Journal of Hazardous Materials 186 (2011) 1141–1146

Contents lists available at ScienceDirect

Journal of Hazardous Materials

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

Process control, energy recovery and cost savings in acetic acid wastewater treatment

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ARTICLE INFO

Article history:
Received 6 July 2010
Received in revised form
11 November 2010
Accepted 27 November 2010
Available online 3 December 2010

Keywords:
Energy recovery
pH control
Anaerobic fixed bed loop reactor
Process control
Acetic acid
Neutralization

ABSTRACT

An anaerobic fixed bed loop (AFBL) reactor was applied for treatment of acetic acid (HAc) wastewater. Two pH process control concepts were investigated; auxostatic and chemostatic control. In the auxostatic pH control, feed pump is interrupted when pH falls below a certain pH value in the bioreactor, which results in reactor operation at maximum load. Chemostatic control assures alkaline conditions by setting a certain pH value in the influent, preventing initial reactor acidification. The AFBL reactor treated HAc wastewater at low hydraulic residence time (HRT) (10–12 h), performed at high space time loads (40–45 kg COD/m^3 d) and high space time yield (30–35 kg COD/m^3 d) to achieve high COD (Chemical Oxygen Demand) removal (80%). Material and cost savings were accomplished by utilizing the microbial potential for wastewater neutralization during anaerobic treatment along with application of favourable pH-auxostatic control. NaOH requirement for neutralization was reduced by 75% and HRT was increased up to 20 h. Energy was recovered by applying costless CO_2 contained in the biogas for neutralization of alkaline wastewater. Biogas was enriched in methane by 4 times. This actually brings in more energy profits, since biogas extra heating for CO_2 content during biogas combustion is minimized and usage of other acidifying agents is omitted.

1. Introduction

The anaerobic treatment of industrial wastewater has become increasingly important in recent years as a result of environmental-protection legislation, increased energy costs and problems with the disposal of excess sludge formed in aerobic treatment processes [1,2]. Acetic acid plays a crucial role for biogas formation in anaerobic digestion. Indeed, early tracer experiments have shown that about 70% of the methane produced in the overall process comes from the degradation of acetic acid [3].

Wastewater composition, temperature, volatile fatty acids (VFAs) and pH strongly affect anaerobic reactor performance by affecting degree of acidification and product formation [2,4,5]. Possibility of process failure is governed by organic loading rate and strength of wastewater [2]. The core problem of either start-up or thereafter efficient anaerobic digestion performance is achieving high space–time yield [5,6]. This is possible in practice if the biomass activity or concentration or both can be increased simultaneously, which can be achieved by effective methods of biomass retention and recycling [1,2,5,6].

Besides optimization techniques in anaerobic digestion, process control is also crucial for achieving high space–time yield [5]. Modern biosensors and controllers are needed for ensuring continuous optimal performance of anaerobic reactors [4,5,7–11]. Ince et al. adjusted space time load during start-up of an anaerobic digester by determining specific methanogenic activity [8]. Inhibitory effects of microbial activity were detected in the influent of anaerobic digesters by [7,10], whereas overloading control was investigated by [4,7,9]. VFAs and COD (Chemical Oxygen Demand) in two anaerobic fixed bed (AFB) reactors were used as the key control parameters [11]. However, pH was already found to be a more important factor than VFA concentration for fast recovery of anaerobic digesters after organic overloading [12]. In any case, an integrated approach of low capital and land area, cost savings and reliable highly efficient operation through efficient start-up and process control is required with regard to industrial anaerobic digestion applications [2,5].

The main wastewater stream in an acetaldehyde industry comes from different parts of the production steps and is characterized by high organic load and low pH, whereas the remaining wastewater streams are alkaline and of lower flow rate. These wastewater characteristics make anaerobic treatment of HAc wastewater very appealing, since acido- and aceto-genesis could be omitted and methane production can start immediately. In fact, the nature of acetic acid wastewater permits substrate removal and methane production rates close to theoretical values of complete volumetric

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doi:10.1016/j.jhazmat.2010.11.115

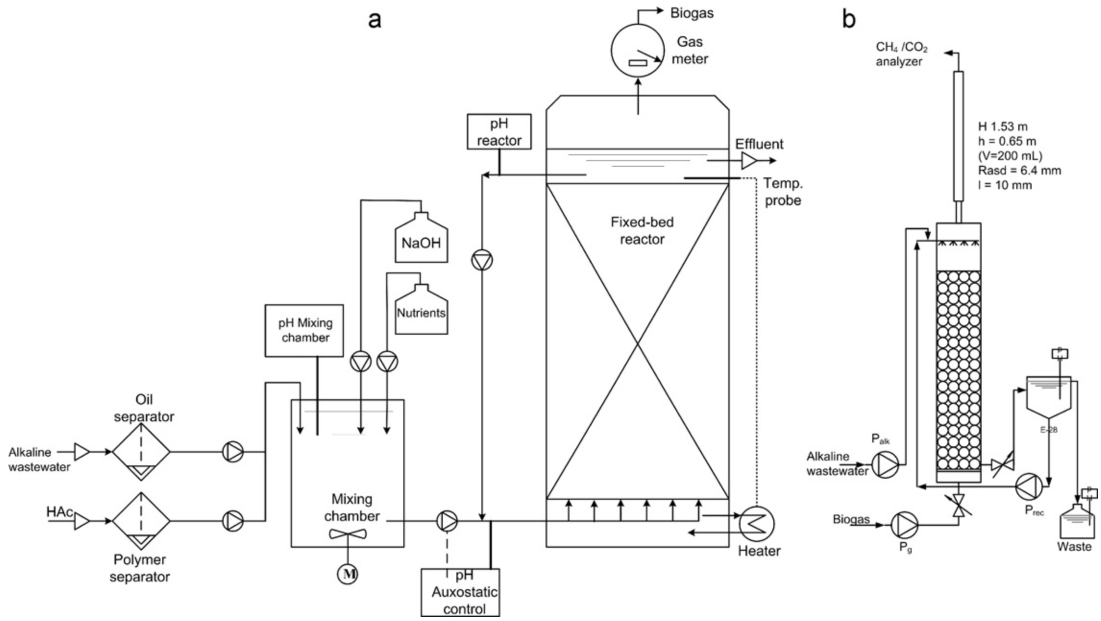

Fig. 1. Schematic diagram of the pilot scale plant for acetic acid wastewater treatment and scrubber for biogas treatment.

COD removal and maximum volumetric methane conversion [12]. However, reports on acetic acid wastewater treatment are scarce [2,12]. Herein, a pilot-scale anaerobic fixed bed loop reactor (Fig. 1) was applied to treat such a wastewater, while low-cost NaOH was added to neutralize wastewater. pH control was implemented by two automatic control concepts [4]. Objectives of this work include:

(1) The control of anaerobic digestion process aiming to achieve short and undisturbed start-up,
(2) The thereafter maintenance of the bioreactor's stabile operation and efficient performance,
(3) Material and cost savings via reducing alkaline agents for neutralization of HAc wastewater,
(4) Energy recovery by enrichment of biogas in methane.

2. Materials and methods

The pilot scale anaerobic fixed bed loop (AFBL) bioreactor that was applied for treatment of HAc wastewater is presented in Fig. 1. The reactor total volume was 1 m^3 with total height of 1.52 m and internal diameter of 0.46 m. Packed-bed took up a volume of 0.95 m^3, which was used to calculate HRT, space time load and volumetric biogas production. Open pored sintered glass in the form of Raschig rings were used as carriers of bacterial consortium for the fixed bed reactor. These glass rings of 25 mm × 25 mm size exhibit up to 70% porosity with 1.6–400 μm pore-size diameters. The colonization of macroporous carriers with high surface area, lowers the risk of biomass detachment, while at the same time substantially increases the amount of biomass that can be accumulated per unit volume [1,5,6]. The pore size of the carrier material permits the flow partially to penetrate the interior of the particles without also detaching the biomass. This has the further advantage that it facilitates the rapid removal of biogas as it is formed. Moreover, this type of bioreactor stands up to high loading rates, whereas requires low energy, capital cost and land [2]. Further information on the AFBL reactor configuration can be found in [13]. The anaerobic reactor was operated at a temperature of 37–39 °C (mesophilic), whereas pH was controlled by two new automatic designs. Effluent treated water and biogas exited from the top of the AFBL reactor. Recirculation takes place through an external loop from the top to the bottom of the reactor to ensure upward flow and completely mixed conditions in the reactor, which characterizes the reactor as AFBL. Biogas produced by the microbial activity is measured and analyzed by an infrared analyzer connected to the upper part of the AFBL reactor. Data of pH, temperature, biogas flow and composition are gathered and stored via a data interface in a computer for operator's use. A proportional pH controller is the one and only device that controls the process. The pH controller commences or ceases feeding of the reactor according to two alternative control patterns; auxostatic and chemostatic. The pH controller measures the influent pH and the pH of the bioreactor. The set-points for the feedback control patterns associated with residence time and temperature are introduced in the computer aiming to maximize the space–time yield. The amount of biogas generated and the pH serve as indication values with this form of control. Actual degradation can be determined on-line on the basis of the residence time and the extent of biogas production.

Synthetic wastewater was prepared to represent real wastewater of an acetaldehyde industry. The main wastewater stream to be treated is characterized by high organic load and low pH, which are attributed to its high concentration of acetic acid (HAc) by almost 100%. The remaining wastewater streams are alkaline and of lower volume. The influent wastewater to the bioreactor is a mixture of acetic acid wastewater and alkaline wastewater in ratio of 4:1, whereas a 45% NaOH solution is also added to adjust pH to a pre-selected value (7–7.3). The feeding solution consisted also of nutrients and trace elements, whose addition was calculated according to the growth requirements of *Methanosarcina barkeri* [14], a well known anaerobic methanogen, to achieve concentrations of 20–30 g COD/L, 72.6 mg N/L (nitrogen), 5.5 mg P/L (phosphorus) and 6.4 mg S/L (sulfur). The nutrients and trace elements solution consisted of NH_4Cl, Na_2SO_4, $(NH_4)_2Fe(SO_4)_2$, KCl, $MgSO_4{\cdot}7H_2O$, $NiCl_2{\cdot}6H_2O$, $ZnSO_4{\cdot}7H_2O$, $MnSO_4{\cdot}7H_2O$, $CuCl_2{\cdot}2H_2O$, $CoCl_2{\cdot}2H_2O$, $Na_2Mo_4{\cdot}2H_2O$, $Na_2SeO_3{\cdot}5H_2O$, KH_2PO_4 and H_3BO_3. The nutrients and trace elements solution exhibited a pH of 2–3 and was prepared in a concentrate of 2.5 L, which was then diluted per 1 m^3 of influent wastewater. There was no need to inoculate the AFBL, as biomass was already present in the filling material (carriers) of the reactor, which was used in a previous trial on anaerobic digestion of domestic wastewater [13]. After feeding, the microbial consortium adapted the synthetic wastewater and most probably the efficient microorganisms were selected.

Biogas that is produced in the anaerobic reactor is afterwards introduced to the gas scrubber in order to investigate reduction of CO_2 content by neutralization of alkaline wastewater. CO_2 content in the biogas is estimated between 40 and 50%, which in standard conditions of 1 atm and 20 °C ranges from 17.8 to 22.3 mol/m^3. This high quantity of CO_2 in the biogas increases operational cost through the extra heating needed for CO_2 quantity during biogas combustion. Alternatively, biogas CO_2 could be used for alkaline wastewater neutralization by interaction in the gas scrubber presented in Fig. 1. The gas scrubber of 1.53 m height is filled with Raschig rings, which take up a volume of 0.54 L reaching a height of 0.65 m (fixed bed height). These Raschig rings are open porous sintered glass with a diameter of 6.4 mm and length of 10 mm. The high porosity of the sintered glass rings facilitates neutralization of alkaline wastewater by biogas, since the two streams are introduced in opposite directions. Biogas is introduced from the bottom of the gas scrubber and moves upwards mixing with the downflowing alkaline wastewater, which is pumped in along with the recycle feed in the upper part of the gas scrubber as shown in Fig. 1. Gas scrubber effluent is pH monitored to assure neutralization from a pH of 10–12 down to 7, which would afterwards control (increase/decrease) the biogas influent flow rate in the gas scrubber's bottom. Biogas flow escaping from the upper part of the gas scrubber is measured and analyzed to monitor CO_2 in the treated biogas by an infrared CH_4/CO_2 analyzer.

3. Results and discussion

3.1. Process control

The control concepts [4] that are potentially applicable to anaerobic degradation can be illustrated in the case of acetic acid. To prevent acidification of the reactor due to insufficient substrate degradation, the substrate pump was controlled via a pH electrode in such a way that substrate feed is interrupted as soon as the pH falls below a certain value. With this pH-auxostatic mode of operation, wastewater feed is governed by a pH controller. The cleavage of acetic acid to CH_4 and CO_2, corresponds formally to a titration with an alkaline solution [4], so as the pH in the reactor rises, this effect must be compensated by the addition of acidic substrate. Use of a proportional controller means that there will always be a difference between the actual pH and the desired value, and the amount of substrate added is proportional to the magnitude of this difference. Since substrate introduction is in effect controlled by the degradation efficiency of the microorganisms, the microorganisms themselves determine how much wastewater will be fed to the reactor.

Seeding of the AFBL reactor was unnecessary and operation started with auxostatic pH control. Applying auxostatic pH control facilitated microbial community adaption into the anaerobic bioreactor, whereas high and stable performance in the pilot-scale unit was accomplished as soon as some process bottlenecks were surpassed. Plant monitoring begun after a short adaptation period of seven days, when initial HRT was 50 h (Fig. 2). Fig. 2 presents the follow-up of HRT, space time load and space time yield in the pilot-scale anaerobic bioreactor, whereas Fig. 3 illustrates COD removal by depicting COD concentrations of the influent acetic acid wastewater, of the influent mixture of acetic acid and alkaline wastewater and of its effluent for the same experimental time. The constantly low HRT is indicative of steady conditions and full adaptation of microbial activity (Fig. 2), which further implies that auxostatic pH control has reached its purpose. In addition, the increase in space time load and yield is also indicative of the reactor's capability to stand up to high loading rates and remove organic compounds efficiently. Polymers, a by-product of the ethanol oxidation to acetaldehyde, and oils were mixed with the feeding due to leakage through permeable points in pipe connections and pumps, and deteriorated plant's performance. Although it is not clear whether polymers and oil affected anaerobic population directly or blocked sites of the porous carriers, HRT was increased whereas space–time load and yield remained low. A polymer trap based on physical separation was introduced in the influent feed having as a result increase in space time load and yield. HRT dramatically decreased to 15–22 h. At this point the plant, receiving organic loading of 17–22 kg COD/m^3, exhibited 80–90% COD removal at a space–time load up to 32 kg COD/m^3 d and a space–time yield of 20–30 kg COD/m^3 d (Figs. 2–3). An oil trap was thereafter installed in the influent and resulted in further HRT decrease and stabilization. Plant was performing at a space–time load and yield of 40–45 kg COD/m^3 d and 30–35 kg COD/m^3 d respectively, whereas finally an HRT of 10–12 h was achieved. Under these conditions COD removal rate was recorded at 80% (Fig. 3). Disregarding technical interference to enhance space–time load and yield, effluent COD always remains lower than 5 kg COD/m^3 (Fig. 3), which could be further post-treated before disposal.

Alternatively to auxostatic control concept, an alkaline pH can be also assured in the reactor by establishing a suitable pH for the influent [4]. After the technical problems in this case were solved and a constantly stable operation (shown as low and constant HRT) was established in the pilot-scale AFBL bioreactor, the plant control was turned into the chemostatic pH control mode. The target pH in the reactor must be sufficiently alkaline so that, in the case of a change in residence time or concentration, a slight decrease in the pH can be associated with an increase in the activity of the microorganisms [4]. In our case, pH was maintained constant in

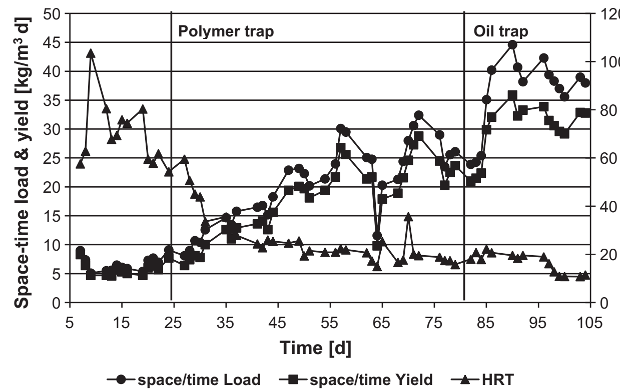

Fig. 2. Hydraulic residence time (HRT), space–time load and space–time yield in the pilot-scale anaerobic bioreactor.

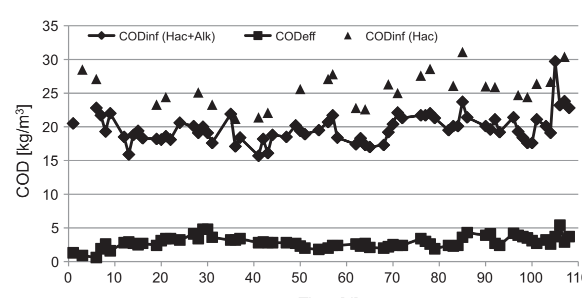

Fig. 3. Chemical Oxygen Demand of the influent acetic acid wastewater [CODinf(HAc)], of the influent mixture of acetic acid and alkaline wastewater [CODinf(HAc + Alk)], and the effluent [CODeff(HAc + Alk)] of the mixed wastewater during the initial 110 days of the pilot-scale anaerobic bioreactor operation.

the range of 7.0–7.3 (alkaline range) both in the AFBL bioreactor as well as in the influent flow rate. This chemostatic concept results in automatic stabilization so long as the operation is conducted on the alkaline side of the pH optimum with respect to the microorganisms. It is advantageous to safeguard the process by ensuring additional pH control on the "acidic side" of the pH optimum to prevent excessive drift into the acidic range. Optimum microbial activity was set at pH of 6.8.

The first approach of pH control has the advantage that the bioreactor always operates at maximum load, but it requires a buffer volume at the inlet to permit the controller to interrupt the substrate feed when needed. On the other hand, the second approach (chemostatic) excludes the possibility of continuous operation corresponding to maximum space–time yield. Nevertheless, it avoids the need for a buffer volume, and there is no risk of uncontrolled preacidification. Therefore, chemostatic control is suggested for full scale applications to avoid both construction cost of the storage tank and operation interruptions. However, in this design the bioreactor cannot be operated at the maximum space–time yield. In any case, both pH control schemes are simplified and effective in comparison to other techniques applied. Their main advantage is the simplicity and reliability of the pH sensor, whereas no further specialized measurements, such as specific methanogenic activity (SMA) [8] or VFA [10,11], are required. Auxostatic pH control permits space–time loads of up to 45 kg COD/m^3 d during reactor start-up, whereas other process control techniques allowed space–time loads of about 1 [8] to 14 kg COD/m^3 d [15].

Table 1 summarizes and compares performance of similar anaerobic reactors treating industrial wastewater. In comparison to the herein presented reactor configuration and wastewater type, there are very few reports on anaerobic fixed bed loop reactors and even fewer on acetic acid wastewater treatment. Acetic acid wastewater treatment has been reported in an anaerobic fixed film (AFF) reactor at HRTs of 0.4–5.7 d and space–time loads of 0.66–17.15 kg COD/m^3 d [12]. At an HRT of 12 h and space–time load of about 10 kg COD/m^3 d, COD removal was 57% and space–time yield was 6.56 kg COD/m^3 d. These authors found that the low accumulation of biomass in the biofilm reactor limited the range of the organic loading rates that could be applied to the reactor which further limits space–time yield. This bottleneck is surpassed in the herein AFBL reactor as biomass was efficiently accumulated in sintered glass porous material allowing the application of high space time organic load. Moreover, Hamoda and Kennedy observed reactor instability at HRTs lower than 2 days [12], whereas the herein AFBL reactor performed most efficiently at HRTs of 10–12 h. However, this downflow AFF reactor could recover fast after organic overloading incidents [12]. High values of space–time load and yield were reported in an upflow anaerobic fixed film loop (AFFL) reactor [15], which, however, treated another type of industrial wastewater. It is well known that wastewater origin and composition strongly affect performance of the wastewater treatment plant. There are reports on AFB reactors, which are quite similar to the one presented herein, because they are both filled with porous sinter glass for biomass immobilization and retention, but both treat different types of industrial wastewater. The one, which treated distillery mash, achieved a high space time yield of 27 kg COD/m^3 d at an HRT of 17 h and a high space–time load of 45 kg COD/m^3 d [6]. The other one treated citric acid wastewater, which, however, consisted also of high concentration of acetic, propionic and butyric acid [16]. This AFB reactor was operated at an HRT of 9 d and space time load of 2.74 kg COD/m^3 d and achieved space time yield of 1.88 kg COD/m^3 d and 63% COD removal. Both AFB reactors treated another type of industrial wastewater rather than acetic acid and resulted to lower space–time yields at higher HRTs than the ones presented in our case. In comparison, Figs. 2 and 3 depict that the presented AFBL reactor can treat acetic acid wastewater at the lowest HRTs (10–12 h), perform at high space–time loads (40–45 kg COD/m^3 d) and results in the highest space–time yield (30–35 kg COD/m^3 d) and COD removal (80%) without operational disturbances. In this case, space–time load and yield are considered very high, which, in agreement to previous findings [2], is related to a high COD removal and biogas production efficiency at very low HRT.

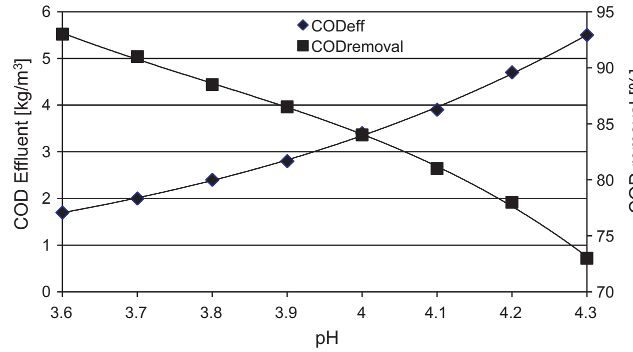

Fig. 4. HRT and alkali consumption in dependence on influent pH.

3.2. Energy and material recovery

The concept of energy recovery and material savings relies on the utilization of both the wastewater characteristics and microbial activity. The mixture of wastewater streams was treated anaerobically and the treated effluent, as shown in Fig. 3, exhibits a COD of about 5 kg COD/m^3. The pH of the influent wastewater mixture was increased to 4.3 by addition of sodium hydroxide (NaOH). This pH increase resulted in NaOH consumption and cost increase. The idea of reducing NaOH requirement utilizing the microbial potential for wastewater neutralization during anaerobic treatment was investigated along with application of favourable pH-auxostatic regulation [4]. With regard to the rest wastewater streams, which are alkaline, neutralization with an acidifying agent should also occur before disposal. An alternative could be the utilization of these alkaline streams for enrichment of biogas in methane (CH_4).

Reduction of NaOH addition for neutralization of the acetic acid wastewater was investigated in the AFBL reactor during the auxostatic pH control in relation to its effects on HRT (Fig. 4). Reduction of NaOH addition resulted to a step reduction of influent pH from 4.3 to 3.6, as shown in Fig. 4. Adjustment of wastewater pH to 4.3 required about 12 kg/m^3 of a 45% NaOH solution, whereas HRT needed for treatment in the AFBL reactor was quite short

Table 1
Literature data comparison of anaerobic fixed film (AFF) or anaerobic fixed bed (AFB) digesters treating acidic or acetic acid wastewater.

Reference	Feed	Reactor	HRT (h)	Space/time load (kg COD/m^3 d)	COD removal (%)	Space/time yield (kg COD/m^3 d)
This work	Acetic acid wastewater	AFB	10–12	40–45	80	30–35
[12]	Acetic acid wastewater	AFF	12	10.64	57	6.56
[16]	Citric acid wastewater	AFB	216	2.7	63	1.88
[6]	Distillery mash	AFB	17	45	60	27
[15]	Acidic whey	AFF	5	14	95	16

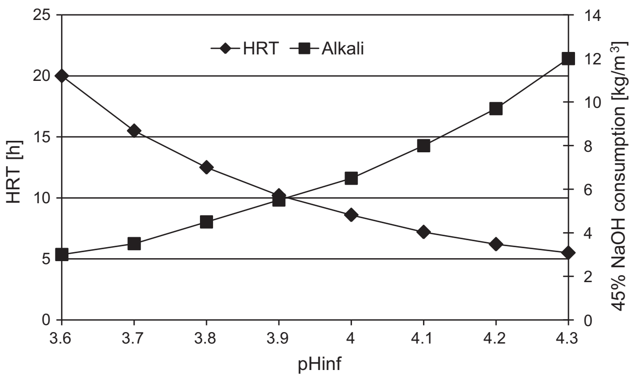

Fig. 5. COD removal and COD effluent versus pH.

Table 2
pH values of neutralized alkaline wastewater and volume of alkaline wastewater neutralized by 1 m^3 of biogas.

Experiment day	Effluent pH	(Neutralized alkaline wastewater) L/(biogas) m^3
147	7.2	244
147	7.3	242
148	7.4	214
153	7.2	260
153	7.3	250
155	7.8	263
155	7.2	294
155	7.3	282
161	7.5	293

at 5 h. Further decrease of pH down to 3.6 reduced NaOH consumption by 75%, whereas HRT increased from 5 to 20 h (Fig. 4). Omitting dilution effects due to, wastewater strength was about 23 kg COD/m^3. Further results of reduction in NaOH requirements are depicted in Fig. 5. COD effluent concentration decreased from 5.5 to 1.7 kg COD/m^3 and COD removal increased from 73 to 93%. Thereafter, the plant operator is facilitated to select the pH of the influent wastewater in relation to the cost and material savings as well as to the applied HRT for efficient treatment.

The utilization of the CO_2 in the biogas for neutralizing the alkaline wastewater stream was also investigated. Fig. 1 depicts the flow chart of the biogas scrubber that was applied for biogas treatment. Results show that 1 m^3 of biogas can neutralize 215–290 L of the alkaline wastewater (Table 2). At the same time, biogas was enriched in methane by 4 times, since biogas content altered from about 57% CH_4 and 43% CO_2 in the influent to 79–89% CH_4 and 14–21% CO_2 in the effluent of the scrubber (Table 3). As a result, CO_2 in the biogas can be used as a cheap acidifying agent for decreasing pH of alkaline wastewaters, replacing the conventional used acids, e.g. NaOH. Thereafter, biogas is enriched in methane, since

Table 3
Indicative volume values of scrubber emissions and CH_4/CO_2 ratio in biogas and scrubber emissions.

Experiment day	Scrubber emission content		CH_4/CO_2 ratio	
	CH_4 (%)	CO_2 (%)	Biogas	Scrubber degas
156	81	19	1.39	4.2
	85	15		5.7
	86	14		6.1
162	89	17	1.26	4.9
	80	20		4.0
	79	21		3.8

CO_2 is removed by neutralization. This enrichment would actually bring in more energy profits, since biogas extra heating of CO_2 content during biogas combustion is minimized. In comparison to these results, biogas CO_2 was also applied efficiently as an acidifying agent of alkaline brewery wastewater prior to anaerobic treatment by an UASB reactor [17]. In this case, 4600–5200 L of the brewery wastewater was neutralized by 1 m^3 of biogas, whereas biogas content in CO_2 reduced from 35% to 20% after neutralization down to 7.3. Biogas was produced either by beer fermentation or by anaerobic digestion of the brewery wastewater.

4. Conclusions

Operation control, performance optimization and transformation of cost to profit are bottlenecks and aims of all scale applications in wastewater treatment. On this basis, an anaerobic fixed-bed loop (AFBL) bioreactor was used for treatment of acetic acid.

- The AFBL bioreactor process was controlled by pH-auxostatic and chemostatic regulation. Auxostatic pH control facilitated bioreactor operation at maximum load, but it requires a buffer volume. On the other hand, chemostatic pH control avoids the need for a buffer volume and there is no risk of uncontrolled preacidification. Therefore, chemostatic control is suggested for full scale applications to avoid both construction cost of the storage tank and operation interruptions.
- Performance results clearly show that AFBL reactor can treat acetic acid wastewater at low HRTs (10–12 h), perform at high space–time loads (40–45 kg COD/m^3 d) and high space–time yield (30–35 kg COD/m^3 d) to achieve high COD removal (80%) without any operational disturbances.
- Material and cost savings were accomplished by utilizing the microbial potential for wastewater neutralization during anaerobic treatment along with application of favourable pH-auxostatic regulation. In this case, NaOH requirement for neutralization was reduced by 75%, but HRT was increased from 5 to 20 h.
- CO_2 in the biogas was used as no cost source for pH neutralization of alkaline wastewater in place of other acidifying agents and thus, their usage is partly or totally omitted.
- At the same time, biogas was enriched in methane by 4 times, since CO_2 was removed by neutralization. This enrichment actually brings in more energy profits, since biogas extra heating of CO_2 content during biogas combustion is minimized.

Definitions

Space–time load = organic loading rate (kg COD_{fed}/m^3 d).
Space–time yield = volumetric reaction rate (kg $COD_{removed}/m^3$ d).

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