Settleability assessment protocol for anaerobic granular sludge and its application

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Abstract

A simple method for settleability assessment of anaerobic granular sludge was proposed and its applicability as an operating parameter was evaluated in a lab-scale UASB reactor treating brewery wastewater. Based on the settleability protocol, the OLR was increased up to 28 kg COD·m^{-3}·d^{-1} (67 kg COD·m^{-3} of granular bed volume·d^{-1}) which corresponds to an HRT of 1 h. The results revealed that the protocol was sufficiently sensitive to define the settleability of the sludge samples and to accurately determine their allowable upflow velocities, resultant organic loading rates, and recycling ratios according to the settleability of the granular bed. Also, a series of graphical procedures with settling tests which are very easy to apply for settleability monitoring was improvised, capable of direct use as an operational and monitoring parameter of the granular bed with laboratory and full-scale reactors, without need for additional sludge bed control such as dosing of chemicals. In addition, this method was also found to be applicable to improve and monitor system performance according to high or low-strength wastewater characteristics. Image analysis of the granular biomass supported the suitability of this graphical method.

Keywords: brewery wastewater, granular sludge, recycling, settleability, UASB, upflow velocity

Introduction

During the past two decades anaerobic wastewater treatment biotechnology was extensively advanced by the development of the innovative upflow sludge bed (USB) type reactor concepts such as upflow anaerobic sludge bed (UASB), anaerobic baffled reactor (ABR), upflow anaerobic solids removal (UASR), hydrolysis upflow sludge bed (HUSB), upflow acidogenic substrate precipitating (UASP), elutriated phased reactor (EPR) as well as expanded granular sludge bed (EGSB) (Bachman et al., 1985; Kim et al., 2001; Lettinga et al., 1997; Zeeman et al., 1997). The success of these anaerobic systems is related to their capacity for accumulation of good settling biomass without the need of a biomass carrier, allowing high solids retention time and process stability with simple and low-cost equipment.

There is a need for an improved settleability monitoring technique that is simple, requires low labour, and is economical, which can directly be used as an operational and monitoring parameter for laboratory and full-scale research. In addition, an assessment of settleability to evaluate the adequate operating upflow velocity as well as the stability of the sludge bed is needed. Restocking granules to replenish those lost due to excessive granular sludge washout is a common problem in higher rate UASB systems (Stover, 2000). The application of a technique to measure operating upflow velocity, particularly in higher rate conditions, would result in production and maintenance of well-defined and densely structured granular sludge as well as enhancement of system performance.

Various direct and indirect methods to quantify the physical characteristics of the anaerobic granular sludge in terms of the size, density, strength and settleability have been presented. Direct granular particle size analysis was performed manually with a portion graticule/lattice ruler (Hulshoff Pol, 1989) or wet sieving using phosphate buffer solution or tap water (Laguna et al., 1999), by automatically using image analysis and computerised data processing (Dudley et al., 1993) and by particle size analysis using a laser (Yan and Tay, 1997). The strength of granules was measured in several studies by examining the effects of shear force, sonication, shaking of the granules, or turbidity (Teo et al., 2000; Quarmby and Forster, 1995; Tramper et al., 1984). Indirect granular sludge density analysis was performed by the measurement of the settling velocities of a sludge sample to extrapolate the corresponding diameters (Hulshoff Pol, 1989; Grotenhuis et al., 1991) or sludge volume index (SVI) (Ahn, 2000; Cuervo-López et al., 1999; Yan and Tay, 1997), and by a settleability profile analysis using an upflow-type settling column (Ahn, 2000; Andras et al., 1989; Moosbrugger, 1994). Unfortunately these methods have several disadvantages in application and cost requirement. Also these methods can only be used to test the sludge particle size distribution and sludge settleability. None can be used directly as an operational parameter in laboratory or full-scale systems to determine the allowable upflow velocity of the anaerobic granular sludge in USB-type reactors.

The purpose of this research is to introduce a settleability assessment protocol for determining the allowable operating upflow velocities of high-rate UASB reactors, and to evaluate its applicability as an operational parameter in laboratory-scale reactors. To assess the suitability of this method, image analysis of each sample was performed, using computerised data processing.

Materials and methods

Settleability assessment protocol

Settling test apparatus

Figure 1 depicts the settling device with a vertical glass tube (effective size: 20 mm diameter and 300 mm height) followed by

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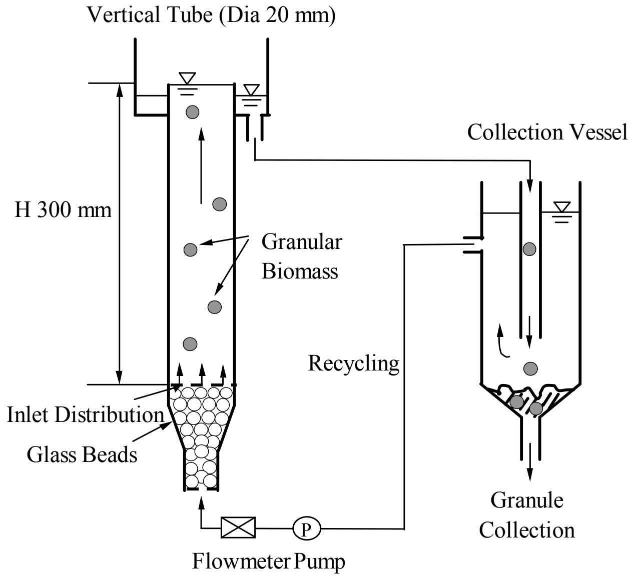


Figure 1
Schematic diagram of settling device

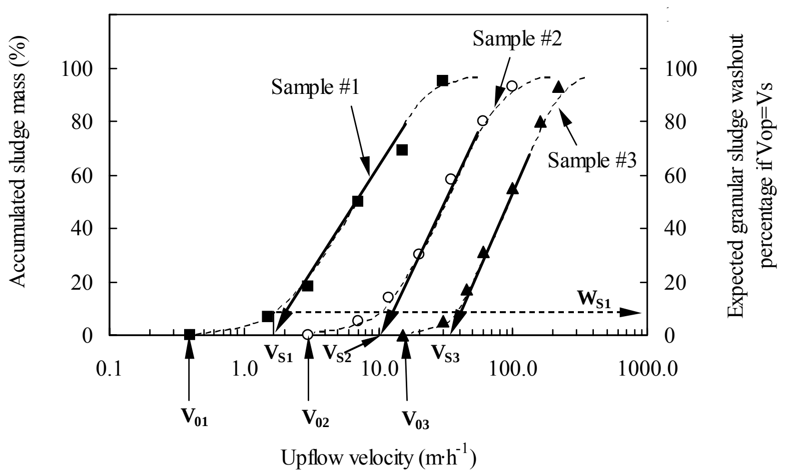


Figure 2
Development of settleability profiles for granular sludge

a collection vessel. The bottom part of the vertical glass tube was packed with glass beads to enhance flow distribution. The inlet line had a flow meter and variable speed pump (Masterflex, Cole Parmer). The settled clear water of the collection vessel was recycled to maintain constant upflow velocity in the vertical tube. Prior to testing, the apparatus was filled with the settled effluent from the main UASB reactor, after which a granular sludge sample being fractionated was put into the vertical tube. For this test, sample size of the granular sludge averaged 50 ml throughout.

Principle and methodology

The settleability assessment test investigated in the study measures settling velocity of granular sludge. For instance, the constant upflow velocity supplied to the bottom of plug-flow type vertical tubes will separate the granular sludge of lower settling velocity. The mass percentage of washed-out granular sludge from the vertical tube to the collection vessel may be recorded by means of this test according to the applied upflow velocity. The increments of differentiated upflow velocities gradually fractionate the entire sludge sample according to its settling velocity. The accumulated sludge mass percentage (or mass) per unit sample mass retained in the settling tube are plotted against the respective upflow velocity on a semi-log scale, as shown in Fig. 2 for these different samples. This curve, which can be represented by the approximation method, is defined as the settleability profile of the granular sludge sample. Measurement of upflow velocity which does not cause granular sludge washout is critical to anaerobic treatment because it may indicate zero washout upflow velocities in the actual reactor operation. In this paper, the maximum upflow velocity which does not cause granular sludge washout is defined as the ***zero washout upflow velocity* (V_0)**. Another important test component is the choice of a representative sludge sample in the laboratory or full-scale UASB reactor because the settleability of the sludge sample will be different according to its location in the reactor. Therefore to determine the representative settleability as well as the stability of the sludge bed, a number of settling tests according to sludge bed depth are required. These results are subsequently compared with the ***actual operating upflow velocity* (V_{op})** of the UASB reactor. However, should determination of only the operating upflow velocity be desired to increase the organic loading rate of the UASB reactor, the representative sludge sample could be taken from the upper part of the UASB reactor alone, since it is the depth which can be expected to have the lowest settling velocity.

From the settleability profile as shown in Fig. 2 it may be seen that several important points can be determined using a graphical approximation method as follows:

V_{0i} : zero washout upflow velocity of i^{th} sample (the crossing point between the settleability profile and the x-axis)
V_{Si} : critical upflow velocity of i^{th} sample to allow some amount of granular sludge washout (=W_{Si}) (the crossing point between the extension of the linear section of the settleability profile and the x-axis
W_{Si}: the expected granular sludge washout percent of i^{th} sample if $V_{opi} = V_{Si}$

V_{opi} can be defined as the i^{th} operating upflow velocity of the main UASB reactor which is operated with certain operating conditions. Using the above data which is determined according to the operating condition of a UASB reactor, the next applicable upflow velocity and the resultant loading increase in the reactor can be approximated. It is possible to make a graph of the relationships between organic loading rate, upflow velocity (such as V_0, V_S and V_{op}), and biomass washout (W_S) which will be discussed in a later section. The next applicable operating upflow velocity (V_{opi+1}) in the reactor can be in the range of $V_{opi} \sim V_{Si}$. However, to ascertain the no washout velocity of the granular sludge (zero washout condition) the upflow velocity for the next step will be in the range of $V_{opi} \sim V_{0i}$. If the reactor has excess capacity against granular sludge washout, the reactor may be operated in an upflow velocity range of $V_{0i} \sim V_{Si}$, yielding a sludge washout of maximum W_{Si} (%).

In addition to the determination of the applicable upflow velocity for a particular feeding condition in the reactor, the allowable (R_{ai}) and critical recycle ratio (R_{ci}) can also be determined by using the upflow velocity data, i.e. $R_{ai} = V_{0i}/V_{opi}$, $R_{ci} = V_{Si}/V_{opi}$.

TABLE 1
Characteristics of brewery wastewater

Item	Concentration (mg·ℓ^{-1})*	
	Range	Average ± S.D.
pH	6.3 ~ 7.0	6.7 ± 0.2
TCOD	920 ~ 1 900	1 290 ± 240
SCOD	680 ~ 1 560	1 040 ± 230
BOD$_5$	730 ~ 1 470	890 ± 170
TSS	60 ~ 380	210 ± 67
VSS	40 ~ 200	140 ± 37
TKN	16.4 ~ 36.4	23 ± 7.4
NH$_3$-N	3 ~ 11.5	6.9 ± 2.7
T-P	5.3 ~ 12.5	9.0 ± 3.1
S-P	2.5 ~ 9.9	6.8 ± 2.3

* unless specified otherwise; S.D, standard deviation

For high-strength wastewater and higher organic loading, V_{opi} should be replaced by total upflow velocity V_{total} (by liquid phase + biogas phase) of ith operating condition due to higher biogas production rate. The determination of the appropriate recycle ratio is dependent upon the wastewater strength and the settleability of the granular sludge in the sludge bed.

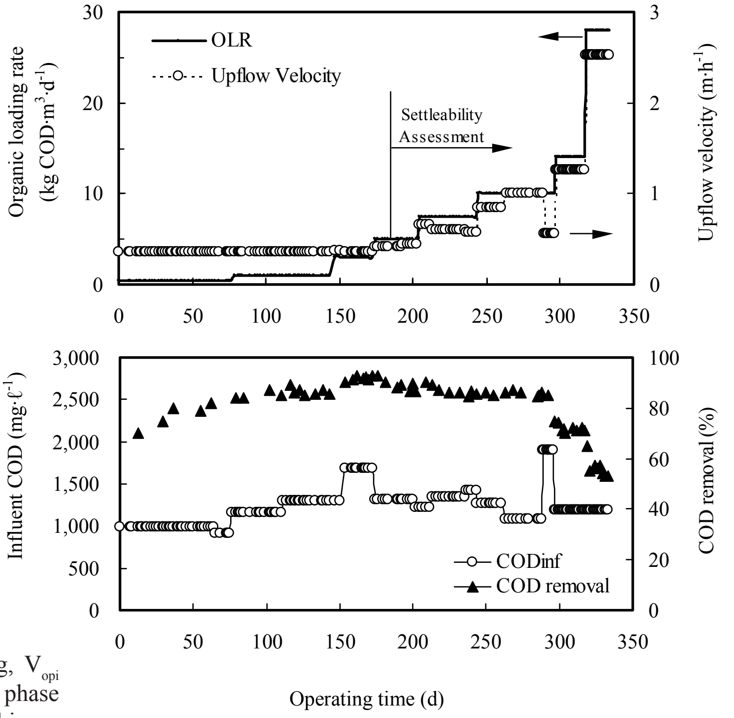


Figure 3
Operating condition and reactor performance of laboratory-scale UASB reactor

Wastewater composition

Table 1 represents the characteristics of the brewery wastewater used in this experiment. The wastewater was collected from the anaerobic pretreatment plant at a brewery industry in the Kumi Industrial Complex, Korea. The average COD concentration of wastewater was 1 300 mg COD·ℓ^{-1} and the insoluble fraction and COD : BOD ratio were 19% and 1.2~1.7, respectively. The average TSS concentration was 210 mg SS·ℓ^{-1} and the VSS fraction (VSS/TSS) was 65%. The COD : N : P of wastewater was 100:2:1, which means that it was not necessary to add nutrients for the growth of anaerobic bacteria. The pH of the raw wastewater was in the range of 4.9 to 8.3 but the influent for the anaerobic sludge bed reactor was neutralised to pH 6.5 to keep a neutral pH in the sludge bed zone.

Reactors and operating conditions

The novel USB-type anaerobic reactor developed by Ahn et al. (2000), was used as the laboratory reactor. The effective reactor volume and the sludge bed volume of this reactor were 8.3 and 3.5 ℓ respectively. 3.5 ℓ granular sludge from a full-scale UASB plant treating brewery wastewater was inoculated into the reactor. The VSS concentration and VSS fraction of the raw seed granular sludge were 29 g·ℓ^{-1} and 70%, respectively.

After start-up the OLR of the reactor was maintained at 7.5 kg COD·m^{-3}·d^{-1} (18 kg COD·m^{-3} of granular bed volume·d^{-1}, HRT 4.6 h) and the operating liquid upflow velocity (V_{op}) in the reactor at approximately 0.6 m·h^{-1}. Increases of organic loading rates based on the settleability protocol were achieved up to 28 kg COD·m^{-3}·d^{-1} (67 kg COD·m^{-3} of granular bed volume·d^{-1}), which corresponds to an HRT of 1 h. Because the influent wastewater concentrations varied a little according to sampling time, the operating upflow liquid velocity at an applied OLR was properly regulated by recycle flow as well as influent. The operating liquid upflow velocity (including influent and recycle) in the laboratory reactor was increased from 0.6 to 2.5 m·h^{-1}. All experiments were performed at 35±2°C in a temperature-controlled room.

Analyses

Analytical procedures were conducted according to *Standard Methods* (1989) unless specified otherwise. pH (Orion Research, Model 420A, USA) and gas production (Sinagawa Seiki Co., Model W-NK-0.5A, Japan) were monitored daily in all reactors by the use of the wet-test gas meter. A gas chromatograph equipped with a TCD detector and silica-gel column (Tremetrics Model 9000, USA) was utilised to determine gas composition. Temperature for the column was kept at 60°C, 85°C for the injector and 75°C for the detector. The helium carrier gas had a flow rate of 22 mℓ·min^{-1}. A Tremetrics integrator was used for data integration. In every experimental run the systems were considered to be stable when the effluent characteristics showed approximately steady state values (i.e. less than 10% variation in concentration).

To substantiate the suitability of the protocol, image analysis with computerised data processing (Image-Pro Plus for Windows 95/NT, Media Cybemetics, USA) was applied to each granular sludge sample after the settling tests. The granular sludge size distribution at different granular bed depth (upper and lower) was examined according to the respective operating upflow velocity in the reactor.

Start-up of laboratory UASB reactor

Figure 3 shows start-up procedure in terms of OLR, influent COD concentration, operating upflow velocity, and COD removal in the laboratory-scale reactor. The COD removal was calculated based on total COD (TCOD). The upflow settling test was first performed

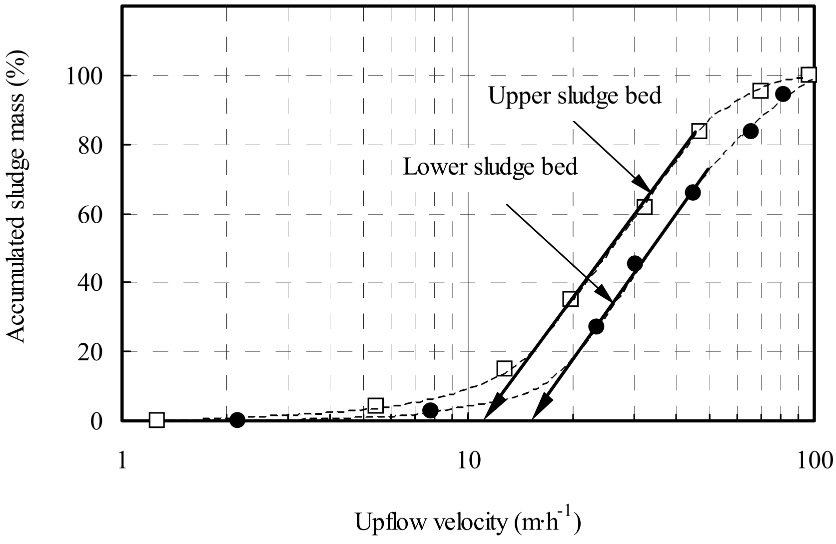


Figure 4
Settleability profile of granular sludge according to location in the sludge bed (OLR = 10 kg COD·m^{-3} of reactor volume·d^{-1})

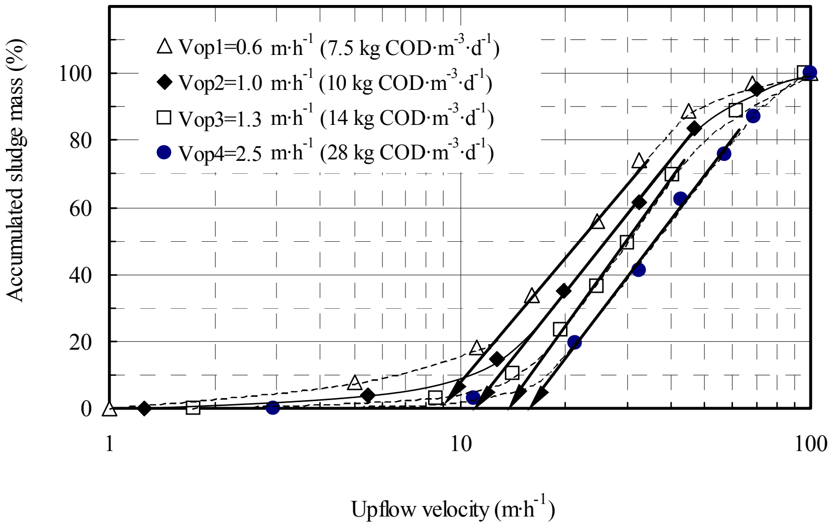


Figure 5
Settleability profile of granular sludge at upper sludge bed zone according to V_{op} and OLR

at the OLR of 7.5 kg COD·m^{-3}·d^{-1} to test for applicability as well as to determine the next operating upflow velocity and resultant organic loading. About 80% COD removal was achieved after 30 d of operation (OLR of 0.5 kg COD·m^{-3}·d^{-1}). After approximately 150 d COD removal increased to 93% at the OLR of 3 kg COD·m^{-3}·d^{-1}, but gradually decreased at the OLR of 5 kg COD·m^{-3}·d^{-1}. Detailed reactor performance subsequent to loading rate increases by the settleability assessment will be discussed in a later section.

Results and discussion

Development of settleability profile

Settling characteristics according to sludge bed depth

Figure 4 shows the varying settleability properties of granular sludge ascertained according to location in the upper or lower sludge bed zone. In this case, the operating OLR and upflow velocity in the reactor were 10 kg COD·m^{-3}·d^{-1} (24 kg COD·m^{-3} of granular bed volume·d^{-1}) and 1 m·h^{-1} respectively. Zero washout upflow velocities (V_0) of the upper and lower sludge bed zone were found to be 1.3 m·h^{-1} and 2.2 m·h^{-1} and the critical upflow velocities (V_S) were 11 m·h^{-1} and 16 m·h^{-1} respectively. If V_S based on the lower sludge bed zone (= 16 m·h^{-1}) were to be used as the actual V_{op} in the reactor, the expected washout granular sludge percentage (W_S) would be about 20% for the upper sludge bed zone and about 8% for the lower sludge bed zone. Whereas if V_S of the upper sludge bed zone (= 11 m·h^{-1}) were used as the actual V_{op}, the W_S would be

TABLE 2
Results of settleability profile analysis

i	OLR (kg COD· m^{-3} reactor volume·d^{-1})	HRT (h)	Upflow velocity(m·h^{-1})			Expected granular sludge washout % if Vopi=Vsi
			Vopi*	Voi	Vsi	
0	5	6.3	0.4	-		
1	7.5(18)	4.6	0.6	1.0	9	16
2	10(24)	3.0	1.0	1.3	11	12
3	14(34)	2.1	1.3	1.7	14	9
4	28(67)	1.0	2.5	2.9	16	7

* by liquid phase only; parentheses, kg COD·m^{-3} granular sludge bed·d^{-1}

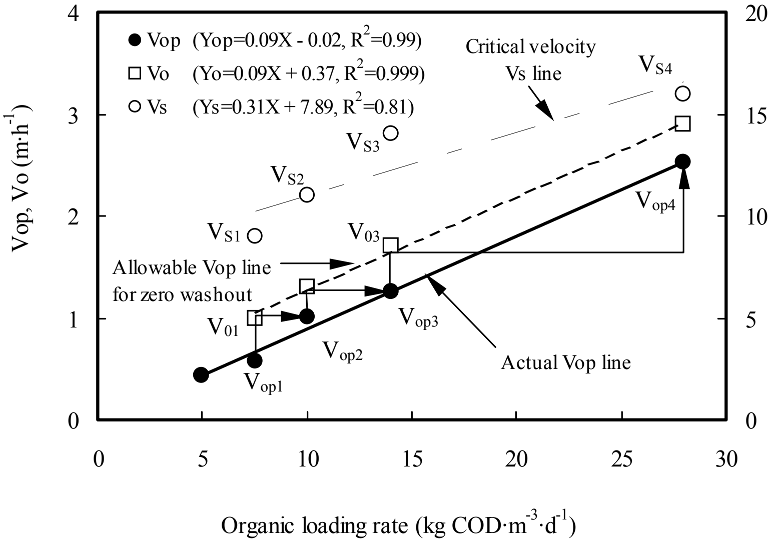


Figure 6
Relationship between V_{op}, V_o and V_s

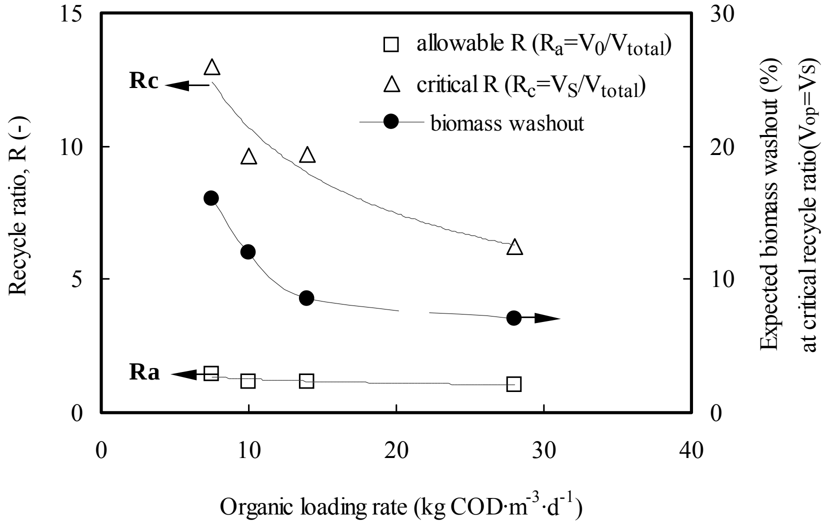


Figure 7
Recycle ratio (R) and W_S according to OLR

considerably lessened to about 12% for the upper sludge bed zone and about 5% for the lower sludge bed zone. These results confirm that the settleability assessment for determining optimum operating upflow velocity and resultant OLR in the reactor must be taken only from the upper sludge bed zone.

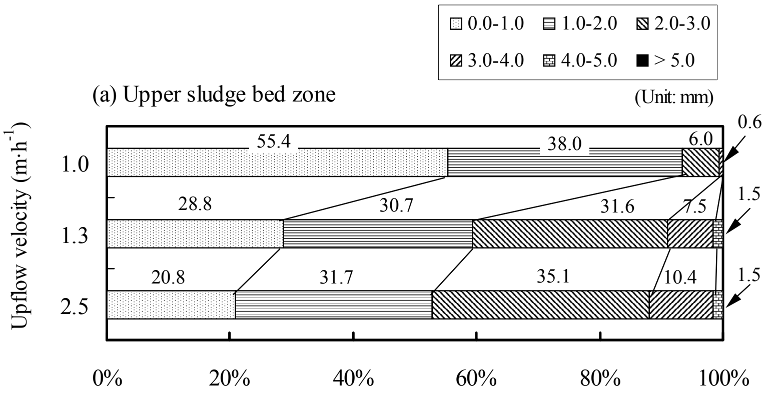
Settling characteristics according to operating OLR

Figure 5 shows increased OLR impact upon the settleability of granular sludge samples. OLR increases were based on the settleability assessment guideline (see next section) of granular sludge samples, all taken from the upper sludge bed zone. The OLR was increased from 5 kg COD·m^{-3}·d^{-1} for V_{op} of 0.4 m·h^{-1} to 28 kg COD·m^{-3}·d^{-1} for V_{op} of 1 m·h^{-1}. Increase of the operating upflow velocity and resultant OLR were changed by 1.5 or 2 times to determine the response of the granular bed under high V_{op} change. The result estimated from the settleability profiles is summarised in Table 2. Even though a granular sludge washout experiment using the UASB reactor was not performed because this was not the main objective of this study, the prediction of the expected granular sludge washout fraction at the critical upflow velocity could be useful to predict the approximate upper limit of upflow velocity.

Determination of V_{op} and OLR

Under stable conditions, in terms of effluent concentration during the operational period at the V_{op1} of 0.6 m·h^{-1} which corresponded to an OLR of 7.5 kg COD·m^{-3}·d^{-1}, the first settleability profile of the operating granular bed was plotted (see Fig. 5). The V_{01}, V_{S1} and W_{S1} determined from this profile were 1 m·h^{-1}, 9 m·h^{-1} and about 16% respectively. The next possible V_{op2} to allow zero biomass washout was 1 m·h^{-1} (=V_{01}) which corresponded to the resultant OLR of 10 kg COD·m^{-3}·d^{-1}. From the second profile determined at this operational condition, the V_{02}, V_{S2} and W_{S2} were 1.3 m·h^{-1}, 11 m·h^{-1} and about 12% respectively. Similarly the next possible V_{op3} to allow zero biomass washout was 1.3 m·h^{-1} (=V_{02}) which corresponded to the resultant OLR of 14 kg COD·m^{-3}·d^{-1}. From the third settleability profile determined at this operational condition, the V_{03} was determined 1.7 m·h^{-1}, 14 m·h^{-1} for the V_{S3}, and about 9% for W_{S3}.

According to a similar iterative procedure, the next possible V_{op4} was also determined from Fig. 5 at the rate of 1.7 m·h^{-1} (=V_{03}) which corresponded to the resultant OLR of 18 kg COD·m^{-3}·d^{-1}. However, to determine the response of the granular bed at two times higher V_{op} than the rate determined by third iteration, the reactor was operated with the V_{op4} of 2.5 m·h^{-1} by extrapolating (shown in Fig. 6). This operating upflow velocity corresponded to the OLR of 28 kg COD·m^{-3}·d^{-1} and HRT of 1 h. The settleability profile measured after the operational period of 15 d under this condition revealed that the V_{04} was 2.9 m·h^{-1}, 16 m·h^{-1} for the V_{S4}, and about



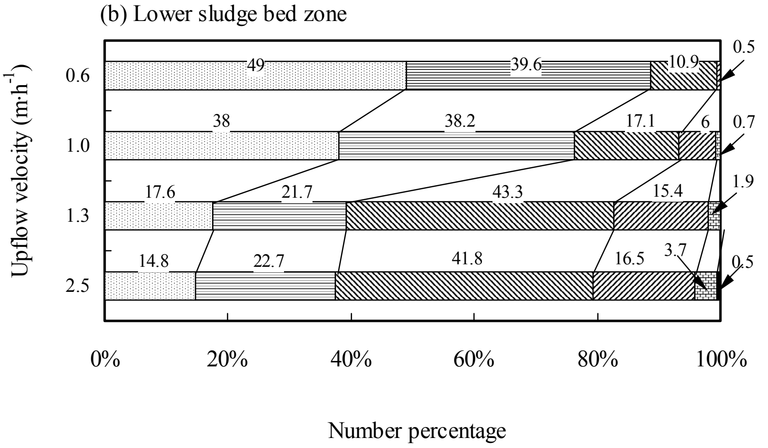


Figure 8
Size distribution of granular sludge by image analysis

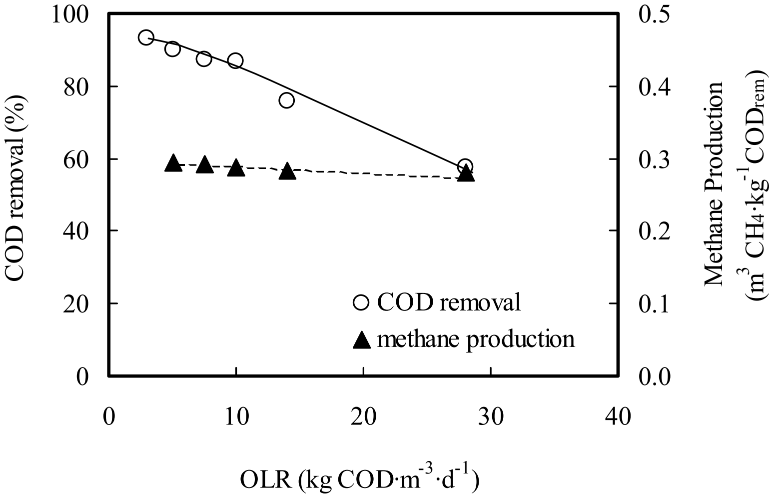


Figure 9
Reactor performance of laboratory-scale UASB reactor

7% for the expected W_{S4}.

From the iterative procedure, the relationship between the V_{op}, V_0 and V_S according the applied OLR in the reactor could be represented in Fig. 6. The slope and the correlation coefficient (R^2) of the V_0 and actual V_{op} plotted against the applied OLR were 0.09 and over 0.99, respectively. Using this figure the V_0 line would be the actual allowable V_{op} line.

In this graphical procedure to determine the V_{op}, the upflow velocity increase by biogas production was not considered because the amount (about 15 to 20% of liquid base upflow velocity) was not significant. This was due to the low-strength wastewater characteristics (average influent COD≈1 300 mg·ℓ$^{-1}$). However, for high-strength wastewater producing a high volume biogas, the determination of the V_{op} should be based on the total upflow velocity (liquid plus biogas phase). Also, the proposed method does not account for process kinetics, thus the estimation of resultant OLR is valid for the given reactor operating conditions in this research. A re-calibration procedure would be required according to various wastewater characteristics and operating conditions.

Determination of recycle ratio

Optimised effluent recycling in anaerobic treatment would have several benefits such as wastewater strength control, reduced alkalinity requirements for influent wastewaters, adequate sludge bed expansion and improved mass transfer. However, determining the number of recycling ratios are necessary for maintaining UASB reactor sludge bed stability has been difficult in the past. Since the V_{op} in UASB reactor can be controlled by effluent recycling and this settleability assessment protocol has successfully determined optimum recycling ratios this difficulty can now be overcome.

Figure 7 plots the relationships between the recycle ratio and granular sludge washout according to OLR. The recycle ratio is based on the total upflow velocity (V_{total}) which indicates the upflow velocity involved in the liquid plus biogas phase in the reactor. From this figure, if the reactor tends to wash out light granular sludge, the recycle ratio will be related to V_S/V_{total}, which means the R_c. In addition the expected mass of granular sludge washout will depend on the V_S. An OLR of 7.5~28 kg COD·m^{-3}·d^{-1} (18~67 kg COD ·m^{-3} of granular bed volume·d^{-1}) was used in this experiment resulting in an R_a to meet the zero sludge washout requirement of about 1 times the influent flowrate, 6~13 times for the R_c, and 7~16% for the W_S.

The assessment protocol described above is an easily applicable settleability monitoring technique. It is simple, economical, and less laborious than prior techniques. The protocol measures maximum upflow rates for both laboratory and full-scale reactors without other floating sludge bed control such as the mechanical grinding and dosing of Fe described by Kosaric et al. (1990) and Yoda and Nishimura (1997). The relationship between the V_{op} and OLR may vary according to sludge bed reactor configuration as well as sludge bed condition. Thus some high-rate EGSB reactors operate with a mixed granular bed (for example, Paques IC® reactor) while others operate with a stratified bed (for example, Biothane's Biobed® reactor). However, the biomass settleability assessment protocol described above could be applied to each reactor configuration.

Image analysis of granular sludge

Granular settleability is imperative for UASB reactor sludge bed stable processing and can be indirectly estimated by the particle size distribution. Fig. 8 depicts the particle size distribution of the granular sludge samples by image analysis based on computerised data of the respective V_{op}. In this experiment even if the settleability test was applied only to the upper sludge bed zone, nevertheless the particle size distribution from both upper and lower sludge bed zones was image-analysed for comparison. The result records the particle size distribution of the granules, showing the influence of sludge bed zone location and V_{op} changes. In the case of the lower sludge bed zone, the granule fraction with a diameter of below 2 mm diameter was reduced at higher V_{op} rates, whereas granules of over 2 mm increased in size under similar conditions. Even though this phenomenon might be due to the expansion of well-defined granules in the lower sludge bed zone, it may also be attributed to an increase in particle size of the whole granular bed, each at the high OLR of 28 kg COD·m^{-3}·d^{-1}.

Reactor performance of the UASB reactor

Although an increase of OLR from 5 kg COD·m^{-3}·d^{-1} (HRT 6.3 h) to 28 kg COD·m^{-3}·d^{-1} (HRT 1 h), caused COD removal to decrease from 93% to 60%, at the same time the unit methane production decreased only slightly to about 0.29 l CH_4·g^{-1} COD removal as shown in Fig. 9. Even if deterioration of effluent quality due to higher OLR should occur, this effect can be decreased if the reactor is operated with a more gradual increase in loading rate, using increments calculated by the settleability protocol. The granular sludge bed was maintained under stable conditions during all operating periods up to the very high operating OLR of 28 kg COD·m^{-3}·d^{-1} (67 kg COD·m^{-3} of granular bed volume·d^{-1}). During these experiments SS concentration in the effluent was 80 to 180 mg SS·l^{-1} and average influent SS concentration was 210 ± 70 mg·l^{-1}, as shown in Table 1.

Conclusions

The settleability assessment protocol for determining the operating upflow velocity and resultant organic loading rate in a laboratory-scale high-rate UASB reactor treating brewery wastewater was introduced and its applicability as an operating parameter was evaluated. Based on the settleability protocol, the OLR was increased up to 28 kg COD·m^{-3}·d^{-1} (67 kg COD·m^{-3} of granular bed volume·d^{-1}) which corresponds to an HRT of 1 h.

The results clearly reveal that the settling protocol is sufficiently sensitive to define the settleability of the granular sludge samples and is also useful to determine the minimum allowable upflow velocity, resultant OLR and recycle ratio according to the settleability of the granular bed. Also, a series of graphical procedures with settling tests is a very easily employed settleability monitoring technique as well as being simple, requiring little labor, and being economical. It can be used directly as an operational and monitoring parameter of the granular bed both with laboratory and full-scale reactors without other floating sludge bed controls. In addition, this method can be applied to improve and to monitor the system performance according to the wastewater characteristics, i.e. high or low-strength. Image analysis of the granular biomass supported the suitability of this graphical method. However, because the proposed method does not account for process kinetics, the estimation of the resultant OLR is valid only for the given reactor operating conditions in this research, and a re-calibration procedure would be required according to various other wastewater characteristics and operating conditions.

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