researchers to understand the effect of two or more independent variables upon a single dependent variable each at two levels. These levels can be quantitative or qualitative (Andoh et al. 2007). The two levels are alluded as “low” and ‘high’, and indicated by ‘- ‘and ‘+’, individually (Andoh et al. 2007). It doesn’t make a difference which of the component qualities is connected with the “+” and which with the ‘- sign. The length of the naming is consistent. At the start of a 2 k factorial design, factors and levels are indicated. When we combine all of them, a design matrix is obtained (Pais et al., 2014).

OPTIMIZATION LEMON GRASS

The research reviews essential oil from lemon grass and also focuses on previous attempts at solving the extraction problem of lemon grass oil for medicinal and other purposes. In this research the direct steam distillation method (DSDM) was adopted because it enables a compound or mixture of compounds to be distilled and subsequently recovered at a temperature substantially below that of the boiling points of the individual constituents (Denny, 2001).With the direct steam refining technique, the plant material is bolstered on a punctured framework over the steam gulf, and steam generated from satellite steam generator is allow to pass through the plant material.

With the direct steam method, lemon grass *(Cymbopogon citratus),* water and LPG gas were the main materials used for the production of the oil. Fresh lemon grasses were harvested from a demonstration farm, washed, weighed and introduced into the boiling tank. The boiler was then closed tightly with bolts and nuts, to prevent steam leakage from the system and ready for the extraction process. The equipment was set up for the extraction, after which the furnace was lit with a liquefied petroleum gas (LPG) as the source of energy. Extraction was done as the steam was generated from the boiling tank and made to pass through the plant material in the boiler forcing the pockets of the lemon grass opened to extract the oil. The stopcock was closed so that the predetermined pressures and temperatures could be attained. After the required pressures and temperatures were reached, the stopcock was then opened and both the oil and water in the form of steam passes through the steam pipe and water runs through the condenser pipe serving as heat exchanger to condense the steam into liquid which drops into the separator. By virtue of density differences the oil is separated from the hydrosol and measured. The procedure is repeated for sixteen times with varying pressures and temperatures (Braimah et al., 2016). Temperature and pressure transducers were used to record the temperature and the pressure at the boiling chamber. Also the burner’s temperature was recorded by a thermocouple. The time duration for the whole process was recorded and the mass flow rate for the LPG used was set to

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flow between 0.8-1.00 kg per hour depending on the set parameters of the experiment. The amount of oil collected was measured, recorded, tabulated and used for the factorial computation of the optimization process (Braimah et al.,2016).

Process optimization is the discipline of adjusting a process so as to improve some specified set of parameters without violating some constraint. The most common goals are minimizing cost, maximizing output, and/or efficiency. This is one of the major quantitative tools in industrial decision making. Process optimization methods provide you with the tools and techniques you will need to successfully optimize your current processes. The programs will provide you with the knowledge, techniques and methods that are used to enhance processes (Braimah et al.,2016).

In Statistics, a full factorial experiment is an experiment whose design consists of two or more factors, each with discrete possible values or "levels", and whose experimental units take on all possible combinations of these levels across all such factors. Montgomery,(2008) characterizes a trial as a test or a progression of tests in which deliberate changes are made to the information variable elements of a procedure or framework with the goal that we might look at and distinguish the purposes behind changes that might be seen in the yield reaction. Measurable configuration of tests alludes to arranging the trial in a way that legitimate information will be gathered and dissected by factual strategies, bringing about substantial and target conclusions (Montgomery, 2008).

The analysis of a complex process requires the identification of target quality attributes that characterize the output of the process and of factors that may be related to those attributes. Once a list of potential factors is identified from subject matter expertise, the strengths of the associations between those factors and the target attributes need to be quantified. A naïve, one-factor-at-a-time analysis would require many more trials than necessary. Additionally, it would not yield information about whether the relationship between a factor and the target depends on the values of other factors (commonly referred to as interaction effects between factors) (Montgomery, 2013).

Exploratory outline has three standards these are: randomization, replication, and blocking. The request of the keeps running in the test outline is arbitrarily decided. Randomization helps in staying away from infringement of autonomy brought on by unessential components, and the supposition of freedom ought to dependably be tried. Replication is an autonomous rehash of every blend of components. It permits the experimenter to acquire an appraisal of the trial mistake. Blocking is utilized to represent the variability brought about by controllable irritation variables, to lessen and dispense with the impact of this component on the estimation of the impacts of hobby. Blocking does not kill the variability; it just confines its belongings.
The test arrangement for factorial design takes the following steps:

(i) defining of objectives of the experiment,
(ii) choosing measures of performance, factors to explore, and factors to be held constant
(iii) designing and executing the experiment.
(iv) analyzing the data and drawing conclusions and
(v) reporting the experiment's results (Barr et al., 1995).

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A 2 k factorial outline includes two factors, each at two levels. These levels can be quantitative or qualitative. The level of a quantitative element can be connected with focuses on a numerical scale, as the span of populace or the quantity of islands. For qualitative elements, their levels cannot be masterminded altogether of greatness, for example, topologies, or methodologies of determination. The two levels are alluded as "low" and 'high', and indicated by '-'and '+', individually. It doesn't make a difference which of the component qualities is connected with the "+" and which with the '- sign, the length of the naming is consistent. At the start of a 2 k factorial design, factors and levels are indicated. When we combine all of them, a design matrix is obtained (Pais et al., 2014). Manipulation of the effective factors results in mathematical models that could be used to monitor or control subsequent extraction processes.

A mathematical model is a description of a system using mathematical concepts and language. The process of developing a mathematical model is termed mathematical modeling. Mathematical models are used in various disciplines. A model may help to explain a system and to study the effects of different components, and to make predictions about behavior (Aris, 1994).

Mathematical models can take many forms, including dynamical systems, statistical models, differential equations, or game theoretic models. These and other types of models can overlap, with a given model involving a variety of abstract structures. In general, mathematical models may include logical models. In many cases, the quality of a scientific field depends on how well the mathematical models developed on the theoretical side agree with results of repeatable experiments. Lack of agreement between theoretical mathematical models and experimental measurements often leads to important advances as better theories are developed.

In the physical sciences, the traditional mathematical model contains four major elements. These Mathematical models are usually composed of relationships and variables. Relationships can be described by operators, such as algebraic operators, functions, differential operators, etc. Variables are abstractions of system parameters of interest, that can be quantified. Several classification criteria can be used for mathematical models according to their structure: (Bender, 2000).

MATERIALS AND METHODS

Factorial Design Experiment

The importance of factorial designs are: (1) per factor of study, few runs are required; (2) They indicate major trends that can determine likely directions for additional experimentation. Three quantitative variables, namely, the mass of the lemon grass (M-kg), the boiler pressure (P-bars) and the LPG flow rates (F-m^3/s) were used to study the response of the extraction. Table 1 shows the experimental matrix and the magnitude of the variables.

Table 1: The Experimental Matrix

Variables	**Lower Level (-)**	**Upper Level (+)**
Mass of grass M (kg)	20	27
Boiler Pressure P (bars)	1.4	2.6
LPG Flow rate f (g/s)	18.5	23.0

Note: (-) represents the lower level of the variables
(+) represents the upper level of the variables

Table 1 shows the experimental matrix, each extracting experiment run uses a combination of factors. These factors could be a combination of a lower factor, upper factor or both upper and lower factors combined.

ANALYSIS OF THE RESULTS

The main effect of each of the process variables reflects the changes of the respective responses as the process variables change from a low to a high level as shown in Table 2.

Table 2 Factor Interactions

Std	m	p	f	mp	mf	pf	mpf
1	-	-	-	+	+	+	-
2	+	-	-	-	-	+	+
3	-	+	-	-	+	-	+
4	+	+	-	+	-	-	-
5	-	-	+	+	-	-	+
6	+	-	+	-	+	-	-
7	-	+	+	-	-	+	-
8	+	+	+	+	+	+	+

Note: (-) represents the lower level of the variables
(+) represents the upper level of the variable

From table 3.2, the average of the four measures is the main effect of the factor (variable) and is given as:

The main effect of the mass is: $E_m = \frac{1}{4}\{ (A_2 + A_4 + A_6 + A_8) - (A_1 + A_3 + A_5 + A_7) \}$ (1)

The main effect of the pressure is: $E_p = \frac{1}{4}\{ (A_3 + A_4 + A_7 + A_8) - (A_1 + A_2 + A_5 + A_6) \}$ (2)

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The main effect of the LPG flow rate is: $E_f = \frac{1}{4}\{ (A_5 + A_6 + A_7 + A_8) - (A_1 + A_2 + A_3 + A_4) \}$ (3)

Two or more of the variables may jointly influence the responses. These joint influences are referred to as interactions. These interactions are given as:
The interaction between the mass and the pressure is defined as:

$$I_{mp} = \frac{1}{4}\{ (A_1 + A_4 + A_5 + A_8) - (A_2 + A_3 + A_6 + A_7) \} \quad (4)$$

The interaction between the mass and LPG flow rate is defined as:

$$I_{mf} = \frac{1}{4}\{ (A_1 + A_3 + A_6 + A_8) - (A_2 + A_4 + A_5 + A_7) \} \quad (5)$$

The interaction between pressure and LPG flow rate is defined as:

$$I_{pf} = \frac{1}{4}\{ (A_1 + A_2 + A_7 + A_8) - (A_3 + A_4 + A_5 + A_6) \} \quad (6)$$

The three-factor interaction is expressed as:

$$I_{mpf} = \frac{1}{4}\{ (A_2 + A_3 + A_5 + A_8) - (A_1 + A_4 + A_6 + A_7) \} \quad (7)$$

The mean of the runs is defined as:

$$E_M = \left[\sum_1^8 A_i / 8 \right] \text{(Andoh et al., 2007)} \quad (8)$$

Where A_i represents the extracting parameters, the estimates for the four parameters i.e. (amount of LPG Q, time T, quantity of oil q, and quality of oil C) are under consideration.

When genuine run replicates are created under a given set of experimental conditions, the variation among their associated observations are used to estimate the standard deviation of a single observation and, hence, the standard deviation of the results. In general, if "g" sets of experimental conditions are genuinely replicated and the n_i replicate runs made at the i^{th} set yield an estimate s_i^2 having $v_i = n_i - 1$ degree(s) of freedom, the estimate of run variance is (Hunter, 1978).

$$s^2 = \frac{v_1 s_1^2 + v_2 s_2^2 + v_3 s_3^2 + \ldots + v_g s_g^2}{v_1 + v_2 + v_3 + \ldots + v_g} \quad (9)$$

With only $n_i = 2$ replicates at each of the g sets of conditions, the formula for the i^{th}

Variance reduces to $s_i^2 = d_i^2 / 2$ (10)

with $v_i = 1$, where d_i is the difference between the duplicate observations for the i^{th} set of conditions.
Thus, Equation 9 will yield:

$$s^2 = \sum (d_i^2 / 2) / g \quad (11)$$

In general, if a total of N runs are made conducting a replicated factorial design, then the variance of an effect is given as:

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$$V(effect) = \frac{4}{N} s^2 \quad (12)$$

and the standard error of the effect is given as:

$$s_e = \sqrt{V(effect)} \quad (13)$$

A full model may consist of three main effects, three two-factor interaction and a three-factor interaction. This is defined as:

$$AT = [\alpha_0 + \alpha_1 m + \alpha_2 p + \alpha_3 f + \alpha_4 mp + \alpha_5 mf + \alpha_6 pf + \alpha_7 mpf] \quad (14)$$

where α_0, α_1,…, α_7 are the constants and m, p, and f are the mass of lemon grass, operating pressure and amount of LPG, respectively. It can be shown that a $\alpha_0 = Ea$, $\alpha_1 = \frac{E_m}{2}$, $\alpha_2 = \frac{E_p}{2}$, $\alpha_3 = \frac{E_f}{2}$, $\alpha_4 = \frac{I_{mp}}{2}$, $\alpha_5 = \frac{I_{mf}}{2}$, $\alpha_6 = \frac{I_{pf}}{2}$ and $\alpha_7 = \frac{I_{mpf}}{2}$ (Andoh et al., 2007).

DISCUSSION OF RESULTS

The Effects of the Processing Parameters

The results from Table 3, suggest that increasing the mass from 20 to 27 kg results in an increase in the quantity of oil of 39.85 cm^3; a decrease of time of 3 minutes; an increase of LPG of 0.41 kg; and a decrease of the quality of oil of1.68%. Increasing the pressure from 1.4 to 2.6 bar results in an increase of quality of oil by 2.46%, a decrease of time of 2.50 minutes, a decrease of LPG of 0.04 kg and an increase of the quantity of oil of 3.67 cm^3. Increasing the flow of LPG from 0.8 to 1.0 m^3/s results in a decrease of LPG 0f 0.39, a decrease of quality of oil of 2.24; a decrease of time of 3.50 minutes; and a decrease of the quantity of oil of -1.42. These results may be confirmed by the application of the experimental error as discussed below.

Table 3 Effects of Process Parameters on the Extraction

	Effects and Interactions			
	LPG (Q) (kg)	**Time (T) (minutes)**	**Quantity (q) (cm^3)**	**Quality (C) (%)**
E_a	3.92	213.25	141.36	36.52
E_m	0.41	-3.00	39.85	-1.68
E_p	-0.04	-2.50	3.67	2.46
E_f	-0.39	-3.50	-1.42	-2.24
I_{mp}	0.09	6.00	-1.21	-1.67
I_{mf}	0.29	5.00	-2.79	2.26
I_{pf}	-0.11	5.50	0.03	-1.04
I_{mpf}	0.01	-4.00	-1.35	-4.20

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From Table 4, it is not clear which of the estimates are important (factors) and which are unimportant (chance). By examining the confidence intervals of each result, it can be determined if each effect or interaction is significant (a factor). However, it is established that, if the range of an effect include zero then, it is by a chance, otherwise it is a factor.

Table 4 The Factorial Experimental results

	Effects and Interactions			
	LPG (Q)	**Time (T)**	**Quantity (q)**	**Quality (C)**
E_a	3.92	213.25	141.25	36.52
E_m	0.41±0.21	-3.00±10.49	39.85±2.05	-1.68±14.81
E_p	-0.04±0.21	-2.50±10.49	3.67±2.05	2.46±14.81
E_f	-0.39±0.21	-3.50±10.49	-1.42±2.05	-2.24±14.81
I_{mp}	0.09±0.21	6.00±10.49	-1.21±2.05	-1.67±14.81
I_{mf}	0.29±0.21	5.00±10.49	-2.79±2.05	2.26±14.81
I_{pf}	-0.11±0.21	5.50±10.49	0.03±2.05	-1.04±14.81
I_{mpf}	0.01±0.21	-4.00±10.49	-1.35±2.05	-4.20±14.81

Table 5 shows the results obtained, the effects and interactions that are significant (factor). The significant effects and interactions are used to develop the empirical model for each response with the use of Equation 14.

Table 5 Factorial Experimental for Factor/Chance results

	Effects and Interactions			
	LPG (Q)	**Time (T)**	**Quantity (q)**	**Quality (C)**
E_a	3.92	213.25	141.25	36.52
E_m	factor	chance	factor	chance
E_p	chance	chance	factor	factor
E_f	factor	chance	chance	chance
I_{mp}	chance	chance	chance	chance
I_{mf}	factor	chance	factor	factor
I_{pf}	chance	chance	chance	chance
I_{mpf}	chance	chance	chance	chance

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Development of the Predicted Model

From Table 3, the mass of the lemon grass, the flow of the LPG and the interaction of the mass of grass and flow of have a significant effect on the amount of LPG used. Hence, the empirical model for the LPG (Q) is

$$Q = \left[3.92 + \left(\frac{0.41}{2}\right)m - \left(\frac{0.39}{2}\right)f + \left(\frac{0.29}{2}\right)mf\right] \qquad (15)$$

Similarly, the model for the quantity of oil (q) and the quality of oil (C) are:

$$q = \left[141.25 + \left(\frac{39.85}{2}\right)m + \left(\frac{3.67}{2}\right)p - \left(\frac{2.79}{2}\right)mf\right] \qquad (16)$$

$$C = 36.52 + \left(\frac{2.46}{2}\right)p + \left(\frac{2.26}{2}mf\right) \qquad (17)$$

Where Q, q, and C are the maximum values for the LPG, quantity of oil, and Quality of the oil responses respectively. Since the process parameters (m, p, and f) are coded, their values are -1 to +1 in these models.

Verification of Model

The value of LPG Q is calculated at each experimental condition and then compared with the mean value. For example, at the lower values of m, p and f,

$$Q = \left[3.92 + \left(\frac{0.41}{2}\right)m - \left(\frac{0.39}{2}\right)f + \left(\frac{0.29}{2}\right)mf\right]$$

$$Q_1 = \left[3.92 + \left(\frac{0.41}{2}\right)(-1) - \left(\frac{0.39}{2}\right)(-1) + \left(\frac{0.29}{2}\right)(-1)(-1)\right] = 4.055$$

This process is then repeated for the remaining process parameters and the results obtained are presented in Table 4.3. The procedure is repeated for the quantity and quality of oil produced, and the results are presented in Table 6.

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Table 6: Comparison of the Measured Mean and the Model Mean

LP Gas			Quantity of Oil			Quality of Oil		
4.05	4.06	-0.12	119.00	118.10	0.76	39.12	36.42	6.91
4.1	4.18	-1.83	161.50	160.74	0.47	32.67	34.16	-4.58
4.05	4.06	-0.12	122.50	121.77	0.60	40.10	38.88	3.05
4.25	4.18	1.76	165.28	164.41	0.53	38.69	36.62	5.35
3.5	3.38	3.57	119.00	120.89	-1.58	31.47	34.16	-8.56
4.1	4.08	0.61	158.61	157.95	0.42	37.92	36.42	3.96
3.25	3.38	-3.85	125.25	124.56	0.55	38.76	36.62	5.52
4.05	4.08	-0.62	159.75	161.62	-1.17	33.47	38.88	**-16.16**

Table 6 shows that the error percentage for the LPG Gas is within a range of 0.12% to 3.85%. This finding means that the two sets of mean LPG are in close agreement. Similarly, the results obtained for the quantity of the oil produced are also within the experimental error except however, one of the results obtained for the quality of oil produced does not agree with the experimental data. Hence that value needs to be investigated if this model may be used for further work.

CONCLUSION

It can be concluded from the findings that, the significant effects of the response variables were used to develop models that relate the operating conditions. These models can be used to monitor, forecast and control the operation parameters or process of the extraction. The research also concluded that, the quantity of LPG used was greatly influenced by the interaction of the mass of lemon grass and the flow rate of liquefied petroleum gas (mf), the mass of lemon grass (m), and the flow rate of the LPG (f), hence, the reason for the development of the empirical model for the LPG (Q).

The operating pressure (P), the mass of lemon grass (m), and the interaction of the mass of the lemon grass and flow rate of liquefied petroleum gas (mf) have substantial influence on the quantity of the oil obtained. Hence, the development of the empirical model for the quantity oil (q).

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The operating pressure (p) and the interaction of the mass of lemon grass and flow rate of liquefied petroleum gas (mf) have substantial influence on the quality of the oil obtained, hence, the need for the development of the empirical model for the quality of oil (C).

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