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Effect of high pressure homogenization on anaerobic digestion of the sludge pretreated by combined alkaline and high pressure homogenization

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ABSTRACT

In order to improve the efficiency of anaerobic sludge digestion, alkaline and high pressure homogenization (HPH) were combined to pre-treat the excess activated sludge. The effect of HPH operating parameters, including homogenization pressure and cycle number, on the performances of anaerobic sludge digestion was studied. The results demonstrated that the performances of sludge disintegration and anaerobic digestion were markedly enhanced by increasing the homogenization pressure. After pretreatment at a homogenization pressure of 60 MPa with one homogenization cycle combined with an alkaline dosage of 0.04 mol/L, the sludge TCOD, VS removal and cumulative biogas production in a mesophilic anaerobic digestion system increased by 24.68%, 18.95% and 95.81%, respectively, in comparison with that with the alkaline pretreatment alone. But the sludge disintegration and biogas production only slightly increased with the increase of homogenization cycle. Considering biogas production and energy-saving, the suitable homogenization operation was selected as homogenization pressure of 60 MPa with once cycle. Relationships between methane yield and sludge disintegration showed that the improved methane production was mainly attributed to the sludge disintegration resulted from combined sludge pretreatment.

Keywords: Anaerobic digestion; High pressure homogenization; Alkaline pretreatment; Biogas production; Combined pretreatment

1. Introduction

Sewage sludge produced from wastewater treatment plants (WWTPs) causes potential environmental risks and high disposal cost. In China, millions of tons of sewage sludge with high water content have been generated annually, which results in a huge burden to environment and society [1]. Anaerobic digestion is considered to be a cost-effective method for sludge stabilization and volume reduction. Additionally, biogas (e.g., methane) generated in the anaerobic digestion process is a source of renewable energy [2,3]. The biogas production in the anaerobic digestion process

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includes three key stages: solids hydrolysis, fermentation of complex organics to hydrogen and volatile fatty acids, and subsequent conversion of these simple compounds to biogas [4]. Usually, slow and incomplete hydrolysis of extracellular polymeric substances (EPS) and microbial biomass is recognized as the rate-limiting step during the anaerobic digestion, which leads to a long sludge retention time and low organic degradation [5,6]. However, the sludge hydrolysis can be enhanced by sludge pretreatment, because the sludge pretreatment can normally disrupt the sludge flocs and microorganism cells and release the extracellular and intracellular materials. As a result, the sludge biodegradability and methane production are enhanced [7]. Several sludge pretreatment approaches have been studied, including chemical [8], thermal [9], mechanical [10], as well as combined pretreatment methods [11].

Alkaline pretreatment is a relatively efficient method, and the order of disintegration efficacy is: NaOH > KOH > $Mg(OH)_2$ and $Ca(OH)_2$ [12]. Alkaline sludge pretreatment can disrupt flocs and cells, release inner organic matters and make the cellular substance more susceptible to enzymes, and consequently improve the performances of anaerobic digestion [13,14]. However, extreme high pH leads to low biodegradability performances, probably due to the formation of refractory compounds through intermolecular interactions between solubilized compounds [15]. In addition, a high concentration of Na^+ or K^+ may inhibit methanogens [16]. Recently, this method has often been combined with other disintegration methods to avoid these problems and to achieve better digestion performances [17,18].

High pressure homogenization (HPH) treatment is an effective approach for cell disruption using mechanical forces. The technology was initially used in the food and dairy processing industries [19]. Application of HPH to sludge pretreatment prior to anaerobic sludge digestion has shown great benefits over the chemical pretreatment methods. In the HPH pretreatment process, the operation is simple, and negligible chemical reaction can take place [20,21]. The homogenization process applies several different stresses on the sludge. When the sludge is pumped through a valve, the pressure sharply increases, and then immediately releases as the sludge passes through, which causes high fluid shear within the sludge flocs. When the external pressure exceeds the internal resistance of sludge flocs or microbial cells, the sludge flocs and cells are broken, releasing the EPS and intracellular substances [22–24]. However, HPH is energy-intensive, which is a great concern of this technology [25].

To overcome such drawback and further improve anaerobic digestion performances, their combination was studied in this study. To our best knowledge, few studies reported this combined pretreatment method, especially the effect of HPH operating parameters (homogenization pressures and cycles) on the sludge digestion has not been studied, and such knowledge is believed to be crucial in determine the cost-effective operational condition of the combined technology [26]. The objectives of this study were thus in 2-fold: (1) to evaluate the impact of HPH operating on performances of anaerobic sludge digestion from the organic removal, biogas and methane production; and (2) to further investigate the relationship between the sludge disintegration degree of the combined pretreatment and efficiency of anaerobic sludge digestion.

2. Materials and methods

2.1. Materials

Waste activated sludge used in this study was taken from the secondary sedimentation tank of a municipal WWTP with an A^2/O process in Beijing, China. The sludge was filtered through a 0.64 mm sieve and then stored at 4°C before use. The average characteristics of sludge used for subsequent experiments were as follows: total solids (TS) of 19,250 mg/L, volatile solids (VS) of 12,170 mg/L, total chemical oxygen demand (TCOD) of 17,602 mg/L, soluble chemical oxygen demand (SCOD) of 472.6 mg/L, alkalinity of 263.5 mg/L (as $CaCO_3$), and pH of 6.85. Inoculum sludge for anaerobic digestion was obtained from an anaerobic digester in the same WWTP, the average alkalinity, pH and methane content in biogas of which was 2,750 mg/L, 6.83 and above 50%, respectively.

2.2. Combined sludge pretreatment

The combined sludge pretreatment process using alkaline and HPH has been thoroughly described in our previous work [27]. A NaOH dosage of 0.04 mol/L was added to a plastic vessel containing the sewage sludge. The dosage was an optimized value based on our previous study [28]. The sludge was mixed at 28°C for 0.5 h using a stirrer with a set speed of 180 r/min. Thereafter, the sludge was treated with a high pressure homogenizer at pressures between 0 and 60 MPa with 1–3 cycles.

The sludge disintegration degree (DD_{COD}) was calculated using Eq. (1) [29]:

$$DD_{COD}(\%) = \frac{SCOD - SCOD_0}{TCOD - SCOD_0} \times 100\% \quad (1)$$

where SCOD and TCOD represent the dissolved COD and the total COD, respectively; $SCOD_0$ and $TCOD_0$ represent the initial SCOD and TCOD concentration, respectively, prior to the pretreatment.

2.3. Anaerobic sludge digestion

Five identical plexiglass reactors of 5 L volume were used to investigate the effect of homogenization pressure on anaerobic sludge digestion. Afterwards, three of them were used to further investigate the effect of homogenization cycle on anaerobic sludge digestion under the optimal homogenization pressure. During the digestion, the temperature in all rectors was controlled at 35°C ± 2°C. The sludge in reactors was continually stirred using an agitator at a speed of 100 r/min. The pretreated sludge was neutralized to a pH of 7 ± 0.2 prior to adding to the reactors. The disintegrated sludge and seed sludge was mixed with a ratio of 6:4. In order to maintain a strict anaerobic condition, nitrogen gas was introduced to the reactor for 5 min before starting the anaerobic digestion. The biogas production was daily measured

Table 1
Performances of anaerobic digestion of sludge without pretreatment

Parameter	Value
SCOD removal (%)	17.37 ± 0.23
VS removal (%)	16.23 ± 0.19
TCOD removal (%)	19.35 ± 0.46
Cumulative biogas (ml)	1,655.56 ± 32.57
Methane yield (ml/g VS)	75.82 ± 0.92
Maximum biogas production rate (ml/d)	265 ± 3.35

using displacement of HCl solution (pH = 3). The SCOD, pH and alkalinity of digestion system were also daily analyzed. When the digestion process became stable, samples of biogas were taken to determine the methane content. The sludge TS, VS and TCOD were measured before and after the anaerobic digestion. All samples were measured in triplicate.

The performances of anaerobic digestion of sludge without pretreatment were listed in Table 1 as control.

2.4. Analytical methods

The COD was analyzed using a COD speed measuring device (CTL-12, Huatong Inc., China). The sludge samples were centrifuged at 5,000 r/min for 30 min and filtered using 0.45 μm membranes prior to analyzing the SCOD. Whereas, for the TCOD measurement, the sludge samples were alkalinized for 24 h with a NaOH dosage of 0.5 mol/L, and thereafter filtered using 0.45 μm membranes prior to the TCOD measurement [6]. The other parameters, such as TS, VS and alkalinity, were analyzed according to the standard methods [30].

The methane content was measured by a gas chromatograph (GC-2014, Shimadzu, Japan) and a column carbon molecular sieve (TDX-01, HRBY Inc., China), following the identical operational conditions that were reported in our previous study [28].

3. Results and discussion

3.1. Effect of HPH on sludge disintegration

The sludge SCOD increases due to solubilization of solid matters, which can be evaluated by DD_{COD} [29,31]. As shown in Fig. 1(a), with one homogenization cycle, both SCOD and DD_{COD} firstly increased with increasing the homogenization pressure rapidly. After the combined pretreatment at 20 MPa, the sludge SCOD and DD_{COD} reached 6,864 mg/L and 37.31%, which increased by about 90.72% and 104.44%, respectively, compared with that by alkaline pretreatment alone. When the homogenization pressure further increased, the increase of sludge SCOD and DD_{COD} became gradual. The maximum sludge SCOD and DD_{COD} of 9,373 mg/L and 51.23% were achieved at 80 MPa with one homogenization cycle, which insignificantly increased compared with that at 60 MPa. The increment of sludge SCOD and DD_{COD} indicated that the sludge flocs were liquidized to soluble fractions or converted to low molecular weight compounds by combined pretreatment.

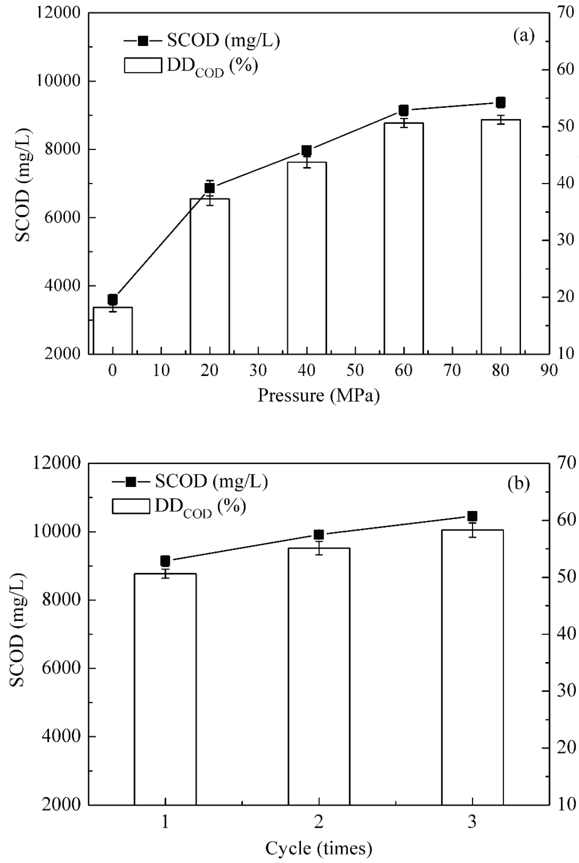

Fig. 1. Effect of homogenization pressure and homogenization cycle on sludge disintegration (NaOH dosage of 0.04 mol/L) (a) Homogenization pressure with one homogenization cycle, (b) homogenization cycle at 60 MPa.

However, increasing the homogenization cycle from 1 to 3 at 60 MPa did not lead to a significant increment in SCOD and DD_{COD} (as shown in Fig. 1(b)). The observation was in agreement with the combined sludge disintegration model reported in our previous work [26]. The results could be reasonably attributed to most of the weak and easily decomposed organic matters were released to a great extent through the first homogenization process and the remaining substances were relatively resistant in the following homogenization cycles. Additionally, sludge mineralization might occur, when the HPH energy consumption reached a certain value [32]. So increasing the homogenization cycle was inefficient.

3.2. Effect of HPH on anaerobic sludge digestion

3.2.1. Organic removal

The sludge SCOD during anaerobic digestion depends on the organic solubilization from sludge solids by exocellular enzymes and their further conversion to micromolecular substances even biogas. A higher initial SCOD was observed after the sludge pretreatment under a higher homogenization pressure because of a higher sludge disintegration degree (as shown in Fig. 1(a)). During the first 3 d of digestion, the SCOD showed insignificant decrease, and thereafter, it started to decrease dramatically [33]. Furthermore, the sludge SCOD decreased faster when the sludge was pretreated with a higher homogenization pressure. After whole anaerobic digestion process, the total SCOD reduction for the combined pretreatment of alkaline and HPH (at 60 MPa with one homogenization cycle) increased by 23.28%, compared with that with alkaline pretreatment alone. The result in this study was lower than that with microwave-alkali pretreatment after 20-d digestion, which might be attributed to the low initial SCOD of 2,700 mg/L and longer digestion period [34], but higher than that with ozone pretreatment [35,36].

Fig. 2(b) presents the effect of homogenization cycle on sludge SCOD change at 60 MPa. The results showed that the reduction of sludge SCOD with three homogenization cycles increased only 3.04%, compared with that for one homogenization cycle, indicating that the effect of homogenization cycle on organic biodegradation efficiency was negligible.

Organic degradation of digestion is usually expressed by sludge TCOD and VS removals [12,37]. The sludge biodegradation was significantly improved when the homogenization pressure increased, and the TCOD removal and VS removal increased by 24.68% and 18.95% at 60 MPa with one homogenization cycle, respectively, compared with that with alkaline pretreatment alone (Fig. 3). The results were higher than that by microwave-alkaline pretreatment or collision treatment [38]. It indicates that, their conversion of sludge solid to soluble organics with homogenization process improved the sludge biodegradation. However, there was no significant difference, comparing the TCOD removal and VS removal at 60 with 80 MPa.

Compared with one homogenization cycle, multi homogenization cycle pretreatment did not remarkably improve the TCOD and VS removal at 60 MPa (as shown in Fig. 3(b)).

3.2.2. Effect of HPH on biogas production

As expected from the organic removal, a more biogas accumulation was observed when the sludge pretreated by combined alkaline and HPH. As shown in Fig. 4(a), the cumulative biogas production increased by 95.81% with pretreatment at 60 MPa, compared with that alkaline pretreatment alone. The combined pretreatment apparently enhanced the organic solubilization as well as the surface area available for enzymatic reaction. A considerable amount of soluble compounds of low-molecular weight released from combined pretreatment were easily biodegraded and converted to the biogas [10,26]. However, there is no significant enhancement

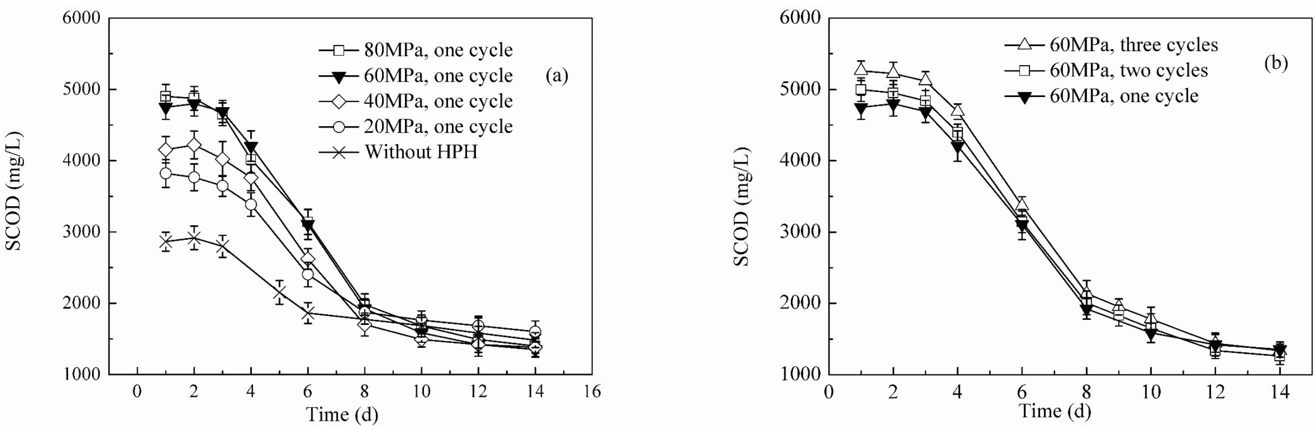

Fig. 2. Effect of homogenization pressure and homogenization cycle on sludge SCOD change (NaOH dosage of 0.04 mol/L) (a) Homogenization pressure with one homogenization cycle, (b) homogenization cycle at 60 MPa.

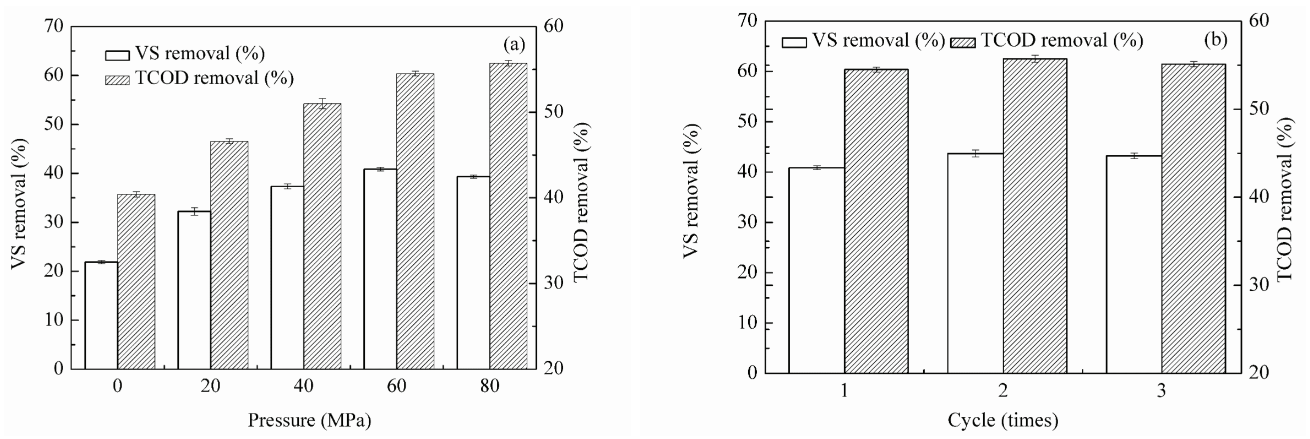

Fig. 3. Effect of homogenization pressure and homogenization cycle on sludge VS and TCOD removal (NaOH dosage of 0.04 mol/L) (a) Homogenization pressure with one homogenization cycle, (b) homogenization cycle at 60 MPa.

between the total biogas production at 60 and 80 MPa with one homogenization cycle, indicating that the homogenization pressure should not excess 60 MPa for energy saving.

As shown in Fig. 4(b), the maximum cumulative biogas production of 5,362 ml was achieved with an alkaline dosage of 0.04 mol/L and a three-cycle homogenization at 60 MPa, however, the cumulative biogas production was only 5.16% higher than that with one homogenization cycle, which agreed with the phenomenon that the sludge disintegration degree had no obvious increase with increasing the homogenization cycle (as shown in Fig. 1(b)). It meant that no more sludge solids could be transformed into the soluble organics. However, the energy input increased by two times, when the homogenization cycle increased from one to three. Considering the biogas production and energy-saving, the suitable homogenization condition was selected as homogenization pressure of 60 MPa with once cycle.

As shown in Fig. 5(a), the biogas production rate was significantly enhanced by the combined pretreatment. The maximum production rate (1,146 ml/d) increased by 91.79% when the sludge was pretreated by the combined pretreatment at 80 MPa with one homogenization cycle. A high biogas production rate may significantly reduce the hydraulic retention time of the digestion. From the Fig. 5(b), we can observe the biogas production rate unremarkably changed when the homogenization cycle varied from one to three. It corroborated with the results that the degradation of organic substances was not remarkably enhanced by increasing the homogenization cycles, as shown in Fig. 3(b).

The methane yield was also affected by the homogenization pressure. As shown in Table 2, the methane yield was in a range of 134.59–473.63 ml/gVS with the combined pretreatment, which was about 43.82% to 406.12% higher than

Table 2
Effect of HPH parameters on methane content in biogas (NaOH dosage of 0.04 mol/L)

Cycle number	Pressure (MPa)	Methane yield (ml/g VS)
1	0	93.58 ± 0.76
1	20	134.59 ± 1.02
1	40	192.41 ± 0.62
1	60	251.18 ± 0.92
1	80	246.49 ± 1.02
2	60	309.71 ± 1.36
3	60	437.63 ± 0.88

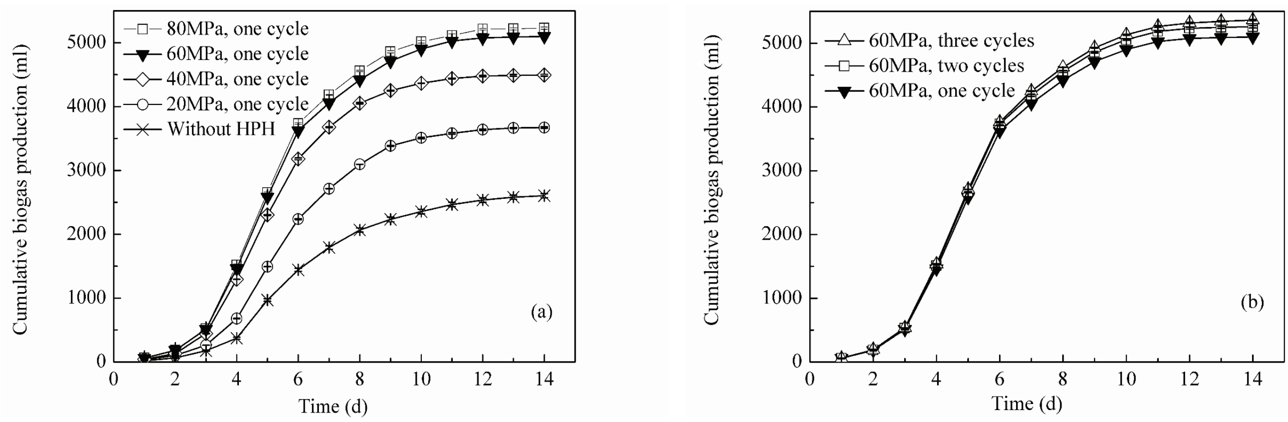

Fig. 4. Effect of homogenization pressure and homogenization cycle on cumulative biogas production (NaOH dosage of 0.04 mol/L) (a) Homogenization pressure with one homogenization cycle, (b) homogenization cycle at 60 MPa.

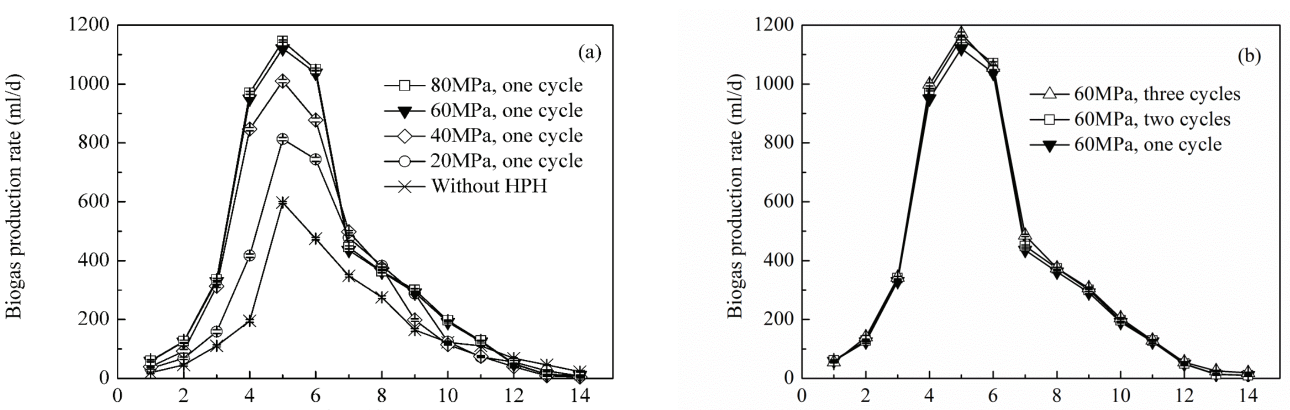

Fig. 5. Effect of homogenization pressure and homogenization cycle on biogas production rate (NaOH dosage of 0.04 mol/L) (a) Homogenization pressure with one homogenization cycle, (b) homogenization cycle at 60 MPa.

that with alkaline pretreatment alone (93.58 ml/gVS). The improvement of methane yield with the combined pretreatment might be attributed to the following two aspects: firstly, the sludge solubilization and anaerobic biodegradability were significantly enhanced when the sludge was pretreated by the combined alkaline and HPH; secondly, the soluble and tiny components generated from the combined pretreatment improved the surface area, which were more availably utilized by methanogens, leading to higher methane content in biogas [21,39].

3.2.3. pH and alkalinity of anaerobic digestion process

The growth of microorganisms during anaerobic digestion is strongly influenced by the sludge pH. It has been reported that the methanogens can function well in a relative narrow pH range of 6.5–8.0 [40]. In this study, the pH always maintained within a range of 6.9–7.5. The pH was negligibly changed under the experimental conditions using different homogenization pressures and cycles. During the first 3 d of operation, the pH was slightly decreased due to the VFA accumulation, and thereafter, it gently increased because of the VFA consumption for methane production [8].

Since the produced VFAs directly consume alkalinity before significant pH change, the alkalinity is usually used as a better alternative to reflect the anaerobic digestion performances [41]. The alkalinity of a steady anaerobic digestion system is between 2,000 and 5,000 mg/L (as $CaCO_3$). In this study, all the alkalinities maintained within a range of 3,000–5,000 mg/L (as $CaCO_3$).

3.3. Relationship between sludge disintegration and anaerobic sludge digestion

As shown in Fig. 6, it's obviously that the methane yield per gram VS removed increased with the increment of DD_{COD}. Within the DD_{COD} range tested, the fitting correlation was defined with the Eq. (2). These results indicated that the organic compounds released from sludge solids by PHP pretreatment can, to a great extent, be biodegraded and converted to methane. As a result, the VS removal was enhanced with the sludge pretreatment using the HPH (Fig. 3). In addition, the methane yield was significantly improved, when the DD_{COD} was higher than 40%. The maximum methane yield was obtained at a DD_{COD} of 58.29% in this study. However, the sludge disintegration was limited, especially after most of the weak sludge cells were disrupted.

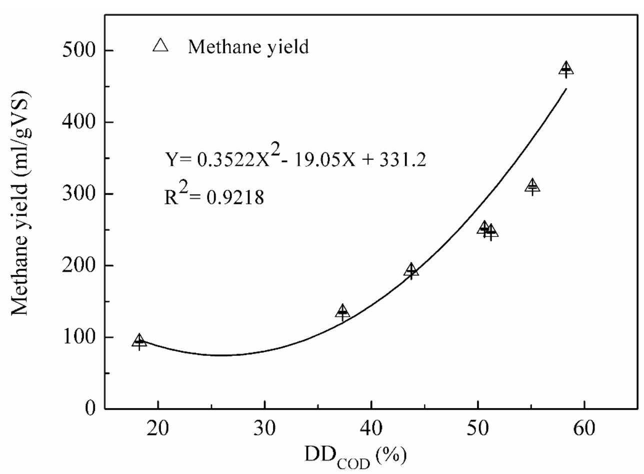

Fig. 6. Change of methane yield with DD_{COD} (%) increase.

$$\text{Methane yield (ml/gVS)} = 0.3522DD_{COD^2} - 19.05DD_{COD} + 331.2 \quad (2)$$

4. Conclusion

In this study, the effect of homogenization of sewage sludge on the digestion efficiency was elucidated. The DD_{COD} was significantly enhanced by the combined pretreatment using HPH and alkaline. As a result, the efficiency in the followed anaerobic sludge digestion was dramatically improved. Meanwhile, the organic removal, biogas production rate and the methane content were greatly improved, because biologically degradable organic compounds were released using the combined pretreatment. Particularly, the homogenization pressure showed remarkable influence on the performances of the followed anaerobic digestion process, while increasing the homogenization cycle showed negligible impact. Considering the biogas production and energy-saving, the suitable homogenization condition was selected as homogenization pressure of 60 MPa with once cycle.

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