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Treatment of Molasses-Based Distillery Wastewater in a Pilot-Scale Anaerobic Sequencing Batch Reactor (ASBR)

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Research Article

Abstract

High-strength distillery wastewater, also called stillage or spent wash, contains high concentrations of organic and inorganic pollutants. Considering the standard allowable limit for effluent discharge, pollutants must be reduced from distillery wastewater before being released into the watercourse. This study examined the use of anaerobic sequencing batch reactor (ASBR) in a pilot-scale treatment of molasses-based distillery wastewater. ASBR was designed and fabricated using a 1,000 L capacity plastic container. The mixture consisted of 65% inoculum and 35% diluted wastewater. Influent was diluted at a proportion of 1 part wastewater and 2 parts water due to the high COD concentration of raw distillery wastewater. The inoculum was anaerobically-treated sludge obtained from an existing wastewater treatment facility. ASBR trials were done with varying reaction (stirring) durations, i.e., 8, 24 and 48 h operated for 24, 40 and 64 h cycle, respectively. Two trials were done per operating conditions. Highest COD (60%) and BOD (86%) reductions were observed in ASBR operation with 8 h react time. Biogas production in ASBR with 8 h react time also had the highest methane content (72%) compared to those with 24 h and 48 h reaction periods having 66% and 50% methane, respectively. Sludge by-product after ASBR operations had 0.91 to 1.02% total NPK (nitrogen, phosphorus, potassium), which can be utilized as soil ameliorant. Post-treatment of ASBR-treated effluent is necessary to attain the allowable discharge limit.

Keywords: Anaerobic sequencing batch reactor; ASBR; distillery wastewater; Biogas.

1. Introduction

Distillery wastewater constitutes one of the greatest concerns of the alcohol production sector. Effluents from molasses-based distilleries mainly contain dark colored molasses spent wash (MSW). MSW is a polluting waste product because of its low pH, high temperature, dark color, high ash content and high percentage of dissolved organic and inorganic matter [1]. The primary sources of pollutants in a distillery plant are the stillage, cooling water from fermenter and condenser, and wastewater from fermenter [2]. For the past years, the search for a sustainable treatment system capable of minimizing energy consumption has encouraged the use of anaerobic biological systems. Anaerobic treatment is an attractive primary treatment option due to its high BOD removal (more than 80%) and energy recovery in the form of biogas [3].

Among the new, high-rate anaerobic plant systems, Anaerobic Sequencing Batch Reactor (ASBR) has been proposed as an alternative to continuous system. ASBR could represent an economical, stable, efficient, easy-to-use process to treat and recover usable energy from industrial & food processing wastewater [4-6]. This technology has been successfully applied in laboratory and semi-commercial scales for treatment of various wastewaters from swine farms, meat industry, dairy industry, olive mill plant, winery and distillery plants and abattoir/slaughterhouse [4-13].

Generally, an ASBR operates in a single reactor vessel. The reactor cycles through four phases of operation, which allows for a high throughput of materials while retaining microorganisms in the reactor. A typical cycle of an ASBR consists of feeding or loading, reaction, settling, and withdrawal [4,6]. During the reaction step, mixing is typically accomplished through biogas circulation, liquid-phase circulation, or mechanical agitation. The reactor content is mixed to allow close contact between organics and microorganisms. Slow agitation avoids disrupting the formation of bacterial flocs. The length of the reaction period depends on substrate characteristics and effluent quality requirements. For wastewater containing high suspended solid concentrations, more contact time between bacteria and substrate is required to complete the hydrolysis of particulates.

When gas production rate decreased to a minimum, reactor content is allowed to settle. Major advantages of ASBR include low capital and operating costs, and minimum daily maintenance. ASBR also allows batch and semi-continuous or intermittent feeding [5].

In the Philippines, no report has been published yet on the application of ASBR for distillery wastewater treatment. Laboratory-scale ASBR treatment of meat processing wastewater was done previously [14]. Reductions of 94% COD and 93% BOD in ASBR-treated effluents were observed after 16 h reaction. Biogas production was 2.7 L per day (L d^{-1}) with about 61% methane content [14]. In the current study, pilot-scale ASBR was applied for treatment of molasses-based distillery wastewater to examine its capability for biogas production and in reducing the organic loads of wastewater.

2. Methods

2.1. Design and fabrication of pilot-scale ASBR

Figure 1 shows the schematic diagram of the pilot-scale ASBR treatment system. The fabricated ASBR and gas holders are shown in Figure 2. The reactor was fabricated using a 1000 L Intermediate Bulk Container (IBC). Polyvinyl chloride (PVC) pipe and ball valve were attached at the top of the container as inlet port of raw wastewater. Mixing of wastewater and sludge inoculum was carried out using a stainless steel paddle (3” × 14”) connected to a 1” stainless shaft. The shaft is connected to a 1 hp gear motor (Fuji Electric FA, Japan; 3 phase) with a ratio of 1: 20. The reactor had an outlet port (2” PVC pipe with ball valve) to draw off treated effluent. A sludge drain port is located at the base of the reactor. Gas outlet is connected to a 145 L capacity gasholder by flexible hose. The design of the gas holder was patterned to the ITDI portable biogas holder [15].

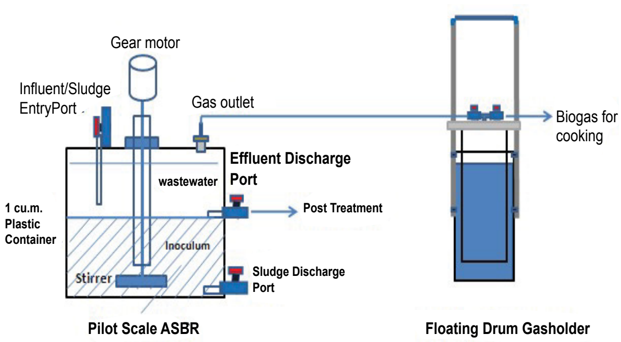

Figure 1. Schematic diagram of the pilot-scale anaerobic sequencing batch reactor (ASBR) system.

Figure 2. Fabricated ASBR and biogas holders. Capacity: ASBR, 1,000 L; gas holders, 245 L.

2.2 Inoculum acclimatization

Inoculum was obtained from an existing anaerobic wastewater treatment plant of a Brewery Plant (Asia Brewery Inc., Sta Rosa, Laguna). The inoculum was blackish, slurry form, with 6.44 pH, 8.24% total solids and 63,000 mg per liter (mg L^{-1}) COD. The inoculum was acclimatized in the target substrate, molasses-based distillery wastewater, collected from a distilling company (Destileria Limtuaco & Co., Inc., Quezon City, Metro Manila). Distillery wastewater had a pH of 4.58, total solids of 15.25% and COD of 171,000 mg L^{-1}. The characteristics of pure distillery wastewater and inoculum are presented in Table 1. The initial mixture used during inoculum acclimatization composed of 50% inoculum and 50% diluted wastewater. Wastewater was diluted at a ratio of 1 part wastewater and 3 parts water due to its high COD content compared to that of inoculum. pH of diluted wastewater was adjusted from pH 4.3 to pH 8.5 by sodium hydroxide (NaOH) solution. The mixture of inoculum-diluted wastewater had a pH of 7.2. ASBR was operated with 8 h (react time) gentle stirring at 80 revolutions per minute (rpm). Total ASBR cycle was 24 h, which consisted of the following phases [4]: 0.5 h refilling, 8 h reaction, 15 h (overnight) settling, and 0.5 h decanting of treated effluent.

Table 1. Characterization of raw distillery wastewater and inoculum.

Parameters	Distillery wastewater	Inoculum
Color	dark brown	black
Odor	burnt sugar	foul odor
pH	4.58 ± 0.00	6.44 ± 0.00
COD (mg L^{-1})	171,000 ± 1.53	63,000 ± 1.52
BOD (mg L^{-1})	42,000 ± 5.66	26,000 ± 2.12
Total solids (%)	15.25 ± 0.01	8.24 ± 0.24
Total volatile solids (%)	81.68 ± 0.61	56.81 ± 0.19
Ash (%)	18.32 ± 0.61	43.19 ± 0.19
Total suspended solids (mg L^{-1})	7,200 ± 0.58	87,850 ± 5.29
Volatile suspended solids (mg L^{-1})	7,000 ± 0.01	53,700 ± 0.42

Biogas production was daily monitored to determine the activity of the inoculum. Water displacement technique was used to quantify the amount of biogas produced. Volume of gas was determined by the volume of water displaced from the reactor into the gas holder as a result of gas pressure build-up inside the reactor [16]. Flame test was conducted for qualitative analysis of the biogas. Methane content is more than 50% when biogas is flammable [17]. Acclimatization of inoculum is complete when sustained biogas production is achieved and COD removal is about 70% for six continuous days [7,18]. Effluent COD and pH were determined after inoculum acclimatization.

2.3. ASBR treatment operations

Initial mixture for anaerobic digestion consisted of 35% diluted wastewater and 65% inoculum. The total volume of mixture was 1,000 L. Distillery wastewater was diluted with water at a ratio of 1 part wastewater to 2 parts tap water. pH of diluted wastewater was adjusted between 8.0 to 9.0 by NaOH solution (40%) prior to loading into the digester.

ASBR treatment of distillery wastewater was done with varying reaction (stirring) durations - 8 h, 24 h and 48 h and operated for 24 h, 40 h and 64 h cycles, respectively. Two trials per treatment cycle were done with the same duration for filling, settling and decanting (Table 2). The mixture was stirred at low speed (80 rpm) to allow contact of microorganisms with the substrate. Batch ASBR operation was done at ambient temperature. ASBR operation with 8 h reaction lasted for 7 days. Biogas production was monitored daily. Methane content was analyzed using a gas chromatograph (GC-2014 NGAS, Shimadzu, Japan) with thermal conductivity detector and capillary columns. Biogas was ignited and burned to dispose collected gas. pH, COD, BOD, total solids (TS), and total volatile solids (TVS) were analyzed in the influents and effluents. Sludge by-products after ASBR operations were collected and characterized for nutrient and organic matter content.

Table 2. ASBR conditions with different reaction durations

Phases (h)	ASBR Cycle		
	24 h	40 h	64 h
Filling	0.5	0.5	0.5
Reaction (Stirring)	8	24	48
Settling	15	15	15
Decanting	0.5	0.5	0.5

2.4. Analytical procedures

COD was analyzed using HACH DR/870 Colorimeter (USA). BOD was determined using Oxi-Top system (WTW, Germany) after five days of incubation. The pH of wastewater was checked by a portable pH meter (pH 600, Milwaukee, Romania), and temperature at the working area was monitored by a digital thermometer (AZ-MT-09). Total solids, total suspended solids, total volatile solids, and ash were determined following the APHA method [19]. Sludge samples were submitted to the Philippine Coconut Authority (Quezon City, Metro Manila) for analysis of nitrogen, phosphorus, potassium, organic matter, and organic carbon.

2.5. Data analyses

The mean percent reductions in concentration of COD and BOD were computed based on the differences between influent and effluent mass concentrations [18]. Means and standard deviations were used in data comparison.

3. Results and Discussion

3.1. Inoculum acclimatization

Inoculum acclimatization was done for 20 days. Acclimatization allows the bacteria to adapt to the new feed material, distillery wastewater. Sustained biogas production was observed from day 1 to 20. Total biogas production was 1,732 L with daily biogas production of 87 L day^{-1}. COD reduction in effluent (pH 7.4) was 77% after 20 days. Effluent COD decreased to 8,800 mg L^{-1} from the initial COD of 38,617 mg L^{-1}. Sustained biogas production and high COD reduction suggest that the collected inoculum was able to adapt to the new substrate and can efficiently treat diluted molasses-based distillery wastewater. Acclimatized inoculum was used in the succeeding ASBR operations.

3.2. ASBR treatment operations

Characteristics of substrate

Pure distillery wastewater was acidic (pH 4.34 to 4.4), with high COD (163,800 to 194,066 mg L^{-1}) and BOD (30,000 to 51,000 mg L^{-1}), total solids of 14.83 to 17.41%, and total volatile solids of 76.30 to 80.51%. A report in literature noted that the BOD and COD of molasses spent wash typically range between 35,000 to 50,000 and 100,000 to 150,000 mg L^{-1}, respectively [20]. Table 3 shows the characteristics of diluted distillery wastewater used as influent for ASBR operations. Inoculum used during the operations had the following characteristics: 7.54 to 7.86 pH, 84,700 to 91,579 mg L^{-1} COD, 11.50 to 12.22% total solids, and 51.2 to 52.42% total volatile solids.

Biogas production

Biogas produced in all three operations was flammable. Highest biogas production was observed in ASBR operation stirred for 48 h (1,454 L day^{-1}). However, methane content of biogas was not correlated with increasing reaction time. Biogas with highest methane content (72%) was observed in ASBR operation with 8 h react time while lowest methane content (50%) was noted in ASBR stirred for 48 h (Table 4). Methane yield was 78 mL g^{-1} VS destroyed in ASBR operated with 8 h reaction time

Table 3. Characteristics of diluted distillery wastewater used during ASBR treatment.

Parameters	ASBR reaction time		
	8 h	24 h	48 h
pH	4.35 ± 0.03	4.42 ± 0.05	4.42 ± 0.03
COD (mg L^{-1})	47,933 ± 7.57	56,800 ± 9.90	48,516 ± 7.78
BOD (mg L^{-1})	17,000 ± 0.70	14,750 ± 0.07	11,750 ±0.35
Total solids (%)	5.19 ± 0.08	5.22 ± 0.97	4.56 ± 0.14
Total volatile solids (%)	80.53 ± 0.25	76.32 ± 0.71	78.04 ± 0.36

Table 4. Biogas production and composition during ASBR operations.
*Data of ASBR with 8 h reaction time was based on the 7[th] day operation

	ASBR reaction durations		
	8 h*	24 h	48 h
Total biogas (L)	2,608	1,305	2,909
Daily biogas (L day^{-1})	395	783	1,454
Biogas Composition			
-Methane (%)	72.0	65.6	50
-Carbon dioxide (%)	28.0	10.6	10
-Nitrogen (%)	0	23.5	39.3
-Hydrogen sulfide (%)	0	0.3	0.7
Methane yield/VS destroyed (mL g^{-1})	78.36	39.63	59.93

whereas that with 48 h reaction time was about 60 mL g^{-1} VS destroyed. For ASBR cycle with 8 h react time, highest biogas production was observed between days 1 to 4 of operation. Based from the composition of biogas (Table 4), increasing the reaction duration to 24 h and 48 h in ASBR tends to stimulate nitrogen production.

Methane content observed in this study was in accordance with other report using distillery wastewater. In literature, methane content in biogas produced during ASBR operation was 40-60% in food processing wastewater, 50-80% in distillery wastewater, and 75% in slaughterhouse wastewater [5,11,21]. Previous study using lab-scale ASBR, with meat processing wastewater as substrate, showed average methane content of 61% [14].

COD and BOD reduction

Reduction in effluents COD was not proportional with increasing ASBR reaction time. Highest COD reduction of about 60% was observed in ASBR operation with 8 h reaction time while 41% and 39% COD reductions were observed in those with 24 h and 48 h reactions, respectively (Figure 3). BOD removal was also highest in ASBR with 8 h reaction time. After 7 days operation with 8 h stirring per day, 86% BOD reduction was observed (Figure 4). For ASBR operations with 24 h and 48 h reaction time,

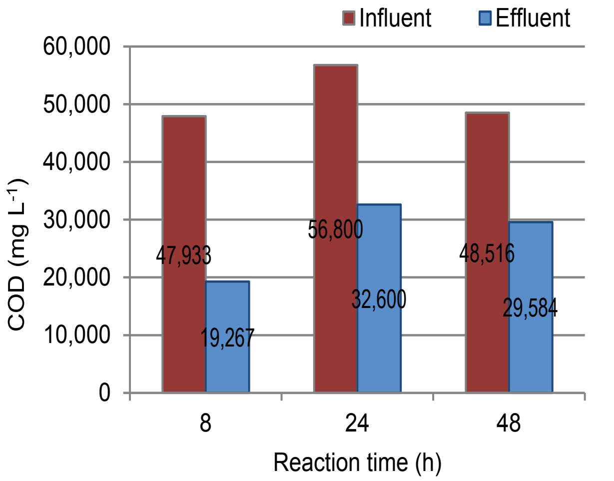

Figure 3. COD values in influent and effluent after ASBR operation with different reaction time. Data in ASBR with 8 h reaction time was based on 7[th] day operation. Data are means from two trials.

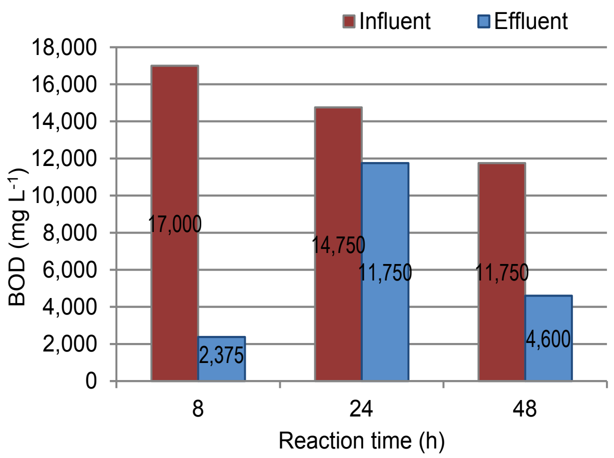

Figure 4. BOD values in influent and effluent after ASBR operation with 8 h, 24 h, and 48 h reaction time. Data in ASBR with 8 h reaction time was based on 7[th] day operation. Data are means from two trials.

about 20% and 61% BOD reductions were observed, respectively. Effluents after ASBR operations had total solids of 3.62 to 4.03% and pH of 7.16 to 7.68. Allowable pH of effluent based on Philippine standard is 6 to 9 [22].

Considering the high COD (60%) and BOD (86%) reductions in ASBR operation with 8 h react time, this condition is recommended for full-scale distillery wastewater treatment. Current data confirm the reported efficiency of anaerobic technique as a good primary treatment of wastewater due to its high BOD removal and energy recovery [3]. In other studies on treatment of low- to medium-strength wastewater by ASBR, greater than 90% organic load removal was observed such as those in winery wastewater (98%), dairy plant wastewater (91%) and meat processing wastewater (93%) [9,12,14].

One advantage of ASBR is the ease to control effluent quality since withdrawal can be done when desired effluent levels are attained [6]. As noted in literature, the length of reaction period depends on substrate characteristics and effluent quality requirements. For wastewaters containing high suspended solids concentrations, more contact time between bacteria and substrate is required for a much complete degradation of organic matter [5,18]. In the present study, increasing the react time to 24 h and 48 h continuous stirring resulted to lower COD and BOD reductions. These long reaction durations seemed to affect the methanogenic activity during ASBR treatment. Less than 24 h react period (e.g. 12 h, 16 h) can be tried to increase the organic load removal. In a previous study using ASBR for meat processing wastewater, 16 h react time yielded the highest organic load removal as compared to operations with 8 h and 12 h reaction durations [14]. Aside from the flexible reaction time, ASBR also allows batch and semi-continuous or intermittent feeding [5]. Batch operation in ASBR allows good effluent quality control and this feature is appropriate for industries that are operating seasonally [11].

Sludge by-product

The characteristics of sludge by-product after anaerobic digestion are shown in Table 5. Total NPK (nitrogen, phosphorus and potassium) of sludge ranged from 0.91 to 1.02%, which is classified as soil ameliorant. Ameliorant refers to substance with total NPK of less than 2.5% and aids in plant growth by improving the physical condition of the soil [23]. Samples with 2.5 to <5% NPK is considered as compost or soil conditioner, while those with 5 to 7% NPK is considered as organic fertilizer [23].

Table 5. Characteristics of sludge by-product after anaerobic digestion operations.

Parameters	Range
Total Nitrogen (%)	0.303-0.426
Total Phosphorus (%)	0.107-0.226
Total Potassium (%)	0.342-0.492
Total NPK (%)	0.906-1.021
Organic Carbon (%)	1.227-1.314
Organic Matter (%)	5.012-5.921
pH	7.6-8.0
Color	black
Odor	none

In previous report using ASBR treating meat processing wastewater [14], sludge by-product had 1.40% organic carbon with lower level of NPK (0.13%) mainly due to the low organic load of the substrate. In the same way, the sludge by-product can be used as soil ameliorant.

Design of full-scale wastewater treatment facility

A design of full-scale distillery wastewater treatment plant was prepared based on the results of pilot-scale study. Current study showed that ASBR technique (with 8 h react time) reduced the COD and BOD of molasses-based distillery wastewater to 60% and 86%, respectively. As expected, the values of COD and BOD of anaerobically-treated effluent are still very high. Therefore, ASBR will serve as the primary treatment and a post-treatment process is necessary for further degradation of organics and to meet the required effluent standard limit. As noted in literature, effluents from anaerobic treatment are often not suitable for direct discharge into receiving waters without further treatment scheme [24]. Molasses spent wash is normally treated by anaerobic digestion followed by aerobic treatment using trickling filter or activated sludge [20]. Resulting effluent after this conventional treatment system needs further processing for the dark colored wastewater still contains compound that maybe toxic to microorganisms [20]. Report of Masse and Masse mentioned that aerobic systems can be used for final purification and nutrient removal after the anaerobic treatment [25]. For the proposed design, modifications will be done on the existing wastewater treatment plant of the cooperator. The existing closed tank was re-designed to work as an ASBR (Figure 5). Aside from the modifications, gas holder tank will be constructed for collection of gas.

The proposed treatment system (Figure 5) is composed of the following: 1) cooling-dilution-neutralization tanks, 2) anaerobic digester (ASBR) and gas holder, 3) covered lagoons (anaerobic), and 4) sand-gravel filter (aerobic). The ASBR has a capacity of 90 m^3 with a biogas holder of 36 m^3. The two covered lagoons, with capacities of 360 m^3 and 400 m^3, will serve as secondary treatment. The remaining open lagoon will be filled with sand and gravel for post-treatment.

Cooling-dilution-neutralization tanks

The two existing cooling tanks will be used to reduce the temperature of the hot distillery wastewater coming out of the fermentation chambers. Capacities of the cooling tanks were 13 m^3 and 15 m^3. These two cooling tanks will serve as the dilution and neutralization tanks of the wastewater treatment plant. Based from the pilot study, the distillery wastewater needs to be diluted to a ratio of 1:2 with 1 part distillery wastewater to two parts water. In addition, the diluted wastewater is still acidic (pH 5.5) and needs to be pH adjusted. A one horse power circulating pump will be installed between the two tanks to mix the

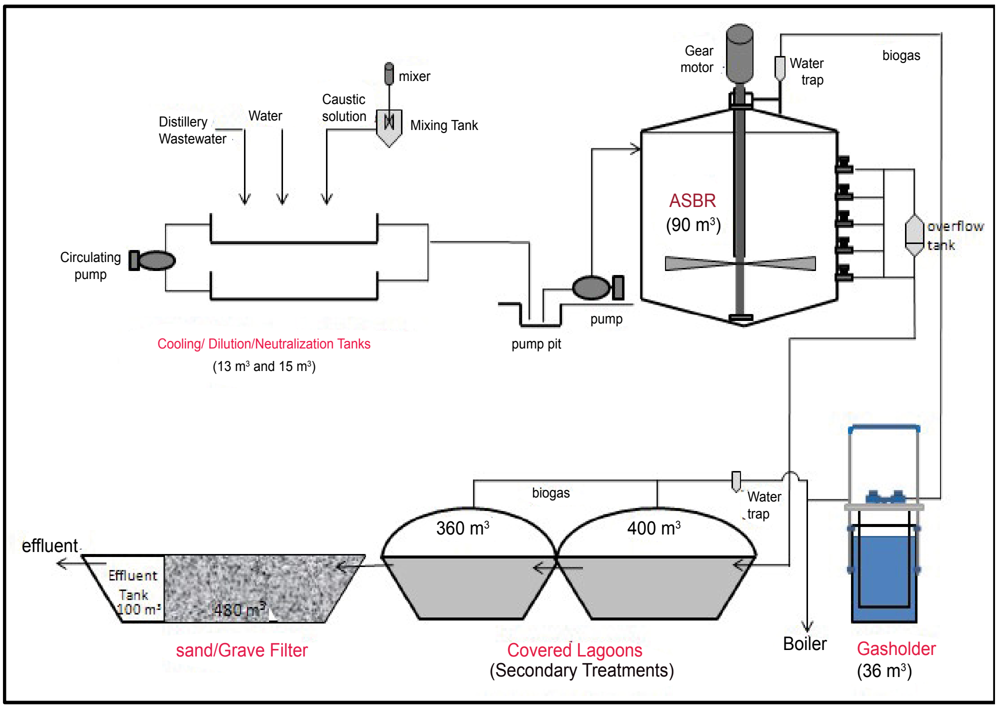

Figure 5. Design of full-scale distillery wastewater treatment plant.

wastewater. A 200 L blue plastic drum (high density polyethylene or HDPE) can be used as mixing tank of sodium hydroxide (NaOH) and water. Mixing can be done either manually or mechanically.

Anaerobic sequencing batch reactor (ASBR) and gas holder

The existing anaerobic digester was re-designed to work as a full-scale ASBR. A gear motor assembly will be installed at the top of the anaerobic digester. The gear motor has a ratio of 1:20 with a speed of about 85-90 rpm. The gear motor assembly includes a stainless shaft and paddle. An oil seal will be installed so that the ASBR will be gas tight. Plunge bearings will be attached to strengthen the hold of the shaft to eliminate swiveling. Solenoid valves will be placed to one side of the anaerobic digester for effluent discharge and sampling ports. Drain ports will be installed at the bottom of the digester for sludge withdrawal and for general cleaning.

A gas holder will be constructed based on the floating gas holder-type design with a holding capacity of about 36 m^3 day^{-1} of biogas. Construction materials can be metal or a combination of concrete and metal.

Covered lagoons

There are three existing open concrete lagoons (Figure 5). Two of these lagoons will be covered with a plastic sheet (HDPE) and will serve as anaerobic digesters. Based on the pilot-scale study, COD and BOD values of the effluents after ASBR were still very high and therefore need further treatment. Covered lagoons are good secondary wastewater treatment and additional biogas can be collected. Capacities of the two lagoons were 400 m^3 and 360 m^3. The two lagoons will have a total hydraulic retention time (HRT) of about 32 days.

Sand-gravel filter

One of the remaining open lagoons will be used as sand-gravel filter. A layer of gravel and sand will be placed at alternating levels. A wall near the effluent discharge will be constructed for effluent tank. This tank will serve as sampling point for effluent analysis. The total capacity of the third lagoon is 580 m^3. This will be divided into sand-gravel filter (480 m^3) and effluent tank (100 m^3).

4. Conclusion

Molasses-based distillery wastewater containing 163,800 to 194,066 mg L^{-1} COD was used as substrate in ASBR operation. Pilot-scale anaerobic digester was fabricated and used successfully in the treatment of diluted distillery wastewater. Highest COD (60%) and BOD (86%) reductions in treated effluents were observed in ASBR operation with 8 h react time. Biogas produced in all operations was flammable. Highest methane content (72%) in biogas was noted in ASBR operation with 8 h react time. Sludge by-product after ASBR operation had 7.6 to 8.0 pH, and with 0.91 to 1.02% total NPK that can be utilized as soil ameliorant.

Based from the results, long reaction time (24 h and 48 h) in ASBR operations are not efficient in reducing the organic load (COD and BOD) of molasses-based distillery wastewater. ASBR run with 8 h react time is recommended for the full-scale operation as this condition resulted to highest COD & BOD reductions, as well as highest methane content. Post-treatment of ASBR-treated effluent is recommended to achieve the allowable discharge limit.

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