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Effect of sodium dodecyl sulfate on waste activated sludge hydrolysis and acidification

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Abstract

The effect of sodium dodecyl sulfate (SDS) on hydrolysis and acidification of waste activated sludge (WAS) was investigated, which has not been documented in the literature. Results showed that SDS improved the solubility of protein and carbohydrate in aqueous phase, and the concentrations of these two substrates increased with the amount of SDS. On the second day of fermentation the enzyme activities of protease and alkaline phosphatase were improved by SDS with its dosage in the range of 0.02–0.3 g/g, but the activities of α-glucosidase and acidic phosphatase decreased at SDS dosage above 0.05 and 0.2 g/g, respectively. The volatile fatty acids (VFAs) production was also enhanced by SDS under room temperature. On the sixth day of fermentation, concentration of total VFAs was 2243.04 mg COD/L at SDS dosage 0.1 g/g, while it was only 191.10 mg COD/L in the blank test. The maximum VFAs production increased with the amount of SDS. However, longer time was needed for WAS treated with higher SDS dosage to reach its own maximum VFAs production. The composition of VFAs was analyzed, and acetic acid was observed the most prevalent product. Further studies showed that methanogenesis was inhibited in the presence of SDS. Almost the same pH variations in the presence and absence of SDS indicated that the improvement of VFAs production and inhibition of methane production in WAS fermentation was more due to the effects of SDS than due to the variations of pH.
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Keywords: Waste activated sludge (WAS); Hydrolysis; Acidification; Volatile fatty acids (VFAs); Sodium dodecyl sulfate (SDS)

1. Introduction

Biological wastewater treatment has been used widely in the world, but large amounts of sludge (including primary sludge and waste activated sludge) are produced in this process. Rapid urbanization in many areas of the world has resulted in a drastic increase of wastewater sludge with a typical person generating over 50 g of dry solids every day [1]. These sludges have to be stabilized in order to reduce biological activity in the sludge and thereby reduce odor production and prevent or slow the release of harmful chemicals into environment.

Anaerobic digestion is a widely applied method for sludge stabilization due to biogas production which can be used as energy. Usually, three stages are involved in anaerobic digestion process, hydrolysis, acidification and methanogenesis. Volatile fatty acids (VFAs) are important intermediate products in the anaerobic digestion. Recently, researchers pay more attention to the formation of VFAs in sludge hydrolysis and acidification process since VFAs are required for biological nutrient removal (BNR) [2–5]. It has been reported that 6–9 mg of VFAs are required for biological removal 1 mg of phosphorus [6]. However, sufficient concentrations of VFAs in the wastewater allow for low phosphorus residuals (<1.0 mg/L as P) in the effluent are not always available in the influent [7], particularly when the wastewater COD is low. Thus, additional supply of VFAs becomes necessary in order to keep high and steady phosphorus removal performance. In addition, the production of VFAs from sludge may also provide a renewable carbon source for the synthesis of polyhydroxyalkanoates, biodegradable plastics [8].

Although direct addition of chemical synthesized VFAs to influent has been proved to be an efficient method to improve BNR performance [9,10], in order to minimize the operating cost of supplementary carbon dosing and reuse the organic compounds in waste sludge for reducing its environment pollution, some researchers conducted the studies of VFAs fermentation from primary sludge or its mixture with waste activated sludge [11–13]. Waste activated sludge (WAS) from wastewater treatment plant contains high levels of organic matter and thus may

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become a plentiful source of inexpensive organic substrate for fermentative VFAs production, by which reduction and stabilization of organic wastes can also be accomplished.

It is believed that the initial hydrolysis of particulate organic matter to soluble substance is the rate-limiting step of anaerobic digestion [14]. If the particulate organic matter contained in an activated sludge is not properly solubilized, only 30–50% of the total COD or volatile solids (VS) in WAS is biodegraded in 30 days [15]. Thus many kinds of treatment processes are used to enhance the efficiency of anaerobic digestion by improving the rate of sludge hydrolysis, which include thermal, thermochemical, mechanical, ultrasonic and enzymatic methods [16].

In the previous studies, it was found in our investigations that some surfactants can alter microorganism cell structure by making cell materials (CM) leave the attached surface and dissolve them in aqueous solution [17–19]. Thus, surfactant may accelerate the rate of sludge hydrolysis, since there are many microorganisms and many CM-like materials adhering to sludge surface. The purpose of this work was firstly to investigate the influence of sodium dodecyl sulfate (SDS) on WAS solubilization and VFAs production during sludge fermentation. Then the variations of activities of several hydrolytic enzymes in WAS fermentation were examined. Finally, the effect of SDS on methanogenesis was investigated. As to our knowledge, the influence of surfactant on WAS hydrolysis and acidification has not been documented in the literature.

2. Materials and methods

2.1. Source of WAS and operation

The WAS used in this study was obtained from the secondary sedimentation tank of a municipal wastewater treatment plant in Shanghai, China. The sludge was concentrated by settling at 4 °C for 24 h, and its characteristics after settlement are shown in Table 1. Apparently, the protein and carbohydrate are the two predominant organic compounds in WAS, which account for about 73% of the total COD (TCOD).

Experiments of influence of SDS on WAS hydrolysis and acidification were carried out in 16 identical reactors, which were made of plexiglass and each had a liquid volume of 2.0 L. All reactors were equipped with stainless steel stirrers with blades for mixing the contents, and were maintained at 21 ± 1 °C. SDS was added to the reactors with its dosage to dry sludge ratio being 0, 0.02, 0.05, 0.1, 0.15, 0.2, 0.25 and 0.3 g/g, respectively. The reactor with no SDS addition was used as the blank test. A duplicate reactor was prepared for each SDS dosage test: one reactor was used for liquid sampling, and another one for methane sampling.

Table 1
Characteristics of the concentrated WAS used in this investigation[a]

Parameter	Value
pH	6.86 ± 0.16
TSS (total suspended solids) (mg/L)	11036 ± 151
VSS (valtile suspended solids) (mg/L)	9531 ± 97
SCOD (soluble chemical oxygen demand) (mg/L)	118 ± 17
TCOD (total chemical oxygen demand) (mg/L)	14890 ± 560
Carbohydrate (mg COD/L)	1085 ± 105
Protein (mg COD/L)	9874 ± 431
Lipid and oil (mg COD/L)	152 ± 6

[a] COD mass equivalent of carbohydrate, protein, and lipid and oil was 1.07, 1.50 and 2.91 g COD/g [20].

To examine whether the degradation of SDS produced VFAs during sludge fermentation, batch fermentation tests with synthetic wastewater using SDS as the carbon source and sludge as innoclum were conducted. SDS of 50, 200, 1000 and 3000 mg was dissolved into 950 mL of tap water, respectively, and 50 mL aliquot of WAS was added to each reactor with a final sludge concentration of 500 mg/L. The batch reactors were maintained at 21 ± 1 °C.

2.2. Analysis

Sludge samples from the reactors were immediately filtered through Whatmann GF/C glass microfiber filter. The filtrate was analyzed for VFAs, carbohydrate and protein, and the filter was assayed for TSS and VSS. The analyses of TSS and VSS were conducted in accordance with Standard Methods [21]. Carbohydrate was measured by the phenol–sulfuric method with glucose as standard [22]. Soluble protein was determined by the Lowry–Folin method with bovine serum albumin (BSA) as standard [23].

Sludge lipid was extracted by the Bligh–Dyer method from the acidified sample, and was then measured gravimetrically after the solvent was evaporated at 80 °C [21]. The total protein content of sludge was estimated from the corresponding TKN concentration by subtracting the inorganic nitrogen concentration and dividing the difference by 0.16, then multiplying the result by 1.5 finally [20]. The activities of sludge enzymes (alkaline and acid phosphatases, α-glucosidase and protease) were measured according to Goel et al. [24].

VFAs were analyzed by HP5890 GC with flame ionization detector and equipped with a 30 m × 0.32 mm × 0.25 mm CPWAX52CB column. Nitrogen was the carrier gas and the flux was 50 mL/min. The injection port and the detector were maintained at 200 and 220 °C, respectively. The oven of the GC was programmed to begin at 110 °C and to remain there 2 min, then to increase at a rate of 10 °C/min to 200 °C, and to hold at 200 °C for 2 min.

Methane concentration was measured by a gas chromatograph (GC-14B, Shimadzu, Japan) equipped with a thermal conductivity detector (TCD) and a 3-m stainless column. The temperature of the injection, column, and detector was set at 40, 50 and 90 °C, respectively. Nitrogen was used as the carrier gas at a flow rate of 30 mL/min. The inhibition rate of methane production in the presence of SDS was calculated by the following formula:

$$\mathrm{IR}\,(\%) = \frac{A - B}{A} \times 100\%$$

where IR is the inhibition rate of methane production (%), A the methane production in the absence of SDS (i.e. in the blank test) on the last day of fermentation (mL/g VSS) and B is the

methane production in the presence of SDS on the last day of fermentation (mL/g VSS).

3. Results and discussion

3.1. Effect of SDS on protein and carbohydrate

Protein, carbohydrate and lipid are the main constituents of domestic sludge [25]. In this study the WAS consisted of almost 66% protein, 7% carbohydrate, 1% lipid, and about 26% unknown components on the basis of sludge TCOD. Thus, protein was the largest constituent of the WAS, and the lipid content could be neglected in this study. Hydrolysis of WAS causes sludge protein and carbohydrate solubilized in aqueous phase, and the variations of soluble protein and carbohydrate concentrations at different SDS dosages were therefore investigated. Figs. 1 and 2 describe the effects of SDS and fermentation time on soluble protein and carbohydrate. It was observed that concentrations of these two substrates increased with the amount of SDS. On the sixth day of fermentation, protein and carbohydrate concentration was, respectively, 341.81 and 51.59 mg/L in the blank test, 827.73 and 157.63 mg/L at SDS dosage 0.1 g/g, and 1372.91 and 220.98 mg/L at SDS dosage 0.3 g/g. The solubilization of sludge particulate organic-carbon was significantly improved by SDS.

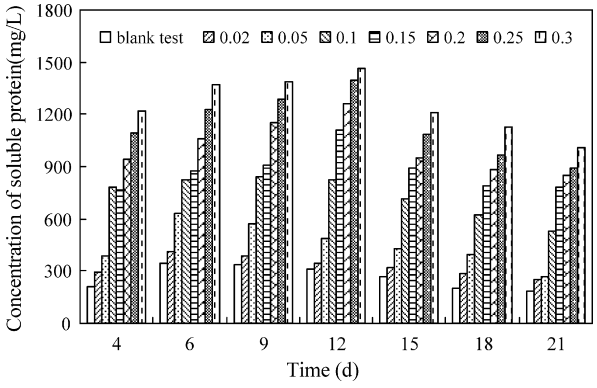

Fig. 1. Effect of SDS dosage on soluble protein concentration at different fermentation time.

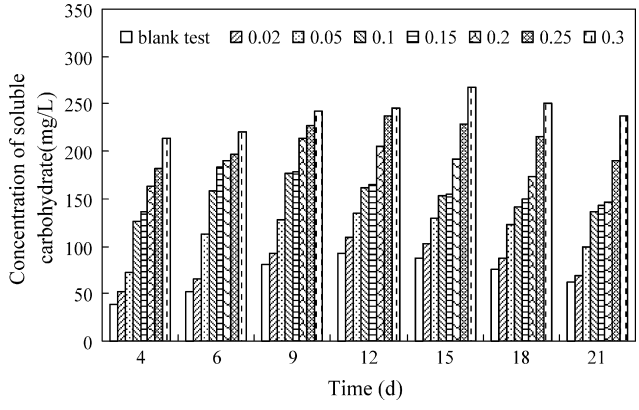

Fig. 2. Effect of SDS dosage on soluble carbohydrate concentration at different fermentation time.

It is well known that sludge components are cemented together by extracellular polymeric substances (EPS), which are mainly composed of microbiologically produced biopolymers, such as carbohydrate and protein [26]. Usually, these sludge protein and carbohydrate are absorbed onto sludge surface, but they can be solubilized by surfactant and then dissolve into aqueous phase because surfactant has the feature of solubilization [27]. Also, the enhanced solubilization of sludge EPS by SDS caused the break-up of sludge matrix, which resulted in more sludge inner protein and carbohydrate released. The improvement of sludge protein and carbohydrate solubilization by surfactant has also been observed in our previous studies [19].

As also seen in Figs. 1 and 2, the concentrations of both protein and carbohydrate increased gradually in the initial stage of fermentation, but decreased in the latter fermentation time. It seems that the initial release rates of these two substrates were higher than their degradation, which made their temporary accumulations, but in the latter stage of fermentation the release rates slowed down and were exceeded by degradation, which resulted in the decrease of observed soluble protein and carbohydrate concentrations.

3.2. Effect of SDS on enzyme activities

The hydrolytic enzymes such as protease and α-glucosidase that break down protein to peptidase and α-1,4-glucosidate link in carbohydrate were monitored in this study. Also acid phosphatase and alkaline phosphatase which hydrolyze phosphate esters and release the phosphate groups were examined. The relative activities of these four enzymes on the second day of fermentation are shown in Fig. 3, in which the relative activity of each enzyme in the blank test was set as 100%. As seen from Fig. 3, protease activity was remarkably improved by SDS with its dosage increasing from 0.02 to 0.3 g/g. The protease activity showed a 1.85-fold increase at SDS dosage 0.05 g/g. This increased to 2.24-fold at SDS dosage 0.2 g/g, while further increasing SDS dosage resulted in a little decrease in protease activity. As to α-glucosidase, lower SDS dosage was benefit for the enhancement of its activity. For example, SDS dosage 0.02 g/g made a 1.12-fold increase in α-glucosidase activity. However, it was observed that α-glucosidase activity

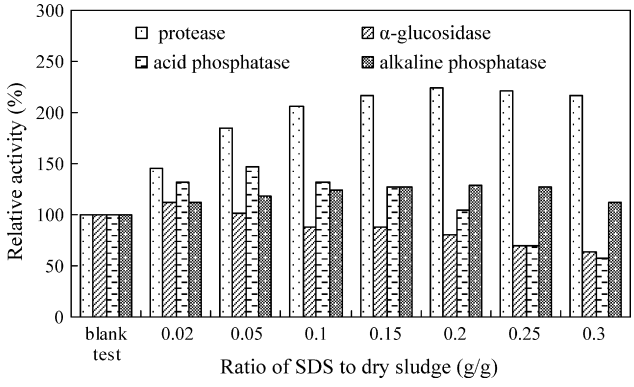

Fig. 3. Enzyme activities of WAS treated with different SDS dosages on the second day of fermentation.

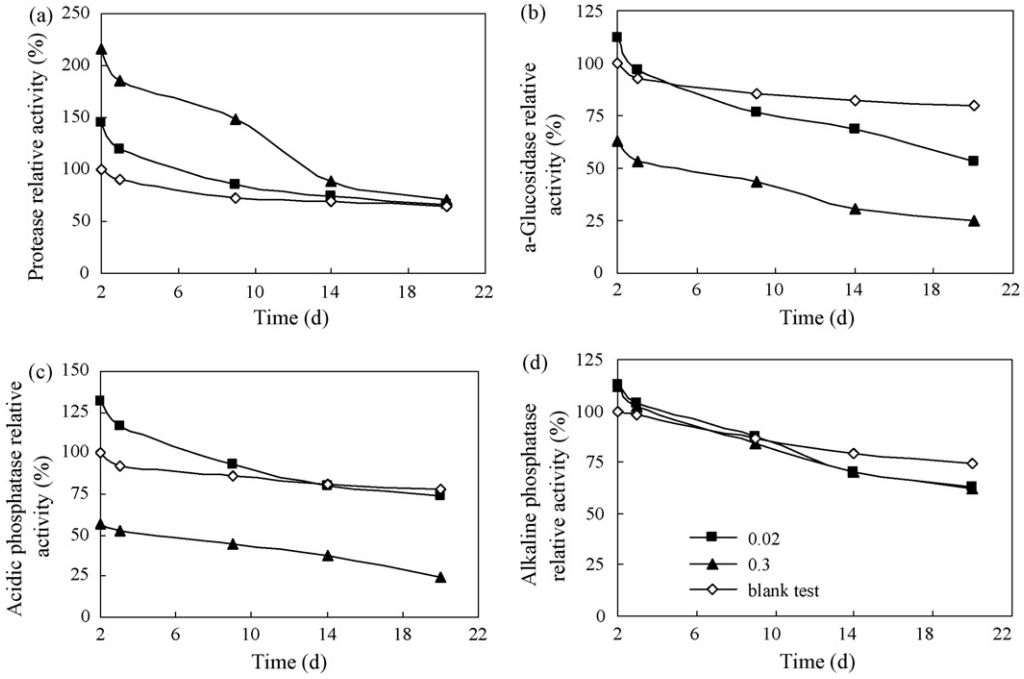

Fig. 4. Time course of relative activities of four hydrolytic enzymes under different fermentation conditions ((a) protease; (b) α-glucosidase; (c) acidic phosphatase; (d) alkaline phosphatase).

was inhibited at SDS dosage above 0.05 g/g, and the activity almost linearly declined from 88.1% at 0.1 g/g to 63.0% at 0.3 g/g. SDS has the same effect on acidic phosphatase as on α-glucosidase, but the inhibition effect occurred at SDS dosage exceeding 0.2 g/g. It is noticeable in Fig. 3 that alkaline phosphatase activity was not significantly influenced by SDS. With SDS dosage increasing from 0.02 to 0.2 g/g, the relative alkaline phosphatase activity increased from 113 to 129%, however, further increasing SDS dosage to 0.3 g/g caused the reduction of relative activity to 112%.

Generally, lower amount of SDS (such as 0.02 g/g) could improve these four enzymes activities. With SDS dosage increasing from 0.05 to 0.2 g/g, the enzyme activities of protease and alkaline phosphatase increased gradually, but activities of α-glucosidase and acidic phosphatase decreased. Too much SDS (such as 0.3 g/g) decreased activities of α-glucosidase and acidic phosphatase seriously.

Variations of these four enzymes activities with fermentation time were also investigated in this study. Here only data with SDS dosage 0.02 and 0.3 g/g were shown as examples (at other SDS dosages the same variations trend were observed). The data in the blank test were also included in Fig. 4 for comparison. Obviously, the hydrolytic enzymes activities of both raw WAS and SDS-treated WAS declined with time, but the latter decreased faster than the former. As to the protease, due to the initial enzyme activity (the second data in Fig. 4a) of SDS-treated WAS was much greater than that of the raw WAS, the former kept a higher activity than the latter till the 14th day of fermentation. In the latter stage of fermentation, as seen in Fig. 4a the protease activity of SDS-treated WAS on the 20th day was almost equal to that of the raw WAS. However, the enhanced activities of α-glucosidase, acidic and alkaline phosphatase were even lower than the activities of the raw WAS in the latter stage of fermentation (Fig. 4b–d). Apparently, the hydrolytic enzymes activities were promoted in the presence of SDS in short time, but the activities decreased faster with the increase of time than those in the blank test, and too long time even caused lower activities.

3.3. Effect of SDS on total VFAs

The effects of SDS dosage and fermentation time on total VFAs production are shown in Fig. 5. The individual VFA was converted to COD using appropriate conversion factors [14]. It was observed that total VFAs production was greatly improved by SDS. The maximum VFAs concentration increased gradually as the ratio of SDS to dry sludge increased from 0.02 to 0.3 g/g. However, longer fermentation time was required to reach the

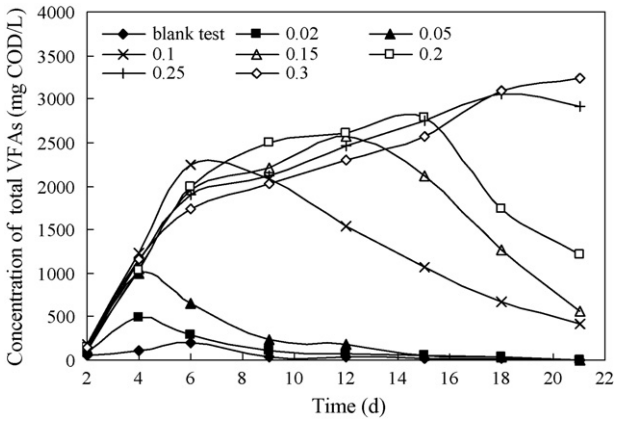

Fig. 5. Effects of SDS dosage and fermentation time on total VFAs production.

maximum VFAs concentration in the case of higher SDS dosage. For example, the maximum VFAs concentration was 191.10 mg COD/L on the 6th day in the blank test, 1000.74 mg COD/L on the 4th day at SDS dosage 0.05 g/g, 2243.04 mg COD/L on the 6th day at 0.1 g/g, and 3066.23 mg COD/L on the 18th day at 0.25 g/g. The less VFAs produced at higher SDS dosage during the initial stage of fermentation might be attributed to the toxic effects of surfactant to acidogenic bacteria [28]. Also, as shown in Fig. 5 an obvious VFAs consumption was observed with the increase of time at SDS dosage less than 0.2 g/g due to the participation of VFAs consumers, such as methanogens. But higher SDS dosage, such as 0.25 and 0.3 g/g, could reduce or inhibit the methanogens activity, which will be discussed in the following text.

3.4. Composition of VFAs

Six kinds of VFAs, including acetic, propionic, *n*-butyric, *iso*-butyric, *n*-valeric and *iso*-valeric acids were detectable in this study. Table 2 shows the composition of VFAs at different SDS dosages on the sixth day of fermentation. Obviously, acetic acid was the most prevalent product. In most cases of this study, *iso*-valeric acid was the second major product, which was followed by propionic acid. These three products represented more than 80% of the total VFAs in all experiments. As seen in Table 2, in the blank test the fraction of acetic > propionic > *iso*-valeric, which has also been observed by other researchers [29]. Wang et al. [29] reported that the accumulation of individual VFA in WAS digestion process was in the following order: acetic > propionic > *iso*-valeric > *iso*-butyric > (*n*-valeric, *n*-butyric), no matter which type of sludge pretreatment method (ultrasonic, thermal or freezing) was used. However, as discussed above the different observation was made when SDS was used in this study. It can be seen in Table 2 that in the presence of SDS the order was acetic > *iso*-valeric > propionic.

It was reported that SDS can be degraded via β-oxidation pathway under anaerobic conditions and the VFAs can also be produced [30]. Thus, it was possible that some of the VFAs produced during WAS fermentation in the presence of SDS came from the degradation of SDS. Right now, however, it is quite difficult to distinguish which VFAs were produced from WAS or from SDS degradation even if the ^{14}C-labeled SDS test were conducted, which is a technology to investigate the microbial metabolism, because there is only one carbon labeled in the available ^{14}C-labeled SDS from the market. After the first β-oxidation, ^{14}C would be in an acetic acid molecular, and then ^{14}C could not give any more information about the degradation of the residual SDS molecular. Hence, batch fermentation tests about whether the degradation of SDS produced VFAs were conducted. The experimental results are shown in Table 3. From the data in Table 3, it seems that little VFAs were produced through the degradation of SDS in our experiments.

Table 3
Amount of VFAs produced from SDS under anaerobic conditions (mg COD/L)

SDS concentration (mg/L)	Time (days)			
	2	6	12	21
50	6.2	13.1	8.9	1.1
200	7.8	16.3	9.5	2.7
1000	9.6	17.4	11.2	8.0
3000	11.7	20.6	18.3	16.9

3.5. Effect of SDS on methane production

Usually, hydrolysis, acidification and methanogenesis all occur in sludge fermentation. VFAs, the products of acidification, are the substrates for methanogenesis and can be easily metabolized to methane by methanogens under proper conditions. Fig. 6 shows the effect of SDS on methanogens activity expressed by inhibition rate of methane production at fermentation time of 21 days. Data in Fig. 6 demonstrate that methane was actually produced in the presence of SDS. However, with SDS dosage increasing from 0.02 to 0.3 g/g, the inhibition rate of

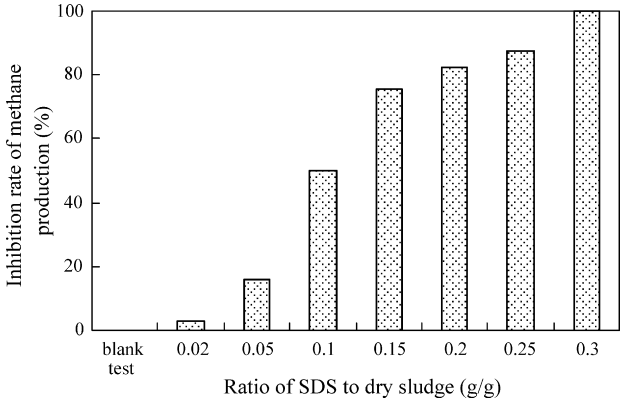

Fig. 6. Effect of SDS dosage on methanogens activity at fermentation time of 21 days.

Table 2
Composition of VFAs at different SDS dosages on the sixth day of fermentation

SDS/dry sludge (g/g)	Acetic (%)	Propionic (%)	*iso*-Butyric (%)	*n*-Butyric (%)	*iso*-Valeric (%)	*n*-Valeric (%)
Blank test	48.38	26.94	6.26	5.13	9.12	4.17
0.02	52.29	12.74	8.79	5.70	17.06	3.43
0.05	53.14	13.62	6.83	6.01	17.22	3.18
0.1	53.33	11.54	4.72	11.56	16.63	2.21
0.15	52.58	10.40	7.15	9.19	18.54	2.14
0.2	57.40	12.67	2.55	9.58	16.12	1.69
0.25	55.65	9.16	4.19	10.42	18.79	1.79
0.3	53.18	13.45	2.85	10.71	17.57	2.24

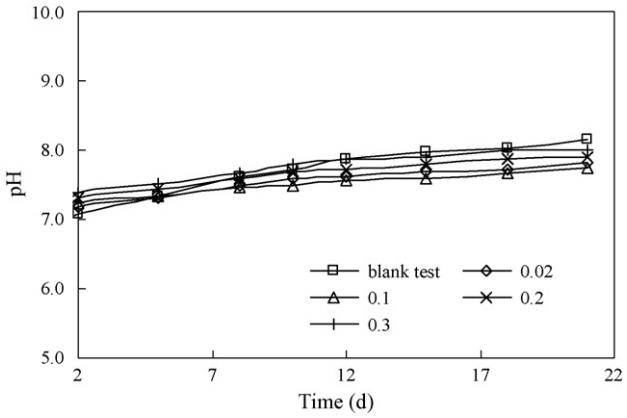

Fig. 7. pH variations at different SDS dosages during sludge fermentation.

methane production increased sharply from 3.00 to 100%. The increase of SDS dosage resulting in the decrease of methane production could be made at any other fermentation time (data not shown). Apparently, the activity of methanogens was inhibited by surfactant, which has also been observed by other researchers [31–33].

3.6. pH variations

It is well known that pH has influence on VFAs production and methanogens, thus pH variations during 21-day time of sludge fermentation were recorded. Data with SDS dosage of 0.02, 0.1, 0.2 and 0.3 g/g and in the blank test are shown in Fig. 7 (at other SDS dosages the same variations were observed). It can be seen from Fig. 7 that pH value of sludge with and without SDS all kept increasing. Although VFAs were produced, the released sludge ammonia (55.93, 68.37, 208.31, 239.41 and 348.26 mg/L in the blank test and in SDS dosage of 0.02, 0.1, 0.2 and 0.3 g/g tests, respectively) compensated for the decrease of pH. Thus, the observed pH values in all tests increased. However, as seen from Fig. 7, pH of sludge in the presence and absence of SDS had almost the same variations. Thus, the improvement of VFAs production and the inhibition of methane production in WAS fermentation was more due to the effects of SDS than due to the variations of pH.

4. Conclusion

The hydrolysis and acidification of WAS were influenced by SDS. It was observed that SDS effectively accelerated the solubilization of WAS, which resulted in the increase of protein and carbohydrate concentrations in aqueous phase. It was also observed that lower amount of SDS (such as 0.02 g/g) could improve the enzyme activities of protease, α-glucosidase, acidic and alkaline phosphatase. Further increasing SDS dosage to 0.2 g/g, the activities of protease and alkaline phosphatase increased gradually, but activities of α-glucosidase and acidic phosphatase decreased. VFAs production was significantly enhanced in the presence of SDS. And the maximum VFAs production increased with the amount of SDS. The reason for large amount of VFAs accumulation in the presence of SDS was due to the improvement of hydrolysis of sludge protein and carbohydrate and the inhibition of methanogenesis by SDS rather than SDS degradation and pH variations.

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