CHAPTER 3

METHODOLOGY

3.1 Wastewater Source

This study was carried out based on POME sampling at the palm oil mill, in Nibong Tebal, Penang. It is owned by the MALPOM Industries Bhd. This mill has the design capacity of 30 tonnes over an hour and currently produces about 80 tonnes CPO a day. Hence, this mill can generate about 500 tonnes wastewater of POME. Before POME wastewaters were discharged for further treatment, these wastewaters were collected from the recovery tank. All the POME wastewaters which collected from the recovery tank were used as the samples for this study to determine its performance.

3.1.1 Collection and Characterization of Wastewater

The raw and anaerobic digested POME samples were collected from the palm oil mill at Nibong Tebal, Penang, Malaysia. Both samples were collected from the recovery tank and anaerobic pond and separately filled in a 10 L clean high-density polyethylene (HDPE) bottles. Then, the HDPE bottles were transported to the laboratory and stored at 4 °C until further detailed analysis could be conducted. The characteristic of the raw POME sample, such as pH, oil and grease (O & G), biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), soluble chemical oxygen demand (SCOD), volatile fatty acid (VFA), alkalinity (Alk), total solids (TS), suspended solids (SS), total volatile solids (TVS)

and ammonia nitrogen (NH_3-N) were determined according to the Standard Methods for the Examination of Water and Wastewater (APHA , 2005).

3.2 Experimental Set-up

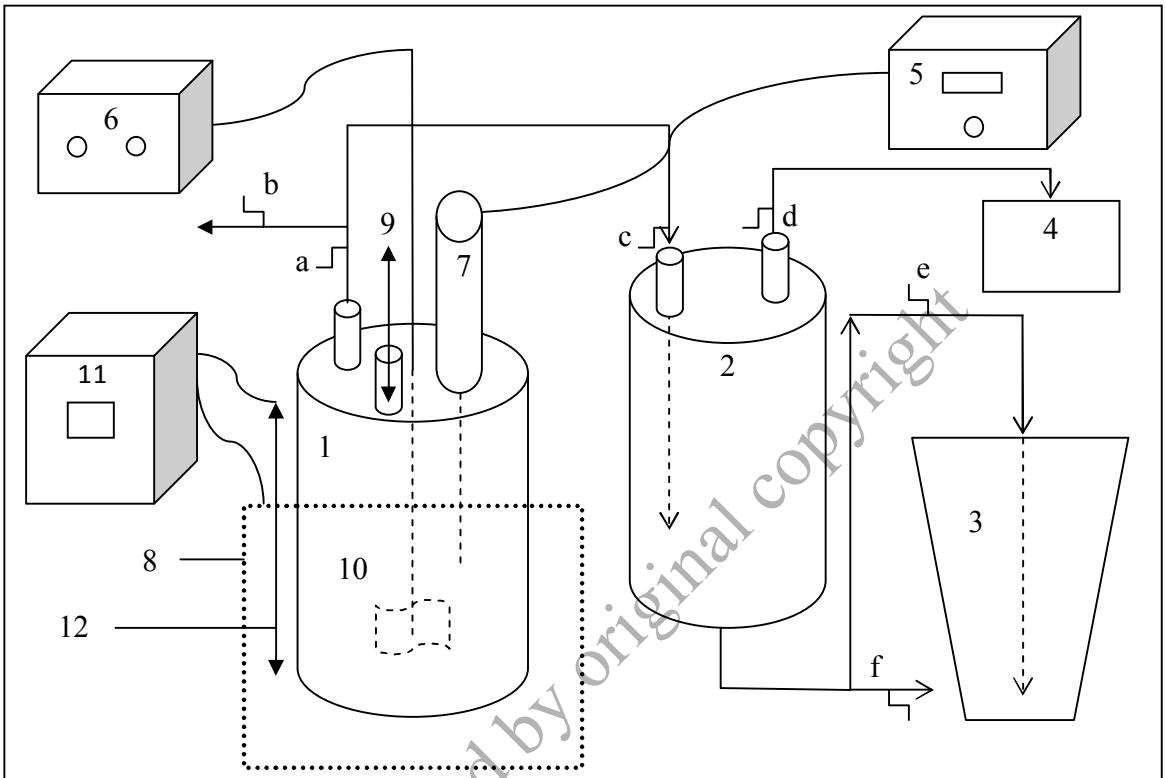
The laboratory scale experimental set-up consists of a suspended closed anaerobic reactor (SCAR) and a gas collection system. The schematic diagram of suspended closed anaerobic system used in this study is shown in Figure 3.1.

3.2.1 Suspended Closed Anaerobic Reactor (SCAR) Configuration

The dimension of SCAR is 0.174 m x 0.269 m (diameter x height). The reactor consists of essentially of a cylinder-shape flexi glass vessel with total and working volumes of 6.4 L and 4.5 L respectively. Moreover, the reactor comprises an integrated on-line pH data recording system which connected to pH probe and a mixer. The operating temperature of the reactor, 35 °C, was maintained constant by circulating hot water through the bioreactor jacket.

3.2.2 Gas Collection System

The SCAR had a gas sampling valve (or clamp) which allowed samples to be taken without interference with head space composition and was connected to a gas collection system of a water displacement tank and a 10 L graduated flask connected in series for gas volume measurement. The water displacement tank was made from a flexi glass with a dimension of 0.146 m x 0.267 m (diameter x height). The total volume of the water displacement tank is 4.5 L. In addition, the water displacement tank had been filled with distilled water of pH 3 or less in order to prevent dissolution of the gas components.



1- Suspended closed anaerobic rector (SCAR),
2-Water displacement tank,
3-10 L graduated flask,
4-Gas collection pump,
5-pH data recording system,
6-Mixer controller,
7-pH probe,
8-Heated water jacket,
9-Inlet or outlet,
10-Mixer,
11-Temperature controller,
12-Thermocouple,
a, b, c, d, e f -Clamps in pipe.

Figure 3.1: The experimental set-up

3.2.3 SCAR Sampling Procedure

In this research study, the sampling procedure had been applied for POME wastewater samples and gas samples. Besides that, gas volume was measured daily using acidified water displacement method. SCAR was controlled and monitored at mesophilic temperature (35 °C) daily in order to ensure smooth operation.

3.2.3.1 POME Wastewaters Sampling

Initially, treated effluent samples were collected daily from the SCAR by the liquid injector. Then mixing was achieved by mixer which was controlled by mixed controller. After 5 minutes, mixed samples would be collected from the SCAR by the liquid injector. Both effluent samples and mixed samples were analysed at each batch of HRT. In this research study, no pH adjustment was applied on the reactor but pH of the raw POME wastewater was controlled at the range of 7-7.3 with 1 N sodium hydroxide (NaOH). After pH adjustment of the raw POME wastewater had been carried out, then the adjusted raw POME wastewater were fed into the reactor.

3.2.3.2 Biogas Sampling and Biogas Volume Measurement

Biogas generated during the reactor operation was collected by water displacement method through an outlet provided at the top of the reactor. As shown in Figure 3.1, while opening clamp a, c and e and closing clamp b, d and f, biogas produced in the SCAR [1] was collected by the water displacement tank [2], and the water in the water displacement tank [2] was expelled and flowed into the 10 L graduated flask [3], so the biogas production was measured by the volume of water displaced from the water displacement tank [2]. When gas was sampled, only clamp d and e were opened whereas the rest of the clamps were closed. The gas in the water displacement tank [2] was forced into three-liter gasbag by pump, and gas samples were collected for analysis of CH_4, CO_2 and H_2 fractions.

3.3 SCAR Operation

3.3.1 Acclimatization of SCAR

The SCAR was initiated with the seeding from the anaerobic pond of MALPOM Industries Bhd wastewater treatment plant and approximately 4.5 L of the anaerobic digested POME were used to acclimatize the laboratory SCAR. Before the feeding was performed, the pH of raw POME was adjusted to 7-7.3. Afterward, the raw POME was fed into the reactor at 6.25-6.67 kg COD/m^3/day of influent organic volumetric loading rate to achieve a HRT of 12 days for 27 days until a steady state reached. A steady state was assumed when the pH, microbial growth, COD of effluent, COD removal efficiency, VFA, Alk, biogas production rate and biogas composition were shown to be constant. After ensuring the steady state of SCAR had been obtained, the next phase of research studies will be preceded.

3.3.2 Operation Procedures

After acclimatization stages had been completed, a series of continuous experiments were carried out. In this research study, the COD and VFA of raw POME wastewater were fixed in the range of 75000 mg/L to 80000 mg/L and 8500 mg CH_3COOH/L to 10000 mg CH_3COOH/L, respectively. The operational temperature of the reactor was 35±1 °C respectively. The pH of the raw POME wastewater was maintained at 7-7.3 by adding 0.1 N NaOH.

This preliminary step was followed by a series of continuous experiments using feed flow-rates of 375 ml/d [run 1], 450 ml/d [run 2], 560 ml/d [run 3], 750 ml/d [run 4], 1125 ml/d [run 5] and 2250 ml/d [run 6] of the wastewater, which correspond to HRTs of 12, 10, 8, 6, 4 and 2 days, respectively. The duration of the experimental runs 1, 2, 3, 4, 5 and 6 was 27, 15, 11, 18, 14 and 14 days, respectively.

For each batch of HRT, the water samples and gaseous samples from SCAR were collected after 24 hours of input raw POME. In order to obtain the information of water sample compositions, the following parameters of the samples were measured such as feed and effluent of COD, VFA and Alk according to American Public Health and Association (APHA) standard methods for water and wastewater analysis (APHA, 2005). To obtain the information of gaseous samples compositions, the following parameters of the samples were measured such as CH_4, CO_2 and H_2 gases content by the Standard Method 2720-C gas chromatographic method. Moreover, the volume of biogas produced was measured manually using the water displacement method.

For each batch of HRT, the SCAR was continuously operated until steady state condition was achieved. The steady-state condition was reached when COD removal efficiency, VFA, biogas production rate and biogas composition were stable for five consecutive days. The steady state value of tested parameter was taken as average value of these consecutive analysis amounts for each batch of HRT. The variation of the actual results from the mean values was <3 %.

3.4 Analysis of Sample

The following parameters were determined in this research study: pH, COD, SCOD, BOD, TS, SS, TVS, MLSS, MLVSS, O & G, NH_3-N, VFA, Alk, CH_4 content, CO_2 content and H_2 content. All analyses were carried out according to the recommendations of the Standard Methods for the Examination of Water and Wastewater (APHA, 2005). The detailed analytical procedures for all parameters are shown at Appendix F-Q. In addition, all the analyses were duplicated, and the results given are mean values. The volume of biogas produced was manually measured using a water displacement method in a 10 L graduated flask.