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Anaerobic Digestion of Pulping Wastewater Using Up-flow Anaerobic Sludge Blanket (UASB) Reactor at Mesophilic Condition

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Abstract: The waste considered for this study was the effluent of the pulping process produced during the manufacture of pulp and paper in the Rakta integrated pulp and paper mill in Alexandria. The pulping process in Rakta mill is conducted under pressure of 7-8 bar, 165°C using 10% NaOH based on straw weight for 2 h. The spent liquor after alkali cooking contains any where 8 to 15% solids in solution and primarily these dissolved solids are lignin and dissolved carbohydrates. The effect of some operating parameters on the performance of the Using Up-flow Anaerobic Sludge Blanket was studied; these include mainly the organic loading rate and hydraulic retention time. The gas production rate increased to 3.43 m^3 biogas/(m^3 reactor volume. day), by increasing the Biochemical Oxygen Demand (BOD) loading rate to 16 kg BOD feed/(m^3 reactor volume. day), with a gas yield of 0.66 m^3 biogas/(kg reduced organic matter). The efficiency was reduced by increasing the loading rate. Thus by increasing the BOD loading rate from 0.4 to 16 kg BOD feed/(m^3 reactor volume. day), the reduction in BOD decreased from 79% to 42%.

Keywords: Anaerobic, Black Liquor, Digestion, Mesophilic Temperature, Using Up-flow Anaerobic Sludge Blanket

Introduction

Anaerobic Digestion (AD), as a stable biological process to deal with various substrates, is increasingly used for treatment of P&P mill wastewaters in recent years. This is mainly due to its several advantages over other conventional techniques such as the reduction of the produced sludge volume by 30-70%, methane production as an energy carrier, design simplicity and non-sophisticated equipment requirement, cost-effectiveness in terms of low capital and operating cost, applicability in different scales and the rate of pathogen destruction, particularly in the thermophilic process (Ekstrand *et al*., 2013; Lin *et al*., 2011; Zwain *et al*., 2013).

The performance of these methods, in terms of optimizing the biogas yields and minimizing the solid wastes production, is directly influenced by various factors. P&P production process, wastewater composition and reactor operating conditions, available inoculums for anaerobic digestion steps and the operational costs can be considered the main influencing factors. The type of P&P making process can significantly affect the total yield of the methane production. Ekstrand *et al*. (2013) investigated the methane potential of 62 Swedish P&P mills wastewater from10 different processes (including KP, TMP, CTMP and NSSC) at seven P&P mills in anaerobic batch digestion conditions. The results of their study illustrated that TMP mill wastewaters samples gave the best average yield (with all six samples) among all studied production methods ranging 40-65% of the theoretical CH_4-yield potential. In terms of raw materials, although alkaline ECF hard wood effluents resulted in higher yields than soft wood effluents, however, no significant raw material dependence has been observed in the case of TCF bleaching effluents at the KP mills.

The ratio between the required nutrients and the pollutant loads can be considered the main wastewater properties, affecting the yield of the AD process. The optimal range of operating C:N ratio, as an effective factor for anaerobic bacterial growth in an AD system, has been recommended between 20/1to30/1 with an optimal ratio of 25/1 (Li *et al*., 2011). The low C:N ratio may result in higher total ammonia nitrogen release and/or high VFA accumulation in the digester which is an important inhibitor of the AD process. Moreover, the high C:N ratio would be responsible for the rapid consumption of nitrogen by methanogens and lower biogas Production (Zeshan *et al*., 2012). Thus, some measures may be required to improve the digestibility of the P&P mill wastewater and the biogas yield. Anaerobic co-digestion is one of these improvements which has been efficiently applied to some types of wastewaters such as co-digestion of sugar-beet processing wastewater and beet-pulp (Alkaya and Demirer, 2011) and olive mill wastewater with olive mill solid waste (Boubaker and Ridha, 2008). This is mainly because of AD co-digestion merits such as making a balance between required nutrients ratio, toxic compounds degradation, buffering capacity supplying and sharing the substrate and equipment between different treatment plants (Alkaya and Demirer, 2011). The results of the study carried out by Yu *et al*. (2004) revealed that a considerable lignocellulose fraction in thermophilic acidogenesis (approximately 6.6 times more) can be achieved by co-digestion of TMP wastewater and glucose. Yang *et al*. (2009) investigated the lignocellulosic structural changes of Spartina alterniflora co-digested with potato. After batch anaerobic digestion for 60 days, the total cumulative biogas yield of co-digestion process was noticeably higher than that of mono-digestion by S. alterniflora.

However, it should be stated that the number of the published works on the P&P mill residues co-digestion with suitable substrates is scarce and they have mainly focused on the P&P primary and secondary sludge co-digested with substrates such as municipal sewage sludge (Hagelqvist, 2013), food waste (Lin *et al*., 2012) and mono sodium glutamate waste liquor (Lin et al., 2011). Wastewater properties can also directly affect the biogas production via, forinstance, the toxic effects on the microbial inoculum which are responsible for anaerobic digestion through hydrolysis, acidogenic, acetogenesis and methanogenesis phases. The results of a recent study indicated that the acidic ECF effluents can significantly reduce the efficiency of the AD process because of the toxic effects on the AD-flora, while most of alkaline ECF bleaching effluents gave positive methane yields (Ekstrand *et al*., 2013). In addition to the pH adjustment, some measures may enhance the inoculums related performance of AD. For instance, rumen fluid has been studied as an effective anaerobic microbial ecosystem, including a complex microbial population (i.e., bacteria, protozoa, fungi and archaea) with a high hydrolytic and acidogenic activity when lignocellulosic substrates are used (Yue *et al*., 2013). Baba *et al*. (2013) conducted the pre-treatment experiments of the waste paper as a model of cellulosic biomass pre-treatments (composed of cellulose, hemicellulose and lignin) using rumen fluid prior to methane production. In their study, VFAs, especially acetate, were produced and the best daily methane yield was obtained by the 6h pre-treatment. The reported amount of produced gas was 2.6 times higher than that of untreated paper, which resulted in 73.4% of the theoretical methane yield.

This paper investigates advantages and performance for Anaerobic Digestion of Pulping Wastewater Using Up-flow Anaerobic Sludge Blanket (UASB) reactor at mesophilic condition by studying the effect of some operating parameters on treatment efficiencies and biogas production rate. Effect of organic loading rate on the bed concentration was monitored during all study periods.

Materials and Methods

Wastewaters Composition

Black liquor was obtained from Rakta mills (Alexandria Governorate in Egypt) after concentration by evaporation. The anaerobic digestion was carried out after dilution of the samples according to the required concentration. The analysis of the concentrated black liquor is given in Table 1. Some samples were taken from the effluent stream of soda pulping section after washing for analysis, the analysis of the black liquor effluent stream is included in Table 1.

Experimental Set-Up

The study was carried out using Fermenter Drive Assembly produced by New Brunswic Scientific. The apparatus consists of:

A glass jar of about 45 cm height and 20 cm internal diameter. The total jar volume is about 14 L. The jar is embedded in a water bath. The reactor contains two similar peristaltic pumps, one of them is used for introducing the feed into the reactor hence acting as nutrient pump, while the other is used for discharging of the treated wastewater from the reactor, thus acting as harvest pump:

- A temperature controlling system, to maintain the operating temperature of the water bath at the required level 37°C
- Level controlling system to adjust the level of the liquid in the reactor at the required value (nearly 12 litre operating volume). The Harvest pump operates automatically, to maintain the liquid level in the reactor near to the required value

Start-Up of the UASB Reactor

Digestor was operated by using old granulated sludge produced during anaerobic treatment of wastewater produced from yeast production. 0.5 L active sludge from a UASB reactor operating on vinasse was added during start-up.

Table 1. Analysis of the Black Liquor

Sample	Strong concentration	Moderate concentration	Diluted stream
pH	11.68	8.64	6.490
Ec dsm^{-1}	47.40	20.90	5.500
Na* %	2.01	0.87	0.090
K* %	0.13	0.13	0.025
Ca* %	0.63	0.25	0.060
Mg* %	0.24	0.36	
N mg/L	807.00	449.00	192.500
P mg/L	82.40	51.60	20.000
TS g/L	120.00	40.00	7.470
TV g/L	55.20	18.50	2.397
COD g/L	117.00	31.70	4.650
BOD g/L	33.00	8.80	1.200
Ash* % (based on TS)	39.00	38.00	42.800

*The units used in Table 1 are weight percent

Table 2. Composition of the biogas produced

No.	Retention time (hrs)	Organic loading rate ($kg/m^3/day$)	COD loading rate ($kg/m^3/day$)	BOD loading rate ($kg/m^3/day$)	$\%CH_4$	$\%CO_2$
1	91.30	1.68	2.06	0.51	83.55	16.45
3	49.10	2.62	4.10	1.21	82.14	17.86
7	12.10	9.57	19.70	5.52	77.90	22.10
9	6.04	19.66	39.00	11.12	68.00	32.00
10	4.11	27.90	56.85	15.97	76.50	23.50

The reactor was started by feeding with vinasse of low concentration (COD=2000ppm). Retention time was fixed around 100 h. Vinasse concentration was increased gradually to reach 13500 ppm COD within four months. Reduction in volatiles was about 80% during operation. After that dosing of black liquor was started using 0.5% based on the total solids fed to the reactor, then an increase in black liquor concentrations was carried gradually by replacing of the vinasse till reaching 100% black liquor, which represented about 1% total solids (the same concentration of the final stream out from pulping section after dilution).The start-up period was carried out at 37°C. Using the aforementioned procedures, the reactor was acclimated fully in 5 months.

Methods of Analysis

Chemical analysis was performed on both the influent and effluent of the system, according to the method recommended by Eaton and Franson (2005). Total Solids (T.S) and volatiles (T.V.S), soluble solids and volatiles, total suspended solids and volatiles, pH and temperature were determined daily. Both total Chemical Oxygen Demand (COD) and The Biochemical Oxygen Demand (BOD) were determined during the period of steady state. Also the total solids and volatiles and suspended solids and volatiles were determined for the bed of the UASB at the end of every experiment.

Analysis of Biogas

During the experimental work, samples of the gas produced were taken to determine the composition of the gas. The apparatus used is a Perkin-Elmer Sigma 2 B gas chromatograph equipped with a hydrogen flame ionization detector and a hot wire detector. The apparatus is also equipped with an electronic integrator (Perkin-Elmer -Sigma-10 Data System).

Results and Discussion

Effect of Operating Parameters on the Gas Production Rates

The effect of some operating parameters on gas production rate was studied using the Upflow Anaerobic Sludge Blanket (UASB) System. These include: the hydraulic retention time, the organic loading rate, the chemical oxygen demand loading rate and biochemical oxygen demand loading rate.

Effect of the Hydraulic Retention Time

Results obtained for the gas production rates at different Hydraulic Retention Times (HRT) are given below. Higher gas production rates were obtained at 4 hrs retention time, reaching 3.43 $m^3/m^3/day$ at the optimum mesophilic temperature of 37°C

Figure 1 shows that the gas production rate decreased by increasing the retention time. The decrease in gas production rates with increasing the retention time is mainly attributed to the associated decrease in the organic loading rate.

Effect of the Organic Loading Rate

The organic loading rate varied in the range 1.68 to 27.9 kg Volatile/m^3.d as shown in Fig. 2. A new distribution in size of bed granules bed was found as a result of increasing the organic loading rate.

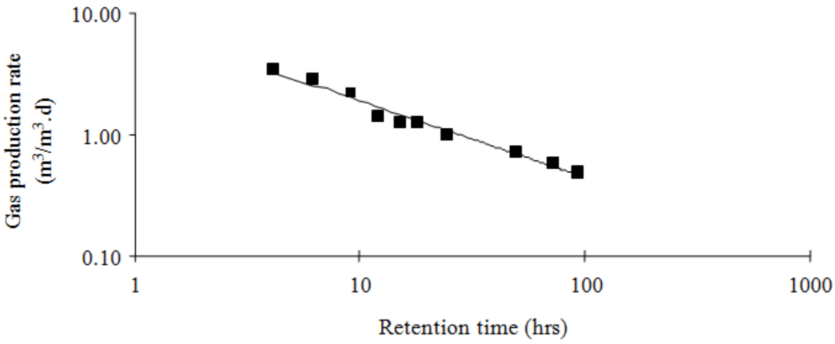

Fig. 1. Effect of retention time on gas production rate

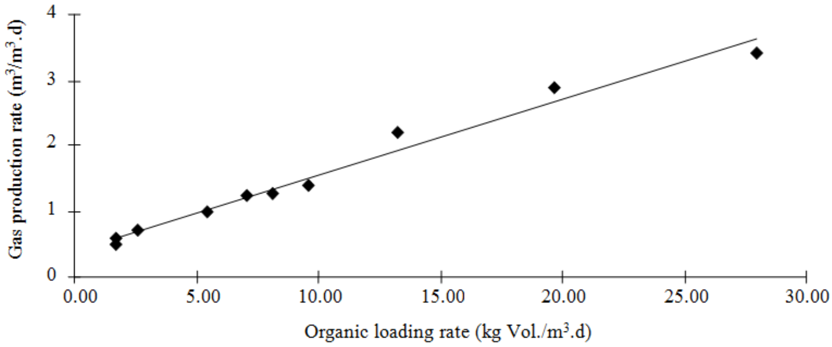

Fig. 2. Effect of organic loading rate on gas production rate

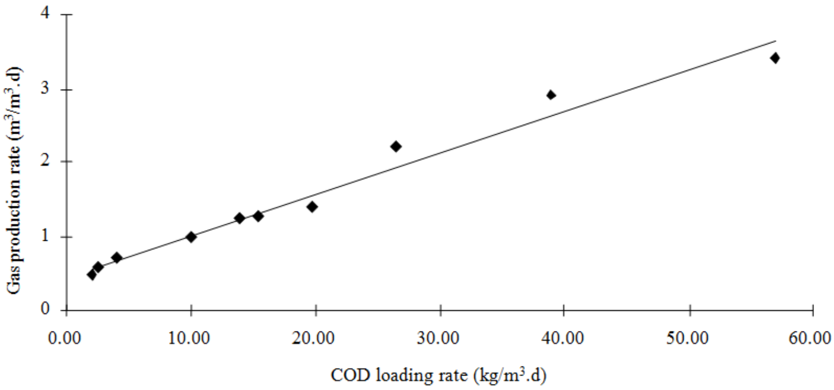

Fig. 3. Effect of COD loading rate on gas production rate

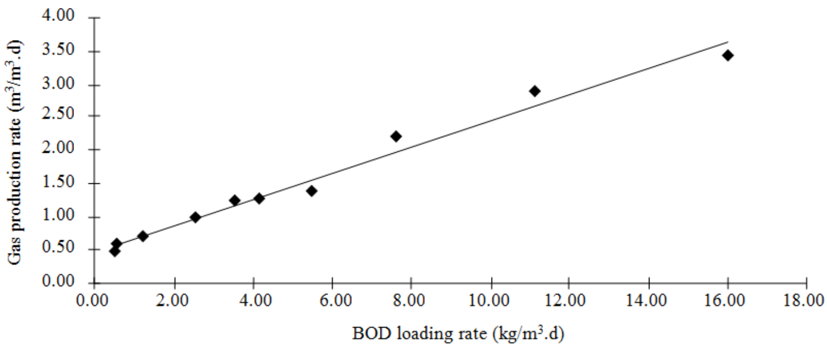

Fig. 4. Effect of BOD loading rate on gas production rate

The gas production rate increased with increasing the organic loading rate as shown in Fig. 2. The increasing in gas production rate with increasing the organic loading rate is mainly due to the increase in the organic matter available for digestion per unit time. The increase in organic loading rates were accompanied by drastic changes in biobed characteristics as well be shown later.

Effect of the COD Loading Rate

The COD loading rate varied in the range 2.06 to 56.85 kg COD/m^3.d, as shown in Fig. 3. The gas production rate increased with increasing the COD loading rate as shown in Fig. 3. The increase in gas production rate by increasing the COD loading rate is mainly due to the increase in the active organic matter available for digestion per unit time.

Effect of the BOD Loading Rate

The BOD loading rate varied in the range 0.51 to 15.97 kg BOD/m^3.d, as shown in Fig. 4. The gas production rate increased with increasing the BOD loading rate as shown in Fig. 4. The increase in gas production rate by increasing the BOD loading rate is due to the increase in the biodegradable organic matter available for digestion per unit time.

In fact the effect of COD loading rate and BOD loading rate on gas production rate are similar to that of organic loading rate. This is quite reasonable, as the COD load and BOD load however different but is directly related to the volatile organic load.

Effect of Operating Parameters on the Gas Yield

The effect of some operating parameters on gas yield was studied. These include: The hydraulic retention time, the organic loading rate, the chemical oxygen demand loading rate and biochemical oxygen demand loading rate.

The evaluation of gas yield was based on reduced volatile, reduced COD and reduced BOD.

Effect of the Hydraulic Retention Time

The results obtained for the gas yield at different hydraulic retention times are given below. The gas yield based on the reduced volatile solids is illustrated in Fig. 5, at different retention times. The maximum gas yield obtained was 0.77 m^3/kg reduced Volatile solids; at retention time equal 91.3 h. As seen from Fig. 5, the gas yield decreased gradually by decreasing the retention time. The decrease in gas yield at lower retention times may be attributed to the incomplete conversion of organic matter as a direct cause of small detention time and high organic load and accordingly overloading of the system.

The gas yield obtained based on the reduced COD and reduced BOD is different to some extent from above mentioned. Thus the maximum gas yield based on reduced COD and reduced BOD of 0.55 m^3/kg reduced COD and 1.28 m^3/kg reduced BOD respectively were obtained while operating at 91.3 h hydraulic retention time. The results obtained are shown in Fig. 5. As seen from Fig. 5, the gas yield decreased by decreasing the retention time, which can be attributed to the incomplete conversion of the organic matter as mentioned before.

Effect of Organic Loading Rate

Results obtained for the gas yield at different organic loading rates are given in Fig. 6. As seen from Fig. 6 the gas yield decreased gradually by increasing the organic loading rate. This may be due to the incomplete digestion of organic matter accompanied by the high organic loads and short retention times.

Effect of the COD Loading Rate

Results obtained for the gas yield at different COD loading rates are given in Fig. 7. The maximum value obtained for the gas yield was 0.55 m^3/kg reduced COD, at COD loading rate of 2.06 kg COD/m^3.day and decreased to 0.25 m^3/kg reduced COD at COD loading rate of 56.85 kg COD/m^3.day. Thus, as seen from Fig. 7, the gas yield decreased by increasing the COD loading rate. The decrease may be due to the incomplete digestion of organic matter accompanied by the high COD loading rates and short retention times.

Effect of the BOD Loading Rate

Results obtained for the gas yield at different BOD loading rates are given in Fig. 8. From this figure the maximum gas yield obtained was equal 1.28 m^3/kg reduced BOD during the operation at 0.51 kg BOD/m^3.day. The gas yield decreased sharply by increasing the BOD loading rate as illustrated in Fig. 8. Values of 0.51-1.28 m^3/kg reduced BOD were obtained.

Effect of Operating Parameters on the Treatment Efficiency

The effect of some operating parameters on the treatment efficiency was studied. These include: The hydraulic retention time, the organic loading rate, the chemical oxygen demand loading rate and biochemical oxygen demand loading rate.

The efficiency was evaluated based on the reduction in volatile solids, COD and BOD.

Effect of the Hydraulic Retention Time

Results obtained for the efficiency at different retention times are given in Fig. 9.

The efficiency, based on the reduction of volatile solids reached 49% at 91.3 h hydraulic retention time. Figure 9 shows that the reduction of volatile solids decreased sharply by decreasing the retention time. This may be attributed to the short time of contact at lower retention time and accordingly, lower reductions in volatiles.

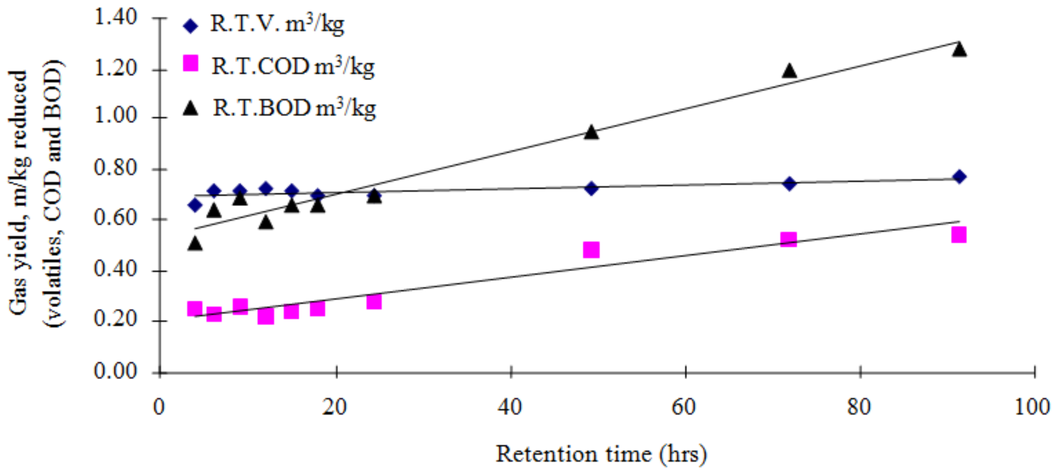

Fig. 5. Effect of retention time on gas yield

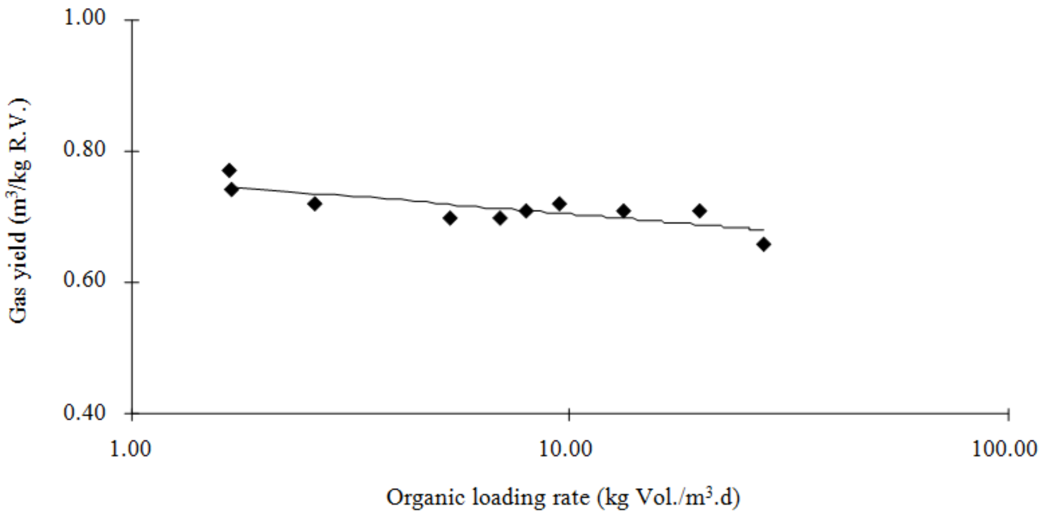

Fig. 6. Effect of organic loading rate on gas yield

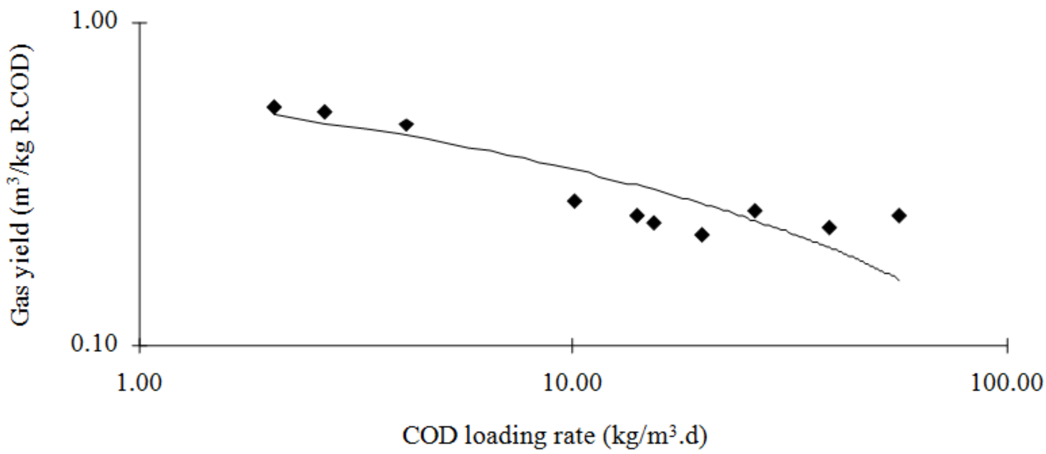

Fig. 7. Effect of COD loading rate on gas yield

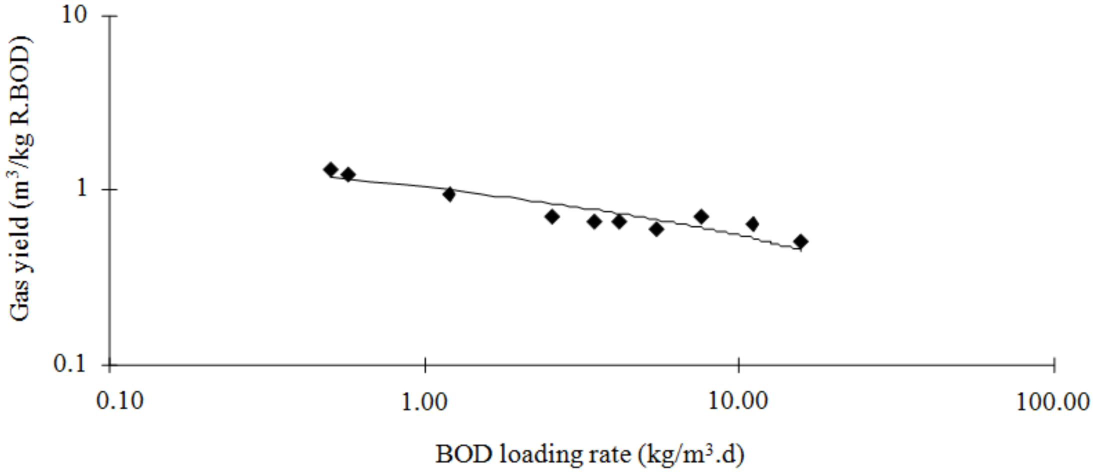

Fig. 8. Effect of BOD loading rate on gas yield

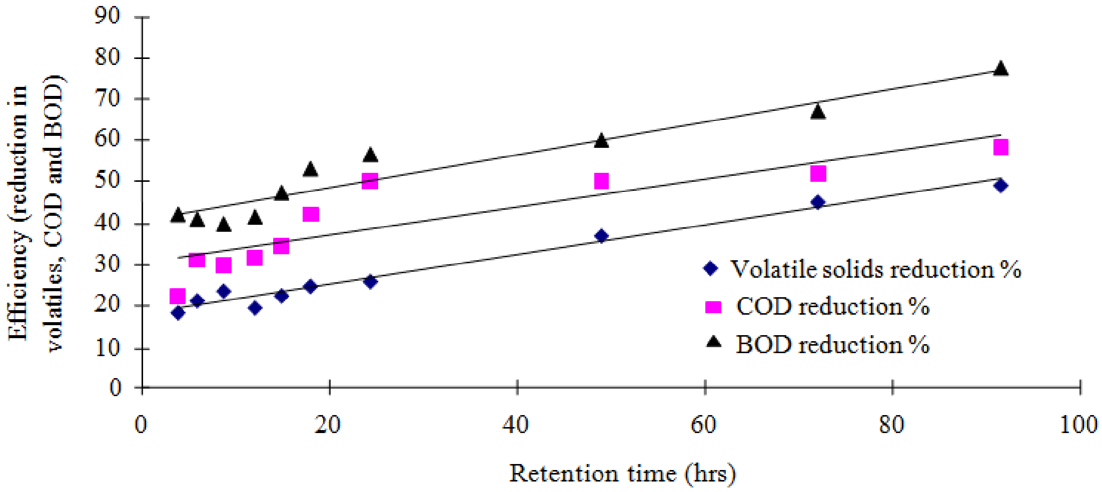

Fig. 9. Effect of retention time on treatment efficiency

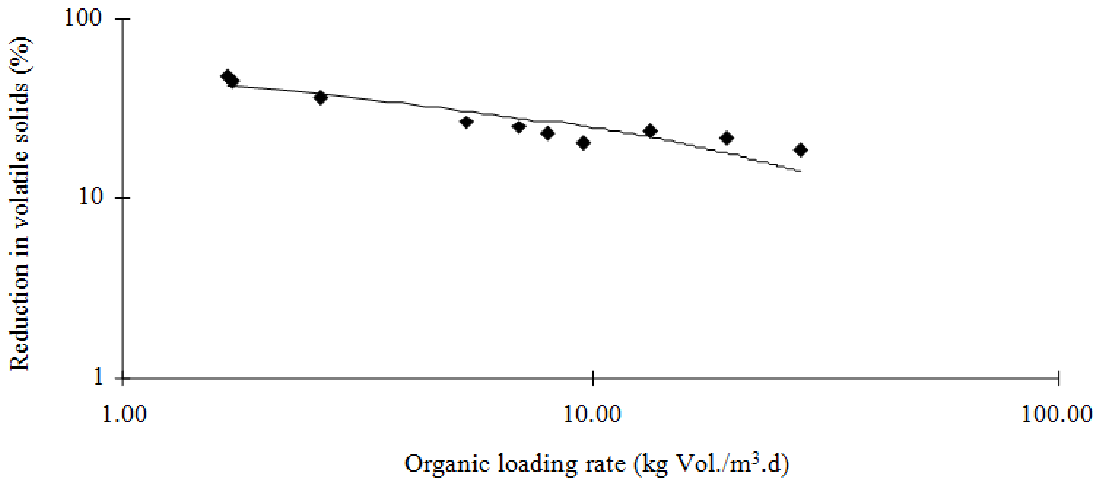

Fig. 10. Effect of organic loading rate on %Reduction in volatile solids

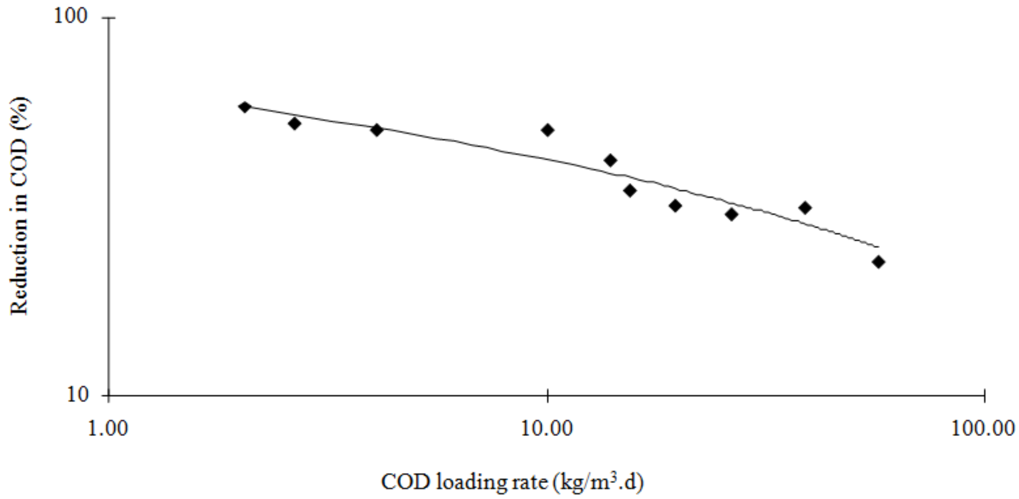

Fig. 11. Effect of COD loading rate on %Reduction in COD

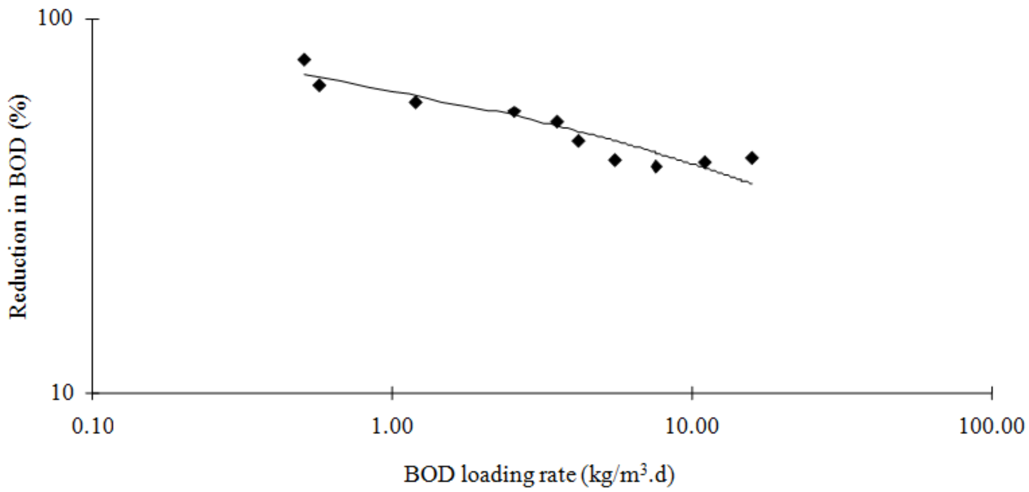

Fig. 12. Effect of BOD loading rate on %Reduction in BOD

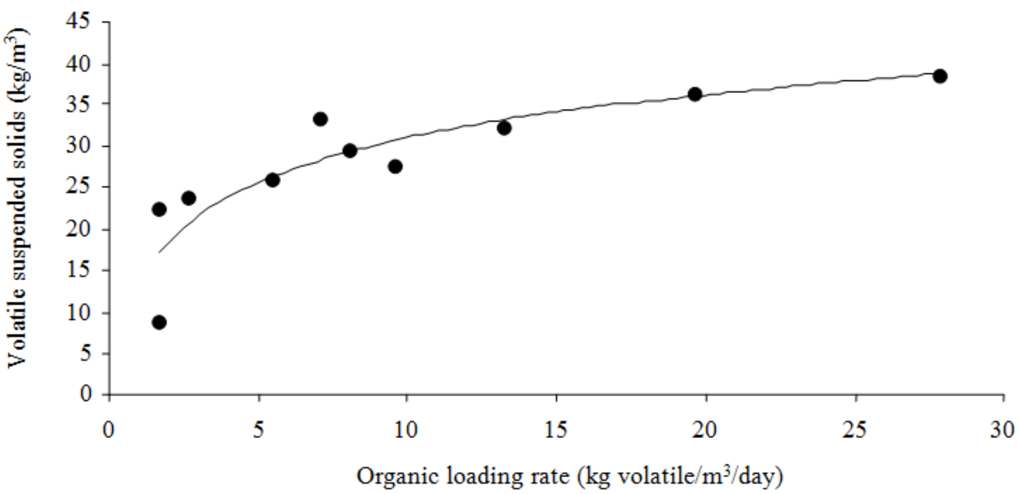

Fig. 13. Effect of organic loading rate on bed concentration

The efficiency, based on the reduction of COD reached 58% at 91.3 h hydraulic retention time. Figure 9 shows that the efficiency (the reduction of COD) decreased by decreasing the retention time. This may be attributed to the short time of contact at lower retention time.

The efficiency, based on the reduction of BOD reached 78% at 91.3 h hydraulic retention time but decreased sharply to reach 42% at 4 hrs hydraulic retention time, as illustrated in Fig. 9. This is due to the short time of contact at lower retention time. The high decrease in BOD reduction at lower hydraulic retention time indicates the difficulty to treat the complex wastewater (black liquor) at low retention time.

Effect of the Organic Loading Rate

The results obtained at different organic loading rates are given in Fig. 10.

The efficiency based on the reduction of volatile solids reached 49% at 1.68 kg volatiles/m^3.day and decreased to 19% at 27.9 kg volatiles/m^3.day, From Fig. 10 it is clear that the reduction of volatile solids(efficiency) decreased by increasing the organic loading rate. This may be due to the decrease in the conversion time associated with the increase in organic loading rate.

Effect of the COD Loading Rate

The results obtained for the treatment efficiency at different COD loading rates are given in Fig. 11. High reduction in COD of 58% was obtained at the lowest COD loading rate of 2.06 kg COD/m^3.day. However, the efficiency was decreased to 23% at COD loading rate of 56.85 kg COD/m^3.day.

The efficiency based on the reduction of COD is illustrated in Fig. 11, from which it is clear that the efficiency decreased by increasing the COD loading rate in the tested range of 2.06-56.85 kg COD/m^3.day.

Effect of the BOD Loading Rate

The results obtained for the treatment efficiency at different BOD loading rates are given in Fig. 12. High reduction in BOD of 78% was obtained at the low BOD loading rate of 0.51 kg BOD/m^3.day.

The efficiency based on the reduction of BOD is illustrated in Fig. 12, from which it is clear that the efficiency decreased by increasing the BOD loading rate. The decrease in efficiency by increasing the BOD loading rate may be regarded to as a result of the incomplete conversion due to the high loading rates.

Effect of Organic Loading Rate on the Bed Concentration

The bed Volatile Suspended Solid (VSS) is an important aspect of UASB reactor design, as it regulates the biosolids concentration and, thereby the maximum organic load which a reactor can assimilate depends on it.

During the study, the sludge bed VSS concentration increased from the initial value of 0.9-3.8% by increasing the organic loading rate from 1.68-27.9 kg volatile/m^3/day; Fig. 13 illustrates the variation of bed VSS as function of organic loading rates. Thus at steady state the bed concentration increased with increasing the organic loading rate.

Gas Composition

Composition of the biogas produced is included in Table 2. The results obtained for biogas produced show high percentage of methane in the range 68-83.5%. The CO_2 content is low and varies in the range 16.45-32%.

The high content of methane may be attributed to the dissolution of appreciable part of CO_2 in the aqueous phase.

Conclusion

Based on the results of experimental study concerning the effectiveness of anaerobic digestion of pulping wastewater at different operating conditions, several recommendations and conclusions can be derived and presented as follows:

- The UASB system used is capable of operating at high loading rate of 16 kg BOD/m^3/day
- High gas production rates were obtained at high BOD loading rates, the gas production rate was3.43 m^3/m^3/day at 16 kg BOD /m^3/day
- The efficiency of treatment decreased by increasing the loading rate. At low BOD loading rate of 0.4 kg BOD/m^3/day the reduction in BOD was 79% and decreased to 42% at 16 kg BOD/m^3/day
- The gas yield values for UASB system were in the range 0.66-0.75 m^3/kg reduced volatiles
- The bed suspended volatile (VSS) of UASB system was proportional to the organic loading rate

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Author's Contributions

Randa M. Osman: Participated in all experiments, coordinated the data-analysis and contributed to the writing of the manuscript and designed the research plan and organized the study.

M. Hamad: Coordinated the data-analysis and contributed to the writing of the manuscript and designed the research plan and organized the study.

Ethics

This article is original and contains unpublished material. The corresponding author confirms that all of the other authors have read and approved the manuscript and no ethical issues involved.

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