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Sewage sludge disintegration by high-pressure homogenization: A sludge disintegration model

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Abstract

High-pressure homogenization (HPH) technology was applied as a pretreatment to disintegrate sewage sludge. The effects of homogenization pressure, homogenization cycle number, and total solid content on sludge disintegration were investigated. The sludge disintegration degree (DD$_{\text{COD}}$), protein concentration, and polysaccharide concentration increased with the increase of homogenization pressure and homogenization cycle number, and decreased with the increase of sludge total solid (TS) content. The maximum DD$_{\text{COD}}$ of 43.94% was achieved at 80 MPa with four homogenization cycles for a 9.58 g/L TS sludge sample. A HPH sludge disintegration model of DD$_{\text{COD}}$ = $kN^aP^b$ was established by multivariable linear regression to quantify the effects of homogenization parameters. The homogenization cycle exponent $a$ and homogenization pressure exponent $b$ were 0.4763 and 0.7324 respectively, showing that the effect of homogenization pressure ($P$) was more significant than that of homogenization cycle number ($N$). The value of the rate constant $k$ decreased with the increase of sludge total solid content. The specific energy consumption increased with the increment of sludge disintegration efficiency. Lower specific energy consumption was required for higher total solid content sludge.

Key words: high-pressure homogenization; sludge disintegration; homogenization pressure; homogenization cycle number; total solid content

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Introduction

Large quantities of putrescible sewage sludge from wastewater treatment plants (WWTPs) cause unpleasant environmental and health problems, and sludge treatment becomes one severe challenge for WWTPs. Recent studies on sewage sludge treatment focus on sludge reduction and utilization (Chu et al., 2009; Dewil et al., 2007; Wang et al., 2010). It is well known that the main part of sewage sludge solids is organics, which can be converted into renewable energy such as methane. Anaerobic digestion of sludge is a promising technology, which can achieve the objective of sludge degradation and energy generation (Dewil et al., 2007; Fantozzi and Buratti, 2009; Rubio-Loza and Noyola, 2010). However, sewage sludge composition generally limits the efficiencies of sludge degradation and energy generation (Lin et al., 2009; Wang et al., 2010). Sewage sludge mainly contains bacterial materials, and direct degradation of bacteria is difficult because the bacterial cell wall forms a barrier to the degradation of intracellular organic materials. Cell disruption is an effective pretreatment method to improve the rate and extent of sewage sludge digestion through disintegration of microbial cells and liberation of intracellular contents. In several countries, e.g., Germany, some mechanical, thermal and other cell disruption methods have been tested and carried out in WWTPs (Ahn et al., 2009; Barjenbruch and Kopplow, 2003; Pham et al., 2010; Zhang et al., 2009).

High-pressure homogenization (HPH) is a well known mechanical method for cell disruption. HPH was mainly developed for the stabilization of food and dairy emulsions, and has opened up more and more new application areas (Paquin, 1999; Wuytack et al., 2002; Jacquel et al., 2008). Recently, HPH has been reported as a sludge pretreatment method, which changes both the rate and extent of sewage sludge degradation in anaerobic digestion processes (Onyechef et al., 2003; Rai and Rao, 2009). HPH pretreatment for sludge disintegration is generally based on the disruption of microbial cell walls and the release of cytoplasm into the liquid phase by the functions of pressure gradient, cavitation, turbulence, impingement, shear stresses, and extensional shear (Floury et al., 2004; Jacquel et al., 2008; Kleinig and Middelberg, 1998; Zhang et al., 2008). HPH treatment has several advantages compared with other sludge disintegration methods, including absence of chemical changes or denaturing during cell lysis, easy operation, and high lysis efficiency (Barjen* Corresponding author. E-mail: panyue_zhang@bjfu.edu.cn
bruch and Kopplow, 2003; Onyech et al., 2003; Li et al., 2008; Rai and Rao, 2009). Furthermore, HPH treatment as a pretreatment can combine with other cell disruption methods such as thermal or alkaline treatment to improve sludge anaerobic digestion efficiency.

The HPH process for cell disruption is mainly influenced by the homogenization pressure, number of homogenization cycles, and cell concentration (Donsi et al., 2009; Middelberg, 1991; Sauer et al., 1989). In a classical HPH process, a moderate homogenization pressure of 20–50 MPa is used in the food, pharmaceutical and cosmetics industries (Thiebaud et al., 2003). The pressure gradient is the main physical cause of cell disruption in the homogenization valve. In addition, pressure energy can convert into heat and transmit to the fluid when the fluid leaves the homogenization valve, raising the fluid temperature (Thiebaud et al., 2003). The number of homogenization cycles is important during a HPH process, since the cell disruption efficiency is low with a single homogenization cycle, and increasing the homogenization cycle number can significantly improve the effectiveness of cell disruption (Anand et al., 2007; Chaves-López et al., 2009). In addition to the HPH operating conditions (homogenization pressure and number of homogenization cycles), medium characteristics, especially the cell concentration, also influence the microorganism disintegration (Sauer et al., 1989). Some researchers reported the effects of homogenization pressure, number of homogenization cycles, and cell concentration on cell disruption using HPH, but previous studies primarily involved the disruption of specific microbes such as Escherichia coli (Middelberg, 1991; Anand et al., 2007; Donsi et al., 2009), and few studies focused on sewage sludge disintegration.

The objective of this study was to investigate the effects of homogenization pressure, homogenization cycle number, and total solid content (TS) on sewage sludge disintegration. The efficiency of sludge disintegration was evaluated by the change of soluble chemical oxygen demand (SCOD), protein and polysaccharide concentrations of the sludge supernatant. A HPH sludge disintegration model based on the test data was established, which can be used to predict the sludge disintegration degree as a function of HPH operating parameters. The energy consumption was also evaluated.

1 Materials and methods

1.1 Sewage sludge

Sewage sludge was obtained from a municipal wastewater treatment plant with a MBR process in Beijing, China. The sewage sludge was concentrated by gravity setting for 24 hr; then sludge samples with a sludge TS of 9.58, 14.95 and 24.60 g/L were respectively obtained through dilution of the concentrated sludge. These sludge samples were stored at 4°C in a refrigerator before use. The characteristics of sludge samples are shown in Table 1. Low SCOD in the sludge supernatant implied that most COD existed in the solid phase.

| Table 1 Characteristics of sludge samples used in experiments |
|---|---|---|---|---|---|---|---|
| TS (g/L) | VS (g/L) | TCOD (mg/L) | SCOD (mg/L) | Proteins (mg/L) | Polysaccharides (mg/L) | pH |
| S1 | 9.58 | 7.13 | 10427 | 93.18 | 6.115 | 16.96 | 6.86 |
| S2 | 14.95 | 11.14 | 14877 | 99.20 | 10.60 | 20.45 | 6.89 |
| S3 | 24.60 | 18.35 | 27925 | 198.15 | 49.70 | 42.37 | 6.92 |

TS: total solid content; VS: volatile solid content; TCOD: total chemical oxygen demand; SCOD: soluble chemical oxygen demand. S1–S3: sludge samples.

1.2 High-pressure homogenization treatment

A high-pressure homogenizer (GJJ-0.03/100, Puzong Inc., China) was used for sludge disintegration with a working pressure range of 0–100 MPa. Without temperature regulation, the sludge HPH treatment was performed with a volume of 1 L sludge sample, and the sludge flow rate was 30 L/hr. The process of HPH treatment is described as follows.

First, the sludge sample was pumped through the homogenization valve by an air driven positive displacement pump, and the homogenization pressure was fixed at a certain value through adjusting the homogenization valve. When the sludge sample went through a convergent section of the homogenization valve, the sludge stream was immediately accelerated to 1000–1500 m/sec.

Then the high-velocity sludge stream impinged on an impact ring and the pressure suddenly dropped from high pressure to low pressure, disrupting the cell walls and liquefying the sludge due to the combined action of pressure gradient, cavitation, turbulence, impingement, shear stresses, and extensional shear.

Finally, the sludge was forced out of the homogenization valve with a lower velocity. Characteristics of the homogenized sludge were then measured.

The homogenization treatment was carried out at a given pressure (20–80 MPa) for one to four cycles. Each experiment was repeated for at least two times.

1.3 Energy consumption calculation

The energy input for HPH treatment is relevant to the homogenization pressure ($P$, Pa) and homogenization cycle number ($N$). The energy consumption per unit sludge volume ($E_v$, J/m³) was defined in Eq. (1) (Anand et al., 2007).

$$E_v = P \times N$$

The specific energy consumption ($E_s$, kJ/kg TS) was calculated by Eq. (2):

$$E_s = \frac{E_v}{TS_0 \times 1000}$$

where, $TS_0$ (g/L) is the TS of the untreated sludge sample.

1.4 Analytical methods

All analyses were performed in duplicate. Total solid content (TS), volatile solid content (VS) and chemical oxygen demand (COD) were determined based on APHA Standard Methods (APHA et al., 1998). Protein concentration was measured using the Coomassie Brilliant Blue...
G-250 method at an absorbance of 595 nm. Polysaccharide concentration measurement was performed by the anthrone method at an absorbance of 630 nm. Two types of COD, soluble chemical oxygen demand (SCOD) and total chemical oxygen demand (TCOD), were examined to study the sludge disintegration after HPH treatment.

For determining the SCOD, the sludge samples were centrifuged at 8000 rpm for 20 min with a centrifuge (TGL-20B, Anting Inc., China), then the supernatant was filtered through a 0.45 μm filter membrane. The filtrate obtained was used to determine SCOD.

For examining the TCOD, the raw sludge samples with a TS of 9.58, 14.95 and 24.60 g/L were respectively solubilized by alkaline treatment with a NaOH dosage of 0.5 mol/L for 24 hr, then filtered through a quantitative paper. The filtrate obtained was used to determine TCOD.

The sludge disintegration degree (DDCOD) was calculated as Eq. (3) (Zhang et al., 2008):

\[
DD_{\text{COD}} = \frac{\text{SCOD} - \text{SCOD}_0}{\text{TCOD} - \text{SCOD}_0}
\]

where, SCOD0 is the SCOD of the untreated sludge.

2 Results and discussion

2.1 Effect of homogenization pressure

The variations of DDCOD, protein and polysaccharide concentrations under different homogenization pressures are shown in Fig. 1. The DDCOD, protein and polysaccharide concentrations increased significantly with the increase of homogenization pressure from 20 to 80 MPa with a single homogenization cycle. At homogenization pressure of 80 MPa, the DDCOD reached higher than 17% for all sludge samples; while at homogenization pressure of 20 MPa, the DDCOD was only about 10%. Both protein and polysaccharide concentrations in sludge supernatant increased by about 140% when the homogenization pressure increased from 20 to 80 MPa. The results demonstrated that larger quantities of sludge organic matters were released from the solid phase into the liquid phase at higher homogenization pressure. High pressure (e.g. 80 MPa) might benefit the sludge disintegration due to the thermal effect in addition to the pressure gradient effect. The sludge temperature change after HPH treatment with a sludge TS of 14.95 g/L is as follows: 21°C at 0 MPa; 30°C at 20 MPa; 33°C at 30 MPa; 40°C at 30 MPa; 43°C at 80 MPa. The sludge temperature after HPH treatment increased with increasing homogenization pressure. Compared with the untreated sludge, the sludge temperature increased by 22°C at a homogenization pressure of 80 MPa. Donsi et al. (2009) reported that the thermal effect resulting from the sludge temperature increase enhanced the sludge disintegration.

2.2 Effect of homogenization cycle number

As shown in Fig. 2, HPH treatment with a single homogenization cycle could not effectively disintegrate sludge and lyse cells, especially at a low homogenization pressure. Therefore, multiple cycle operation of HPH treatment was necessary to improve the sludge disintegration efficiency. The effects of homogenization cycle number on DDCOD, protein and polysaccharide concentrations at 80 MPa are shown in Fig. 2. Multiple cycle operation was more effective for sludge disintegration and cell lysis than the single cycle process. Gradual DDCOD, protein and polysaccharide concentration increases were observed as the homogenization cycle number increased from one to four. The DDCOD mainly increased during the first two cycles, and further DDCOD increment was less than 10% during the following two homogenization cycles for sludge samples. Meanwhile, both protein and polysaccharide...
concentrations showed similar increases with increasing homogenization cycle number, and the average protein and polysaccharide concentrations in sludge supernatant increased by 53% and 64%, respectively. The effect of homogenization cycle number on sludge disintegration was much lower than that of homogenization pressure. The behavior could be reasonably attributed to selective cell disruption. The weaker and more sensitive cells were selectively disrupted in the previous homogenization cycles and the resistant cells were disintegrated in the following homogenization cycles (Donsi et al., 2009). When most of the weak sludge cells were disrupted, no significant sludge disintegration could be further achieved even if the number of homogenization cycles increased.

Table 2 shows that increase of homogenization cycle number achieved the same result in DD$_{COD}$ as increasing homogenization pressure did. The DD$_{COD}$ after four homogenization cycles at 30 MPa was equivalent to that after a single homogenization cycle at 80 MPa for the 9.58 g/L TS sludge sample, which meant that the same sludge disintegration efficiency could be achieved through high pressure and multiple homogenization cycles.

Table 2

<table>
<thead>
<tr>
<th>Cycle number</th>
<th>Pressure (MPa)</th>
<th>DD$_{COD}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30</td>
<td>12.61</td>
</tr>
<tr>
<td>4</td>
<td>30</td>
<td>24.21</td>
</tr>
<tr>
<td>1</td>
<td>80</td>
<td>24.67</td>
</tr>
</tbody>
</table>

2.3 Effect of sludge TS

From Figs. 1 and 2, it can be seen that sludge TS also played an important role in sludge disintegration. The DD$_{COD}$ for 9.58 g/L TS sludge were always the highest at the same homogenization pressure and with the same number of homogenization cycles, while that for 24.60 g/L TS sludge were always the lowest. The results indicated that the increase of sludge TS had a negative effect on sludge disintegration under all the conditions studied, showing as a decrement in DD$_{COD}$. However, both protein and polysaccharide concentrations in the sludge supernatant increased with increasing sludge TS. As shown in Fig. 1, when the sludge TS increased from 9.58 to 24.60 g/L, the DD$_{COD}$ decrement was only 3% at 20 MPa, but was 8% at 80 MPa after a single homogenization cycle. Figure 2 shows that the DD$_{COD}$ decrease was 11% at 80 MPa after four homogenization cycles, when sludge TS increased from 9.58 to 24.60 g/L. The DD$_{COD}$ decrement could be attributed to the interaction among the sludge cells in the homogenization valve. Fewer cells could be disrupted due to the protection of more peripheral cells when more concentrated sludge went through the slit of the homogenization valve, thus a lower DD$_{COD}$ was obtained (Donsi et al., 2009). It was found that a threshold TS value of about 25 g/L existed for the high-pressure homogenizer used in the research, above which the effectiveness of HPH treatment drastically reduced and the normal operation of the homogenizer was significantly affected due to blockage in the homogenization valve. Considering protection of the high-pressure homogenizer, TS of about 25 g/L was adopted as the highest TS in this research.

2.4 High-pressure homogenization sludge disintegration model

The HPH for E. coli cell disruption was modeled by Eq. (4) (Sauer et al., 1989):

$$\ln \frac{1}{1 - R} = kN^aP^b$$

(4)

where, $k$ is the rate constant of sludge disintegration; $a$ and $b$ are the homogenization cycle exponent and homogenization pressure, respectively.
tion pressure exponent respectively; $R$ is the fraction of cells disrupted, which was calculated by the ratio between the protein content in the supernatant after disruption and that after complete disruption.

Although the HPH operation for *E. coli* cell disruption was excellently described by Eq. (4), no direct relationship between the cell disruption and the operating conditions was revealed. In this study, $D_{\text{COD}}$ was used to represent the sludge disintegration degree, and the correlation between $D_{\text{COD}}$ and homogenization pressure or number of homogenization cycles complied basically with the power function, as shown in Figs. 1 and 2. Therefore, the above Eq. (4) was modified to Eq. (5) for HPH sludge disintegration:

$$D_{\text{COD}} = kN^aP^b$$  \hspace{1cm} (5)

When the logarithm of both sides was taken, Eq. (6) was obtained:

$$\ln D_{\text{COD}} = \ln k + a\ln N + b\ln P$$ \hspace{1cm} (6)

$D_{\text{COD}}$ values, which were obtained by factorial design for five groups of homogenization pressures, four groups of number of homogenization cycles and three groups of sludge TS, were divided into three groups according to differences in sludge TS. The data of the three groups were separately analyzed by the multivariable linear regression method to calculate the homogenization cycle exponent $a$, the homogenization pressure exponent $b$ and the rate constant $k$, and to verify Eq. (5). The regression coefficients of $a$, $b$ and $k$ are shown in Table 3. A correlation test and $t$-test were conducted between the dependent variable and independent variables in the regression models. The corresponding parameters of the tests are listed in Table 4.

It can be seen from Table 3 that both the homogenization cycle exponent $a$ and the homogenization pressure exponent $b$ were similar for all sludge samples, which suggested that sludge TS had little influence on the homogenization cycle exponent $a$ and homogenization pressure exponent $b$. Therefore, the homogenization cycle exponent $a$ and the homogenization pressure exponent $b$ in Eq. (5) could be taken as the average values of 0.4763 and 0.7324, respectively. Furthermore, the computed data for the homogenization cycle exponent $a$ and the homogenization pressure exponent $b$ could estimate which homogenization parameter was more significant for sludge disintegration.

For a power function, the larger the exponent of an independent variable was, the larger its impact on the dependent variable. Table 3 shows that the homogenization pressure exponent $b$ was larger than the homogenization cycle exponent $a$ for HPH disintegration in this study, indicating that the effect of homogenization pressure was more significant than that of the number of homogenization cycles on sludge disintegration and cell lysis.

From Table 3, it is known that the HPH disintegration of three sludge samples followed the model of $D_{\text{COD}} = kN^{0.4763}P^{0.7324}$, and the value of constant $k$ was $10.19\times 10^{-3}$, $8.63\times 10^{-3}$ and $7.16\times 10^{-3}$ for the sludge sample with a TS of 9.58, 14.95 and 24.60 g/L, respectively. Apparently in Figs. 1 and 2, sludge TS impacted the sludge disintegration and cell lysis during the homogenization process. The rate constant $k$ might be related to sludge TS. The values of the rate constant $k$ for sludge disintegration decreased with increasing sludge TS, which meant that the efficiency of sludge disintegration and cell lysis was reduced when sludge TS increased.

As shown in Table 4, the partial correlation coefficients ($r$) between $D_{\text{COD}}$ and homogenization pressure or number of homogenization cycles were all higher than 0.96, which indicated that the independent variables $P$ and $N$ were well correlated with the dependent variable $D_{\text{COD}}$. In addition, the regression models of Eq. (6) had a good linear correlation with high multiple correlation coefficients $R^2$ (more than 96%), which meant that the degree of fit between the regression models and data samples was excellent. Moreover, values of $t$ passed the $t$-test with a degree of confidence of 95%, which showed that both homogenization pressure $P$ and homogenization cycle number $N$ were significant impact factors on the sludge disintegration (Li et al., 2010; Wang et al., 2005). All these results demonstrated that the model of $D_{\text{COD}} = kN^aP^b$ was excellent to study the effect of homogenization pressure and number of homogenization cycles on sewage sludge disintegration during the HPH process.

### 2.5 Energy consumption

The energy consumption of HPH treatment is important for its application. Figure 3 shows the correlation between the $D_{\text{COD}}$ and the special energy consumption ($E_s$). The $E_s$ steadily increased with the increase of $D_{\text{COD}}$, indicating that higher energy input was required for more effective sludge disintegration, releasing more organic components into the solution phase. Energy consumption of about 8450 kJ/kg TS was needed to achieve the highest $D_{\text{COD}}$ of 24.7% for the sludge sample with TS 9.58 g/L. Moreover, it was found that the specific energy consumption of the HPH treatment decreased with the sludge TS increase, showing
that HPH treatment was more energy-efficient for higher TS sludge.

It is widely known that some sludge disintegration methods are energy intensive, especially mechanical and thermal treatments. Table 5 shows a special energy consumption comparison of sludge pretreatment with HPH, ultrasound and microwave. Ultrasonication and microwave irradiation consumed 236% and 395% energy more than HPH treatment, to achieve similar sludge disintegration (SCOD/TCOD) for similar sludge (Ahn et al., 2009; Feng et al., 2009). The energy consumption comparison demonstrated that HPH treatment was an excellent sludge disintegration method with respect to energy conservation.

Table 5 Comparison of special energy consumption between HPH treatment and other sludge treatment methods

<table>
<thead>
<tr>
<th>Treatment method</th>
<th>TS (g/L)</th>
<th>SCOD/TCOD</th>
<th>$E_s$ (kJ/kg TS)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultrasonication</td>
<td>14.38</td>
<td>22%</td>
<td>18000</td>
<td>Feng et al., 2009</td>
</tr>
<tr>
<td>Microwave irradiation</td>
<td>25.70</td>
<td>17%</td>
<td>16000</td>
<td>Ahn et al., 2009</td>
</tr>
<tr>
<td>HPH</td>
<td>14.95</td>
<td>23%</td>
<td>5351</td>
<td>This study</td>
</tr>
<tr>
<td>HPH</td>
<td>24.60</td>
<td>17%</td>
<td>3252</td>
<td>This study</td>
</tr>
</tbody>
</table>

$E_s$: specific energy consumption.

3 Conclusions

HPH treatment of excess sewage sludge was significantly influenced by homogenization pressure, number of homogenization cycles and sludge TS. The increase of homogenization pressure or number of homogenization cycles effectively improved sludge disintegration, while the increase of sludge TS significantly decreased the sludge disintegration. The HPH sludge disintegration model $DD_{COD} = kN^{0.4763}p_0^{0.7324}$ was successfully established to analyze the effects of the relevant impact factors. The homogenization pressure was proved to be more effective than number of homogenization cycles on sludge disintegration, and the effect of sludge TS was reflected in the rate constant $k$. The HPH was an energy-efficient treatment method for sludge disintegration.

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