Bioresource Technology 101 (2010) 8616–8622

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Evaluation of the effect of temperature, NaOH concentration and time on solubilization of palm oil mill effluent (POME) using response surface methodology (RSM)

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ARTICLE INFO

Article history:
Received 2 April 2010
Received in revised form 16 June 2010
Accepted 24 June 2010
Available online 16 July 2010

Keywords:
Thermo-alkaline pre-treatments
Palm oil mill effluent (POME)
Solubilization
Response surface methodology (RSM)
Optimization

ABSTRACT

In this study, palm oil mill effluent (POME) was solubilized by batch thermo-alkaline pre-treatments. A three-factor central composite design (CCD) was applied to identify the optimum COD solubilization condition. The individual and interactive effects of three factors, temperature, NaOH concentration and reaction time, on solubilization of POME were evaluated by employing response surface methodology (RSM). The experimental results showed that temperature, NaOH concentration and reaction time all had an individual significant effect on the solubilization of POME. But these three factors were independent, or there was insignificant interaction on the response. The maximum COD solubilization of 82.63% was estimated under the optimum condition at 32.5 °C, 8.83 g/L of NaOH and 41.23 h reaction time. The confirmation experiment of the predicted optimum conditions verified that the RSM with the central composite design was useful for optimizing the solubilization of POME.

1. Introduction

Palm oil is one of the main agricultural products of Malaysia which constitutes 41.37% of the total world production for the year 2007 (MPOB, 2009). The production of palm oil from the fruit of *Elaeis guineensis* results in enormous generation of palm oil mill effluent (POME), which has 100 times more oxygen depleting potential as domestic sewage (MPOB, 2009). Thus, the palm oil industries are facing remarkable challenges to comply with the increasingly stringent environmental regulations.

Anaerobic digestion system is suitable for the POME treatment as this system is environmental friendly and allows the energy recovery from methane gas production. Recent researches investigated the performance of anaerobic digestion of POME where 62–98% of COD reduction and 39–84% of methane production was reported depending upon system operation, feed rate and substrate concentration (Megat et al., 1989; Borja et al., 1996; Ugoji, 1997; Pechsuth et al., 2001; Najafpour et al., 2006; Choorit and Wisarnwan, 2007).

However, the hydrolysis reaction is the rate-limiting step in the overall anaerobic digestion process (Kim et al., 2003; Park et al., 2005). The inhibition is due to the raw feed which contains large particle size organic matter which is also insoluble (Kim et al., 2003; Park et al., 2005; Bougrier et al., 2008; Liu et al., 2008). In order to enhance this biological treatment technology, pre-treatment is required to convert the complex and biorefractory particulate organic materials to readily biodegradable low-molecular-weight compounds (Lin et al., 1998; Park et al., 2009).

The most common and convenient pre-treatment methods are thermal and alkaline pre-treatment which can effectively accelerate sludge solubilization, reduce the particle size and subsequently improves the overall anaerobic digestion (Heo et al., 2003; Kim et al., 2003; Lin et al., 2009). The significance of solubilization of waste on anaerobic digestion has been proven by many previous researches (Heo et al., 2003; Kim et al., 2003; Park et al., 2005; Bougrier et al., 2008; Lopez and Espinosa, 2008). Some studies reported that the thermal and alkaline pre-treatments were successful to solubilize waste activated sludge and microbial biomass and achieved COD solubilization ranged from 25% to 83% (Penaud et al., 1997,1999; Lin et al., 1998; Heo et al., 2003; Kim et al., 2003; Valo et al., 2004). Reaction time is another important factor which significantly affects solubilization (Lin et al., 1999; Heo et al., 2003; Valo et al., 2004). However, most of the researches were done with a reaction time less or equal to 24 h (Lin et al., 1999; Penaud et al., 1999; Kim et al., 2003; Heo et al., 2003; Valo et al., 2004).

On the basis of the above reasons it seems possible that, thermo-alkaline pre-treatment can be applied to improve the COD solubilization of POME prior to enhance anaerobic digestion. But, there were insufficient researches regarding the effect of thermo-alkaline pre-treatments on POME. Therefore, the objectives of this study are:

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0960-8524/$ - see front matter
doi:10.1016/j.biortech.2010.06.101

i) to investigate the effect of temperature, NaOH concentration and reaction time on solubilization of POME,
ii) to determine the corresponding optimum condition.

2. Methods

2.1. Palm oil mill effluent (POME)

POME was obtained from a local palm oil mill in Nibong Tebal, Penang, Malaysia. The POME was collected in a carboy container then brought to the laboratory and stored at 4 °C until it was used. The chemical and physical characteristics are presented in Table 1.

2.2. Analytical methods

The samples were analyzed to determine the pH, total solids (TS), total suspended solids (TSS), volatile solids (VS), volatile suspended solids (VSS), total chemical oxygen demand (TCOD) and the soluble chemical oxygen demand (SCOD) (Table 1). The measurement of the pH was conducted using a pH meter (HACH sensION3). The TS, TSS, VS and VSS determinations were referred to Standard Methods for the Examination of Water and Wastewater (APHA, 2005). The TCOD and SCOD concentration were analyzed using closed reflux-colorimetric method. The spectrophotometer (HACH Odyssey DR/2500) at the wavelength 620 nm was used to measure the absorbance of the COD in samples after digestion in the COD reactor (HACH DBR 200). The samples were filtered through a glass microfiber filter paper (Whatman 934-AH) to analyze SCOD. The ratio of SCOD to TCOD (in %) always used to reflect the extent of hydrolysis (Lin et al., 1999). Percentage of COD solubilization was calculated using Eq. (1) (Penaud et al., 1999, Kim et al., 2003; Valo et al., 2004):

$$\text{COD solubilizaion} = \frac{\text{SCOD}}{\text{TCOD}} \times 100\% \quad (1)$$

2.3. Thermo-alkaline pre-treatments

In order to investigate the effect of the temperature, NaOH concentration and reaction time on COD solubilization, a series of experiments were performed under various conditions. NaOH (1 M) was used as an alkali agent to achieve the alkaline condition. About nine 250 mL Erlenmeyer flasks were used in each set of the batch experiments. The desired amount of NaOH was added into each flask after 100 mL of POME was added. All flasks were purged with nitrogen gas for 30 s to obtain the anaerobic condition; then closed with a glass stopper and sealed with parafilm to make them gas tight. The sample flasks were incubated in an incubator shaker (Lab Companion SI-300R) at a desired temperature and time. All the experiments were duplicated.

2.4. Preliminary study of palm oil mill effluent (POME) solubilization process

A preliminary study was done to investigate the experimental factors and to narrow the corresponding ranges before the application of statistical design. Penaud et al. (1997) observed that COD solubilization was maximal for temperature ranging from 35 to 55 °C; another research (Penaud et al., 1999) reported a nearly steady COD solubilization trend was obtained at NaOH concentration higher than 13.9 g/L. Hence, a total 36 experimental sets were carried at 30, 40, 50 and 60 °C with NaOH concentration ranged from 0 to 16 g/L. The reaction time was temporarily set at 24 h as prolonged incubation time may initiate the production of biogas which may lead to breakage or leakage of the gas tight reaction flasks. Each set of experiment was conducted in triplicate.

2.5. Experimental design and statistical analysis

After the preliminary study, a three-factor central composite design (CCD) was obtained by using MINITAB 14. It was employed to investigate the interactive effects of three variables, viz. temperature, sodium hydroxide (NaOH) concentration and reaction time on POME solubilization. The design matrix contained a 2^3 factorial design augmented by six axial points coded ±α and three central points (all factors at zero level). The value of α was calculated by equation below:

$$\alpha = 2^{n/4} \quad (2)$$

where n is the number of factors in the design. Therefore, α is equal to $2^{3/4}$ = 1.682 according to Eq. (2). The range and levels of all factors (temperature, x_1; NaOH concentration, x_2; reaction time, x_3) in coded and real values which presented in Table 2 were calculated with the following equations:

$$x_i = \frac{X_i - X_{ic}}{\Delta X_i} \quad (3)$$

where x_i is the coded value of the ith test variable; X_i is the uncoded value of the ith test variable, X_{ic} is the value of X_i at the center point of the studied area, and ΔX_i is the step size.

As shown in Table 3, the response variable (COD solubilization) was fitted using a predictive function of variables in order to correlate to the three independent variables. The general form for COD solubilization (%) can be written as a linear or quadratic equation, i.e.,

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3, \quad (4)$$

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \quad (5)$$

where Y is the response (COD solubilization), x_1, x_2 and x_3 are the coded levels of the three variables respectively, and β_0, β_1, β_2, β_3, β_{11}, β_{22}, β_{33}, β_{12}, β_{13} and β_{23} are the model coefficients calculated from the experimental data. The responses and variables (in coded values) were analyzed by the response surface function to obtain the values of the coefficients of Eq. (4) and Eq. (5).

The quality of fit of the polynomial model equation was expressed by the coefficient of determination, R^2. The model terms were selected or rejected based on the p-value with 95% confidence level. The responses were completely analyzed using analysis of variance (ANOVA). The simultaneous interaction of the three independent variables was investigated by constructing the response

Table 1
Characterization of raw POME sample (n = 5).

Parameters	Range	Average ± standard deviation
pH	4.10 – 4.70	4.38 ± 0.30
TS, mg/L	34,116 – 60,796	48,191 ± 12,990
TSS, mg/L	16,000 – 32,600	24,960 ± 7146
VS, mg/L	27,972 – 53,996	41,678 ± 12,645
VSS, mg/L	15,200 – 30,600	22,040 ± 7260
TCOD, mg/L	54,100 – 94,300	74,340 ± 17,310
SCOD, mg/L	22,000 – 32,400	27,480 ± 3767

Table 2
Levels of factors used in this study.

Variables	Label	Level				
		−1.682	−1	0	1	1.682
X_1	Temperature, °C	26.6	30.0	35.0	40.0	43.4
X_2	NaOH concentration, g/L	1.27	4.00	8.00	12.00	14.73
X_3	Incubation time, h	7.64	24.00	48.00	72.00	88.36