EXTRACTION PARAMETERS OPTIMIZATION IN IMPROVING THE PRODUCTION RATE OF SPENT BLEACHING EARTH OIL

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DEDICATION

This dissertation is dedicated to my beloved wife and kids for their unconditional love, which serve as my greatest motivation and inspiration to complete this study.

"All the perfect praise belongs only to the Almighty God"

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ABSTRACT

Spent bleaching earth (SBE) is classified as an industrial waste generated by almost all crude palm oil refineries around the globe. SBE contains oil content in the range of 20-40% by weight, which is being extracted out by a solvent extraction process plant to cater for biodiesel production. Most of the current studies are focusing on the optimization of various plants and seeds extraction parameters based on a laboratory scale. Hence, a study on the optimization of the solvent extraction parameters conducted on an actual industrial-scale production plant is the key element for an enhanced SBE oil extraction process plant management. This study shall pioneer in optimizing the extraction parameters to enable plant managers to monitor the inputs and outputs of each plant equipment to operate the plant efficiently. In this study, the extraction parameters, namely, settling rate, *n*-hexane temperature, and slurry concentration were optimized by response surface methodology using Box-Behnken Design (BBD) to improve the extraction oil rate. The independent variables, settling rate (13 ml, 11 ml, and 8 ml), *n*-hexane temperature (45 °C, 50 °C and 55 °C), and slurry concentration (30%, 35%, and 40%) were selected for optimization at three-factorial levels and their values were selected based on the extraction plant current operating condition and limitation. The BBD consisted of 17 experimental actual production plant runs with 3 hours of continuous process control with a steady-state operation for each run. A second-order polynomial model was used for predicting the responses' outcomes. Analysis of variance of the conducted experimental runs concluded that 96.9% of the variation was explained by the models. The optimized extraction parameters were 13 ml, 55 °C, and 30%, for settling rate, *n*-hexane temperature, and slurry concentration, respectively. Under the optimized extraction parameters, the values of the corresponding response was 2.080 tons per hour. Mass balance computation, which was conducted with the aid of SuperPro Designer® software, resulted an improved extraction oil rate of 2.086 tons per hour which was an improvement of 9% as a result of the optimization conducted the studied production plant.

ABSTRAK

Tanah peluntur terpakai (SBE) diklasifikasikan sebagai sisa industri yang dijanakan oleh hampir semua penapisan minyak sawit mentah di seluruh dunia. SBE mengandungi kandungan minyak dalam julat 20-40% mengikut berat, yang disarikan oleh loji proses penyarian pelarut untuk menampung pengeluaran biodiesel. Kebanyakan kajian terkini tertumpu kepada pengoptimuman parameter proses penyarian pelbagai tumbuhan dan bijian berdasarkan skala makmal. Oleh itu, suatu kajian tentang pengoptimuman parameter penyarian pelarut yang dilakukan dalam suatu loji pengeluaran yang berskala industri adalah sangat penting supaya pengurusan loji proses penyarian minyak SBE dapat dilakukan dengan lebih baik. Kajian ini akan menjadi perintis dalam mengoptimumkan parameter penyarian supaya pengurus loji dapat memantau masukan dan keluaran setiap peralatan loji untuk mengendalikan loji dengan lebih cekap. Dalam kajian ini, parameter penyarian, iaitu, kadar pengenapan, suhu *n*-heksana, dan kepekatan buburan dioptimumkan oleh kaedah sambutan permukaan dengan menggunakan rekabentuk *Box-Behnken Design* (BBD) untuk mempertingkatkan kadar penyarian minyak. Pembolehubah tidak bersandar, iaitu kadar pengenapan (13 ml, 11 ml dan 8 ml), suhu *n*-heksana (45 °C, 50 °C dan 55 °C) dan kepekatan buburan (30%, 35%, dan 40%) telah dipilih untuk pengoptimuman di tahap tiga-faktoran dan nilai-nilai tersebut dipilih berdasarkan keadaan semasa dan had operasi loji penyarian. BBD terdiri daripada 17 eksperimen di loji pengeluaran sebenar yang dijalankan dengan 3 jam kawalan proses berterusan dengan operasi yang stabil bagi setiap eksperimen. Model polinomial tertib kedua telah digunakan untuk meramal hasil jawapan. Analisa varians untuk eksperimen yang dijalankan menyimpulkan bahawa 96.9% daripada variasi telah dijelaskan oleh model. Parameter penyarian yang dioptimumkan masing-masing adalah 13 ml, 55 °C, dan 30%, untuk kadar pengendapan, suhu *n*-heksana, dan kepekatan buburan. Berdasarkan parameter penyarian yang dioptimumkan, nilai yang diperoleh adalah 2.080 tan per jam kadar penyarian minyak. Selanjutnya, pengiraan kesimbangan jisim yang dikira dengan menggunakan perisian SuperPro Designer® menghasilkan kadar penyarian minyak sejumlah 2.086 tan per jam, iaitu peningkatan sejumlah 9% hasil daripada pengoptimuman yang telah dilakukan di loji pengeluaran yang dikaji.

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LIST OF ABBREVIATIONS

ANOVA	-	Analysis of Variance
B10	-	Biodiesel 10%
B20	-	Biodiesel 20%
B30	-	Biodiesel 30%
B5	-	Biodiesel 5%
BBD	-	Box-Behnken design
CCD	-	Central composite design
CPO	-	Crude Palm Oil
DOE	-	Design of experiment
DOSE	-	Deoiled spent earth
EN	-	European Engineering Standard
FAC	-	fatty acid composition
FAME	-	Fatty Acid Methyl Ester
GHG	-	Green House Gas
GSR	-	Good settling rate
Gtoe	-	Giga ton oil equivalent
HVO	-	Hydrogenated Vegetable Oil
IEC	-	International Electrotechnical Commission
IP	-	Ingress Protection
IPA	-	Isopropanol Alcohol
KJ	-	Kilo Joules
MJ	-	Mega Joules
MSDS	-	Material Safety Data Sheet
MSR	-	Moderate settling rate
PHE	-	Plate heat exchanger
PLC	-	Programmable logic control
PORAM	-	Palm Oil Refinery Association of Malaysia
PSR	-	Poor settling rate
RSM	-	Response surface method
SBC	-	Spent Bleaching Clay

SBE	-	Spent Bleaching Earth
SR	-	Settling rate
STHE	-	Shell and tube exchanger
SWS	-	Solvent water separator
tph	-	Ton per hour
ULO	-	Used Lubricating Oil
VIF	-	Variance Inflation Factor
WBE	-	Waste bleaching earth
WHO	-	World Health Organization

LIST OF SYMBOLS

%	-	Percentage
MS_{lof}	-	Media of square due to lack of fit
MS_{pe}	-	Media of square due to pure error
MS_{reg}	-	Media of square due to regression
d_i	-	Deviation
y_i	-	Observation
y_{ij}	-	Replicates
x_1	-	Independent variables
β_1	-	Linear coefficient
β_o	-	Model constant
/	-	Divide
+	-	Plus
<	-	Less than
>	-	More than
±	-	Plus-minus
°C	-	Degree Celsius
µm	-	Micrometer
Å	-	Angstrom
cm^3/g	-	Meter cubic per gram
cp	-	Centipoise
c_p	-	Replicate number
cST	-	Centistokes
CV	-	Coefficient of variance
d.f.	-	Degree of freedom
g/ml	-	Gram per mililiter
K	-	Factor number
kg/m^3	-	Kilogram per cubic meter
kg/mt	-	Kilogram per metric ton
KJ/kg-C	-	Kilo Joules per kilogram Celsius
m^2/g	-	Meter square per gram

MJ kg^{-1}	-	Mega Joules per kilogram
mm	-	Millimeter
mmH_2O	-	Milimeter water
mPas	-	Mili Pascal second
N	-	Number of experiments
R^{2-}	-	Coefficients
R_{adj}^2	-	Adjusted coefficients
SS_{reg}	-	Sum of square due to regression
SS_{res}	-	Sum of square due to residuals
SS_{tot}	-	Total sum of squares
W/m-C	-	Watt per meter Celsius
wt%	-	Weight by percentage
X	-	Multiple
α	-	Alpha

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Today's globalized and competitive market demands not only to force a manufacturing plant management to react reactively on accommodating the challenges but also to formulate a concrete result-driven action plan to achieve excellence in their business [1]. The palm oil industry, like any other manufacturing industries, is constantly experiencing the growing demand from consumers from all over the world. In 2019, the oil palm planted area increased to 5.90 million hectares, an increase of 0.9% as against 5.85 million hectares in the previous year [2]. CPO production increased marginally by 1.8%, to 19.86 million tonnes as against 19.52 million tonnes recorded in 2018. The increase was due to higher FFB processed, up by 0.5 % arising from higher FFB yield which increased by 0.2%. Total Malaysian exports of oil palm products in 2019 was recorded at 27.88 million tonnes, higher by 12.1% from 24.88 million tonnes exported in 2018 [2].

India maintained its position as the largest Malaysian palm oil export market for the sixth consecutive year since 2014, with the intake in 2019 at 4.41 million tonnes or 23.9% of total Malaysian palm oil exports. Secondly was China at 2.49 million tonnes or 13.5%, the EU 2.09 million tonnes or 11.3%, Pakistan 1.09 million tonnes or 5.9%, Turkey 0.71 million tonnes or 3.8%, the Philippines 0.63 million tonnes or 3.4% and Vietnam 0.60 million tonnes or 3.2%. These top seven markets accounted for 12.01 million tonnes or 65.0% of total Malaysian palm oil exports in 2019 [2].

The crude palm oil (CPO) produced by the plantation's upstream division is primarily supplied to the palm oil refinery, whereby the CPO is processed into refined, bleached and deodorized palm oil (RBDPO). In 2019, 16.39 million tonnes of RBDPO was produced to be either exported or further fractionated into palm olein and palm stearine [2]. Malaysian palm refinery consists of two types of refining method, namely, physical and chemical refining. Given lower processing cost, minimal oil loss and less volume of wastewater for further treatment, physical refining is preferred. Physical refining consists of two main sections, bleaching and deodorizing. Bleaching section functions as impurities removal by the bleaching earth, which is a strong acid-activated adsorbent. Impurities such as chlorophyll, heavy metals, chlorides, and phosphatides are adsorbed by the bleaching earth when the CPO is vigorously mixed under 20-50 mbar vacuum condition for 45 minutes [3]. Deodorizing is mainly carried out by utilizing sparing steam to remove the odour, free fatty acid and colour pigments. In ensuring low colour and odourless RBDPO, the bleaching section must be operated within its optimal conditions or risk producing darkened refined oils [4].

The bleaching earth is dosed in the range of 9 – 15 kg per tonne CPO in the bleaching vessel via a hopper and pneumatic system [5]. Based on the 2019 Malaysian Palm Oil Board (MPOB) statistic on the volume of RBDPO produced, with an average of 12 kg of bleaching earth consumed for each tonne of CPO, refineries in Malaysia consumed an estimated 210,000 tonnes of bleaching earth in 2019. After the bleaching process, the bleaching earth is considered industrial waste and known as spent bleaching earth (SBE), which contains 18-40% of oil by weight [6].

Recently, a serious concern is rising among environmentalists and common people on environmental pollution which may arise from the inappropriate disposal of the SBE. The remnant oil from the SBE might cause a leachate issue when a large amount of SBE is exposed to rainfall at the landfill disposal area [7]. Those leachates may heavily pollute rivers which lead to alarming issues for residency water treatment plant and direct life-threatening impact to the various macro and microorganism [8].

Hence, solvent extraction was found to be the most efficient process to recover the oil residue in SBE [9]. Higher yield and better quality of oil are the main justification for niche industry players to build a solvent extraction plant to extract the oil contains in the SBE [10]. The extracted oil from SBE is widely consumed by biodiesel production plants around the world as one of the primary raw material. Besides gaining on the economical aspect, the de-oiled SBE is further decomposed by burning it in a customized fluidized-bed combustion steam boiler to generate steam for the plant [11]. The ashes as the product of the combustion are sold to cement factories to be blended in a particular ratio with the cement to enhance the strength and function as an activator [12].

Biodiesel is an alternative fuel in place of conventional fuels due to a reduction in fuel reserves and rising environmental issues. Nowadays, several countries have encouraged the use of alternative fuel like biodiesel fuel utilizing governmental initiatives and mandate. Palm oil is certainly a good source of energy for the production of biodiesel with the oil extracted from SBE shall be a reliable and suitable ingredient for a palm oil-based biodiesel plant [13].

Almost all the solvent extraction plant is consuming *n*-hexane as the solvent to extract out the adsorbed oil content in the SBE. In spite that *n*-hexane is relatively expensive, hence, it requires an optimized process control for improved recovery of extracted oil to ensure the production cost of the solvent extraction plant is well-controlled based on the budgetary figures.

However, *n*-hexane consumption is not the only extraction parameter that contributes to the optimization of the extracted oil production. Various independent variables that significantly contribute to the production rate needs to be studied to operate the plant most efficiently. A solvent extraction plant that shares similar crucial necessity as other manufacturing industries in boosting up the production volume on daily basis, is in the desire needs in improving the plant’s efficiency to ensure the production rate is maintained at an optimum level.

The need to reduce the cost of operations and the increase in global competition amid tough economic climates, particularly during and post the COVID-19 pandemic's adverse effects, are the ultimate drivers for most of the manufacturing business owners to eliminate non-value adding operations, reinvent the process control and increase production volume and yield. Hence, business owners who enduring themselves in the fierce and stiff competition are zooming into operational excellence to enable them to stand firm on their profit ground with broader market capitalization. By definition, operational excellence is the state of any business organization that the organization achieves through the development of technology and innovation in the service and product development as well as their distributions [14].

Operational excellence becomes a major concern for the manufacturing sector around the world to improve product yield, increasing production rate, and reducing production costs. While operational excellence predominantly deals with production process optimization, market and customer orientation, and production efficiency, process plant managements around the globe have geared up with the enormous amount of effort and resources to ensure their respective manufacturing plants can sustain and compete efficiently in their respective business field [15].

In many typical production process plants, the production rate of a desired plant and machinery is generally assumed to be inflexible and predetermined [16]. An optimized production rate shall translate into an economic scale, mainly on improving the business profitability and lowering unit costs [17]. Concerning the optimized production rate, other essential operations, and business key elements such as capital costs, operating costs, size of processing plant and infrastructure are eventually a function of production rate [18].

To optimize the production rate, an optimization activity has to be conducted on the critical parameters which contribute significantly to the daily production rate and yield. The extraction production plant studied contains a set of extraction parameters that need to be optimized to improve the production rate and yield of the SBE oil. Those parameters need to be manipulated according to a workable range that suits the production plant's equipment capability and limitation.

Optimization needs to be carried out by utilizing computational software that is available in the market such as Design Expert, Aspen HYSYS and MATLAB. Any of that software can conclude the most optimized parameters which produce an improved production rate based on the actual experimental runs outcomes [19]. A mass balance computation for the whole optimization process control shall provide a quick check-and-balance for the plant process engineers to forecast the production rate and based on the optimized parameters.

1.2 Problem Statement

The solvent extraction plant studied in this research work has a great potential for an improvement of the extracted oil rate. However, the extraction parameters, which are in place and inherited from the previous generation of engineers and plant managers, have not been optimized to improve the production rate. Currently, the optimized average SBE oil extraction achieved for the studied production plant is 1.90 tons per hour.

Being the plant manager and engineer of the studied extraction plant, there is very limited research reference that they can be referred to in optimizing the extraction parameters. Most of the studies were conducted mainly on extraction optimization of various parameters of plants and seeds. Those studies were done on a lab-scale approach rather than on industrial plants. Besides, none of the studies was done on the optimization of SBE extraction parameters on an industrial scale to improve the extracted oil production rate.

Besides optimizing the parameters, the establishment of mass balance for the whole extraction process is considered the desired step to ensure quality control done on various parameters is reflected in the actual operations of the plant. Besides, the mass balance shall help engineers to monitor closely the efficiency of each piece of equipment involved in the extraction plant. On the other hand, a check and balance on the process control shall be well-established with the aid of mass balance.

Subsequently, any losses on extracted oil production at any process flow stages shall be promptly detected. To date, no study was done on mass balance for a SBE extraction plant. Therefore, a detailed and comprehensive study on optimization of SBE oil extraction parameters to improve the extracted oil production rate shall be a novelty study to assist extraction plant management to manage and operate their respective plant in a most efficient manner.

1.3 Objective

The objective of this study is to optimize the extraction parameters to improve the SBE oil extraction rate in the studied extraction production plant.

1.4 Scopes of Study

To achieve the objectives, the following scopes have been identified in this research, which is: -

1. Identification of extraction parameters (settling rate, solvent temperature and slurry concentration) that need to be optimized to improve the SBE oil extraction rate.
2. Optimization of extraction parameters (settling rate, solvent temperature and slurry concentration) using Response Surface Methodology and Design Expert Version 12.0 software to improve the SBE oil extraction rate.
3. Development of mass balance for the entire process flow of the SBE oil extraction plant using SuperPro Designer® Version 10.3.

1.5 Significant of Study

This research study shall contribute significantly to both engineering and science in assisting SBE extractor plant manufacturers, owners, management, and engineers on solutions to improve the SBE oil extraction rate from SBE. The improvement in the SBE oil extraction rate may increase product sales revenue and assist the plant owner in positive cash flows. Nevertheless, from the environmental-friendly point of view, the improvement in the oil extraction rate shall prevent any potential harm arising from the remnant of oil residue from the SBE.

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