OPTIMIZATION OF PATCHOULI OIL EXTRACTION USING DESIGN OF EXPERIMENT (DOE) METHOD

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Thesis submitted to the Faculty of Chemical and Natural Resources Engineering in Partial Fulfillment of the Requirement for the Degree of Bachelor of Chemical Engineering

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MAY, 2008

I declare that this thesis entitled *"Optimization of Patchouli Oil Extraction By Employing Design of Experiment (DOE) method"* is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Date	**: 15 MAY 2008**

DEDICATION

Special dedication to my beloved mother and father, Saniyah bt Hj Ideris and Ismail b Abd Rahman, my aunty and uncle and all my family members that always inspire, love and stand besides me, my supervisors, my beloved friends, my fellow colleague, and all faculty members

For all your love, care, support and believe in me. Thank you so much.

ACKNOWLEDGEMENT

First and foremost, I would like to express my high gratitude to Allah s.w.t for his guidance and blessing I received during good time and hard time in completing this research.

I wish to convey my deepest appreciation and sincere to my previous supervisor, Cik Norlisa bt Harun, for his guidance, advice and criticism for my work during early stage of research. I'm very much indebted with her. I also would like to dedicate an appreciation to my current supervisor, Cik Rohaida bt Che Man for her help, guidance, patience and building criticism for my work until it fully complete.

Special thanks go to postgraduate student Cik Asdarina bt Yahya for his excellent supervise and guidance on my work especially during experiment stage and regarding the equipment and material used for this research. Special thanks also to my research colleague Cik Dewi Haryani bt Sulaiman for her help during experiment work.

I also would like to express my deepest gratitude to my family especially for my beloved mother and father, Saniyah bt hj. Ideris and Ismail bin Abd Rahman for their moral support over the completion of this research.

Last but not least, I would like to extend my gratitude to all my colleagues and friend, Fauzi, Hakim, Nabil, Zafaruddin, Saifuddin, Munzir, Izzuddin and all member of my degrees class for their friendship and cooperation during my research and undergraduate study.

ABSTRACT

Patchouli oil is one of the essential oil that has a wide variety of application in cosmetic and aromatherapy industry which used as one important ingredient in perfume and soap. Patchouli also has a potential to be used as an insect repellent and antiseptic product in future. Patchouli oil is obtained using a steam distillation where an aromatic chemical is extracted from patchouli leaves by steam and condensed which provide relatively high yield of product and low cost of running the process. This study is aim to improve a yield of patchouli oil extraction using steam distillation by manipulating the process factor such as extraction time and mass of raw material. The optimization of extraction of patchouli oil is carried out using Response Surface Methodology (RSM) based on Central Composite Design (CCD). Response Surface Methodology is a branch of Design of Experiment (DOE) which a response of interest is influenced by several variables of the process and the main objective of using this method is to find the optimum condition. The optimize condition for steam distillation of patchouli oil was found at 2.38 kg of patchouli leaves and 6.84 hour of extraction time. The yield of 0.6289 percent (weight percent) was achieved using this optimized condition. This gave 67.7 percent increment of yield percentage in comparison to the initial experiment which gives 0.3751 percent of yield.

ABSTRAK

Minyak patchouli adalah salah satu daripada jenis minyak wangi yang mempunyai kegunaan yang meluas dalam industri kosmetik dan aromaterapi dimana ianya digunakan sebagai bahan yang penting untuk pembuatan minyak wangi dan sabun. Minyak patchouli juga mepunyai potensi untuk digunakan sebagai bahan penghalau serangga dan sebagai produk antiseptik pada masa hadapan. Minyak Patchouli juga diperolehi dengan menggunakan proses penyulingan stim dimana bahan kimia aromatik diekstrak dari daun patchouli oleh stim dan dikondensasikan dimana proses ini secara perbandingannya mempunyai hasil yang tinggi daripada proses yang lain dan mempunyai kos yang rendah. Kajian ini bermatlamat untuk memperbaiki hasil minyak patchouli menggunakan proses penyulingan stim dengan memanipulasi faktor proses pengekstrakan seperti masa pengekstrakan dan juga berat bahan mentah. Proses pengoptimuman penghasilan minyak patchouli dijalankan menggunakan kaedah gerak balas permukaan (RSM) berdasarkan kepada rekabentuk komposit berpusat (CCD). Kaedah gerak balas permukaan merupakan salah satu cabang rekabentuk eksperimen (DOE) dimana gerak balas dipengaruhi oleh beberapa faktor kepada proses dan objektif utama menggunakan kaedah ini adalah untuk mencari keadaan optimuman proses tersebut. Proses pengekstrakan dijalankan menggunakan loji pandu penyulingan stim .Didapati keadaan optimuman untuk penyulingan stim bagi minyak patchouli adalah pada berat 2.38 kg bagi daun patchouli dan 6.84 jam masa pengekstrakkan. Hasil sebanyak 0.6289 peratus telah diperolehi menggunakan keadaan optimuman ini. Ini meberikan peningkatan 67.7 peratus terhadap peratusan hasil berbanding sebelum ekperimen iaitu 0.3751 peratus hasil.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Introduction

Patchouli oil is one of the essential oil which getting a strong interest due to its wide variety of application including in cosmetic and pharmaceutical industries. It has been used as a scent in several of product such as paper towels, laundry detergents and air fresheners. Patchouli oil can be found as a major ingredient of most high end perfume as it has an ability to retain other scent for longer time. Patchouli oil has a number of benefits including in health aspect such as balancing emotion, fighting depression and anxiety. It also able to act as an antiseptic and promote a faster healing of wound. Most of patchouli oil is obtained through a steam distillation of leaves which produces a high yield of oil compared to other method.

Design of Experiments (DOE) is an analysis tool that used by researchers, field engineers and scientists to find the optimize point for their process. Design of Experiments was developed by Sir Ronald Fisher back in 1920s at the Rothamsted Agricultural Field Research Station in London, England. His initial experiment concerning about determining effect of various fertilizer on different plot of plant. Sir Ronald Fisher used DOE to differentiate the effect of fertilizer and other factors. Since then DOE has been accepted as analysis tool for researcher. The important key in DOE is to find the relationship between input variables and output performances or output quality of the process. For example injection molding process, the input variables such as molding temperature, pressure, type of raw material is customize to find the best output performances such as length of molded part (Anthony,2003). One of the common approaches used by scientists, researchers and engineers is One-Variable-At-a –Time (OVAT), where we vary one variable at a time keeping all other variables in the experiment fixed, this approach depends on guesswork, luck and intuition for its success. OVAT often unreliable, inefficient, time consuming and yield an inaccurate optimum for the process.

The potential of DOE in manufacturing processes includes:

1) Improve yield and stability
2) Improve profits and return on investment
3) Improve process capability
4) Reduce manufacturing costs
5) Reduce process design and development time

Since 1990s the complex statistical analysis of DOE has been simplified using computer software that specially developed to assist the researcher in optimizing the process. A software like Design Expert® has reduce the difficulty of statistical analyzing for moderate skill researcher and field engineer and widen the usage of this method and all the potential of DOE could be manipulated to benefits the manufacturer from various aspects in processing and manufacturing of product.

1.2 Problem Statement

In this research, a steam distillation method is employed to extract important aromatic ingredient. The yield of essential oil from this method is low which is around 0.5% to 2% from raw material weight.

The yield could be further optimized by varying possible input variables from the process such as flow of team, solid to solvent ratio, extraction time and others. The reason of choosing Design of Experiment (DOE) as a method to optimize this process because it is only involving a simulation from input data using computer software thus saving cost, time consume without jeopardize the integrity of result.

1.3 Objective

The main objective of this research is to optimize the process of patchouli oil extraction by employing Design of Experiment (DOE) method.

1.4 Scope of Research

To achieve the above objective, the following research scope has been identified:

i) To identify the major input variables which are extraction time and mass of raw material that affects the yield of patchouli oil from extraction process.

ii) To determine the optimum value of input variables and to optimize the output/ yield of patchouli oil.

CHAPTER 2

LITERATURE REVIEW

2.1 Patchouli Oil

Patchouli (also patchouly or pachouli) is both a plant and an essential oil (patchouli oil) obtained from the leaves of a plant of the same name. The scent of patchouli is heavy and strong. It has been used for centuries in perfumes and is grown in the East and West Indies.

Patchouli oil and incense underwent a surge in popularity in the 1960s and 1970s. During the Vietnam War, American soldiers used patchouli to mask the smell of the graves of enemy soldiers killed in combat. Despite its common association with an alternative lifestyle, patchouli has found widespread use in modern industry. It is a component in about a third of modern, high-end perfumes, including more than half of perfumes for men. Patchouli is also an important ingredient in East Asian incense. It is also used as a scent in products like paper towels, laundry detergents and air fresheners. The essential oil is obtained by steam distillation of the dried leaves of the plant which provides a relatively high yield of the oil. An important component of the essential oil is patchoulol (Maude, 1995).

During the 18th and 19th century silk traders from China traveling to the Middle East packed their silk cloth with dried patchouli leaves to prevent moths from laying their eggs on the cloth. Many historians speculate that this association with opulent eastern goods is why patchouli was considered by Europeans of that era to be a luxurious scent. This trend has continued to the present day in modern perfumery.

The plant and oil have a number of claimed health benefits and its scent is used with the aim of inducing relaxation. The patchouli plant is a bushy herb reaching two or three feet in height. The plant grows well in southern climates. It enjoys hot weather but not direct sunlight. If the plant withers due to lack of watering it will recover well and quickly once it has been watered. The seed-bearing flowers are very fragrant and bloom in late fall. The tiny seeds may be harvested for planting, but they are very delicate and easily crushed. Cuttings from the mother plant can also be rooted in water to produce further plants. Patchouli is a tropical member of the mint family, grown in the East and West Indies (Maude, 1995). Leaves are harvested several times a year, dried and exported for distillation of the oil, although the highest quality oil is usually produced from fresh leaves, distilled close to the plantation. Figure 2.1, 2.2 and 2.3 showed a patchouli flower, patchouli plant and dry patchouli leaves.

Figure 2.1: Patchouli flower

Figure 2.2: Bushy of patchouli leaves

Figure 2.3: Patchouli leaves (cut)

2.1.1 Application of Patchouli Essential Oil

Patchouli oil has grounding and balancing effect on the emotions and banishes lethargy, while sharpening the wits, fighting depression and anxiety. It is also said to create an amorous atmosphere. It is effective for fungal and bacterial infection and is of great help for insect bites. It could also be used as an insect repellant and is also used as a support for dealing with any substance addiction. With its excellent diuretic properties, it is effective in fighting water retention and to break up cellulite, easing constipation and helping to reduce overweight (Foster, 1996). Furthermore, it has a great deodorizing action and helps when feeling hot and bothered, and assisting with wound healing. On the skin, this oil is one of the most active and is a superb tissue regenerator, which helps to stimulate the growth of new skin cells. In wound healing, it not only promotes faster healing, but also helps to prevent ugly scarring when the wound heals.

2.2 Essential Oil Extraction Method

There are various ways to extract essential oil from aromatic plant. The type of botanical plant material that determine the method. The common methods are solvent extraction and steam distillation. Beside type of material, cost available and scale of the process also affect the selection of extraction method.

2.2.1 Steam Distillation

To extract the essential oil, the plant material is placed into a still where pressurized steam passes through the plant material. The heat from the steam causes globules of oil in the plant to burst and the oil then evaporates. The essential oil vapor and the steam then pass out the top of the still into a water cooled pipe where the vapors are condensed back to liquids. At this point, the essential oil separates from the water and floats to the top.

2.2.2 Solvent Extraction

A hydrocarbon solvent is added to the plant material to help dissolve the essential oil. When the solution is filtered and concentrated by distillation, a substance containing resin, or a combination of wax and essential oil remains. From the concentrate, pure alcohol is used to extract the oil. When the alcohol evaporates, the oil is left behind. This is not considered the best method for extraction as the solvents can leave a small amount of residue behind which could cause allergies and effect the immune system.

2.3 Design of Experiment

Design of Experiments is a good tool to analyze the potential of process to be further optimized. There a lot of researcher and engineer using this method in various fields such as biotechnology, botanical, pharmaceutical and others field of engineering (Lazic, 2004). Design of experiment also a common method in medical science, design of experiment was used by James Lind, a surgeon on HM Bark Salisbury to discover the remedies for scurvy among sailor. He was experimenting with five potential remedies using six pair of patient until he found that citrus fruits are the answer for the disease (Hunter, 2005). The modern technique of DOE was invented by Sir Ronald A. Fisher in 1920s and since then there a lot of improvement in this method such as applying mathematical and statistical model and development of computer software.

2.3.1 Fundamentals of Design of Experiment

In order to properly understand Design of Experiments (DOE), it is important to have a good understanding of the process. Basically, a process is transformation of inputs to outputs under appropriate circumstances. In context of manufacturing, input is a process variable such as peoples, materials, methods, environment, machines, procedures, etc and output could be a performances or quality of a product. Output also can refer as a responds. In performing a Design of Experiment, input variables or machine variables is intentionally changed in order to observe corresponding changes in the output variables. The information gained from properly plan, execute, and analyzed experiments can be used to improve functional performance of product (Lazic, 2000).

In Figure 2.4, output is performance characteristic which are measured to access product and process performance. Controllable variables can be varied easily during experiments and such variables have a key role to play in process characterization. Uncontrollable variables are difficult to control during an experiment. These variables are responsible for variability in product performances or product inconsistency. It is important to determine the optimal setting of controllable variables in order to minimize the effect to uncontrollable variables.

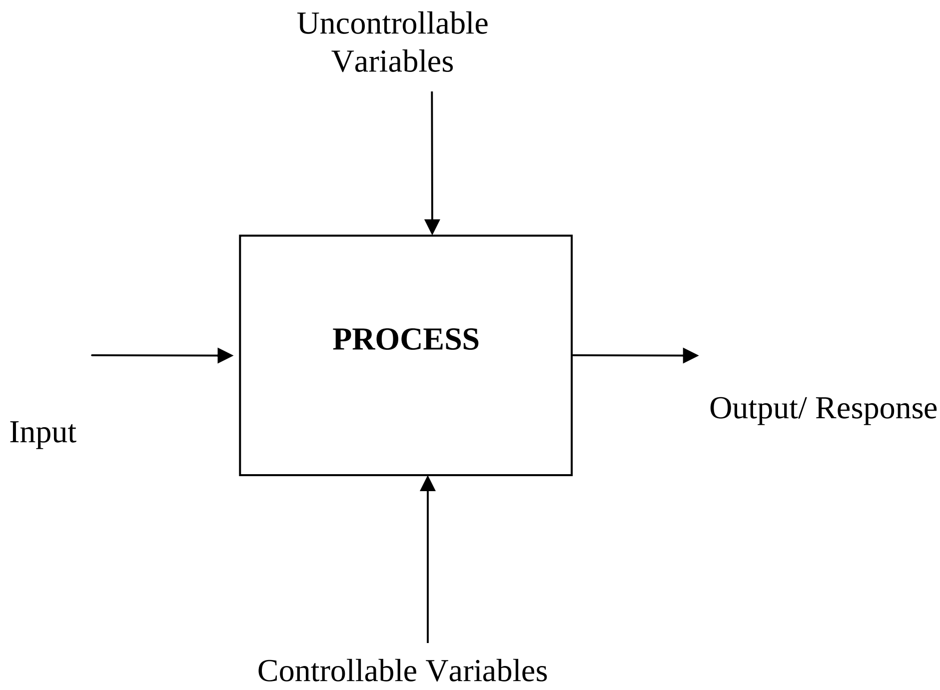

Figure 2.4: General model of process

It is important to define the specific objective to obtain from experiment before starting to use Design of Experiment The objective will determine a type of Design of Experiment that suitable for the study. The objectives for the experiment are best determined by a discussion if experiment is conduct in group. The group should discuss which objectives are the key ones, and which ones are quite good but not really necessary. Prioritization of the objectives helps to decide which direction to go with regard to the selection of the factors, responses and the particular design (Lazic, 2000). Sometimes prioritization will force to start over from beginning when realize that the experiment decided to run does not meet one or more objectives.

Types of designs are listed here according to the experimental objective that needs to be met:

Comparative objective: One or several factors under investigation, but the primary goal of experiment is to make a conclusion about one important factor and the question of interest is whether or not that factor is significant, this is a comparative problem and need a comparative design solution.

Screening objective: The primary purpose of the experiment is to select or screen out a few important main effects from the many less important ones. These *screening designs* are also termed main effects designs.

Response Surface (method) objective: The experiment is designed to allow estimate interaction and even quadratic effects and therefore give an idea of the (local) shape of the response surface. For this reason, they are termed R*esponse Surface Methodology (RSM) designs*. RSM designs are used to find improved or optimal process settings, troubleshoot process problems and weak points, make a product or process more *robust* against external and non-controllable influences. Robust means relatively insensitive to these influences.

Optimizing responses when factors are proportions of a mixture objective: A factors are proportions of a mixture and the objective is to know what the best proportions of the factors to maximize (or minimize) a response, then it is a mixture design.

Optimal fitting of a regression model objective: Model a response as a mathematical function of a few continuous factors and want a good model parameter estimates, then it is a regression design.

There are several experimental design methods that available which are:

1) Completely randomized design
2) Randomized block design
3) Full factorial design
4) Fractional factorial design
5) Plackett- Burmann design
6) Response surface design
7) Three level full factorial design
8) Three level, mixed level, and fractional factorial

The guideline to choose design methods based on objectives and number of factors is summarized in Table 2.1:

Table 2.1: Design selection guideline

Number of Factors	**Comparative Objective**	**Screening Objective**	**Response Surface Objective**
1	1 Factor completely randomized design	-	-
2-4	Randomized Block Design	Full or Fractional factorial	Central composite or Box- Behnken
5 or more	Randomized Block Design	Fractional factorial or Plackett- Burman	Screen First to reduce number of factors

2.3.2 Response Surface Methodology (RSM)

Response surface methodology (RSM) explores the relationships between several explanatory variables and one or more response variables. The main idea of RSM is to use a set of designed experiments to obtain an optimal response. Box and Wilson suggest using a first-degree polynomial model to do this. They acknowledge that this model is only an approximation, but use it because such a model is easy to estimate and apply, even when little is known about the process (Wilson, 1951).Response surface methodology or RSM is a collection of mathematical and statistical technique that are useful for the modeling and analysis of problem in which a response of interest is influenced by several variable and the objective is to optimize this response.

2.3.3 Blocking in Design of Experiment

Blocking is generally a method of eliminating the effect of variation due to noise factors and thereby improves the efficiency of experimental design. The main purpose is to eliminate unwanted sources of variability such as batch to batch, day to day or shift to shift. The block is as set of homogenous experimental condition. The block can be batches of raw material, different operators and different vendors. Experiment in same condition like day, shift fall under same block. Variability between block must be eliminated from experimental error, which significantly increase in the precision of the experiment (Anthony, 2003).

2.3.4 Case Study of Design of Experiment (Applying DOE to Microwave Popcorn)

One of the common cases to demonstrate the practices of DOE in optimization is using DOE in microwave popcorn production (Handerson, 1972). The response or output performances are to reduce unpopped corn kernel, burn corn and improving taste. Before starting the experiment, an independent variables is identified which is raw material brand, cooking time, microwave oven temperature, preheat time and tray elevation. Two brand of the corn is bought from the local store which is international brand and a cheaper local brand. The objective is to relate the quality of the final popcorn with the brand of raw material. A quick study of the instruction on every corn packaging say almost the same thing which is cooking time between 3 to 7 minutes, and oven temperature between medium and high setting. Two instructions were inconsistently appear in every packaging which is preheating and elevation of the corn from floor of oven. Two level factorial designs are used since every variable only involving two values.

Table 2.2: Factors and levels of popcorn

Factor	**Low Level(-)**	**High Level (+)**
Price	Generic	Brand
Time	4 Min	6 Min
Power	Medium	High
Preheat	No	Yes
Elevate	No	Yes

A statistically desirable array of combinations of the low and high levels was built, for a total of 16 runs, half the total number 32 of combinations possible. Such a fractional factorial design is sufficient to learn all we needed to know about popping popcorn. In fact, making more runs would not add to our know-ledge. It is not necessary to run all 32 combinations to study the interactions between factors. The runs were randomized to protect the study against lurking variables, such as changes in the environment that could otherwise confound the study. To simplify the administration of such a study, Design-Ease® software is used for design of experiments. It handled randomizing the samples and the statistical analysis. To measure the effects of the variable factors in each run, three response factors were considered. First the unpopped kernels (bullets) were weighed and the weight recorded. Likewise, burnt popcorn was collected from each sample run and weighed. However, this response turned out to be unreliable. The third response is which taste was subjective, but finding people willing to serve on a judging panel was not difficult in this case. Taste evaluations were recorded using a scale from 1-10, with 10 being high or good. Observed values ranged from 1.0 to 9.0. Observations from the 18 runs were then entered in the Design-Ease package. The software calculated the effect each independent variable and combination of variables had on the responses.

The software automatically produced a graph, called the normal plot of effects that helped isolate the factors that were key to determining the yield – the percentage of unpopped bullets. Figures 2.5 and 2.6 shows the main effects and two factor interactions for the two measurable responses. The trivial many factors, which had no influence, fall on a straight line near the zero effect level.

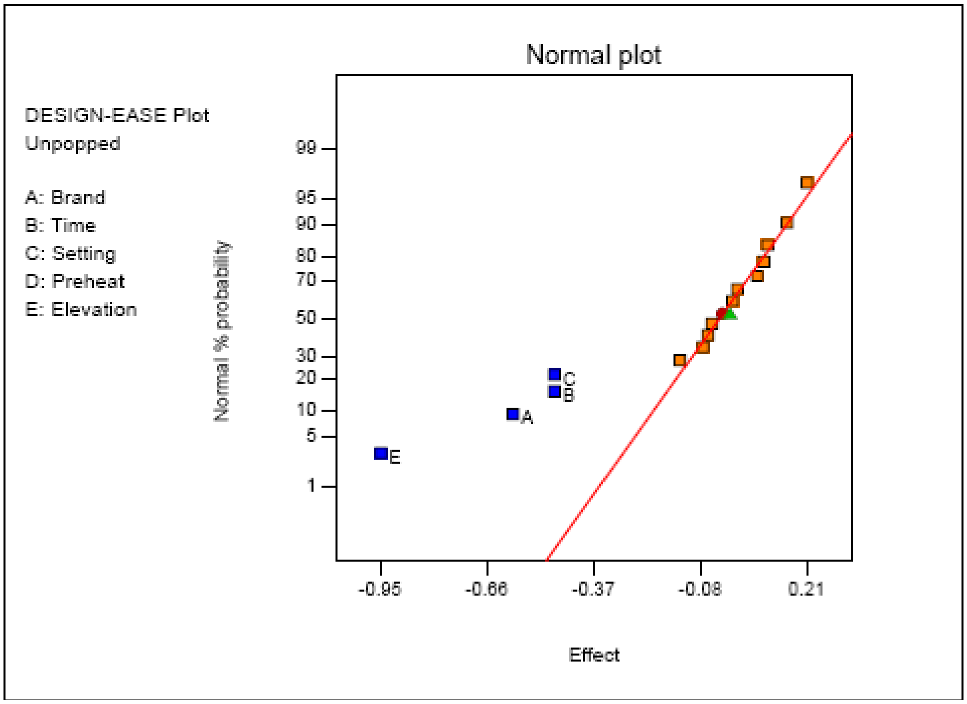

Figure 2.5: Analysis of unpopped kernels

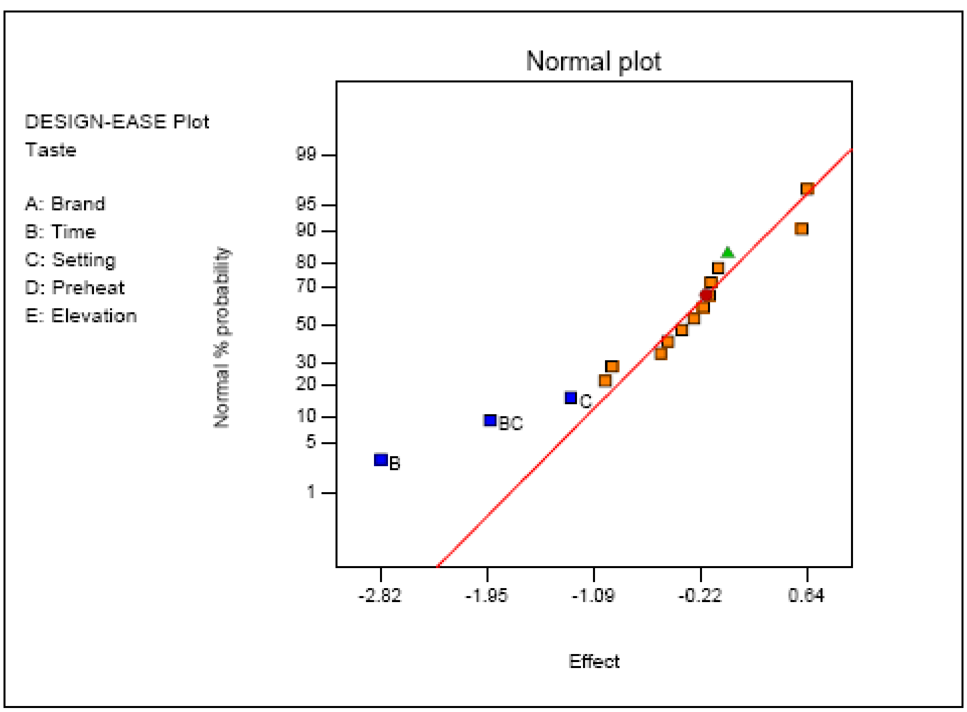

Figure 2.6: Analysis of popcorn taste

One of these factors was the preheating step. Preheating thus had no impact the responses. This is an important outcome because it means we don't have to wait an extra minute for the popcorn. The four remaining factors (brand, time, temperature, and elevation) significantly affected the bullets. Residual analysis by Design-Ease™ revealed the possibility that run 2 was an outlier for bullets. This experiment produced an unusually low amount of popcorn, but since no special cause could be attributed to this, and it did not greatly affect the findings, it's included in the results. Figure 2.6 shows the normal plot of effects for the taste response. It reveals highly significant interaction between time and temperature. The biggest effect comes from the time alone, but its impact depends on the level of temperature. The result is to cook the raw material at moderate setting temperature, a little bit off the floor, and using international brand to produce less bullets and tastier popcorn. It is also concluded that preheating does no effect to popcorn production.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This research involving a data simulation to optimize the extraction process. A raw data is collected from respectable resource such as operating condition, input variable value which is extraction time and mass of raw material, output or response value which is yield percentage in weight percent. Then a data is enter to DOE software (Design Expert™ 7) to simulate the process and identify major input variable as well as provide with suggestion of number of experiment that need to be run to perform analysis. After all experiment is completed, the corresponding yield data is key in into Design Expert to finalize the analysis and come out with suggestion of optimum value for this process. Figure 3.1 shows the summary flow of this research.

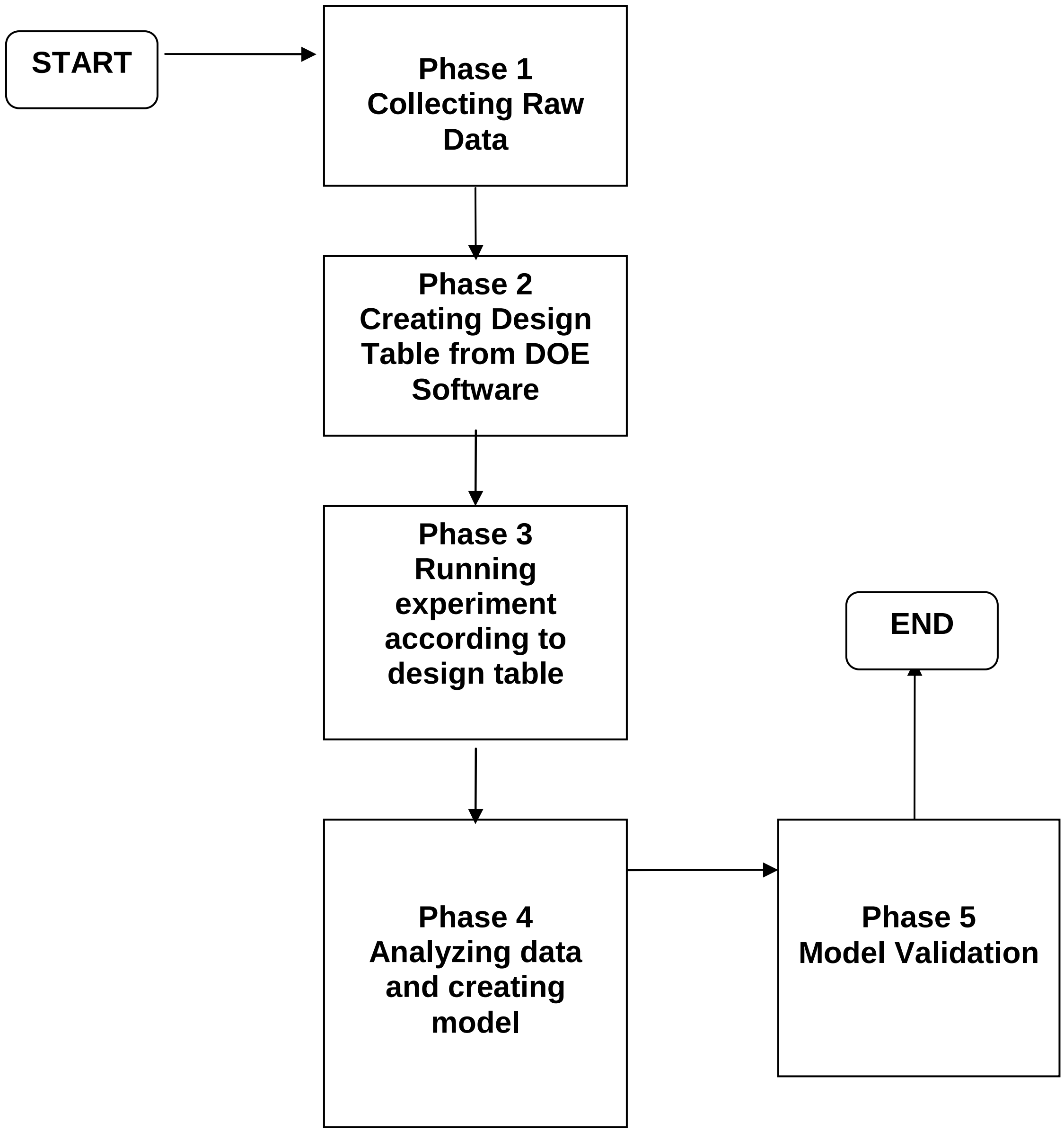

Figure 3.1: Flow of Research

3.1.1 Phase 1: Collecting Raw Data

Raw data is obtained to set a possible range of input parameter that need to be optimized. In this research there are two input parameter that need to be optimized which is extraction time and mass of raw material.

a) Extraction time- An optimum extraction time that able to extract a maximum essential oil without wasting utilities used for process like steam.

b) Mass of raw material- An optimum mass of patchouli leaves inside pilot plant without affect distribution of steam.

3.1.2 Phase 2: Creating Design Table

In this phase, a method of Design of Experiment (DOE) is decided according to type of objective of the experiment. In this experiment the objective is to optimize the chosen parameter as well as to maximize the yield of oil. The suitable method of Design of Experiment is Response Surface Methodology (RSM) which complies with experiment objective. Response surface methodology is selected in Design of Expert 7.1 along with number of parameter and number of respond or output. High level and low level for every parameter is set according to range that has been verified possible to the experiment. High level and low level is maximum and minimum level for every input parameter. In this experiment, two parameter is taken as input parameters are optimize which is extraction time (hr) with range 3 hour to 6 hour and Raw material mass (kg) with range 2 kg to 4 kg. Design Expert 7 will suggest a number of experiments along with corresponding value of input parameters for every experiment.

3.1.3 Phase 3: Experiments

An experiment is conducted according to number of experiment and value of input parameters suggested by software. In this experiment a steam distillation pilot plant is used to extract essential oil from patchouli leaves. Experiment methodologies are summarized in Figure 3.2 below.

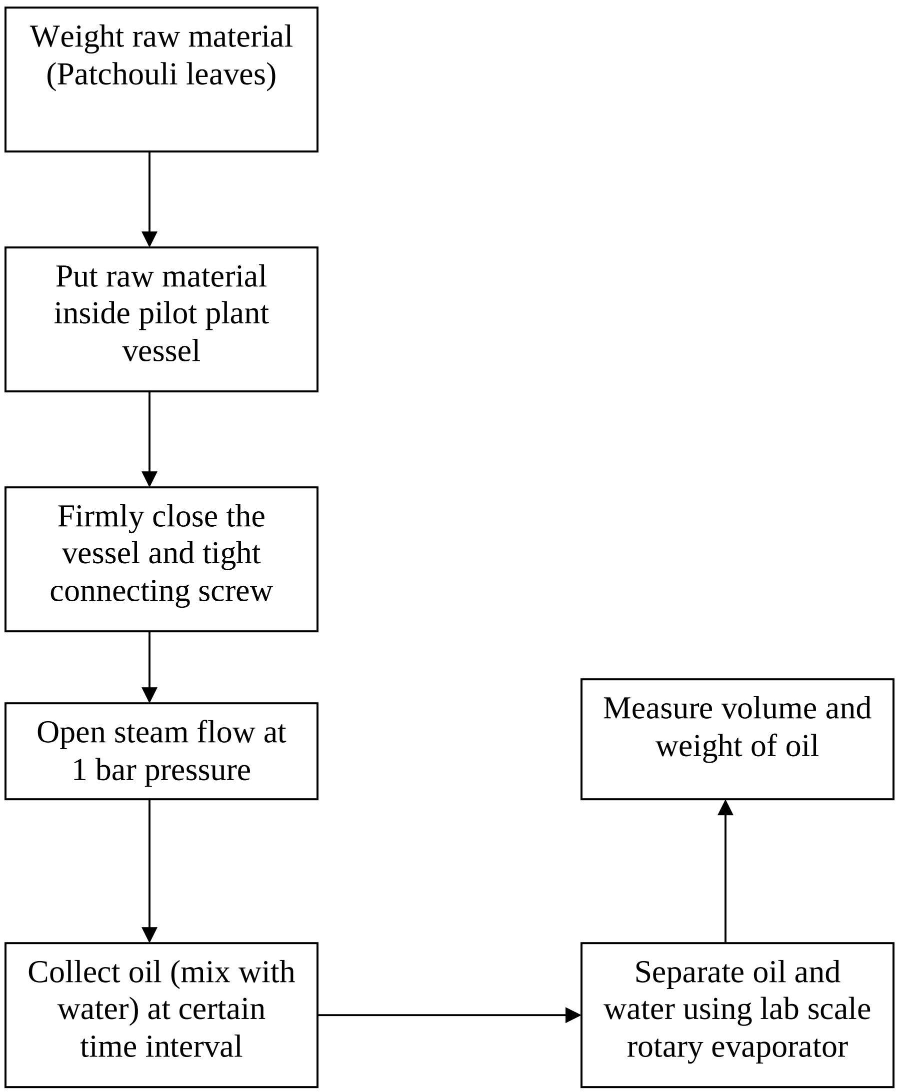

Figure 3.2: Experiment Methodologies

3.1.4 Phase 4: Analysis

Analysis is performed after all experiment is completed and all result needed by software is available. Analysis is performed by using several features from Design Expert 7 according to Design of Experiment statistic method.

3.1.5 Phase 5: Model Validation

Validation of model is made by running an experiment with predicted optimum value of input from analysis before. A model is validated if experiment value show the respond is in 10 percent range error.

3.2 Steam Distillation Pilot Plant

To ensure the consistency and reliability of data, all experiment is carried out using same pilot plant. Steam distillation pilot plant is consisting of several parts which are vessel or chamber, condenser, oil collection system. Most of part is made of stainless steel to ensure the durability. The chamber inside vessel contains three level of tray and all trays are consistently used through out the experiment to ensure there is no effect on the extraction yield. Figure 3.3 shows the picture of pilot plant.

Figure 3.3: Steam Distillation Pilot Plant

Figure 3.4: Condenser unit

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

The objective of this research is to find an optimum value of input parameters of extraction of patchouli essential oil using newly developed pilot plant of steam distillation. Before that, there are two input parameters that possible to be optimized from the pilot plant. First is weight of raw material and the second input parameter is extraction time. The optimum value of weight of raw material is has a direct impact to the amount of essential oil extracted since a large amount of patchouli leaves able to limit the quantity of steam reach to the condenser at the top of raw material chamber. While the extraction time also has an impact, logically the longer time of extraction the more essential oil produced. However the maximum extraction time could lead to utilities waste like steam and chilling water because the rate of essential oil produces could drop down to negligible figure.

4.2 Experimental Result of Patchouli Leaves Essential Oil Extraction

An experiment is carried out to find the respond value (weight percent of essential oil) of combination of input parameters required by Design Expert 7. Table 4.1 shows the result of in weight percent of essential oil by different combination of raw material mass and extraction time.

Table 4.1: Experimental Result of Steam Distillation

Mass of raw material (kg)	Extraction Time (hr)	Volume of Essential Oil (ml)	Weight of Essential Oil (g)	Weight Percent Of Essential Oil (%)
2.00	3.0	7.0	6.3588	0.3179
2.00	4.0	9.0	8.1756	0.4088
2.00	5.0	11.3	10.2649	0.5133
2.00.	6.0	12.5	11.3550	0.5678
2.00	7.0	13.1	11.9000	0.5950
3.00	3.0	12.0	10.9018	0.3634
3.00	4.0	14.0	11.7801	0.3927
3.00	5.0	17.1	14.5961	0.4865
3.00	6.0	20.6	17.7755	0.5925
3.00	7.0	22.8	19.7740	0.6591
4.00	3.0	8.0	7.2679	0.1817
4.00	4.0	10.5	9.1011	0.2275
4.00	5.0	13.5	11.8263	0.2957
4.00	6.0	15.5	13.6431	0.3411
4.00	7.0	17.0	15.0057	0.3751
1.59	5.0	7.30	6.8884	0.43323
4.41	5.0	14.8	13.5228	0.30664

From table 4.1 result, the percentage of yield increase as time of extraction increase but there are slightly different pattern observed with mass of raw material the percentage of yield show a peak while running with three kilogram and decreasing both with two and four kilogram.

4.3 Design Table of Patchouli Essential Oil Extraction Experiment Using Response Surface Method (RSM)

Optimization of input parameters is carried out using Response Surface Methodology (RSM). The parameter involved is extraction time (hr) and mass of raw material (kg) with range 3 hour to 7 hour and 2 kg to 4 kg. By using Central Composite Design (CCD), the table of experiment arranged by Design Expert is completed to further analyze the data. Table 4.2 showed a design table response from experiment.

Table 4.2: Design Table of Experiment

Std	Run	Block	Factor 1: Mass of Raw Material (kg)	Factor 2: Extraction Time (hr)	Response 1: Yield Weight %
13	1	Block 1	3.00	5.00	0.4731
12	2	Block 1	3.00	5.00	0.4865
1	3	Block 1	2.00	3.00	0.3179
7	4	Block 1	3.00	2.17	0.2256
11	5	Block 1	3.00	5.00	0.4864
9	6	Block 1	3.00	5.00	0.4865
4	7	Block 1	4.00	7.00	0.3751
2	8	Block 1	4.00	3.00	0.1817
3	9	Block 1	2.00	7.00	0.5950
8	10	Block 1	3.00	7.83	0.6001
5	11	Block 1	1.59	5.00	0.4332
6	12	Block 1	4.41	5.00	0.2003
10	13	Block 1	3.00	5.00	0.4865

The result of response of every combination of factor 1 (mass of raw material) and factor 2 (extraction time) is taken from raw experimental result from previous section.

The objective of the study is to maximize the yield percentage while searching the optimum value of two input parameter. Before proceed to optimization, a prediction value of respond by software must be compared to actual value from experiment. Table 4.3 shows the comparison between both values.

Table 4.3: Central Composite Design Matrix, the predicted and experimental value obtained for Patchouli Oil extraction

Std	Run	Coded Value		Response (%)	
		X1 (kg)	X2 (hr)	Actual Value	Predicted Value
13	1	3.00	5.00	0.47	0.48
12	2	3.00	5.00	0.49	0.48
1	3	2.00	3.00	0.32	0.31
7	4	3.00	2.17	0.23	0.24
11	5	3.00	5.00	0.49	0.48
9	6	3.00	5.00	0.49	0.48
4	7	4.00	7.00	0.38	0.38
2	8	4.00	3.00	0.18	0.18
3	9	2.00	7.00	0.60	0.60
8	10	3.00	7.82	0.60	0.59
5	11	1.59	5.00	0.43	0.43
6	12	4.41	5.00	0.20	0.20
10	13	3.00	5.00	0.49	0.48

The difference between predicted value and actual value from Table 4.3 is plotted on graph in Figure 4.1. From the graph, the distribution of plot is quite even which close enough to straight line.

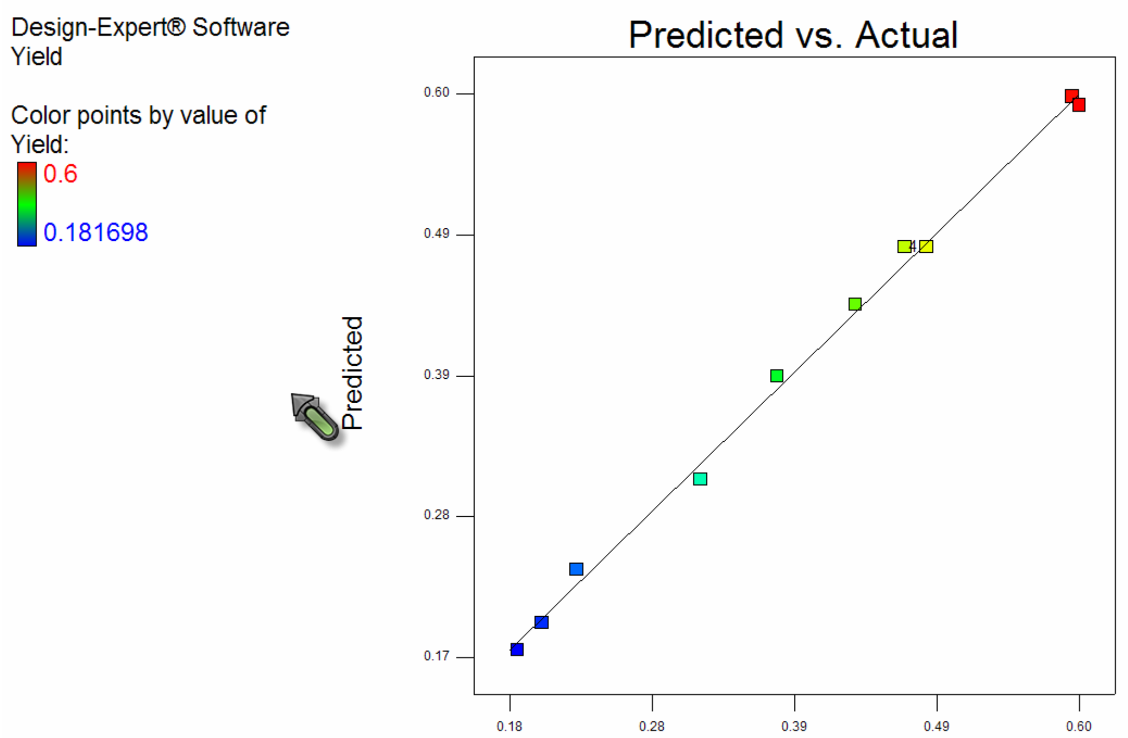

Figure 4.1: Predicted Values of Responds Vs Actual Value of Response

Result from Table 4.2 was further analyzing using Analysis of Variance (ANOVA) as appropriate to the experimental design used. Table 4.4 showed the ANOVA for Response Surface quadratic model of Patchouli Oil extraction.

Table 4.4: ANOVA for Response Surface Quadratic Model of patchouli oil extraction

Source	Sum of Squares	Degree of Freedom	F Value	p-value Prob Value > F
Model Significant	0.24	5	440.22	< 0.0001
A-Mass of Raw Material	0.059	1	546.29	< 0.0001
B-Extraction Time	0.12	1	1159.45	< 0.0001
AB	1	1.75E-03	0.005	
A^2	1	0.047	< 0.0001	
B^2	8.23E-03	8.23E-03	< 0.0001	
Residual	7.54E-04	7		
Lack of Fit Significant	5.37E-04	3	3.31	0.139
Pure Error	2.17E-04	4		
Cor Total	0.24	12		

When testing the significant of model, it was found that p- value obtain were small which is less than 0.0001 compared to desired significant which is 0.05. This show that regression model were accurate in predicting the significant to the yield production. To investigate the optimum point of the two factors of patchouli oil extraction, the response surface methodology was used and three dimensional of plot were drawn. Figure 4.2 showed the response surface curve for the two variables in the extraction of patchouli oil. The response surface representing a yield of oil as a function of raw material mass and extraction time and it was easy to comprehend the interaction of this two point and also locate the optimum level of response.

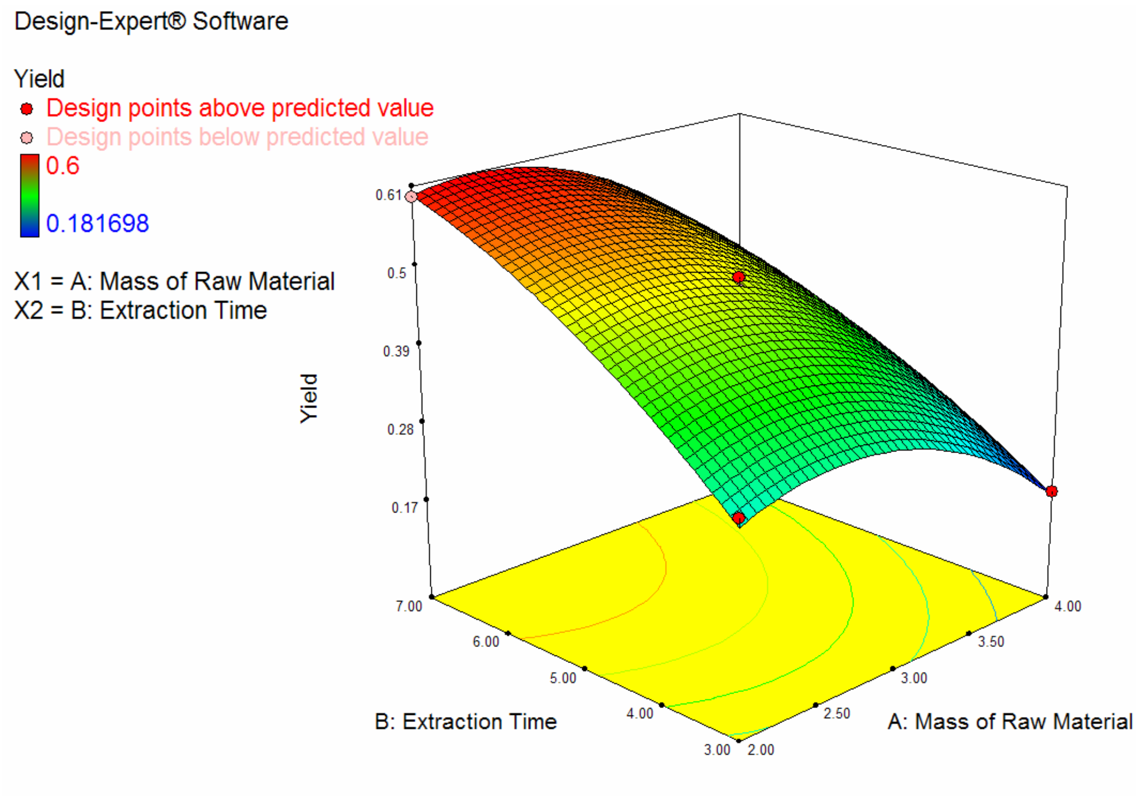

Figure 4.2: Response surface plot of patchouli oil yield: Extraction time Vs mass of raw material

The extraction time gives a significant effect on yield of oil. The interaction between response and extraction time show that increasing value of response. The mass of material show a different pattern where a significant drop is observed when raw material reached around 4 kg.

4.4 Optimization of Patchouli Oil Extraction Using Response Surface Methodology (RSM)

It was found that the predicted optimize input variable is 2.38 kg for mass of raw material and 6.84 hr of extraction time with predicted yield 0.6115 %. This model were verified by running the experiment with predicted optimize value. After running the experiment, it was found that the response is 0.6289 percent which is about 3 percent off from predicted value and the model is acceptable. Before the optimization the process, 4 kg of mass of raw material and 7 hr of extraction process will produce 0.3751 percent of essential oil. It showed that the response of the process has been increase 68 percent which is from 0.3751 percent to 0.6289 percent. For input variable, mass of raw material is reduced from 4 kg to 2.38 kg and extraction time is cut from 7 hour to 6.84 hour. Table 4.5 below shows the summary of optimization process of this research.

Table 4.5: Summary of Optimization Process

Condition	**After optimization**			**Before optimization**	
	Value	**Response (%)**		**Value**	**Response (%)**
		Predicted	Experimental		
Mass of Raw Material	2.38	0.6115	0.6289	4.00	0.3751
Extraction Time	6.84			7.00	

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Patchouli oil was successfully extract using steam distillation pilot plant with a number of response or yield depending on the condition of the process.

Optimization is successfully carried out using Response Surface Methodology (RSM) based on Central Composite Design (CCD). The optimized condition for the process is 2.38 kg of mass of raw material and 6.84 hour of extraction time. Production was 0.6289 percent of yield using this optimized condition. This gave 67.7 percent increment compared to value before optimization which is 0.3751 percent with 4 kg of raw material and 7 hour extraction time.

In conclusion, Design of Experiment (DOE) through Response Surface Methodology (RSM) is reliable method to optimize patchouli oil extraction using steam distillation.

5.2 Recommendations

This research could be further improved by changing several design weakness on the pilot plant used to ensure the consistency of the process. This improvement could reduce the noise factor which disturbed the stability of the process. The pilot plant needs a proper pressure gauge that able to measure the flow of steam to the chamber inside pilot plant vessel. Inconsistent flow of steam will affect integrity of the data obtained.

Vessel seal also need to be redesign to avoid steam leakage during extraction process. Leakage steam will reduced the amount of essential oil extracted as the steam which carries oil escaped outside of vessel. Pressure release valve also need to be install on pilot plant in case of the emergency of overpressure which could endanger a person running the process.

Outside disturbance also need to be considered to avoid noise factor. Inconsistency flow of chilled water and steam from sources could vary the performance of extraction process. The flow of utilities for process should be constant, to ensure the yield of process only affected by varying the interested input variable.

It is also recommend that the optimization process is carried out by adding a number of input variables that varying a yield of process. The condition of raw material like grinding, a different flow of steam and a number of trays used inside vessel also has a significant impact on the process. By taking all factor into consideration, the optimize process could achieve a higher value than before.

REFERENCES

Anthony. Jiju (2003), *Design of Experiment for Engineer and Scientist*, Butterworth Heinamann Publication.

Box,G. E, Hunter, W.G., Hunter, J.S., Hunter,W.G. (2005), *Statistics for Experimenters: Design, Innovation, and Discovery*, 2nd Edition, Wiley, ISBN: 0471718130.

Box, G. E. P. and Wilson, K.B. (1951) *On the Experimental Attainment of Optimum Conditions (with discussion)*. Journal of the Royal Statistical Society Series.

Guenther,E. (1949).*The Production of Essential Oils. In: The Essential Oils*. New York: D. Van Nostrand Company, Inc.218.

Grieve. Maude (1995), *A modern Herbal*, Prentice Hall.

Haagen-Smit,A.J.(1949). *The Chemistry, Origin and Function of Essential Oil in Plant Life. In:Guenther, E.The Essential Oils*.New York: D.Van Nostrand . Company, Inc.18

Kamel, Rekab (2005), *Statistical Design of Experiment with Engineering Application*, Chapman &Hall.

L.F. Hu., Li, S.P., Cao.H., Liu, J.J., Gao, J.L., Yang, F.Q., Wang, Y.T. (2005). *GC-MS Fingerprint of Pogostemon cablin in China*. China: Elsevier.

Montgomery. Douglas (2001), *Design and Analysis of Experiment*, Wiley International Publication.

R. Lazid and Zivorad (2004), *Design of Experiment in Chemical Engineering*, Mc Graw Hill Publication.

S. Foster (1996), C*ommon natural ingredients used in food, drugs and cosmetics,* JohnWiley and Sons.

W.G. Hunter (2005), *Statistics for Experimenters: Design, Innovation, and Discover 2nd Edition,* Wiley International.

APPENDIX A

Figure A-1 Raw Material Supplies

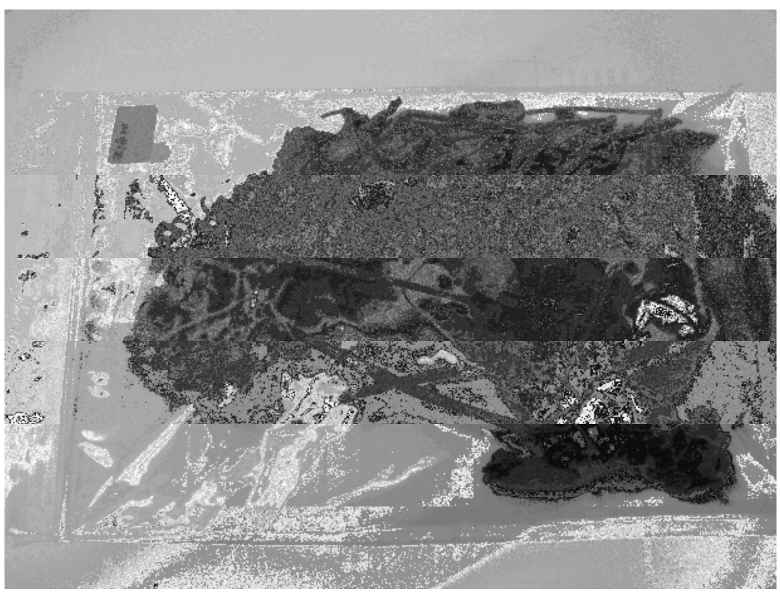

Figure A-2 Dried Patchouli Leaves

Figure A-3 Steam Distillation Pilot Plant

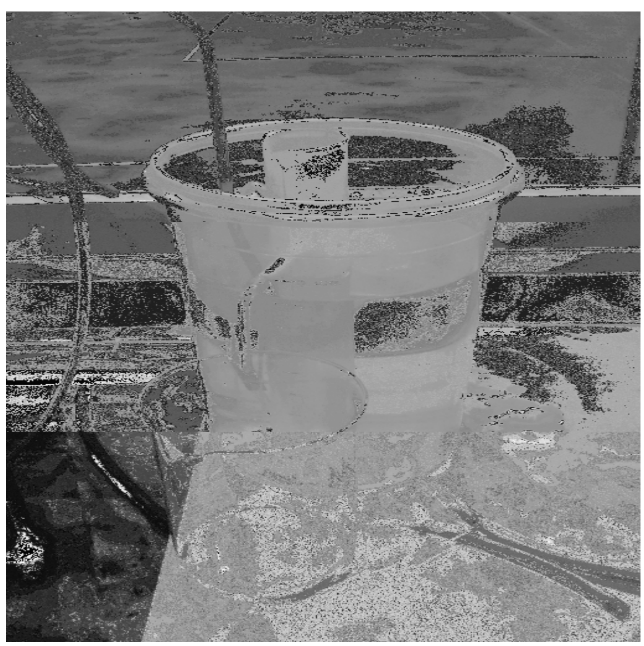

Figure A-4 Oil Collection System

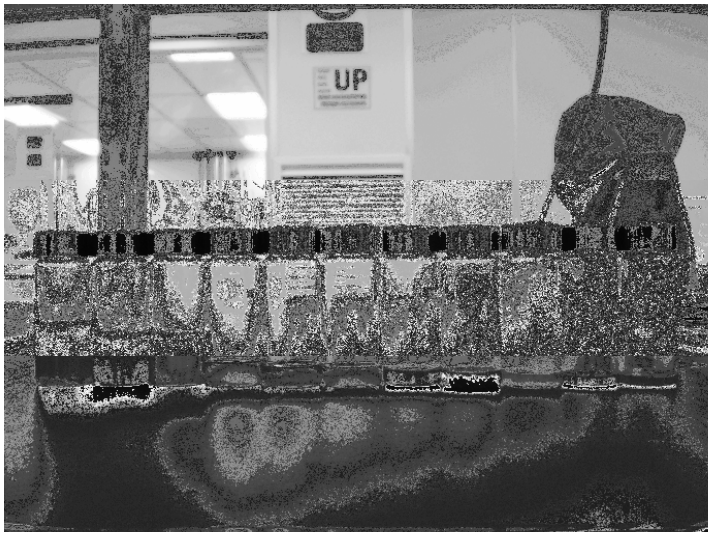

Figure A-5 Patchouli Oil Collected From Experiment

APPENDIX B

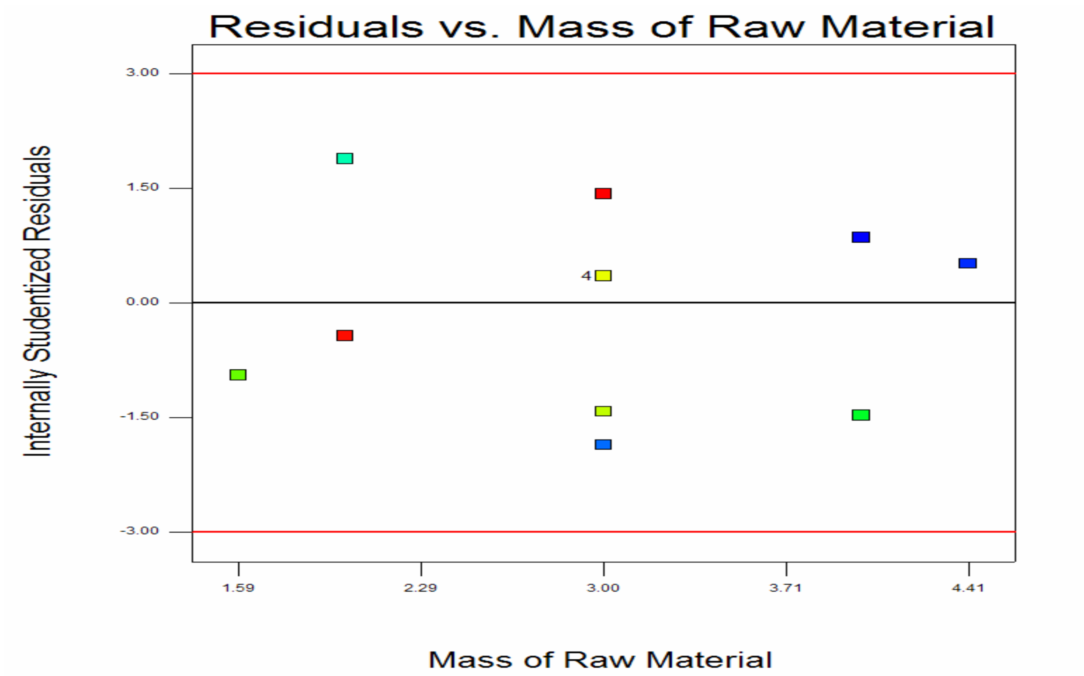

Figure B-1 Residual of Data Vs Mass of Raw Material

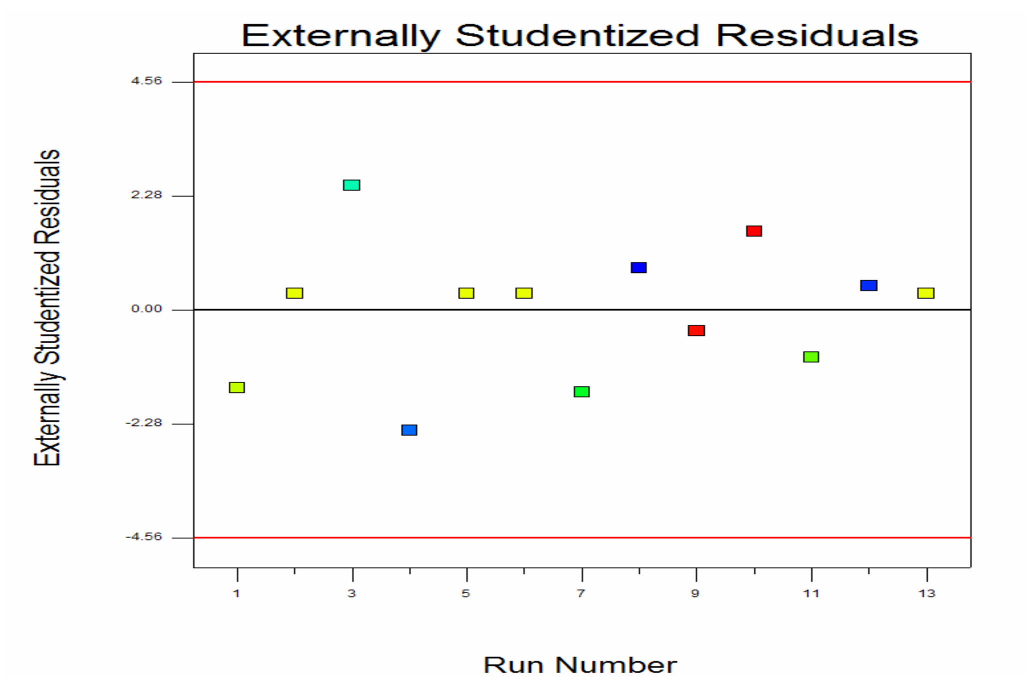

Figure B-2 External Residual of Data Vs Number of Experiment

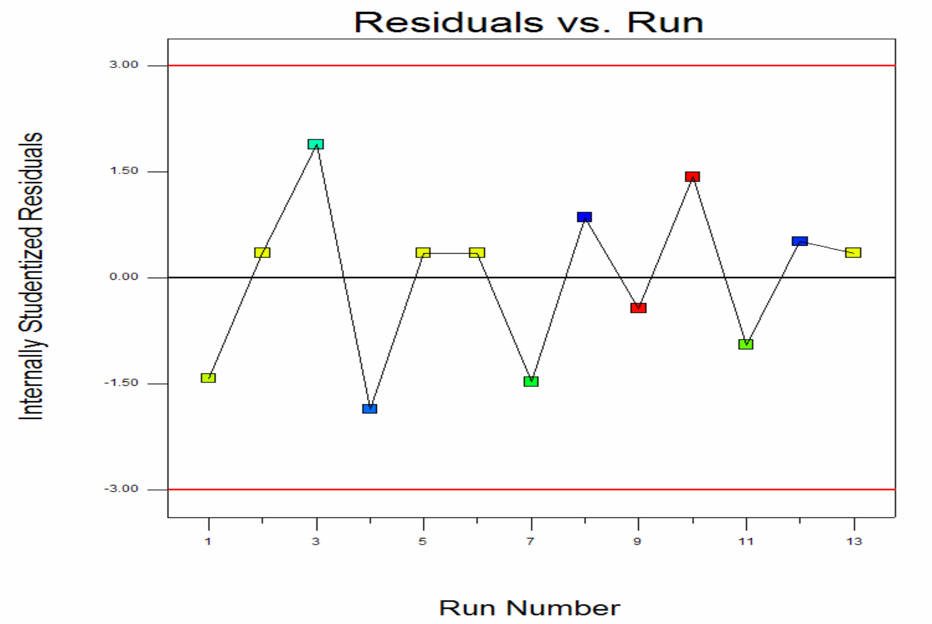

Figure B-3 Residual of Data Vs Number of Experiment

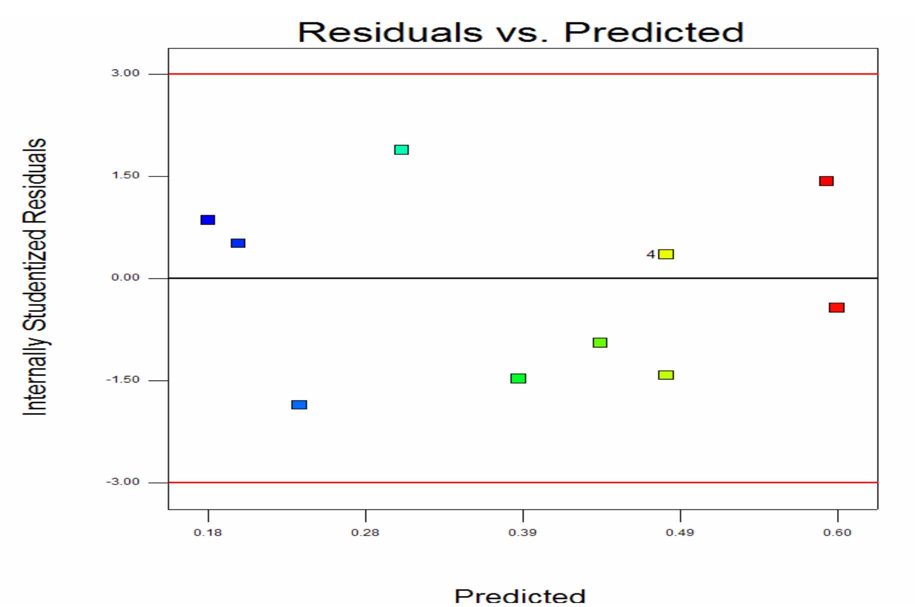

Figure B-4 Residual of Data Vs Predicted Value

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