Journal of Environmental Science and Engineering B 4 (2015) 111-118
doi:10.17265/2162-5263/2015.03.001

Overview on Application of Response Surface Methodology (RSM) in Treatment of Palm Oil Mill Effluent (POME)

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Abstract: There are many factors affecting the performance of a treatment system especially in the treatment of palm oil mill effluent (POME) as its contains high amounts of suspended solid, low pH, high salt content and high chemical oxygen demand (COD). However, one factor at a time approach is complicated method in establishing relationship between multiple parameters. Response surface methodology (RSM) is a recommended approach as it is widely used to analyze and study the interactions between multiple parameters and provides optimum output as well as minimizing the defects which result in good treatment system. This paper overviews the recent and current research in the application of RSM in optimizing the treatment development of POME.

Key words: Palm oil mill effluent (POME), multiple parameters, response surface methodology (RSM).

1. Introduction

Malaysia was recognized as one of the world's largest exporters of palm oil. In 2012, Malaysia palm oil has accumulated 5.0 million hectares of land, producing 18.79 million tonnes of palm oil and 2.16 million tonnes of palm kernel oil [1]. Consequently, this also means the polluting wastewater, known as palm oil mill effluent (POME) has produced directly proportional to the large amount of palm oil production [2]. It is estimated that 5-7 tonnes of water are required to extract 1 tonnes of crude palm oil production and more than 50% of the water will end up as POME [3]. Many treatment and removal methods of POME have been investigated and proposed in order to eliminate the pollution caused by the palm oil industry. However, each treatment process depends on many process variables and optimization of these processes may increase their efficiency [4].

Response surface methodology (RSM) is a recommended approach as it is widely used to analyze and study the interactions between multifactors [5]. It is based on collection of mathematical and statistical techniques which are useful for developing, improving, and optimizing of processes involved. Basically, a response of interest is influenced by several variables and the objective of RSM is to optimize this response. In order to analyze the effects of the independent variables, this experimental methodology will create a mathematical model which explains the chemical and biochemical processes [6, 7]. In recent years, the application of RSM has been applied for optimization in numerous chemical and biochemical processes and this also includes the treatment of POME. This paper discussing the general application of RSM which is used for modeling and optimizing the treatment of POME towards better treatment process.

2. Materials

2.1 Palm Oil Mill Effluent (POME) Characteristics

Palm oil mill effluent (POME) which obtained from the clarification process of oil mill contains of various

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suspended compounds, including cell walls, organelles, short fibre, series of carbohydrates ranging from hemicelluloses to simple sugars, a range of nitrogenous mixtures from protein to amino-acids, free organic acids and a compilation of minor organic and mineral constituents [8]. However, the contents in the POME are non-toxic, but release an unpleasant odor [9]. POME is generated from three main processes and these includes sterilizer condensate, hydrocylone waste and separator sludge [10]. POME is a thick, more to acidic with a pH value between in 4.0-5.0, and brownish in color liquid with the temperature ranging from 80 ℃ to 90 ℃ during discharged. The properties of POME are 95%-96% water, 0.6%-0.7% of oil and grease and 4%-5% of total solids [11]. Table 1 shows the typical characteristics of raw POME [12-14].

Due to the high quantity of pollution source in raw POME, the needs of proper treatment system is important. Most palm oil mills and refineries have their own treatment systems in treating POME and normal application is ponding system as it was applied to more than 85% of palm oil mill in Malaysia [15]. In general, ponding system is recognized as stabilization ponds and oxidation ponds where the term oxidation also refers as aerobic and anaerobic ponds [16].

2.2. Theory and Fundamental of Response Surface Methodology (RSM)

In the appplications of RSM, the use of experimental design and multiple regression-based methods can be applied in order to evaluate the process where several independent variables (factors) might influence dependent variables (responses) [17]. There are three steps involved in the application of RSM, these include: (1) design of experiment; (2) modeling of RSM through regression and (3) optimization of RSM.

2.2.1 Design of Experiment

The important section in the RSM is a design of experiment where its objective is to select the points in order to evaluate the response. There are three popular experimental designs, namely (1) three-level factorial design; (2) Box–Behnken design (BBD) and (3) central composite design (CCD).

The three-level factorial design consists of all possible combinations of -1, 0 and +1 which indicate as minimum level, intermediate level and maximum level respectively. The number of runs in the three-level factorial design depends on 3^k, where, k is the number of available independent variables.

The Box-Behnken design is a fractional 3^k factorials, which are performed by combining two-level factorial

Table 1 Characteristics of raw palm oil mill effluent.

Parameter	Standard by DOE (mg/L)	Concentration (mg/L)
Chemical oxygen demand	100	50,000
Biochemical oxygen demand	50	25,000
Oil and grease	10	4,000-6,000
Total solids	NA	40,500
Suspended solids	400	18,000
Total volatile solids	NA	34,000
Calcium	NA	439
Potassium	NA	2,270
Phosphorus	NA	180
Boron	4.0	7.6
Iron	5.0	46.5
Manganese	1.0	2.0
Copper	1.0	0.89
Magnesium	NA	615
Zinc	1.0	2.3

design with incomplete block designs [18]. In BBD, the level of one of the factors is fixed at the center while all possible combinations of other factor are applied [19]. The number of runs in BBD is given by $2k\ (k\text{-}1) + c_p$, where, k is the number of factors and c_p is the number of central points [20].

CCD was introduced by Box and Wilson in year 1951which consists of a two-level factorial design, a center point and an axial point. Generally, CCD requires 5 levels of each factor which are called –α, -1, 0, 1, and +α. The total number of experimental runs of CCD is given by the expression $2^k + 2k + n_c$, where n_c is the number of center points [21].

2.2.2 Modeling of RSM through Regression

The objective of the RSM is to build the regression model which acts as an approximation model in order to establish results as a true regression model. The first order of the response surface model can be expressed as:

$$y = f\left(x_1, x_2, x_3, ..., x_k\right) \quad (1)$$

Where, y is the response and x_i are factors.

The aim of RSM is to optimize the response y, since the first order are not applicable in analyzing the interaction effect between different parameters and cannot determine the critical points, the second order or the response surface model is generally used in RSM which can be expressed as:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k=1} \sum_{j=2}^{k} \beta_{ij} x_i x_j + \varepsilon \quad (2)$$

Where, x_i and x_j are the input factors which influence the response y; β_0, β_i, β_{ii}, and β_{ij} are regression coefficients for intercept, linear, quadratic and interaction coefficients respectively and ε is a random error.

2.2.3 Optimization of RSM in the Treatment of POME

The response surface design can be used to investigate the optimum value of the response over entire variable space and in its region when it reaches the optimum value. In RSM, desirability function is commonly used to determine the optimal of multiple responses [22]. For each response, a desirability function assigns numbers between 0 and 1 to the possible values of response. If the desirability function is equal to 0, it represents a completely undesirable value of the response, and if the desirability function is equal to 1, it represents a completely desirable or ideal response value.

3. Results and Discussion

3.1. Application of RSM in the Treatment of POME

In recent years, there are a large number of studies reported on the use of RSM for analysis of different processes used in the treatment of water and wastewater including POME. Some of these are discussed in this section.

3.2 Anaerobic Treatment

An anaerobic process in treating industrial wastewater such as POME is highly recommended which results in higher treatment efficiency, lower energy requirement and minimizes the amount of sludge production [23, 24]. The common application of anaerobic process in treating POME is open tank digester and extended aeration systems [25]. Since the anaerobic process is influenced by multiple factors such as feed concentration, alkalinity, biomass concentration, flow rate, and so on, the use of RSM can be advantageously as it is applicable in findings the optimum results. Zinatizadeh et al. [26] used the application of RSM in analyzing the optimum conditions of POME treatment using an up-flow anaerobic sludge fixed film bioreactor. Feed flow rate and up-flow velocity were the factors considered, while total chemical oxygen demand (TCOD) removal, soluble COD (SCOD) removal, effluent pH, effluent total volatile fatty acid (TVFA), effluent bicarbonate alkalinity (BA), effluent total suspended solids (TSS), methane percentage in biogas, methane yield (Y_M), specific methanogenic activity (SMA), food-to-microorganism ratio (F/M), sludge height in the UASB portion and solid retention time (SRT) were the responses studied. The experiments of 13 runs were conducted based on a central composite face-centered design (CCFD) and analyzed using response surface methodology (RSM). The optimum conditions were obtained based on 6 critical responses which are TCOD removal, pH, BA, methane yield, SRT and effluent TSS as these criteria are the most important parameters in optimizing anaerobic treatment process. The experimental result shows that the maximum feed flow rate 2.45 L/d and up-flow velocity 0.75 m/h give the optimum conditions for POME treatment which produces 92.62% of TCOD removal, 93.16% of SCOD removal, 7.53 of effluent pH, 43.1 mg acetic acid/l of effluent TVFA, 1,520 mg $CaCO_3$/L of BA, 200 mg/L of effluent TSS, 70.83% of CH_4 percentage, 0.313 $lCH_4/gCOD_{rem}d$ of methane yield, 54.4% of sludge height and 226.3d of SRT. The application of RSM in this study shows that the experimental value is as close as model prediction which works well in identifying the optimum condition.

Similar studies are also done by Zinatizadeh et al. [27] in the treatment of POME using anaerobic process. However, this research has focused on the effect of pretreatment. Two types of pretreatment namely physical and chemical pretreatment are applied before the anaerobic process in order to remove or reduce the amount of suspended solid in the POME. Further, the application of RSM was applied for the optimum analysis based on a central composite face-centered design (CCFD) with two independently operating variables; feed flow rate and up-flow velocity. 13 runs were conducted in each experiment and 6 dependent parameters were measured as responses, namely total COD (TCOD) removal, effluent pH, effluent total volatile fatty acid (TVFA), effluent bicarbonate alkalinity (BA), methane yield (Y_M) and solids retention time (SRT). Based on the experimental performance, the physical pretreatment shows a better result compared to chemical pretreatment in terms of anaerobic treatment process. Both optimum conditions were feed flow rate of 1.65 L/d, up-flow velocity of 0.6 and feed flow rate of 2.45 L/d, up-flow velocity of 0.75, respectively.

In other research, Sompong O-Thong et al. [28] used RSM with CCD to investigate the removal of chemical oxygen demands (COD) in the anaerobic treatment of POME by microorganism as seed sludge. The seed sludge is identified as *Thermoanaerobacterium*. Twenty runs of experiments were performed where iron concentration, C/N ratio, and C/P ratio were factors considered. COD removal was a single response in term of the anaerobic process of POME. Generally, normal application of anaerobic process requires a lot of retention time in order to reduce the content of COD. However, by applying the seed sludge, specific microorganism will enhance the performance of the treatment and increase the process efficiency. The optimum conditions were found at iron concentration of 200 mg/L, C/N ratio of 70 and C/P ratio of 550 where COD removal efficiency of 56.4%. It was observed that the application of RSM was useful for optimizing COD reduction from POME with *Thermoanaerobacterium* as seed sludge.

3.3 Coagulation–Flocculation Process

Coagulation-flocculation is a common process used in water and wastewater treatment in order to destabilize the colloidal particles where ferric chloride and/or polymer are added as destabilization agents [29]. Throughout the process, many factors can influence its efficiency, such as dosage of coagulant-flocculant, pH, temperature, turbidity, retention time and so on. The use of RSM to optimize this factor may significantly increase the performance of the process. Ahmad et al. [30] had conducted a series of experiment on the treatment of POME based on RSM where treatment optimization was performed using jar tests. A CCD was applied to justify the effect and interaction of three factors; coagulant dosage, flocculent dosage, and pH, while water recovery and turbidity were acting as a response. A CCD of 20 runs, including six replicates at the centre point was used in the RSM. The results showed that 78% of water recovery with a 20 NTU turbidity was the optimum value which can be obtained at a coagulant dosage of 15,000 mg/L, a flocculant dosage of 300 mg/L, and pH 6. The verification experiments demonstrate that the application of RSM works well for optimizing the coagulation–flocculation process. The optimum conditions obtained from the compromise regression equation were turbidity of 19 NTU and water recovery of 76%.

The RSM was used by Bhatia et al. [31] to optimize the suspended solids and sludge recovery in regards to the treatment of POME by using a natural coagulant known as *Moringa oleifera*. A most common coagulant is actually a chemical based, however the increasing in price and environmental concern, natural coagulant which was friendly to the environment presented as an alternative for the treatment of wastewater such as POME. A 2^4 factorial design of 30 experiments with eight star points and six replicates at the central points were employed in the RSM. After screening the experiments, pH, settling time, *M. oleifera* dosage (after oil extraction), and flocculant dosage were selected as factors, while suspended solids and sludge recovery was the response studied. The optimum conditions were obtained at pH 5, settling time of 114 min, *M. oleifera* dosage of 3,469 mg/L and flocculant dosage of 6,736 mg/L, where the suspended solid value was 181 mg/L and percentage recovery of the sludge was 87 wt%. The use of *M. oleifera* alone is highly economical as it can reduce the cost of chemicals used for pH adjustment and also reduce the amount of sludge volume as well as the sludge handling cost.

3.4 Solubilization

Optimization of thermo-chemical treatment of POME using RSM was studied by Chou et al. [32]. The input variables included NaOH concentration (1.27-14.73 g/L), incubation time (7.64-88.36 h) and temperature (26.6-43.4 ℃), while COD solubilization was the single response. This study concentrated on the effects of temperature, NaOH concentration and reaction time on the solubilization of POME, while a CCD was applied to determine the corresponding optimum condition. Six axial points and three central points were augmented in the design with a total number of experiments were 20 runs. The COD solubilization was calculated based on the ratio of soluble chemical oxygen demand (SCOD) to total chemical oxygen demand (TCOD in percentage. The optimum condition obtained is 8.83 g/L of NaOH, at 32.5 ℃ and 41.23 h reaction time in response to the maximum COD solubilization of 82.63%. The experiment also runs in triplicate in order to confirm the prediction and the result obtained is close to the predicted value. This also indicates that the solubilization of POME can be optimized to the application of RSM.

Table 2 shows the summary of the experimental design used to optimize the treatment of POME. It is noted that the central composite design is the single model used by most POME studies for utilizing the analytical procedures. The main reason CCD was always used in many researches is because it can utilize and optimize a large number of factors and all experiments can also be conducted at the same time instead of one after another [33]. This method will also reduce the time consumed for each experiment and have a shorter research project duration.

4. Conclusion

The application of response surface methodology in modeling and optimizing the treatment development of POME is a highly efficient approach because it is applicable in identifying the most significant operating factor to optimize the response with minimum effort and time. Furthermore, it also helps to evaluate the effect of multiple variables on the response and their interactions which result in efficient treatment systems.

Table 2 Summary of the experimental design for the treatment of POME.

POME treatment	Factor	Response	Ref.
Coagulation-flocculation process incorporated with membrane separation technology.	coagulant dosage flocculent dosage pH	water recovery turbidity	[30]
Coagulation–flocculation process for POME treatment using *Moringa oleifera* seeds extract.	pH settling time *M. oleifera* dosage (after oil extraction) flocculant dosage	suspended solids sludge recovery	[31]
Pre-treated POME digestion in an up-flow anaerobic sludge fixed film bioreactor.	feed flow rate up-flow velocity	total COD (TCOD) removal effluent pH effluent total volatile fatty acid effluent bicarbonate alkalinity methane yield solids retention time	[27]
POME treatment using an up-flow anaerobic sludge fixed film bioreactor.	feed flow rate up-flow velocity	total COD soluble COD removal effluent pH effluent total volatile fatty acid effluent bicarbonate alkalinity effluent total suspended solids CH4 percentage in biogas methane yield specific methanogenic activity F/M ratio sludge height in the UASB portion solid retention time	[26]
Removal of chemical oxygen demands in the anaerobic treatment of POME by microorganism as seed sludge.	iron concentration C/N ratio C/P ratio	COD removal	[28]
Effect of temperature, NaOH concentration and time on solubilization of POME.	NaOH concentration incubation time temperature	COD removal	[32]

As shown in this paper, the central composite design is a most common experimental design used for the development of analytical procedures. This is because its ability to integrate with higher numbers of variables and its efficiency in optimizing the process which leads to the increasing number of published works in this field. In order to improve the application of RSM in optimizing the treatment process of POME, more studies and researches are needed with the possibility of combining or comparing with other modeling techniques such as artificial neural network (ANN) where its provides non-linear modeling for response surfaces and optimization in the treatment of POME.

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