Sewage sludge solubilization by high-pressure homogenization

Yuxuan Zhang, Panyue Zhang, Jianbin Guo, Weifang Ma, Wei Fang, Boqiang Ma and Xiangzhe Xu

ABSTRACT

The behavior of sludge solubilization using high-pressure homogenization (HPH) treatment was examined by investigating the sludge solid reduction and organics solubilization. The sludge volatile suspended solids (VSS) decreased from 10.58 to 6.67 g/L for the sludge sample with a total solids content (TS) of 1.49% after HPH treatment at a homogenization pressure of 80 MPa with four homogenization cycles; total suspended solids (TSS) correspondingly decreased from 14.26 to 9.91 g/L. About 86.15% of the TSS reduction was attributed to the VSS reduction. The increase of homogenization pressure from 20 to 80 MPa or homogenization cycle number from 1 to 4 was favorable to the sludge organics solubilization, and the protein and polysaccharide solubilization linearly increased with the soluble chemical oxygen demand (SCOD) solubilization. More proteins were solubilized than polysaccharides. The linear relationship between SCOD solubilization and VSS reduction had no significant change under different homogenization pressures, homogenization cycles and sludge solid contents. The SCOD of 1.65 g/L was solubilized for the VSS reduction of 1.00 g/L for the three experimental sludge samples with a TS of 1.00, 1.49 and 2.48% under all HPH operating conditions. The energy efficiency results showed that the HPH treatment at a homogenization pressure of 30 MPa with a single homogenization cycle for the sludge sample with a TS of 2.48% was the most energy efficient.

Key words | energy efficiency, high-pressure homogenization, sludge solubilization, soluble organics, volatile suspended solids

INTRODUCTION

Sewage sludge reduction and utilization has become one of the most urgent and complex problems in municipal wastewater treatment plants for the production of large quantities of sewage sludge (Pousada-Ferradás et al. 2012). It is well known that sewage sludge mainly consists of bacterial materials. In recent years, research on efficient sludge treatment has been rapidly increasing; the research commonly tries to explore alternative processes to degrade bacterial materials for sewage sludge minimization and subsequent utilization. Anaerobic digestion is an appropriate technology for sewage sludge treatment before final disposal, but it cannot be efficiently and extensively applied due to poor release of biodegradable organics. In general, some macromolecular substances such as proteins and carbohydrates, which mainly consist of cytoplasm and extracellular polymeric substances (EPS), are more degradable compared to other bacterial materials. However, floc structure and tough and rigid cell walls form physical and chemical barriers to avoid degradable substances release (Ahn et al. 2009). In order to solve this problem and reduce the excess sludge, it is necessary to develop various promising pretreatment methods for further sludge anaerobic digestion (Rajesh Banu et al. 2011). These pretreatment technologies are usually referred to as sludge disintegration, which disrupts sludge floc structure and cell walls and releases intracellular materials and EPS from sludge solids into liquid phase (Zhang et al. 2009). The effect of pretreatment on sludge solids is usually assessed by the reduction of total suspended solids (TSS) and volatile suspended solids (VSS) (Laurent et al. 2009; Salsabil et al. 2010). Released
organic matter is represented with chemical oxygen demand (COD), proteins and polysaccharides and so on, and is used to assess the sludge solubilization (Zhang et al. 2008; Feng et al. 2009; Laurent et al. 2009; Salsabil et al. 2010).

High-pressure homogenization (HPH) is well known for its microbial cell disruption and emulsification properties, and is generally suitable for treating a variety of bacteria, yeast and mycelia except highly filamentous microorganisms. HPH technology has been studied extensively on product emulsification, dispersion, mixture and process in chemical, pharmaceutical, food and biotechnology industries since the beginning of the last century and has evolved to explore new application areas (Paquin 1999; Cruz et al. 2007; Wang et al. 2008). Recently, several studies have been reported that HPH was applied to disintegrate sewage sludge for improving anaerobic digestion (Onyeche et al. 2003; Rai & Rao 2009). In HPH, sludge is forced through a narrow homogenization valve at high velocity, and bacteria cells are disrupted as a result of a combination of the large pressure drop, highly focused turbulent eddies and strong shearing forces when the sludge travels across the valve gap. When the sludge stream flows out, the intracellular organics and EPS are released from solids into solution (Kleinig & Middelberg 1998; Floury et al. 2004; Casoli et al. 2010).

The operating parameters of HPH including homogenization pressure and homogenization cycle number mainly affect the sludge disintegration degree. Sauer et al. (1989) set up a dynamic model of HPH based on the relationship between the cell disruption efficiency and operating parameters. It is generally accepted that microbial cell disruption is more efficient using higher homogenization pressure or multiple homogenization cycles. However, mechanical methods for cell disruption are energy intensive, which means that more energy input is needed for the operation at higher homogenization pressure and with multiple homogenization cycles. Therefore, it is necessary to optimize the operating conditions to obtain desired disintegration efficiency with less energy input.

Furthermore, the sludge disintegration efficiency can be affected by the sludge characteristics such as total solids (TS) of the sludge.

The objective of this study was to investigate sludge solubilization using HPH treatment. Sludge solid reduction and organic matter solubilization was studied through the change of TSS, VSS, soluble COD (SCOD), protein and polysaccharide concentrations. The correlation between VSS reduction and SCOD solubilization was discussed to confirm the repartition of organics between sludge supernatant and solids. Furthermore, energy efficiency of HPH sludge disintegration was calculated to optimize the operating parameters with low energy consumption.

**METHODS**

**Sludge characteristics**

Samples of sewage sludge were collected in June 2011 from a municipal wastewater treatment plant in Beijing, China. The sewage sludge was gravitationally thickened for 24 h and the supernatant was moved; then sludge TS was adjusted to 1.00, 1.49 and 2.48% through adding the supernatant. The three experimental sludge samples were stored in a refrigerator at 4°C before use. Characteristics of the three sludge samples are presented in Table 1.

**HPH sludge treatment**

The sludge samples of 1.00 L were subjected to HPH treatment in a high-pressure homogenizer (GJJ-0.03/100, Puzong Inc., China), which is equipped with two pressure valves, and the flow rate was 30 L/h. The homogenization pressure range was from 0 to 100 MPa. The HPH treatment was conducted at a homogenization pressure of 20, 30, 40, 60 and 80 MPa with one to four homogenization cycles in this research.

Approximately 0.20 L of the homogenized sludge were taken and stored at 4°C for component analysis. The duration of each cycle under the different pressures was about

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Characteristics of sludge samples</th>
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<tr>
<td>Sample</td>
<td>TS (%)</td>
</tr>
<tr>
<td>1</td>
<td>1.00</td>
</tr>
<tr>
<td>2</td>
<td>1.49</td>
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<tr>
<td>3</td>
<td>2.48</td>
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240 s. The homogenization pressure was gradually adjusted from low pressure to high pressure by manual operation.

**Component analysis**

TS, total COD (TCOD), TSS, VSS, SCOD, proteins and polysaccharides were examined to study the sludge solubilization after HPH treatment. All measurements were carried out in duplicate. TS, TSS, VSS and COD were analyzed according to American Public Health Association Standard Methods (Eaton et al. 2005). For separating the supernatant from the sludge solid phase, the sludge samples were centrifuged at 8,000 r/min for 20 min, then the supernatant was filtered through a 0.45 μm filter membrane. The obtained filtrate was used to determine SCOD, proteins and polysaccharides. Protein concentration was measured using the Coomassie Brilliant Blue G-250 method at an absorbance of 595 nm (Marion 1976). Polysaccharide concentration measurement was performed by the anthrone method at an absorbance of 630 nm (Riesz et al. 1985).

**Energy efficiency calculation**

Energy input for HPH treatment is relevant to homogenization pressure and homogenization cycle number. The energy consumption per unit volume was defined as Equation (1) (Anand et al. 2007):

\[
E = PN
\]

where \( E \) is the energy consumption per unit volume (MJ/m³); \( P \) stands for homogenization pressure (MPa); \( N \) represents homogenization cycle number.

The energy efficiency of sludge solubilization (EESCOD) was calculated by Equation (2):

\[
EE_{SCOD} = \frac{SCOD - SCOD_0}{E}
\]

where \( SCOD_0 \) is the SCOD of the untreated sludge sample (mg/L).

**RESULTS AND DISCUSSION**

**Effect of HPH on sludge solid reduction**

Sludge samples were subjected to HPH, so that sludge cells were disrupted and organic materials were transferred from sludge solids into solution, leading to sludge solid reduction. Figure 1 presents the variation of sludge TSS and VSS before and after HPH treatment for the sludge sample with a TS of 1.49%. Figure 1 shows that both TSS and VSS decreased with increasing homogenization pressure and number of homogenization cycle. The TSS and VSS reduced almost linearly with the homogenization pressure, indicating stable sludge solid reduction was achieved by gradual increase of homogenization pressure. The improvement of sludge VSS reduction benefited from multiple homogenization cycles. It was noticed that the VSS reduction was mainly achieved by the first two homogenization cycles, which meant that more homogenization cycles could not further significantly reduce the sludge solids. The increase of homogenization pressure represents enhancing the compression of microbial cells, and the increase of number of homogenization cycle represents multiple disruptions of microbial cells (Donsi...
et al. 2009). During the HPH treatment, organics were released from the solid to liquid due to the disruption of microbial cells (Zhang et al. 2013). The TSS and VSS of the untreated sludge sample were 14.26 and 10.58 g/L, respectively, which decreased by 30.52 and 37.04% after HPH treatment at 80 MPa with four homogenization cycles, showing that sludge solids was effectively reduced by HPH treatment.

Sludge TSS reduction or VSS reduction represented the difference of TSS or VSS between the untreated and treated sludge. The correlation between TSS reduction and VSS reduction under different operating conditions (homogenization pressure and number of homogenization cycles) for the sludge sample with a TS of 1.49% was fitted, as shown in Figure 2. A linear relationship between TSS reduction and VSS reduction was found with a slope of 0.8615, which indicated that the VSS reduction accounted for about 86.15% of the TSS reduction under all operating conditions. For HPH sludge disintegration, the VSS were the main portion in the solid material reduction, therefore HPH sludge disintegration mainly led to the transfer of organic materials from the sludge solids into supernatant.

Effect of HPH on organic matter solubilization

HPH treatment disrupted sludge floc structure and cell walls, releasing organics into sludge supernatant, which led to a repartition of COD, proteins and polysaccharides between sludge solids and supernatant as shown in Figure 3. It is obvious that SCOD, protein and polysaccharide concentrations in the sludge supernatant increased with increasing homogenization pressure and homogenization cycle number. The raw sludge sample with a TS of 1.49% contained

Figure 2 | Correlations between TSS reduction and VSS reduction under different HPH operating conditions (sludge TS, 1.49%; homogenization pressure, 20–80 MPa).

Figure 3 | SCOD (a), protein (b) and polysaccharide (c) changes using HPH treatment at different operating conditions (sludge TS, 1.49%).
bits of organics (99.20 mg/L SCOD, 10.60 mg/L proteins, 20.45 mg/L polysaccharides) in sludge supernatant (see Table 1), while the SCOD, protein and polysaccharide concentrations respectively reached 5,660.26, 614.12 and 372.17 mg/L after HPH treatment at 80 MPa with four homogenization cycles. The result indicated that HPH treatment effectively disintegrated sewage sludge, and quantities of organics were released into solution (Zhang et al. 2013).

In this study, the solubilization of organic compounds was defined as the organic concentration difference before and after sludge HPH disintegration (Zhang et al. 2009). For example, the SCOD solubilization represented the difference between the supernatant SCOD of treated and untreated sludge samples. As shown in Figure 4, protein and polysaccharide solubilization increased with the increase of SCOD solubilization. A linear relationship between protein or polysaccharide solubilization and SCOD solubilization was found with a slope of 0.1034 or 0.0591, respectively, indicating much more protein was solubilized than polysaccharide with the sludge HPH disintegration. Furthermore, good correlation coefficients of the fitted lines showed that both the changes of protein and polysaccharide solubilization were proportional to the SCOD solubilization after HPH treatment under different operating conditions.

**Correlation between sludge solid reduction and organic matter solubilization**

The repartition of organics between particulate and soluble fractions occurred after HPH treatment due to sludge disintegration. Therefore, the increase of soluble organics could be directly linked to the organic sludge particulate reduction. Figure 5 presents the correlation between VSS reductions and SCOD solubilization at the homogenization pressures from 20 to 80 MPa with one to four homogenization cycles for the sludge samples with TS of 1.00, 1.49 and 2.48%. It was observed that an excellent linear relationship existed between SCOD solubilization and VSS reduction with a good correlation coefficient ($R^2 = 0.9899$). The slope of the fitted line was 1.6493, indicating that the SCOD increased by 1.65 g/L in the sludge supernatant corresponding to one unit VSS reduction (1.00 g/L). The results demonstrated that the increase of organics in the sludge supernatant resulted from sludge solid reduction through HPH treatment. Moreover, the linear relationship implied that the relationship between VSS reductions and SCOD solubilization was not changed with the change of HPH operating conditions and sludge solid content, although these factors could influence the change of VSS reduction and SCOD solubilization.

**Energy efficiency of HPH sludge disintegration**

Energy efficiency plays an important role in the cost-benefit analysis of a process. Figure 6 shows the effects of homogenization pressure and homogenization cycle number on the energy efficiency for sludge disintegration. Clearly, the energy efficiency firstly increased with the increase of homogenization pressure from 20 to 30 MPa, then decreased with the homogenization pressure in the range of 30–80 MPa. Namely, the maximum energy efficiency of sludge solubilization was obtained at a homogenization pressure of 20 MPa.
pressure of 30 MPa for all sludge samples. Though high pressure and multiple cycles were of benefit for the sludge disintegration, they also increased the energy consumption. As shown in Figure 1 and Figure 5, the sludge disintegration efficiency increased more and more slowly with increasing homogenization pressure and number of homogenization cycle. However, the energy consumption had a linear relationship to the homogenization pressure or number of homogenization cycle with a certain number of homogenization cycle or homogenization pressure, respectively, as shown in Equation (1). Therefore, a higher energy efficiency of the HPH treatment was probably obtained at a lower homogenization pressure with a single homogenization cycle. On the other hand, the energy efficiency was probably not very high at a very low homogenization pressure (such as 20 MPa) with a single homogenization cycle due to low sludge disintegration efficiency.

In addition, it was found that the increase of homogenizing cycle number resulted in energy efficiency reduction for SCOD solubilization, indicating that the operation with multiple homogenization cycles was energy intensive. The maximum energy efficiencies for SCOD solubilization were 46.92, 55.31 and 77.18 g/MJ at a homogenization pressure of 30 MPa with a single homogenization cycle for the sludge samples with a TS of 1.00, 1.49 and 2.48%, respectively.

In Figure 6, under the same homogenization pressure and homogenization cycle number, energy efficiency for sludge solubilization increased as the sludge TS increased within the range from 1.00 to 2.48%, which indicated that HPH treatment of high TS sludge was more energy efficient. However, a threshold value of TS (about 2.50%) was found for the high-pressure homogenizer used in the research, above which the sewage sludge could not be effectively disintegrated with the HPH treatment. Therefore, it was necessary to optimize the HPH process parameters, when HPH was applied to disintegrate sewage sludge with low energy consumption. On the other hand, some methods may be used together with HPH to improve the HPH sludge disintegration with higher sludge concentration, such as the combined treatment of alkaline and HPH (Zhang et al. 2012).

**CONCLUSIONS**

HPH treatment was effective for sludge disintegration, which led to sludge solid reduction and organics solubilization. The TSS, VSS reduction and SCOD, protein, polysaccharide solubilization were highly dependent on homogenization pressure and homogenization cycle number. The ratio between VSS reduction and TSS reduction was 86.15% for the sludge sample with a TS of 1.49%. Both the protein and polysaccharide solubilization showed a linear increase with the SCOD solubilization, and more proteins were solubilized than polysaccharides.
The VSS reduction and SCOD solubilization had an excellent linear relationship, and 1.65 g/L SCOD solubilization corresponded to per unit VSS reduction (g/L). Energy efficiency of sludge disintegration reached a maximum at 30 MPa in the homogenization pressure range of 20–80 MPa. The operation of multiple homogenization cycles led to a drop in energy efficiency. The higher the TS of the sewage sludge, the higher the energy efficiency was under the same operating conditions.

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