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Waste activated sludge hydrolysis and short-chain fatty acids accumulation in the presence of SDBS in semi-continuous flow reactors: Effect of solids retention time and temperature

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ABSTRACT

In the presence of surfactant sodium dodecylbenzene sulfonate (SDBS), the effects of sludge retention time (SRT) and temperature on the hydrolysis and short-chain fatty acids (SCFA) accumulation during waste activated sludge (WAS) anaerobic fermentation were investigated in semi-continuous flow reactors. Results showed that the presence of SDBS remarkably increased WAS hydrolysis and SCFA accumulation. A longer SRT or higher temperature accelerated WAS hydrolysis. The SCFA accumulation was increased as SRT increased from 3 to 12 d, but decreased as SRT further increased to 18 d. The presence of SDBS increased the total SCFA concentration remarkably, but did not change the SCFA composition significantly. The SCFA production was improved with the enhancement of temperature from 10 to 35 °C. During WAS fermentation in the presence of SDBS, the concentration of released NH_4^+-N increased linearly with SRT or temperature, but that of PO_4^{3-}-P remained relatively stable. Further investigation revealed that the presence of SDBS caused not only more total SCFA accumulation than the blank (1149.8 against 246.9 mgCOD/L), but also more volatile suspended solids reduction (33.8% versus 17.9%).

1. Introduction

It has been well recognized that both the biological removals of phosphorus and nitrogen from wastewater require carbon source, such as short-chain fatty acids (SCFA). When the influent COD is not enough for efficient wastewater nutrient removal, additional supply of carbon source is needed. Recently, the use of anaerobic fermentation liquid of waste sludge to improve biological nutrient removal (BNR) performance has been reported in the literature [1–6], by which both the reduction of sludge and the production of SCFA are accomplished. It is well known that all the following three stages, hydrolysis, acidification and methane production occurred during sludge anaerobic fermentation. In order to obtain more SCFA for BNR, the sludge hydrolysis need be improved, and at the same time the consumption of SCFA for methane production should be prevented.

During WAS anaerobic fermentation in short-term batch experiments, it has been observed that the production of SCFA from waste activated sludge (WAS) was improved significantly when surfactant (sodium dodecylbenzene sulfonate (SDBS) or sodium dodecyl sulfate (SDS)) was present [5,7]. However it is unclear whether the SCFA can be accumulated stably if the WAS fermentation is operated continuously because the wastewater treatment plant (WWTP) usually requires the continuous addition of SCFA to its facility.

The sludge retention time (SRT) and temperature are the two most important design parameters when sludge was treated anaerobically. Elefsiniotis and Oldham [8] observed that the distribution of SCFA was to some extent affected by the change of SRT during primary sludge digestion. The study of Miron et al. [9] revealed that the hydrolysis of lipids and carbohydrates of primary sludge increased with SRT. de la Rubia et al. [10] found that SRT has a considerable effect on the population levels of methanogens and the total VFA increased when SRT decreased. The investigation of Mahmoud et al. [11] showed that temperature had a significant influence on the hydrolysis of proteins, carbohydrates and lipids in primary sludge, and the hydrolysis at 35 °C was significantly higher than that at 25 °C.

The aim of this research was to investigate the influences of SRT and temperature on WAS hydrolysis and SCFA accumulation in the presence of surfactant SDBS during WAS anaerobically treated in semi-continuous flow reactors. As the waste activated sludge, instead of primary sludge was used in this article, significant amounts of phosphorus and nitrogen release were observed during

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Table 1
Characteristics of the concentrated WAS.

Parameter	Value
pH	6.55
TSS (total suspended solids) (mg/L)	12,121
VSS (volatile suspended solids) (mg/L)	8,289
SCOD (soluble chemical oxygen demand) (mgCOD/L)	135
TCOD (total chemical oxygen demand) (mgCOD/L)	12,035
Total protein (mgCOD/L)	7,277
Total carbohydrate (mgCOD/L)	909
Lipid and oil (mgCOD/L)	131

WAS fermentation in the presence of SDBS, the influences of SRT and temperature on the releases of orthophosphate (PO_4^{3-}-P) and ammonia nitrogen (NH_4^+-N) were examined. Finally, the volatile suspended solids (VSS) reduction in the presence and absence of SDBS was compared.

2. Materials and methods

2.1. Source of WAS

The WAS used in this study was obtained from the secondary sedimentation tank of a municipal WWTP in Shanghai, China. The sludge was concentrated by settling at 4 °C for 24 h and its main characteristics are listed in Table 1. Obviously, protein and carbohydrate are the two predominant organic compounds in WAS.

2.2. Reactor

The investigations of SRT and temperature on WAS hydrolysis and SCFA production in the presence of SDBS were carried out in a series of identical reactors, which were made of plexiglass and each had a liquid volume of 4.0 L. All reactors were equipped with stainless-steel stirrers with blades for mixing the contents. The first day of the experiments, 4.0 L fresh sludge was added to each reactor and SDBS was supplied with its dosage to dry fresh sludge ratio being 0.02 g/g according to our previous study [5]. The reactors were then operated semi-continuously, i.e. every day the WAS was wasted one time from the reactors and the same amount of fresh WAS was added according to the SRT. Also, the SDBS was supplied at a dosage of 0.02 g/g of dry fresh sludge.

To investigate the influence of SRT on WAS hydrolysis and SCFA accumulation as well as nitrogen and phosphorus releases, five semi-continuous flow reactors, which were maintained at room temperature (20 ± 1 °C), were operated. From reactor 1 to 4, the SRT was controlled respectively at 3, 6, 12 and 18 d. The reactor 5, used as the blank test (control), was operated at SRT 12 d with no SDBS addition.

To study the influence of temperature on WAS hydrolysis, SCFA accumulation and nitrogen and phosphorus releases, four semi-continuous flow reactors were operated at 15 ± 1, 20 ± 1, 30 ± 1, and 35 ± 1 °C, respectively. The SRT in all four reactors were controlled at 12 d.

After all reactors were run for more than 52 d, constant soluble protein and polysaccharide concentrations and SCFA level were observed and then the data were reported.

2.3. Analytical methods

The analyses of carbohydrate, protein, lipid, COD, PO_4^{3-}-P, NH_4^+-N, methane, suspended solids (SS) and VSS were the same as described in our previous publications [12,13].

For the quantification of SCFA, the filtrate was collected in a 1.5-mL gas chromatography (GC) vial, and acidified with 3% H_3PO_4 to pH 4.0 before assayed on an Agilent 6890N GC with flame ionization detector and DB-WAXETR column (30 m × 1.0 μm × 0.53 mm). The sample injection volume was 1.0 μL. Nitrogen was the carrier gas and the flux was 25 mL/min. The injection port and the detector was maintained at 220 and 250 °C, respectively. The oven of GC was programmed to begin at 110 °C and to remain there for 2 min, then to increase at a rate of 10 °C/min to 220 °C, and to hold at 220 °C for an additional 2 min.

3. Results and discussion

3.1. Effect of SRT on WAS hydrolysis in the presence of SDBS

It can be seen from Table 1 that protein and carbohydrate are the two predominant organic compounds in WAS. The effect of SRT on WAS hydrolysis expressed by the variations of observed soluble protein and carbohydrate concentrations is shown in Fig. 1. The concentrations of soluble protein and carbohydrate in the experiment with SRT of 12 d and SDBS addition were respectively 604.2 and 106.2 mgCOD/L (Fig. 1), whereas it was 187.8 and 45.4 mg/L in the blank experiment. The presence of SDBS therefore remarkably increased the hydrolysis of WAS in the semi-continuous flow reactor.

During anaerobic treatment of primary sludge, it has been reported that more sludge hydrolysis was observed at longer SRT [9,11]. As shown in Fig. 1, the concentrations of soluble protein and carbohydrate were respectively 470.2 and 65.5, 552.5 and 82.3, 604.2 and 106.2, and 659.5 and 121.2 mgCOD/L at SRT 3, 6, 12 and 18 d, which indicated that the effect of SRT on WAS hydrolysis was significant, and the increase of SRT resulted in the improvement of WAS hydrolysis. Further analysis showed that the influence of SRT on the observed soluble protein and carbohydrate concentrations could be expressed by Eq. (1) and (2), respectively.

$$y_{\text{protein}} = 96.9\ln(\text{SRT}) + 365.9 \quad (R^2 = 0.952) \tag{1}$$

$$y_{\text{carbohydrate}} = 29.2\ln(\text{SRT}) + 33.1 \quad (R^2 = 0.971) \tag{2}$$

The components of activated sludge are cemented together by extracellular polymeric substances (EPS), which are mainly composed of microbiologically produced polymers, such as carbohydrate and protein. It is well known that surfactant SDBS has the features of solubilization, which caused sludge protein and carbohydrate solubilized into the liquid phase. With the increase of SRT, SDBS had more time to contact with sludge EPS, and more sludge protein and carbohydrate were released. Thus, the hydrolysis of WAS in the presence of SDBS increased with SRT.

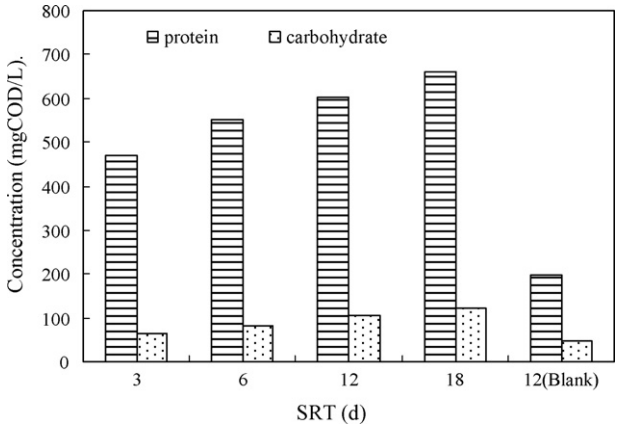

Fig. 1. Effects of SRT on the observed soluble protein and carbohydrate concentrations during WAS fermentation in the presence of SDBS.

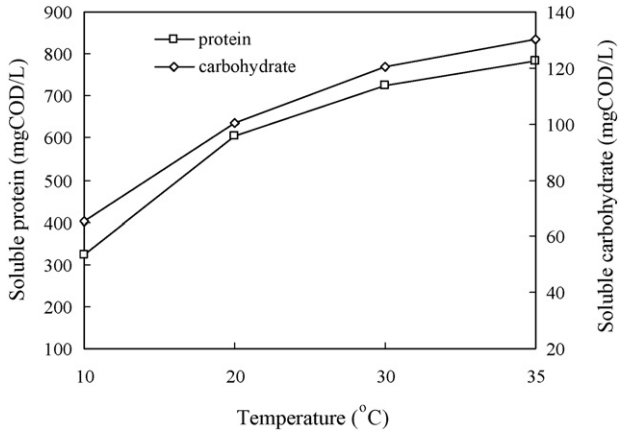

Fig. 2. Effects of temperature on the observed soluble protein and carbohydrate concentrations during WAS fermentation in the presence of SDBS.

3.2. Effect of temperature on WAS hydrolysis in the presence of SDBS at SRT 12 d

At SRT 12 d, the effect of temperature on the observed soluble protein and carbohydrate concentrations is shown in Fig. 2. With the increase of temperature from 10 to 20 °C, the concentrations of protein and carbohydrate were increased respectively from 323.1 to 604.2 mgCOD/L, and from 65.2 to 100.1 mgCOD/L. The increase rate was 28.1 mgCOD/L °C with protein, and 3.5 mgCOD/L °C with carbohydrate. When the temperature was raised from 20 to 30 °C, the increase rates of protein and carbohydrate were respectively 12.1 and 2.0 mgCOD/L °C. A further enhancement of temperature from 30 to 35 °C resulted in the improvement of soluble protein and carbohydrate concentrations, but the increase rates of both protein (11.7 mgCOD/L °C) and carbohydrate (1.9 mgCOD/L °C) showed a slight decrease compared with those between temperature 20 and 30 °C. It is obvious that the hydrolysis of WAS in the presence of SDBS was accelerated at higher temperatures, which was the same as that reported in the literature when primary sludge was hydrolyzed in the absence of SDBS. Ferreiro and Soto [14] observed that during the hydrolysis of primary sludge the concentration of soluble COD increased with temperature. Mahmoud et al. [11] also reported that primary sludge hydrolysis at 35 °C was significantly higher than that at 25 °C.

3.3. Effect of SRT on SCFA production in the presence of SDBS

In the presence of SDBS, the total SCFA concentration was observed to change with SRT during WAS continuous fermentation. The total SCFA (expressed by mgCOD/L) includes acetic, propionic, *n*-butyric, *iso*-butyric, *n*-valeric and *iso*-valeric acids, whose COD conversion factors are 1.07, 1.51, 1.82, 2.04, 1.82 and 2.04, respectively. Results showed that the total SCFA concentration was 834.7 mgCOD/L at SRT 3 d, which was respectively 1022.4 and 1149.8 mgCOD/L at SRT 6 and 12 d. Nevertheless, at SRT 18 d the total SCFA concentration decreased to 932.5 mgCOD/L. Thus, a pertinent increase of SRT improved the total SCFA production, and the suitable SRT for maximal SCFA accumulation during WAS anaerobic fermentation in the presence of SDBS was 12 d.

The total SCFA concentration in the blank test was 246.9 mgCOD/L, while in the presence of SDBS the total SCFA concentration was respectively 3.4 (SRT 3 d), 4.1 (SRT 6 d), 4.7 (SRT 12 d), and 3.8 (SRT 18 d) folds of that in the blank test. The result was in accordance with our previous study in short-term batch experiments [5]. The data in this study indicated that even in the long-term semi-continuous flow experiments the addition of SDBS to WAS anaerobic fermentation system could significantly improve SCFA accumulation.

According to the above study at different SRTs it is easy to understand that the reason for SCFA accumulation increased with SRT at a certain range (3–12 d) was that more soluble protein and carbohydrate (the substrates for fermentation to produce SCFA) were provided at longer SRT. However, with a further increase of SRT from 12 to 18 d, it was interesting to note that although the soluble protein and carbohydrate increased (Fig. 1), the SCFA concentration slightly declined. During anaerobic sludge digestion, de la Rubia et al. [10] also observed that the total SCFA decreased when SRT increased from 7 to 15 d. It might be attributed to the participation of SCFA consumers, such as methanogens at longer SRT. Miron et al. [9] reported that at SRT $\leq$ 8 d acidogenic conditions prevail while at SRT $\geq$ 10 d methanogenic conditions prevail. In this study it was observed that the production of methane at SRT 18 d (31.4 mL/g-VSS) was much greater than that at SRT 12 d (8.6 mL/g-VSS).

In the literature, acetic acid was reported as the main SCFA component during anaerobic fermentation of primary sludge [10,15,16]. In this study, the percentage of individual SCFA (acetic, propionic, *n*-butyric, *iso*-butyric, *n*-valeric and *iso*-valeric acids) accounting for total SCFA at different SRTs is shown in Fig. 3. It can be seen from Fig. 3 that acetic acid ranked the first at any SRT investigated, and its percentage increased with SRT. At SRT 3 d, the acetic percentage was 45.2%. With SRT increasing from 6 to 12 d, acetic fraction improved from 48.0 to 56.3%. When SRT was 18 d, the percentage of acetic increased to 58.8%. Eq. (3) shows the relationship between acetic percentage and SRT.

$$y_{\text{acetic-percentage}} = 8.1\ln(\text{SRT}) + 35.4 \quad (R^2 = 0.961) \tag{3}$$

As seen in Fig. 3, *iso*-valeric acid was the second SCFA, which accounted for 25.8, 24.2, 21.1 and 23.3% of total SCFA at SRT 3, 6, 12, and 18 d, respectively. Both the percentages of propionic and *iso*-butyric acids were around 9%. The lowest two SCFA were valeric and butyric acids. Their percentages were less than 5% at any SRT investigated. It seems that the influence of SRT on the fraction of other SCFA (propionic, *n*-butyric, *iso*-butyric, *n*-valeric and *iso*-valeric acids) was not significant.

From Fig. 3 it can also be seen that the fraction of branched SCFA (*iso*-butyric and *iso*-valeric) was much greater than their corresponding straight SCFA (*n*-butyric and *n*-valeric) at any SRT. The main reason might be that the decomposition rate of SCFA with a straight-chain (C2–C5) was greater than that of their respective isomer with a branched chain [17]. In short-term batch experiments it was also observed that the accumulation of *iso*-butyric

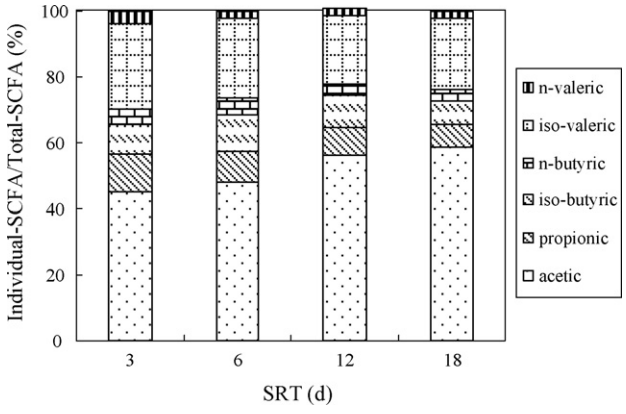

Fig. 3. Percentage of individual SCFA at different SRTs during WAS fermentation in the presence of SDBS.

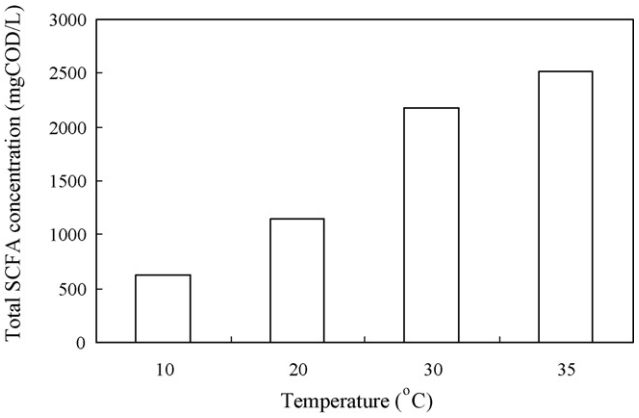

Fig. 4. Effect of temperature on total SCFA concentration during WAS fermentation in the presence of SDBS.

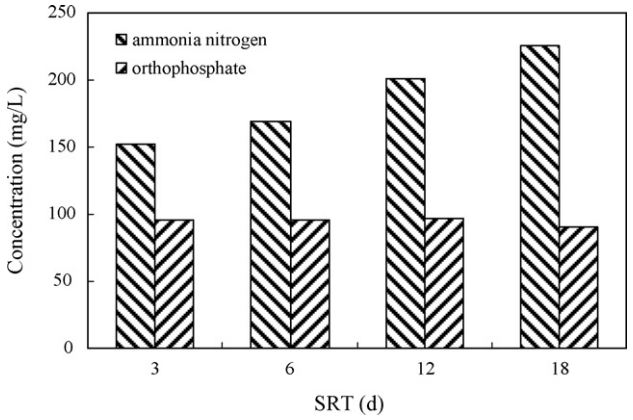

Fig. 5. Releases of ammonia nitrogen and soluble phosphorus during WAS fermentation in the presence of SDBS at various SRTs.

and *iso*-valeric acids were significantly higher than that of *n*-butyric and *n*-valeric acids after 6 d of WAS anaerobic fermentation in the presence of SDBS [5].

Acetic, propionic, *iso*-butyric and *n*-butyric acids may be formed directly from the fermentation of carbohydrates and proteins, but the higher molecular weight SCFA, such as *iso*-valeric and *n*-valeric acids are largely relevant to the fermentation of protein [18]. The content of protein in WAS was around 8-folds of that of carbohydrate in this study (Table 1), which suggested that a great deal of *iso*-valeric and *n*-valeric acids should be produced during WAS fermentation. As it has been discussed above that *n*-valeric acid was more easily biodegraded than *iso*-valeric acid in the anaerobic fermentation system, significant amount of *iso*-valeric was therefore accumulated.

In the blank test, the percentage of each SCFA was acetic 52.5%, propionic 8.4%, *iso*-butyric 9.7%, *n*-butyric 7.1%, *iso*-valeric 18.8% and *n*-valeric 3.5% (data not shown in Fig. 3). From Fig. 3 it can be seen that in the test with SRT 12 and SDBS addition, the percentage of acetic, propionic, *iso*-butyric, *n*-butyric, *iso*-valeric and *n*-valeric was 56.3, 8.5, 9.5, 3.4, 21.1 and 2.3%, respectively. Thus, the use of SDBS did not change the composition of SCFA significantly. Nevertheless, as discussed above, the presence of SDBS increased the total SCFA concentration remarkably (from 246.9 mgCOD/L in the blank test to 1149.8 mgCOD/L in the test with SRT 12 and SDBS addition).

3.4. Effect of temperature on SCFA accumulation in the presence of SDBS at SRT 12 d

At SRT 12 d and in the presence of SDBS the effect of temperature on the concentration of total SCFA accumulated in the semi-continuous flow system is shown in Fig. 4. The total SCFA was respectively 622.5, 1149.8, 2171.8, and 2522.4 mgCOD/L at 10, 20, 30 and 35 °C, which indicated that a higher temperature benefited SCFA production. It is well known that the formation of SCFA during sludge anaerobic treatment was associated with the fermentation of soluble organic substrates. As seen in Fig. 2, the soluble protein and carbohydrate concentrations were greater at higher temperature. Thus, the SCFA production increased with temperature.

In the absence of SDBS, other researchers also reported a greater SCFA accumulation at a higher temperature during anaerobic digestion of wastes. Maharaj and Elefsiniotis [19] studied the effect of temperature on the acid-phase anaerobic digestion of municipal and industrial wastewaters mixture, and observed that the SCFA production rate increased significantly with temperature between 8 and 25 °C. The study of Banerjee et al. [16] showed that the SCFA accumulation at 30 °C was greater than that at 22 °C when a mixture of primary sludge and starch-rich industrial wastewater was digested.

3.5. The releases of phosphorus and ammonium during WAS fermentation in the presence of SDBS at different SRTs and temperatures

During WAS fermentation in the presence of SDBS in the semi-continuous flow reactors, the releases of sludge phosphorus and ammonium were observed. Fig. 5 shows the influence of SRT on the observed concentrations of ammonia nitrogen and orthophosphate at 20 ± 1 °C. The concentration of NH_4^+-N at SRT 3, 6, 12 and 18 d was respectively 152.2, 169.4, 201.5 and 225.8 mg/L, which indicated that the NH_4^+-N release increased linearly with SRT ($y = 4.9\ SRT + 139.2$, $R^2 = 0.994$). In the anaerobic fermentation ammonia is produced by biological degradation of nitrogenous substrates, mostly in the form of sludge protein in this study, which is hydrolyzed to amino acids and further degraded to ammonia. As the WAS hydrolysis was enhanced by the extension of SRT (see Fig. 1), the release of NH_4^+-N was therefore increased with SRT.

The above investigation has shown that with the increase of SRT the hydrolysis of WAS was enhanced. The phosphorus release should be increased with SRT. However, the observed orthophosphate concentration at different SRTs remained relatively stable

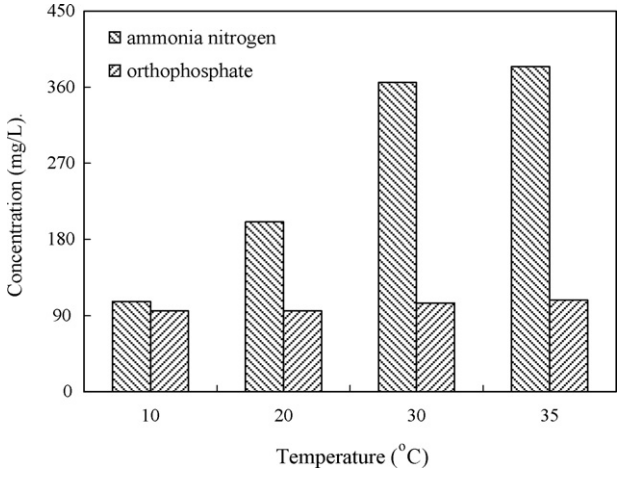

Fig. 6. Releases of ammonia nitrogen and soluble phosphorus during WAS fermentation in the presence of SDBS at various temperatures.

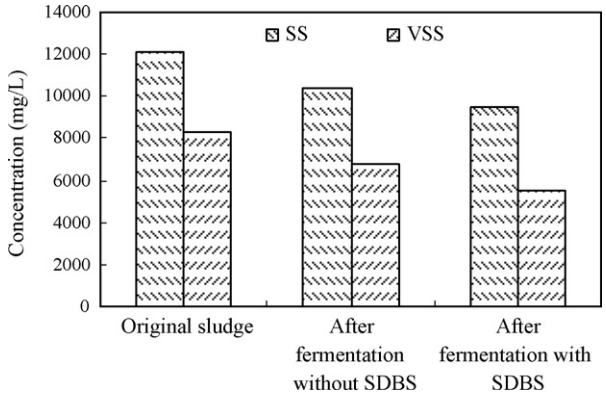

Fig. 7. Comparison of SS and VSS reduction during WAS fermentation in the presence and absence of SDBS at SRT 12 d and 20 °C.

(Fig. 5). One possible reason for this observation was that some heavy metals, which also released during WAS fermentation, reacted with the released phosphorus [20].

Fig. 6 illustrates the effect of temperature on the releases of ammonia nitrogen and soluble phosphorus during WAS fermentation in the presence of SDBS at SRT 12 d. The changes of NH_4^+-N and PO_4^{3-}-P concentrations with temperature were the same as those with SRT. The increase of temperature caused a significant NH_4^+-N release, but gave little impact on the observed PO_4^{3-}-P concentration.

It is generally believed that ammonia is one of inhibitory substances when it is present in substantial concentrations in anaerobic reactor [21]. In this study, the average of released ammonia varied between 106.5 and 385.2 mg/L at all runs. It seems that the released ammonia might not negatively influence the anaerobic microorganisms. The inhibitory ammonia concentrations are from 1.7 to 14 g/L at different environmental (temperature, pH, etc.) conditions [22], and ammonia concentrations below 200 mg/L are even beneficial to anaerobic process as an essential nutrient for anaerobic microorganisms [23]. The coming studies, we will examine the influence of released ammonia and phosphorus on SCFA accumulation during WAS fermentation in the presence of SDBS.

3.6. The VSS reduction at SRT 12 d and room temperature with or without SDBS addition

When WAS was anaerobically fermented in the presence of SDBS in the semi-continuous flow reactor, the reductions of SS and VSS were observed. Fig. 7 shows the SS and VSS variations before and after WAS fermentation in the presence and absence of SDBS at SRT 12 d and temperature 20 °C. The SS and VSS of original sludge were 12,121 and 8289 mg/L, respectively. After fermentation in the presence of SDBS, the SS was 9478 mg/L, and VSS was 5486 mg/L, which were respectively 10,402 and 6801 mg/L in the absence of SDBS. The reduction of VSS in the presence of SDBS was 33.8%, whereas it was 17.9% in the blank test. It can be said that during the anaerobic fermentation of WAS the presence of SDBS not only improved the production of SCFA, but also accelerated the reduction of WAS.

4. Conclusion

In the semi-continuous flow reactors, the effects of SRT and temperature on WAS hydrolysis and SCFA accumulation in the presence of SDBS were investigated. With the increase of either SRT or temperature the hydrolysis of WAS was increased. A pertinent increase of SRT improved the total SCFA production, and the maximal SCFA accumulation was observed at SRT 12 d, which was 4.7-fold of the blank test (in the absence of SDBS). Also, it was observed that the total SCFA was increased with temperature.

Acetic acid was the most prevalent product at any SRT, and its percentage increased with SRT. The influence of SRT on the fraction of other SCFA was not significant. *iso*-Valeric acid was the second SCFA, and the lowest two SCFA were valeric and butyric acids. The fraction of branched SCFA (*iso*-butyric and *iso*-valeric acids) was much greater than their corresponding straight SCFA (*n*-butyric and *n*-valeric acids) at any SRT. Nevertheless, compared to the blank test, the use of SDBS did not change the composition of SCFA significantly.

The increase of SRT or temperature caused a significant increase of NH_4^+-N release, but gave little impact on the observed PO_4^{3-}-P concentration. Furthermore, it was found that 33.8% of VSS was reduced in the presence of SDBS at SRT 12 d and 21 °C, whereas the VSS reduction was only 17.9% in the blank test. According to this study it can say that the anaerobic fermentation of WAS in a semi-continuous flow reactor in the presence of SDBS can accomplish both the SCFA production and WAS reduction efficiently.

Acknowledgements

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