



## Research Journal of Pharmaceutical, Biological and Chemical Sciences

### Performance of an Anaerobic Stage Reactor (ASR) Treating Synthetic Wastewater during Start-Up Phase Using Palm Oil Mill Effluent (POME) Sludge

Mahat SB<sup>1</sup>, Chelliapan S<sup>1\*</sup>, Yuzir A<sup>2</sup>, Md Din MF<sup>2</sup>, Anwar AN<sup>2</sup>, Othman N<sup>1</sup> and Shamsuddin S<sup>1</sup>

<sup>1</sup>UTM Razak School of Engineering and Advanced Technology, Universiti Teknologi Malaysia, Jalan Semarak, 54100, Kuala Lumpur, Malaysia.

<sup>2</sup>Department of Environmental Engineering, Faculty of Civil Engineering, Universiti Teknologi Malaysia, 81310, Johor Bahru, Malaysia.

#### ABSTRACT

Start-up of an anaerobic reactor depend on various factors, such as wastewater composition, organic loading rate (OLR), hydraulic retention time (HRT), seed sludge, temperature, and reactor configuration. Accordingly, the present study describes results of an investigation into start-up performance of an anaerobic stage reactor (ASR) using palm oil mill effluent (POME) sludge. The average chemical oxygen demand (COD) removal efficiency of the reactor system was 55% when the ASR was operated at an OLR of 0.82 kg CODm<sup>3</sup>d<sup>-1</sup>. However, when the OLR was increased further from 1.22 to 2.45 kg CODm<sup>3</sup>d<sup>-1</sup>, the COD removal efficiency declined to an average value of 35%. The pH profile (pH 3.9 - 5.5) was not stable in all stages of ASR system, confirming accumulation of acids in the ASR stages, and the inhibition of methanogens. In addition, a lower level of methane composition was also observed in all ASR stages (4.8 to 36.1%, at OLR of 0.82 - 2.45 kg CODm<sup>3</sup>d<sup>-1</sup>). The OLR played an important role during the start-up period using the POME sludge. The load values applied during the start-up should be low for seed sludge acclimatization.

**Keywords:** Anaerobic stage reactor (ASR), anaerobic start-up, organic loading rate, palm oil mill effluent sludge, seed sludge

*\*Corresponding author*

## INTRODUCTION

One of the few problems that recur in the application of anaerobic bio-treatment to wastewater is the start-up period. It is widely observed in the literature that a significant amount of down time is involved in the initial start-up of anaerobic reactor systems. The main difficulty appears to be the development of the most suitable microbial culture for the waste streams, which is still in question. The sensitive nature of the majority of anaerobic bacteria and the extreme oxygen liability of the enzyme systems of obligate anaerobes render the reactor population more susceptible to slight fluctuations [1]. Start-up of an anaerobic system is consequently more time consuming than initiation of an anaerobic process.

During anaerobic reactor start-up, the biomass is acclimatized to new environmental conditions, such as substrate, operating strategies, temperature and reactor configuration. Moreover, the methanogens and certain acetogens may be greatly outnumbered by the fast-growing acidogens. Consequently, an accumulation of volatile fatty acids (VFAs) and dissolved  $H_2$  will occur. Start-up procedures will depend on various factors, including wastewater composition and strength, available inoculum, reactor operating conditions, and reactor configuration [2]. Shorter start-up time can be obtained by using wastewater low in particulate organics. For example, for up-flow anaerobic sludge bed (UASB) reactors, the objective of start-up is to develop an active granular biomass with good settling capacity. The OLR should be increased when the COD and VFA concentrations have been reduced by 80% [3].

Quick start-up and improved operational control of the anaerobic processes are important factors to increase its efficiency [4]. In general, high-rate anaerobic processes can be operated with organic loads much higher than those of the conventional anaerobic reactors, but frequently these highly efficient processes require longer start-up periods, better operational control and more qualified operators. Systematized operational procedures are very important, mainly during the start-up of high-rate systems [5]. The start-up of anaerobic reactors is determined by the initial transient period, marked by operational instabilities.

There are few reports on the treatment of anaerobic reactor during start-up phase. Majority of them had studied the methods which can be carried out in order to enhance and improve the efficiency of the reactor system during the start-up period. Cresson [6] reported that, with increasing of OLR from 0.5 to 20 g CODL<sup>-1</sup>d<sup>-1</sup>, the COD removal was maintained at 80% during the start-up period, and the reactor was operated with low HRT to favor bio-film accumulation. Sowmeyan and Swaminathan [7] carried out a study on the performance of inverse anaerobic fluidized bed reactor for treating high strength organic wastewater during start-up phase and showed that the system was capable of achieving 84% COD removal and high biogas production (13.22 Ld<sup>-1</sup> at OLR of 3.5 kg CODm<sup>-3</sup>d<sup>-1</sup>). Vadlani [8] demonstrated the performance of UASB during start-up operation using synthetic mixed acid waste. These workers reported that, seed sludge from a distillery waste treatment plant improved the performance of the UASB reactor due to a predominance of active biomass. More recently, Zwain [9] demonstrated the start-up performance of modified anaerobic baffled reactor in treating recycled paper mill wastewater at batch and continuous phases and showed a COD

removal efficiency up to 71%. Nevertheless, they reported that the pH was slightly decreased (from 7.3 to 6.2) during this phase.

In summary, start-up is often considered being the most unstable and difficult phase in anaerobic digestion. Therefore, the main objective of this study was to observe and evaluate the start-up performance of an ASR system treating synthetic wastewater using anaerobic digested palm oil mill effluent (POME) sludge. The reactor performance was evaluated in terms of chemical oxygen demand (COD) removal efficiency, biogas composition (methane and carbon dioxide) and pH.

## MATERIALS AND METHODS

### Anaerobic Stage Reactor (ASR)

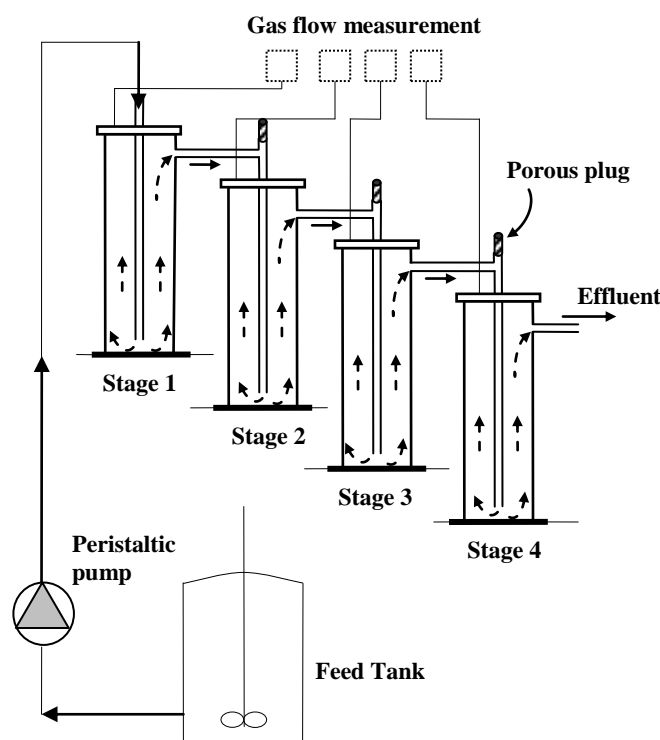


Figure 1: ASR system and flow regime.

The ASR system, comprising four identical cylindrical Plexiglas compartments (stages), linked in series. The operational set-up, flow diagram and the reactor design are presented in Figure 1. Each stage of the reactor had a 3-phase separator baffle, placed below the effluent ports, to prevent floating granules from being washed out with the effluent. The design concept of ASR is similar to the anaerobic baffled reactor (ABR), where each stage represents separate compartments. By having four stages, the ASR could behave like an ABR system, where phase separation is created for better removal of organic substances. The influent wastewater entered through a down comer tube in the head plate and allowed feed to flow upward through the sludge bed. Effluent from each stage of the reactor flowed by gravity to the next,

as each stage was placed on a stepped platform. Temperature controller and heater were installed to maintain the reactor temperature at 37°C. Peristaltic pumps (Masterflex L/S, Easy Load II Pump Head) were used to control the influent feed rate to the first stage of the ASR.

### Reactor Operation

The start-up of ASR was carried out in two (2) major steps; continuous and intermittent feeding using anaerobic digested POME sludge. The reactor was operated in continuous mode of operation at a hydraulic retention time (HRT) of 4 d and OLR of 1.07 kg CODm<sup>3</sup>d<sup>-1</sup> for a period of 34 d. It was found that the continuous feeding method during reactor start-up could not achieve the desired COD removal efficiency. There were several problems occurred during the start-up of ASR system using POME sludge. It was concluded that the POME sludge contained a significant amount of inert solids and dead or inactive microorganisms, which had contributed to the poor performance of ASR during the start-up phase. In addition, there may have been a community imbalance in the anaerobic biomass since the digester was not designed to run exclusively on glucose. As a result, it was thought that the reactor could perform better at intermittent feeding; hence the ASR was started again using POME sludge. The reactor was fed intermittently (9 hours per day), from day 35 until 88 with gradual increment in the OLR (0.82 - 2.45 kg CODm<sup>3</sup>d<sup>-1</sup>).

### Feed and Nutrients

The start-up of ASR was accomplished using glucose. Glucose was used since it is readily degradable, soluble carbohydrate and does not limit the rate of anaerobic biodegradation [1]. In addition, it produces readily measurable intermediary metabolites in anaerobic digestion and is commonly used as a carbonaceous substrate in many experimental studies. Nutrients requirement was adjusted using macronutrients N100 (from Bio-Systems Corporation Asia Pacific Sdn Bhd).

### Seed Sludge

The ASR was seeded with anaerobic digested POME sludge (FELDA Plantation, Gemas, Negeri Sembilan). This was sieved pass 2.0 mm mesh, giving solid contents of 53,750 mg TSSL<sup>-1</sup> and 41,500 mg VSSL<sup>-1</sup>. Each stage of the reactor was added with 7.5 L of the sieved sludge. The remaining volume of the reactor was then filled with tap water. After seeding, the head plates were attached, and the headspace above each reactor was flushed with nitrogen gas to displace residual air in the system before introducing the feed. The reactor was allowed to stabilize at 37°C for 24 hours in 7 d without further modification.

### Sampling and Analysis

Supernatant liquor, gas and sludge samples were taken separately from each stage of the ASR for analysis. In addition, gas composition rate was determined separately for each stage. Routine analysis such as chemical oxygen demand (COD), Mixed Liquid Suspended Solid

(MLSS), Mixed Liquid Volatile Suspended Solid (MLVSS) and pH were carried out in accordance with Standard Method [10]. Reactor gas composition (CO<sub>2</sub> and CH<sub>4</sub>) was determined using Gas Analyzer (GeoTech 2000).

## RESULTS AND DISCUSSION

Figure 2 shows the COD profile in each stage of the ASR treating synthetic wastewater. It can be seen that the COD fluctuated in the ASR due to variability of OLR (1.07 kg CODm<sup>3</sup>d<sup>-1</sup> for continuous and 0.82 to 2.45 kg CODm<sup>3</sup>d<sup>-1</sup> for intermittent feeding). Most of the COD removal in the reactor system occurred in Stage 1, with minor amounts occurring in the subsequent stages, which is a general pattern in staged anaerobic treatment [11, 12, 13]. The influent COD applied to ASR was varied in order to create a different OLR (1.07 kg CODm<sup>3</sup>d<sup>-1</sup> for continuous and 0.82 to 2.45 kg CODm<sup>3</sup>d<sup>-1</sup> for intermittent feeding). The COD profile across the reactor followed the order of Stage 1 > Stage 2 > Stage 3 > Stage 4.

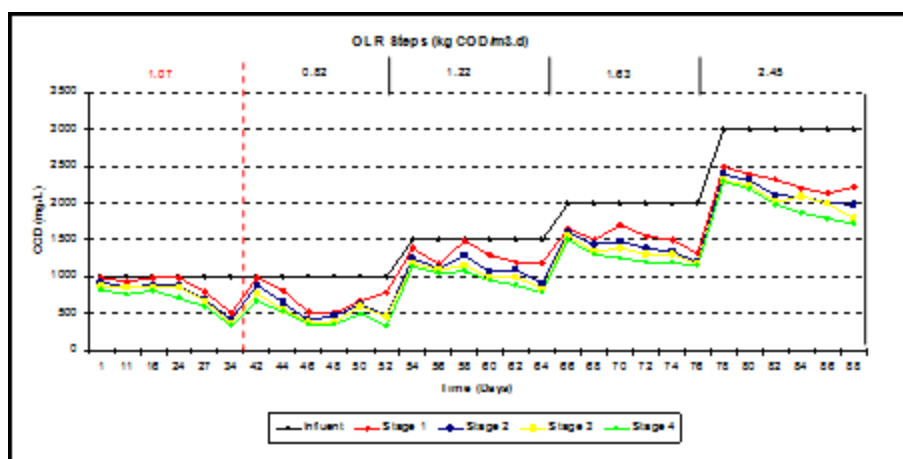
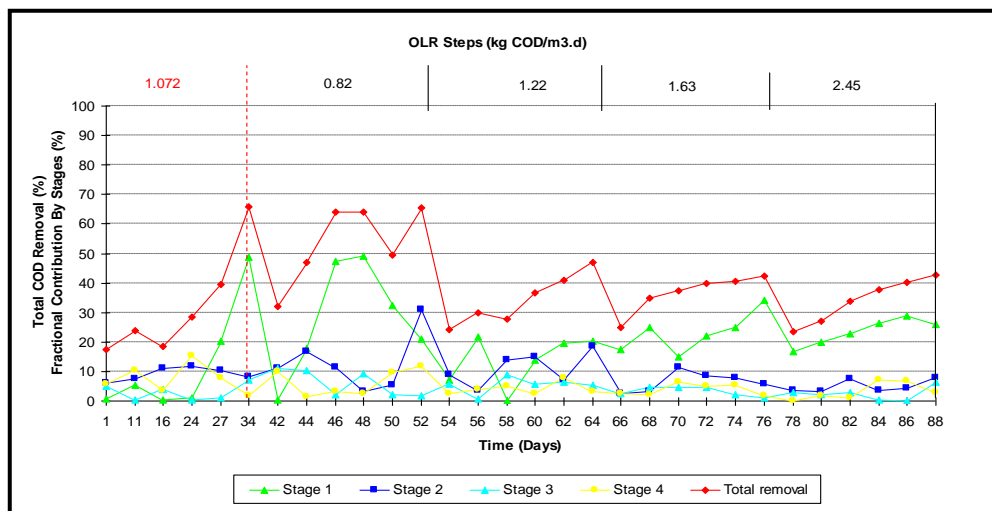
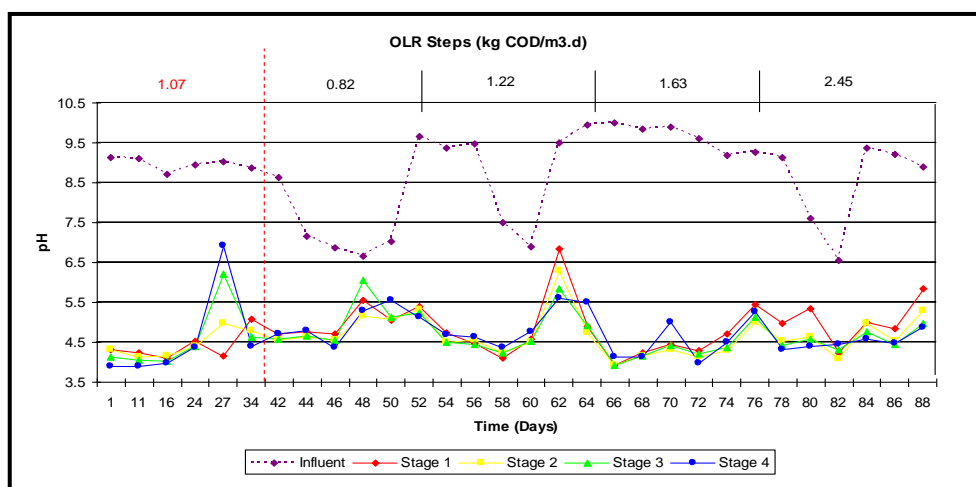


Figure 2: COD profile in each stage of ASR at different OLR (the value 1.07 kg CODm<sup>3</sup>d<sup>-1</sup> (highlighted in red) shows continuous feeding).

Figure 3 shows temporal changes in the total COD removal and fractional contribution by each stage of the ASR treating synthetic wastewater. When the reactor was fed by continuous feeding (OLR 1.07 kg CODm<sup>3</sup>d<sup>-1</sup>), the total COD removal efficiency was low (average 26%). However, when the ASR was fed by intermittent feeding, the COD removal efficiency showed some slight improvement at an OLR of 0.82 kg CODm<sup>3</sup>d<sup>-1</sup> (average removal efficiency was 54%). Nevertheless, when the OLR was increased further from 1.22 – 2.45 kg CODm<sup>3</sup>d<sup>-1</sup>, the COD removal efficiency declined to an average value of 35%. It can be concluded that continuous and intermittent feeding does not improve the performance of the ASR system during the treatment using synthetic wastewater using POME sludge.



**Figure 3: Total COD reduction (%) of ASR and fractional contribution (%) to the total COD reduction by each stage at different OLR .**



**Figure 4: pH profiles in each stage of ASR at different OLR .**

Microbial groups involved in anaerobic degradation have a specific pH region for optimal growth. The desired pH for anaerobic treatment is between 6.6 and 7.6 [14]. Values outside this range can be quite detrimental to the process, particularly to methanogenesis. Therefore, maintaining a suitable and stable pH within the digester should be a major priority for ensuring efficient methanogenic digestion. Figure 4 shows pH levels in all stages of ASR system and it was not stable (pH 3.9-5.5). The pH profile stability across the reactor followed the order of Stage 1 < Stage 2 < Stage 3 < Stage 4. The order follows general view of pH profile across stage reactors, where initial stages or compartments predominated by acids resulting in reduced pH values. In order to maintain the pH levels, sodium hydroxide (NaOH) was added to the reactor system; however, this did not help recover the required pH values. This is because the anaerobic microorganisms which mediate the biodegradation of glucose grow at slow rates, even under ideal conditions of neutral pH. If the rate of acid production in an anaerobic reactor is not matched by the rate of methanogenesis, acids may accumulate and lead to a drop in pH. Methanogenic bacteria, in particular, are severely inhibited by pH levels below 6.5 [14]. In

addition, anaerobic sludge used in a reactor should be properly acclimatized to select the microorganisms that are best suited to degrade the VFAs produced by the acid formers [15]. Therefore, the accumulation of acids in the ASR stages may have contributed to the pH reduction. Moreover, the methanogens may possibly have reduced further leading to even higher accumulations of acids and lower pH levels.

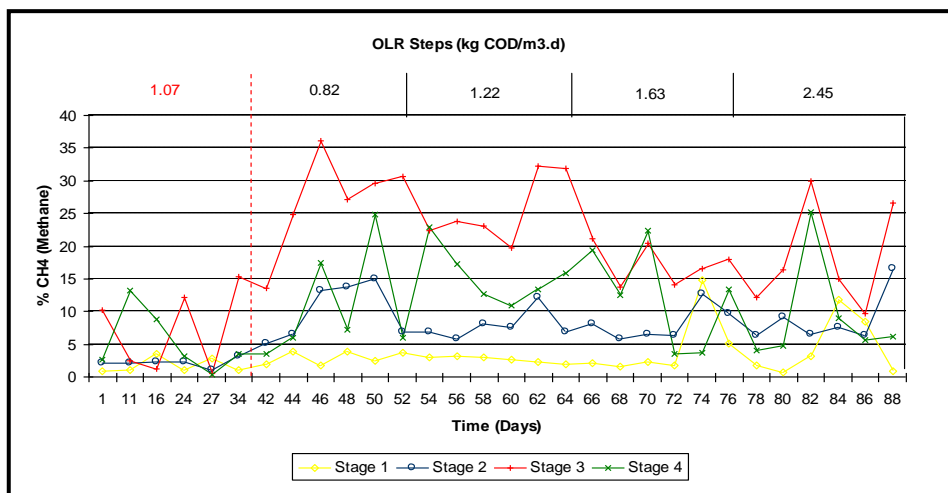


Figure 5: Proportion of CH<sub>4</sub> (%) in the biogas in each stage of ASR at different OLR.

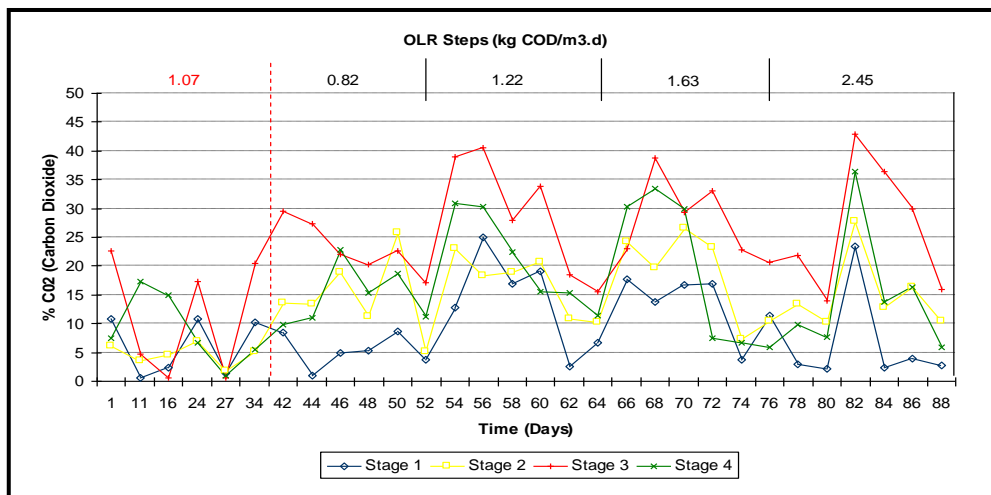


Figure 6: Proportion of CO<sub>2</sub> (%) in the biogas in each stage of ASR at different OLR.

Figure 5 shows the methane composition in each stage of ASR during the start-up phase. The methane composition fluctuated in all stages, with Stage 1, having lowest (less than 5%) compared to other stages. The highest methane composition was produced in Stage 3 (36.1%; OLR 0.82 kg CODm<sup>3</sup>d<sup>-1</sup>). Carbon dioxide (CO<sub>2</sub>) composition was presented in Figure 6 and shows fluctuation in all stages during the start-up period. The highest value of CO<sub>2</sub> composition was 42.9%, occurred in Stage 3 at OLR 2.45 kg CODm<sup>3</sup>d<sup>-1</sup>. The presence of CO<sub>2</sub> (methanogenesis phases) in the reactor up to 30% will increase the acid concentration in sludge and may reduce the pH value [16]. Moreover, higher CO<sub>2</sub> content may results from lack of proper balance

among food supply, temperature and digestion time [1]. The lower levels of methane composition may be due to the effect of pH in the reactor system, which was not stable.

One important observation from the biogas composition profile is that the predominance of methane gas in Stage 3 and 4 of the ASR system (Figure 7 and 8). It can be seen that the methane gas was less than 14.5% in Stages 1 and 2 of the ASR system, confirming this stages were dominated by acidogens; an apparent trend in staged reactor system. In can be concluded that phase separation occurred in the ASR system during this period, with acidogenesis in the first two stages (Stage 1 and 2) and methanogenesis, in later stages (Stage 3 and 4).

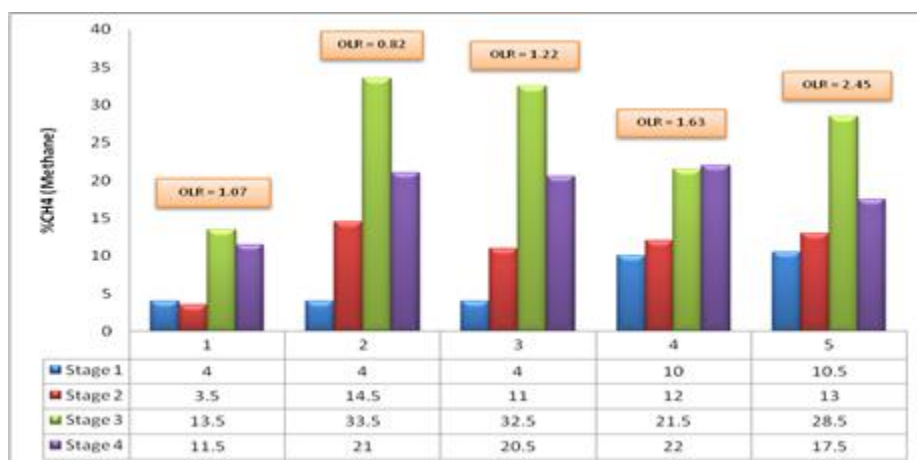


Figure 7: Typical CH<sub>4</sub> (%) trend in each stage of ASR at different OLR.

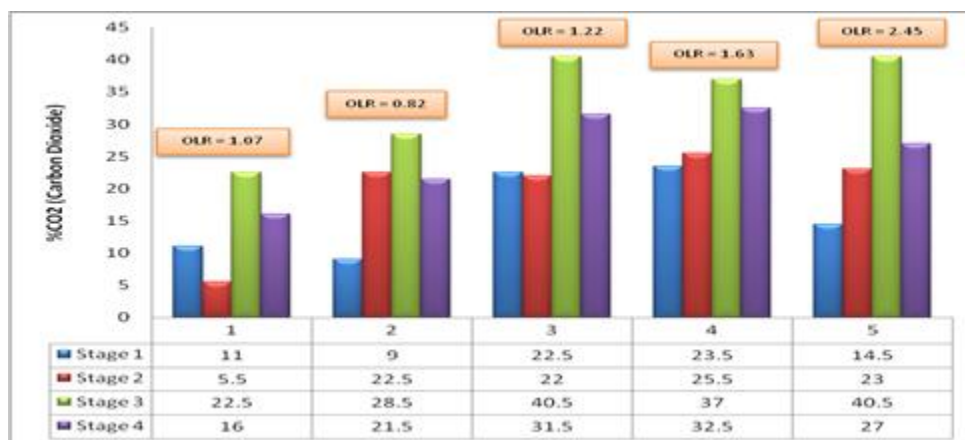


Figure 8: Typical CO<sub>2</sub> (%) trend in each stage of ASR at different OLR.

From the results, it can be concluded that OLR plays an important role in the performance of the ASR system. The load values to be applied during the start-up depend on the seed sludge employed and on its acclimatization to the wastewater to be treated. The initial load should be gradually increased according to the treatment efficiency of the system. In the current study, the COD removal efficiency was only 26% (average) when the reactor was operated at an OLR of 1.07 kg CODm<sup>3</sup>d<sup>-1</sup> and 54% (average) at an OLR of 0.82 kg CODm<sup>3</sup>d<sup>-1</sup>,



suggesting that the OLR should not be increased further since the reactor was not stable. It is always a good practice to allow adequate time for the reactor to stabilize (in terms COD removal efficiency) before increase the OLR. A steady state of COD removal of more than 80% is considered acceptable for anaerobic reactor start-up and acclimatization. In another word, the OLR should be increased when the COD concentrations have been reduced by 80% [3].

## CONCLUSIONS

The reduction of the period necessary for the start-up and improved operational control such as OLR are important factors to increase the efficiency and the competitiveness of anaerobic systems. Systematized operational procedures are very important, mainly during the start-up of anaerobic reactor systems. The start-up of anaerobic reactors is determined by the initial transient period, marked by operational instabilities. Accordingly, the poor performance of the ASR system during start-up period using POME sludge could be due to many reasons. One reason could be due to the adaptability of the POME sludge to the influent wastewater. It is generally known that if seed sludge not adapted to the wastewater to be treated, the start-up of the system goes through an acclimatization period, including a microbial selection phase. This process will take some time since acclimatization is necessary for the new microorganisms. Another reason could be due to the OLR applied. The load values to be applied during the start-up depend on the type of seed sludge employed and on its acclimatization to the wastewater to be treated. The initial load should be gradually increased according to the efficiency of the system. Once the COD removal efficiencies are above 80% and remain there, then the loading rate can be increased.

## ACKNOWLEDGEMENTS

The authors thank Universiti Teknologi Malaysia and Ministry of Education for funding this research under Research University Grant, Project Vote Number Q.K130000.2540.03H64.

## REFERENCES

- [1] Stronach SM, Rudd T, Lester JN. Anaerobic Digestion Process in Industrial Wastewater Treatment 1986.
- [2] Weiland P, Rozzi A. Water Sci Technol 1991; 24: 257-277.
- [3] Lettinga G, van Handeel. Antonie van Leeuwenhoek 1995; 67: 3-28.
- [4] Chernicharo CAL. Anaerobic Reactors. Biological Wastewater Treatment Series 2007.
- [5] Chernicharo CAL, Nascimento MCP. Water Sci Technol 2001; 44: 221-228.
- [6] Cresson R, Carrere H, Delgenes JP, Bernet N. Biochem Eng J 2006; 30: 55-62.
- [7] Sowmeyan R, Swaminathan G. Biores Technol 2008; 99: 6280-6284.
- [8] Vadlani, PV, Ramachandran KB. Biores Technol 2008; 99: 8231-8236.
- [9] Zwain HM, Hassan SR, Zaman NQ, Aziz HA, Dahlan I. J Environ Chem Eng 2013; 1: 61-64.
- [10] Greenberg AE, Trussell RR, Clisceri LS. Standard Methods for Examination of Water and Wastewater. American Public Health Association (APHA). 6<sup>th</sup> Edition 1998.
- [11] Chelliapan S, Wilby T, Yuzir A, Sallis PJ. Desalination 2011; 271: 257-264.



- [12] Chelliapan S, Wilby T, Sallis PJ. Brazil J Chem Eng 2001; 28: 51-61.
- [13] Chelliapan S, Wilby T, Yuzir A, Sallis PJ. Water Sci Technol 2011; 63: 1599 - 1606.
- [14] Speece RE. Anaerobic Biotechnology for Industrial Wastewater. Archae press 1996.
- [15] Aguilar A, Casas C, Lema JM. Water Res 1995; 29: 505-509.
- [16] Gerardi MH. The Microbiology of Anaerobic Digesters 2003.