Anaerobic digestion of heat treated sludge liquor by pilot scale mesophilic EGSB reactor

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Abstract: Biogas recovery from heat treated sludge liquor was tested by a 4 m^3 mesophilic expanded granular sludge bed (EGSB) reactor within 206 days. Before pilot experiment, the heat pretreatment effects on hydrolysis and biogas production potential on three sludges from different wastewater plants were evaluated using bench test. One of these sludges was used in the subsequent pilot heat treatment and EGSB reactor. The biogas conversion potential of this sludge filtrate was 79%. Seeded with granular sludge, the EGSB reactor could be quickly activated and adapted to the new filtrate substrate. The EGSB influent COD was 20.0-35.0 g/L, and the effluent COD was lower than 10.0 g/L. The COD removal rate was 60%-70% at an organic loading rate (OLR) of 10.0 kg COD/(m^3 d). The fresh supernatant with low pH resulted in the high biogas production in EGSB. The effluent with high nitrogen and phosphor should be considered for further treatment.

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1 Introduction

The sewage sludges produced from wastewater treatment plants are poor handling waste, representing a considerable pollution threat for the environment. In China, increasing sludge generation is a very prominent problem[1]. On the other hand, sewage sludge is considered a renewable biomass source. The European Union has a fixed goal of supplying 20% of the energy demands from renewable energy source by 2020. Moreover, 25% of the bio-energy will come from biogas through the anaerobic digestion of biomass waste[2].

Anaerobic digestion is an appropriate technique and is employed worldwide for bio-energy recovery from sludge. Four major anaerobic digestion steps can be distinguished: hydrolysis, acidogenesis, acetogenesis and methanogenesis. Hydrolysis is the rate-limiting step during the anaerobic digestion of wastewater rich in organic solids, especially for primary and/or secondary sludge[3]. Moreover, the sludge was composed of microbe cells and had colloid structure which greatly limited the hydrolysis rate of sludge in anaerobic digestion[4]. Disrupting the sludge bacterial cells and releasing the inside organisms were regarded as a useful way to improve biogas production. Consequently, pretreatment such as physical, chemical, thermal, and oxidation methods were studied to disrupt sludge cell. It

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has been proved that the heat treatment (HT) of sludge at temperature (100-275°C) could significantly increases the disintegration and solubilization of solid fraction of sludge and thus improves the sludge biodegradability by Müller[5]. Brooks[6] reported that heating sludge to temperatures of 100-275°C greatly disrupted sludge cell and liquefy the solid particulates. The organic loading rate (OLR) could reach to 5.4 kg VS/(m^3 d) by HT[7]. Chauzy et al.[8] reported a volatile solid (VS) conversion rate of 50% with a HRT of 3 d.

The largest HT combined with mesophilic anaerobic digestion was 37 000 tons of dry sludge per year operated by Cambi company[9]. In China, the first pilot-scale HT unit was built in Beijing[10].

The liquefied small molecule weight organic materials could be readily anaerobic degraded. Neyens et al.[11] reported that the reduced viscosity benefit the separation of high filtrate and the solid. Therefore, the liquid fraction of heat treated sludge by high rate anaerobic process such as EGSB was possible[12]. Yoneyama et al.[13] use a UASB reactor to treat the liquid streams of heat treated cow manure and get a COD removal up to 75.9% with OLR of 11.7 kg/(m^3 d). The UASB normally provided advantages as high removal rate and shorter HRT compared to conventional CSTR reactor[14]. Qiao et al.[15] reported that an OLR of 18.0 kg COD/(m^3 d) and a COD removal of 70% in a UASB reactor treating the heat treated sludge liquid.

Expanded granular sludge bed (EGSB) reactor was further developed to improve methane production. The high upflow velocity (>4 m/h) of EGSB reactor achieved the expansion of the granular sludge and the elimination of the dead zones[16]. A comparison between the UASB and EGSB in terms of treating potato juice reported by Fang et al.[17] showed that the EGSB reactor could generate a higher methane yield than UASB. The fresh leachate from the municipal solid waste could be converted to biogas with 88%-97% COD removal efficiency using an EGSB reactor[18].

In this study, a pilot EGSB reactor combined with a HT unit was constructed to treat the liquid fraction of sludge. The process efficiency was evaluated through the long term experiment.

2 Materials and methods

2.1 Materials

The thickened sludge with a suspended solid (SS) concentration of 3-5 g/L used in bench test was taken from the second sedimentation tank in three wastewater treatment plants in Shuofang, Chengbei and Taihu in Wuxi city. The capacities of these plants are 4×10^4 tons, 25×10^4 tons, and 18×10^4 tons wastewater per day. The effect of HT was tested through bench-scale experiments. The pilot HT unit and the EGSB reactor were built in Shuofang plant.

2.2 Bench and pilot heat treatment

The bench HT was conducted using eight stainless vessels with single volume of 1 L. The vessels were placed in an electrically heated oil bath. A concentrated sludge volume of 600 mL was put into the vessels and then heated to 130°C, 150°C, 175°C and 190°C respectively. The residence time was set at 30 min, 60 min and 90 min according to previous research[19]. The pilot HT system had three high-temperature reactors and was operated by batch mode. The steam generated from an electric boiler was injected into the reactor to heat sludge. The parameters of the pilot HT unit were 175°C/60 min. The dewatered sludge was batch feed into a preheating tank, and diluted to fluid by back processing water, and then pumped into the HT reactor. Then the heated sludge was flashed into next parallel reactor to recover energy in steam which went to the preheating tank. Finally, the treated sludge was dewatered. The cake moisture was lower than 50%. The filtrate from the belt press was used as the EGSB feeding.

2.3 Pilot EGSB reactor

Figure 1 shows the pilot HT unit and EGSB process. The total volume of the EGSB was 4 m^3 with a working volume of 2 m^3, diameter of 0.8 m and height of 5.2 m. There were seven sampling ports located along the reactor height. The sludge filtrate from the HT unit gave adequate energy keeping the reactor at 35-37°C. Electricity heating wires wrapped on the external panel of the reactor as a backup heating method. A 1 m^3 tank was used to receive the EGSB effluent which was then recycled to obtain an upflow velocity. The EGSB reactor was seeded with 1 m^3 granular sludge from a mesophilic internal circulation (IC) digester treating citric acid processing wastewater.

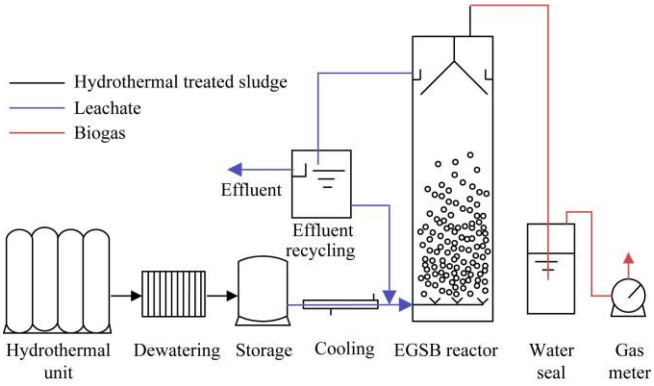


Figure 1 Scheme of pilot EGSB process

2.4 Analytic methods

The soluble COD (SCOD) was determined by the potassium dichromate/ferrous ammonium sulfate method using an ET3150B analyzer. The volatile fatty acids (VFAs) were measured using a Shimadzu-2010 gas chromatograph with a flame ionization detector. Ammonia and total phosphorus (TP) were measured in accordance with the national environmental protection agency (EPA) standard methods[20]. The pH was determined using a FE20 Mettler–Toledo pH analyzer. Gas flow meters (BSD 0.5) were used to record the daily biogas volume. The total solid (TS) and SS were measured by drying at 105°C for 24 h. The VS and volatile suspended solid (VSS) were tested by burning at 600°C for 2 h. A biochemical methane potential (BMP) test was conducted according to the literature to evaluate the methane conversion of the heated sludge filtrate[21]. The serum bottles (250 mL) at a temperature of (37±1)°C water batch were seeded with mesophilic anaerobic sludge. The adding volume of filtrate into the serum bottles was controlled to contain 1.5 g of COD.

3 Results and discussion

3.1 Effects of bench and pilot heat treatment

The sludge characteristics varied considerably primarily depending on the wastewater composition and the wastewater treatment process. In this research, three wastewater treatment plants were located in a rapidly developed district of Wuxi city. The wastewater treatment plant treats the mixture of industry and domestic sewage.

Table 1 shows the three plants used different bioprocess and give a different sludge production yields. The moisture of the dewatered sludge was approximately 80%. The organic content of the sludge was lower compared to the sludge from municipal sludge treatment plant[22]. The samples were the concentrated sludge from the second sedimentation tank in the bench heat pretreatment test. The TS of the samples was lower than the dewatered sludge used in the pilot experiments. Table 2 shows that the SS of the raw sludge ranged from 28.33 g/L to 43.43 g/L. The organic contents of three raw sludges were 61.9%, 41.9% and 39.2%. The treated sludge organic contents were 48.4%, 38.6% and 33.7%. The organic content of the treated sludge was reduced due to the liquefaction of VS. The organic matters moving to the liquor apparently increased the SCOD concentration in liquid. The VFA/SCOD ratios in the leachate were 24.5%, 14.8% and 21.4%. The high VFA/SCOD ratio indicated the filtrate was readily degradable substrate. In the pilot HT, analyzing the effect of the HT parameters was necessary. Table 3 shows that the biogas conversion at 130℃ of HT was not analyzed because of the difficulty in separating the leachate from the hydrolysized sludge. The low temperature had no effect on the sludge settlement and dewatering performance, which could also be seen from the treated sludge high moisture content. At a higher temperature of 150℃, 175℃ and 190℃, the moisture content of the dewatered treated sludge was lower than 60%. Even in Everett research, to obtain an easily dewatered sludge, temperatures in excess of 190℃ had to be used within 40 min[23].

Sludge reduction was defined as the reduced wet sludge amount before and after HT. The moisture and SS dissolution contributed to sludge amount reduction. The mean sludge reduction after treatment at 130℃ to 190℃ was 53.9%, 65.9%, 72.9% and 73.7%. The methane conversion of liquid fraction of heat treated sludge by BMP test was 70.7%, 75.3% and 71.7% at 150℃, 175℃ and 190℃, respectively. Higher temperature and longer heating time facilitated the liquefying of particular sludge but generated refractory compounds which were difficult for degradation.

Table 1 Sludge of three plants

	Sewage source	Biotreatment process	Capcity/ $\times 10^4$ t d^{-1}	Dry sludge production/t d^{-1}	Dewatering process	Test method
Shuofang sludge	Municipal sewage + industry wastewater	A^2O+SBR	4	13	Belt press	Bench+pilot
Taihu sludge	Municipal sewage + industry wastewater	A^2O	18	23	Belt press	Bench
Chengbei sludge	Municipal sewage + industry wastewater	Oxidation tank	25	55	Belt press	Bench

Note: A^2O is the abbreviation of Anaeroxic-Anoxic-Oxic process of wastewater treatment.

Table 2 Characteristics of heat treated sludge

	Shuofang		Taihu		Chengbei	
	Raw sludge	Treated sludge	Raw sludge	Treated sludge	Raw sludge	Treated sludge
Moisture/%	96.74	96.82	95.17	95.67	95.84	96.13
TS/g L^{-1}	32.45	31.29	46.71	42.85	40.48	38.29
VS/g L^{-1}	18.51	16.48	19.27	18.51	18.36	17.9
SS/g L^{-1}	28.33	22.69	43.43	34.94	38.89	32.83
VSS/g L^{-1}	17.55	10.98	18.19	14.49	15.24	11.07
TCOD/g L^{-1}	39.37	32.74	36.43	32.60	32.57	30.43
SCOD/g L^{-1}	1.09	7.54	2.22	11.66	2.11	11.62
VFA/g L^{-1}	0.54	1.85	1.34	1.72	1.19	2.49
Zeta/mV	−7.04	−7.71	−12.01	−11.45	−11.49	−4.61
TP/mg L^{-1}	-	221	-	216	-	219

Note: ‘‘–’’means the data is not detected.

Table 3 Effect of different pilot heat treatment parameters

	COD /mg L^{-1}	Moisture /%	Organic content /%	SS dissolution /%	Sludge reduction /%	Methane production /mL CH_4 (g COD)$^{-1}$	Filtrate biogas conversion/%
Raw sludge	-	83.4	-	-	-	-	-
130°C/30 min	11 051	68.5	44.0	5.1	49.9	-	-
130°C/60 min	11 332	67.7	43.4	13.0	55.3	-	-
130°C/90 min	13 352	66.0	43.8	11.0	56.6	-	-
150°C/30 min	13 464	53.4	44.9	12.5	68.8	238.0	68.0
150°C/60 min	15 618	61.8	41.8	12.5	62.0	259.0	74.0
150°C/90 min	17 593	57.8	40.9	16.1	67.0	245.0	70.0
175°C/30 min	17 784	56.0	41.1	18.8	69.4	255.5	73.0
175°C/60 min	20 252	52.2	39.3	23.9	73.6	255.5	73.0
175°C/90 min	22 552	54.9	37.6	34.2	75.8	280.0	80.0
190°C/30 min	23 355	52.2	39.9	21.7	72.8	276.5	79.0
190°C/60 min	26 311	51.3	38.5	24.6	74.3	241.5	69.0
190°C/90 min	23 057	53.6	37.1	27.3	74.0	234.5	67.0

Note: “–’’means the data is not detected.

3.2 Biogas production in the pilot EGSB reactor

Bench HT research normally uses fluid thickened sludge with low SS feed stock[12,24]. In this pilot HT system, dewatered sludge was diluted by flashed steam from high temperature reaction tank and by hot process water to SS of 10%. The characteristics of the raw sludge, pretreated sludge and filtrate are shown in Table 4. The filtrate was an acidic substrate with a mean COD concentration of 24.0 g/L and pH of 6.74. The BOD/COD ratio of filtrate was 0.46, indicating the readily biodegradability. The COD/N ratio was 9.2. The EGSB reactor runs for 206 days in four stages. The first stage was the start up of the reactor using 12 d. The initial OLR was a high value of 3-4 kg COD/(m^3 d). At this stage, the influent COD was unstable because of the HT unit debugging. The second stage was short period from day 13 to 20, the OLR increased to 6.0 kg COD/(m^3 d) by increasing the influent COD concentration. The digester steadily operated at this stage. The COD removal efficiency was between 60% and 75%. At the long third stage from day 21 to 136, the OLR was 8.0 kg COD/(m^3 d). The COD removal efficiency was between 40% and 72%. From day 90 to 136, COD removal was stabilized at around 62%. At the fourth stage, the OLR increased to about 10.0 kg COD/(m^3 d) and did not remarkably change the COD removal rate.

Table 4 Basic properties of heat treated filtrate

	Sludge				Filtrate			
	SS/%	VS/%	VS:SS/%	pH	COD/g·L^{-1}	TN/g·L^{-1}	NH_3-N/g·L^{-1}	TP/mg·L^{-1}
Raw sludge	16.4	8.5	51.7	5.86-7.74	10.4-30.6	1.9-3.1	1.0-2.2	11.2-182.0
Beat treated sludge	7.4	3.1	41.1	6.74	24.0	2.62	1.72	61.0

Figure 2 shows the COD of the influent and effluent, OLR and COD removal efficiencies. Throughout the experiments, the COD removal was lower than that of the bench mesophilic UASB reactor[15]. The VFA in feeding was 6068.6 mg/L and 1772.0 mg/L in the effluent, indicating that 71% of the easily degradable organics was removed from the digester. Eskicioglu et al.[12] reported that VFA concentration increased by about 250% to raw sludge by heating to 70°C.

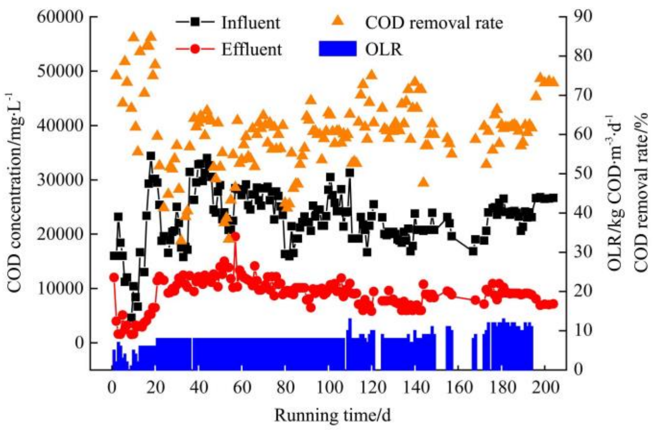


Figure 2 Reactor performance

During the experiment, the daily biogas fluctuated considerably, particularly in the start up of the digester. Figure 3 shows the biogas production curve. The heat pretreatment unit was operated using batch mode. Heat treated sludge liquor was storage in a tank before feeding. From day 42 to 48, a peak in the biogas production was observed. In these days, the heat pretreatment unit has been continuously operated for 168 h, and the influent flowing into the EGSB was fresh. After that, the filtrate for feeding was stored for a long time. The high influent pH and the low biogas production were then observed in Figure 3. From day 50 to 180, the low-pH influent resulted in a high biogas production, whereas the high-pH influent resulted in a low biogas production. The results from 17 days show that the filtrate storage could increase pH and thus decrease biogas production. During the storage, the clear brown filtrate became dark brown. When the EGSB reactor was kept in a stable operation state, the daily biogas amount was about 400 L. The average content of methane was around 85%. The theoretical biogas production was calculated on 0.35 m^3 CH_4/kg COD removed. The observed daily biogas was consistent with the theoretical calculation results.

The pH is an indicator of the EGSB stability. At the initial stage, the pH of the influent fluctuated, and the effluent fluctuations were lesser. After 50 d, the influent pH was stable and was lower than 7.0 with the stable operation of the HT unit. The pH of the effluent was constantly maintained at 8.0. The influent pH significantly changed because of the diversity of different sludge samples, heat pretreatment operation and sludge liquor storage.

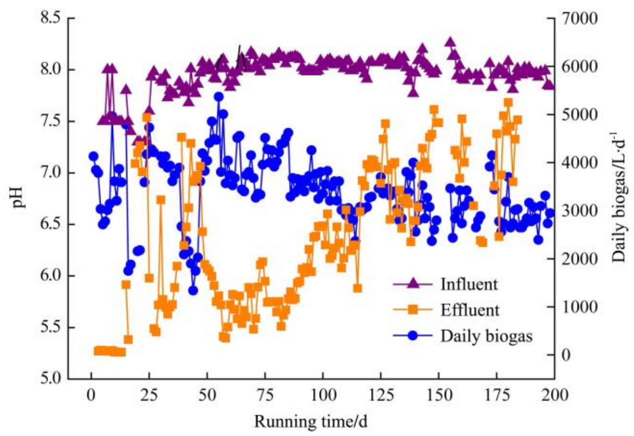


Figure 3 Daily biogas and pH variation

3.3 Ammonia, phosphorus and pH variation

The nitrogen and phosphorus in sludge was dissolved into liquid fraction by high temperature heating. The distribution of nitrogen and phosphorus in the EGSB should be analyzed for further effluent treatment. As shown in Figure 4, the ammonium was relatively high both in the effluent and influent. The mean influent ammonium was 1715 mg/L, and the effluent ammonium was 1590 mg/L. The ammonium to total nitrogen ratio in the influent and effluent was 0.66. Sometimes, the ammonium of effluent was higher than that of influent because of the degradation of organic nitrogen compounds in feeding. As mentioned by Appels et al.[4], ammonium is produced during the degradation of nitrogenous matter, mainly proteins and urea.

Ammonium (NH_4^+) and free ammonia (NH_3) are the two most predominant forms of inorganic nitrogen present. Ammoniums (< 200 mg/L) are beneficial to anaerobic digestion because nitrogen is an essential nutrient for the micro-organisms[25]. Free ammonia of 560-568 mg NH^3-N/L can cause a 50% inhibition of methanogenesis at pH 7.6 under thermophilic conditions[26]. Throughout the EGSB operation, the concentration of TP was mostly lower than 100 mg/L. Ammonium could be dissociated and combined with CO_2, which absorbed the CO_2 from the biogas and resulted in the decrease of CO_2 in biogas.

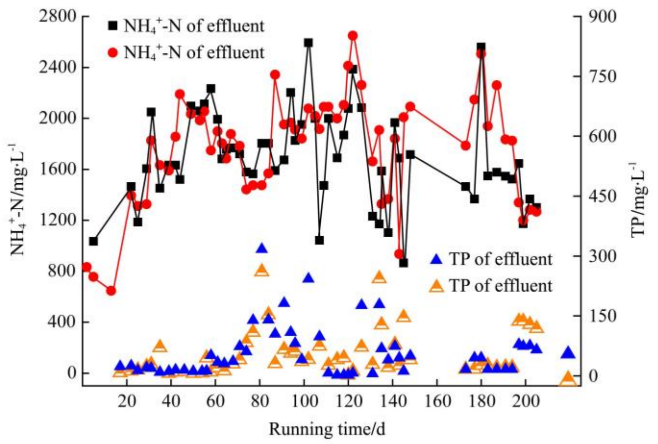


Figure 4 NH_4^+-N and TP variation

3.4 Effect of upflow velocity on granular sludge

The sedimentation performance of granular sludge was very important in retaining adequate microorganism. In the EGSB reactor, the sludge bed behaved as a completely mixed tank. At different stages, the sludge distribution and the content of the organic matter were detected along the EGSB reactor, as shown in Figure 5a. When the upflow velocity was 2 m/h, the sludge bed was stable, and 50% of the effective reactor volume was occupied by granules. On the other hand, the high upflow increased the risk of the washing out of bio-granules from the reactor.

The upflow stream was adjusted by increasing or decreasing the recycling effluent amount. In the first period, the velocity was set at 1 m/h. In the following stages, the velocity was controlled at 2, 3 and 4 m/h. In a long period from day 19 to 52 and from day 68 to 122, the reactor was operated at a velocity of 2 m/h. After 31 d, a clear sludge bed interface was observed. In the upper part of the reactor, the sludge concentration was approximately 10.0 g/L. In the granule bed, the concentration was greater than 35.0 g/L. From day 52 to 68, the upflow velocity was still adjusted to 4 m/h. The increment of upflow velocity did not change the sludge distribution.

In Figure 5b, the organic content of the granules was analyzed between 131 and 167. Within this period, the upflow velocity was adjusted to 1, 2 and 3 m/h. When the upflow rate changed from 1 m/h to 3 m/h, the sludge concentration remained stable. Compared with the first 54 days, although the interface still existed, the sludge concentration difference between the fixed sludge bed and the upper suspended phase decreased. Sometimes, the press filter dewatering worked abnormally, resulting in an increment in the SS concentration in the influent, which was then detected by sampling from the EGSB. As seen in Figure 5, higher upflow rate elevated the EGSB sludge bed.

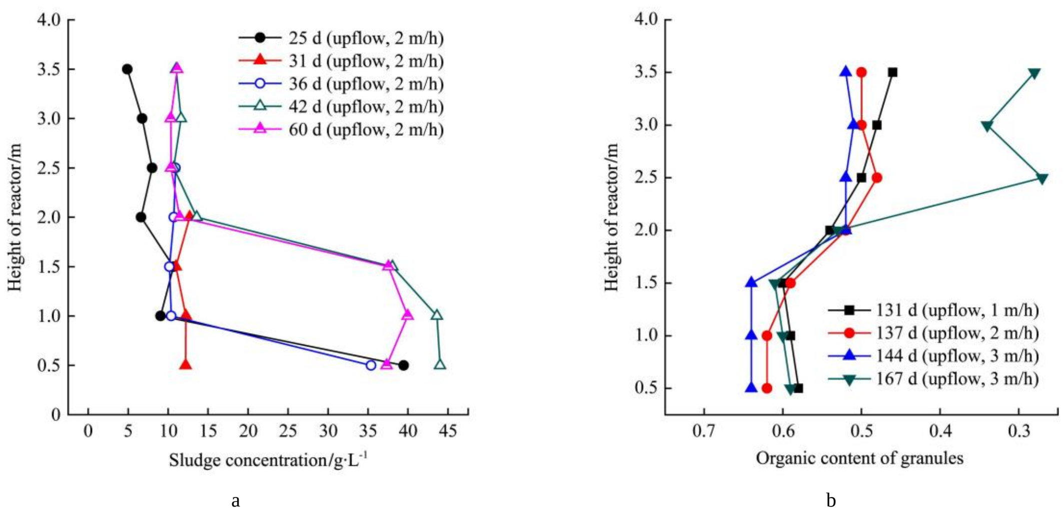
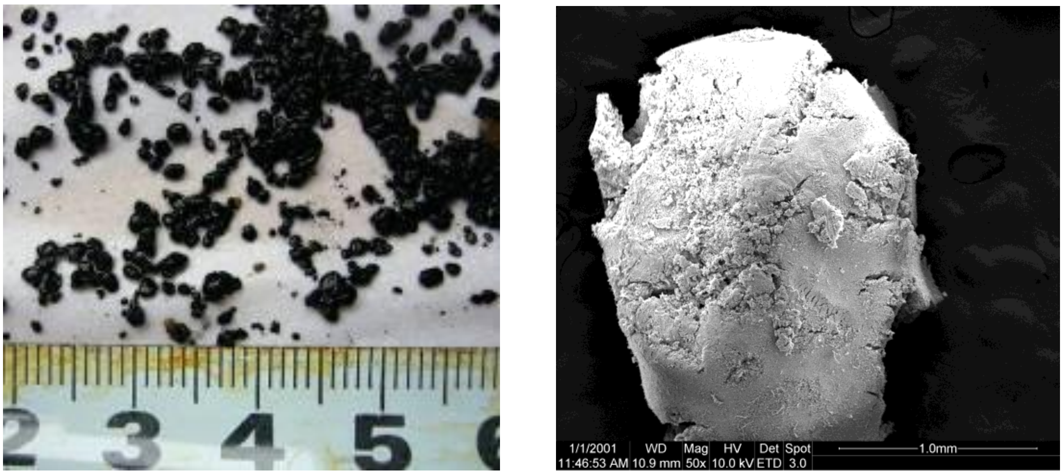


Figure 5 Distribution of sludge in EGSB reactor

In the lower half of the reactor, the organic content of granules was approximately 0.6, whereas in the upper reactor, the organic content of the suspended solids was 0.5. The high upflow stream movement and the gas escaping from the sludge bed hoisted parts of the sludge. Simultaneously, parts of organic and inorganic particles were washed out from the reactor instead of bio-sludge. Throughout the experiments, the solid/liquid interface was stable under different upflow velocities. The high-bioactivity sludge was kept in the reactor. Limited by the effluent pump flow, the effect of high upflow velocity on the sludge distribution in the reactor was not studied.

3.5 The properties of granular sludge

The composition and the structure of granular methanogenic sludge depended on the type of influent substrate and different microorganisms[27]. Fang et al.[28] and Molina et al.[29] investigated the characteristics of the anaerobic granules formed by the treatment of different types of wastewater. Two different granular arrangements were found. In the first case, the granules developed from a substrate rich in carbohydrates and presented a three-layer structure. In the second case, the granules grown on complex substrates and volatile fatty acids showed a random distribution of microorganisms. In recent research, the granular sludge could be formed accepting the heat treated sludge liquor seeded by suspended growth sludge[15]. Hence, the direct granular sludge inoculums were expected to achieve a quick and stable start up of EGSB reactor. There was an opportunity of the granular inoculums from the nearby IC digester in this pilot system. The seeded granular sludge acclimatized to the new sludge leachate substrate and achieved certain removal efficiency even under unstable conditions. The fresh raw sampled granules comprised black particles of approximately 1 mm. After 98 days experiments, the granule sludge was extracted and photographed. No significant change was apparent in the sludge, as shown in Figures 6a and 6b. During the experiments, the granules were extracted from the reactor and transported to laboratory as new bench mesophilic UASB reactor inoculums and provide a high activity.



a b

Figure 6 Picture of granular sludge (a) photo of granules at 98 day; (b) SEM photo of granules at 98 day

4 Conclusions

1) The biogas production from heat treated sludge liquor by the mesophilic EGSB reactor was stable and the methane conversion rate was approximately 60%-70%.

2) The granular sludge inoculums from citric acid anaerobic digester could be quickly activated and adapted to the sludge filtrate.

3) The EGSB effluent contained high ammonia and phosphorus which need further treatment.

Acknowledgements

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