Ultrasound pre-treatment of waste activated sludge

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Abstract
Waste activated sludge (WAS) is more difficult to digest than primary sludge due to rate limiting cell hydrolysis. High-power ultrasound can effectively disintegrate the bacterial cells and thus enhance the subsequent digestion. This research examines the effectiveness of ultrasound pretreatment on WAS disintegration at different specific energy inputs, ultrasonic densities and total solids (TS) contents. The results show that the cut diameter ($d_{50}$) for WAS with 2% TS content declined nearly 6.5-fold at an ultrasonic density of 0.67 W/ml. For higher TS contents of 4 and 6%, higher densities of 1.03 and 0.86 W/ml, respectively, were needed to achieve the same degree of particle size reduction. The efficacy of ultrasonic disintegration measured as soluble chemical oxygen demand (SCOD) release was primarily governed by ultrasonic density (W/ml); whereas ultrasonic density did not show a significant effect on protein release at all TS levels. SCOD release of about 320 mg SCOD/g TS was obtained at a TS content of 2% and specific energy input of 5 kWs/gTS. The SCOD release, however, decreased to 160 and 90 mgSCOD/gTS at 4 and 6% TS contents, respectively. The highest protein release of 73 mg/gTS was obtained at a TS content of 2% and specific energy input of 10 kWs/gTS. The sludge disintegration efficiency declined significantly at higher TS content. Thus, there is a limiting TS concentration that could be effectively disintegrated by ultrasound, and this is governed by the capability of an ultrasonic unit in producing cavitation. The degree of disintegration also depends on types of ultrasonic unit used.

Keywords Digestion; sludge disintegration; specific energy input; ultrasonic density; ultrasound pre-treatment; waste activated sludge

Introduction
Biological methods such as aerobic and anaerobic digestion are widely used for sludge stabilization. Biological stabilization reduces the quantity of sludge to be disposed of, minimizes the offensive odor, and produces useful by-products, such as methane gas (during anaerobic digestion) and soil conditioners. Aerobic or anaerobic digestion of waste activated sludge (WAS), however, is often slow due to the rate limiting cell lysis step (Li and Noike, 1992). This is because the cell wall and the membrane of prokaryotic organisms consist of complex organic materials such as peptidoglycan, teichoic acid, and complex polysaccharides, which are recalcitrant to biodegradation (Pelczar et al., 1993) and requires a long retention time of 30 to 60 days during biological treatment. Thus, the WAS needs to be pretreated to enhance the digestibility and reduce the retention time. The aim of such pretreatment is to rupture the cell wall and membrane to release the intracellular matter into the aqueous phase for subsequent degradation. Pretreatments include physical (e.g. ball milling, ultrasonic, etc.), chemical (ozone, hydrogen peroxide, acid and base), thermal or biological (e.g. enzymatic hydrolysis). Lately, ultrasonic...
pretreatment has become popular for sludge disintegration due to several merits (Khanal et al., in press).

Ultrasound is a sound wave at a frequency above the normal hearing range of humans (>20 kHz). When the ultrasound wave propagates in a medium such as sludge, it generates a repeating pattern of compressions and rarefactions in the medium. The rarefactions are regions of low pressure, where microbubbles are formed. As the wave fronts propagate, microbubbles oscillate under the influence of positive pressure, thereby growing to an unstable size before violently collapsing. The sudden and violent collapse of huge numbers of microbubbles generates powerful hydro-mechanical shear forces in the bulk liquid (Kuttruff, 1991), resulting in rupturing of cell wall and membranes.

The sonication conditions (e.g., ultrasonic density, sonication time, specific energy input, etc.) need to be optimized to maximize sludge disintegration for efficient sludge digestion. The quantitative evaluation provides valuable information needed for design and process optimization of an ultrasonic system (Khanal et al., in press). Such information is not readily available in the literature. Therefore, the goal of this research was to examine the effects of different sonication conditions on WAS disintegration and to quantify sludge disintegration efficiency.

Methods

Waste activated sludge (WAS)

WAS samples were collected from the Boone Wastewater Treatment Plant, Boone, IA, USA on a biweekly basis and stored at 4°C prior to use. The 2.1 MGD (8000 m³/day) plant serves the city of Boone with a population of 12,000 and comprises an extended aeration type activated sludge process with solids retention time (SRT) of 25 to 28 days.

Ultrasonic pretreatment

The WAS samples were thickened by centrifugation to obtain three different total solids (TS) contents of 2, 4 and 6%. The important characteristics of TWAS at different TS contents are presented in Table 1. Sludge samples (500 ml) were sonicated in a batch mode using a laboratory-scale ultrasound unit - Branson 2000 Series (Branson Ultrasonics, Danbury, CT, USA). The ultrasound system has a maximum power output of 2.2 kW and operates at a frequency of 20 kHz. Sonication tests were carried out at four different amplitudes of 25, 50, 75 and 100% of the maximum power input. At each power level (or amplitude) and TS content, the sludge samples were sonicated for different durations of 0 (control), 10, 30, 60, 120 and 240 seconds. A cooling water stream

Table 1 Characteristics of thickened waste activated sludge (TWAS) at different TS levels

<table>
<thead>
<tr>
<th>Parameters</th>
<th>TS content</th>
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<tbody>
<tr>
<td></td>
<td>2%</td>
</tr>
<tr>
<td>Total chemical oxygen demand (COD), g/L</td>
<td>20.5–23.3</td>
</tr>
<tr>
<td>Soluble chemical oxygen demand (SCOD), mg/L</td>
<td>126–189</td>
</tr>
<tr>
<td>Volatile solids (VS), %</td>
<td>1.3</td>
</tr>
<tr>
<td>VS/TS ratio</td>
<td>0.72–0.74</td>
</tr>
<tr>
<td>Protein, mg/L</td>
<td>242–279</td>
</tr>
<tr>
<td>Ammonia, mg N/L</td>
<td>20–30</td>
</tr>
<tr>
<td>Total Kjeldahl nitrogen (TKN), mg N/L</td>
<td>600–660</td>
</tr>
<tr>
<td>Specific oxygen uptake rate (SOUR), mgO₂/gVSS-hr</td>
<td>29–30</td>
</tr>
<tr>
<td>pH</td>
<td>6.8–7.1</td>
</tr>
</tbody>
</table>
(temperature of 10°C) was constantly recirculated around the sonication chamber to maintain a constant temperature of 25 to 30°C during sonication.

**Calculation of energy/power input**

The specific energy input to the sludge was calculated for each sonication duration and TS content using Equation (1).

\[
E = \frac{P \cdot t}{v \cdot TS}
\]

where, \( E \): specific energy input (kWs/gTS); \( P \): power input (kW); \( t \): sonication time in seconds (s); \( v \): volume of sludge sonicated (L); and \( TS \): total solids (g/L).

The ultrasonic density was estimated using the following relationship:

\[
Q_{avg} = \frac{P_{avg}}{V}
\]

where, \( Q_{avg} \): average ultrasonic density (W/ml).

**Evaluation of sludge disintegration efficiency**

The sludge disintegration efficiency was judged based on microscopic examination, reduction in particle size, increase in SCOD and protein content, and SOUR.

**Analytical methods**

The WAS samples were analyzed for TS, VS, SCOD, TKN and NH₃ before and after sonication as per *Standard Methods* (1998). The protein concentration in the supernatant was determined spectrophotometrically with a UV/Visible spectrophotometer at an absorbance of 280 nm. The SOUR of TWAS was determined before and after sonication as discussed elsewhere (Khanal et al., 2006). The particle size distribution of TWAS before and after ultrasonic treatment at different power inputs was determined using a Malvern particle size analyzer (Mastersizer, 2000, Malvern Inc., Worcestershire, United Kingdom). Similarly, light microscopy images of nonsonicated and sonicated samples were taken to physically observe the sludge morphology.

**Results and discussion**

**Light microscopy examination of WAS**

Prior to sonication, several large floc structures (dark patches) were observed at all TS levels (Figure 1A, 1C and 1E). These patches were a few \( \mu \text{m} \) to several hundreds \( \mu \text{m} \) in sizes. During 240 seconds of sonication at ultrasonic density of 0.86 W/ml, the structural integrity of flocs was significantly disrupted with nearly complete destruction of floc-like structure for both 2 and 4% TS contents (Figures 1B and 1D). At 6% TS content, however, the floc structures were not entirely disintegrated as judged by the presence of several dark patches (Figure 1F). The disintegration efficiency appeared to decline at higher TS content. This could be due to hindrance in the formation of cavitation bubbles at higher solids levels and poor dissipation of ultrasound in the aqueous phase.

**Particle size distribution at different specific energy inputs, TS levels and ultrasonic densities**

The reduction in particle size reflects the efficacy of sonication on sludge disintegration. The particle size varied from 4 to 1000 \( \mu \text{m} \). The peaks centered at particle sizes of 230, 40, 35, 25, 22 and 18 \( \mu \text{m} \) at specific energy inputs of 0 (control), 1.7, 5.9, 10.19, 20.37 and 40.75 kWs/gTS, respectively.

The cut diameter (\( d_{50} \)) (that is 50% of the particles (by volume) with a diameter of \( d_{50} \) or lower) at different TS contents and ultrasonic densities is shown in *Table 2*. As...
apparent from the table, the \( d_{50} \) for 2% TS content decreased nearly 6.5-fold at an ultrasonic density of 0.67 W/ml. Thereafter, the \( d_{50} \) remained fairly constant. For TS contents of 4 and 6%, higher ultrasonic densities of 1.03 and 0.86 W/ml were needed to achieve a similar degree of particle size reduction. It is important to point out that during anaerobic digestion, hydrolysis is a rate-limiting step for the waste containing a considerable amount of particulate matter (e.g., sewage sludge). The rate of methane production in a mature digester is proportional to the net rate of particle solubilization (Gujer and Zehnder, 1983), and the rate of solubilization is governed by the size of particles in the wastes. Thus, the breaking down of bioflocs into finer particles will significantly improve the digestibility of WAS.

**SCOD release at different specific energy inputs, TS levels and ultrasonic densities**

The increase in SCOD is commonly adopted as a measure of ultrasonic disintegration efficiency. TS content is considered to be one of the most important parameters affecting disintegration efficiency. This study examined high TS levels, and the specific SCOD release was calculated at all TS levels. The SCOD release (mg/gTS) at different specific energy inputs (kWs/gTS) for various TS contents and ultrasonic densities is shown in Figure 2. As apparent from the figure, the SCOD release showed an increasing trend with increase in both specific energy input and ultrasonic density at all TS contents. At lower

<table>
<thead>
<tr>
<th>TS Level</th>
<th>Ultrasonic Density (W/ml)</th>
<th>( d_{50} ) (μm)</th>
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<tbody>
<tr>
<td>2%</td>
<td>0</td>
<td>209.1</td>
</tr>
<tr>
<td></td>
<td>0.67</td>
<td>32.4</td>
</tr>
<tr>
<td></td>
<td>1.28</td>
<td>28.5</td>
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<tr>
<td></td>
<td>2.2</td>
<td>22.6</td>
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<tr>
<td></td>
<td>3.22</td>
<td>18.1</td>
</tr>
<tr>
<td>4%</td>
<td>0</td>
<td>217.3</td>
</tr>
<tr>
<td></td>
<td>0.44</td>
<td>185.1</td>
</tr>
<tr>
<td></td>
<td>1.03</td>
<td>36.7</td>
</tr>
<tr>
<td></td>
<td>1.87</td>
<td>33.4</td>
</tr>
<tr>
<td></td>
<td>2.24</td>
<td>38.2</td>
</tr>
<tr>
<td>6%</td>
<td>0</td>
<td>225.2</td>
</tr>
<tr>
<td></td>
<td>0.45</td>
<td>120.7</td>
</tr>
<tr>
<td></td>
<td>0.86</td>
<td>38.4</td>
</tr>
<tr>
<td></td>
<td>2.12</td>
<td>29.8</td>
</tr>
<tr>
<td></td>
<td>2.14</td>
<td>33.4</td>
</tr>
</tbody>
</table>
TS content of 2%, SCOD release increased by just 90 and 120 mg/gTS with respect to control at specific energy input of 5 kWs/gTS for the lower ultrasonic densities of 0.62 and 1.19 W/ml, respectively (Figure 2A). The SCOD release increased to nearly 320 mg/gTS with respect to control at the same energy input with further increase in ultrasonic density to 2.03 and 3.05 W/ml. The SCOD release, however, slowed down at higher specific energy inputs for all ultrasonic densities. The higher TS contents of 4 and 6% also showed a similar trend with respect to SCOD release. With the same energy input of 5 kWs/gTS, the maximum SCOD releases were only about 160 and 90 mg/gTS, respectively at TS contents of 4 and 6% under maximum ultrasonic density (Figure 2B and 2C). Thus, for efficient sludge disintegration, ultrasonic density plays a more important role than the specific energy input.

Better sludge disintegration was achieved at a longer sonication time, and thus at a higher specific energy input. This is because at longer sonication times, there was ample opportunity for cells and debris to come under perpetual attack of collapsing cavitation bubbles. Interestingly, SCOD release decreased with increase in initial TS content. This finding contradicts earlier studies that reported significant improvement in SCOD release (Grönnroos et al., 2005; Wang et al., 2005; Khanal et al., 2006). Previous research, however, examined rather low TS levels of 1.0 to 3.0%, and SCOD released was expressed as mg/L, instead of specific SCOD release (mgSCOD/gTS), which made it difficult to compare the SCOD release data. At higher TS content, the propagation of ultrasound energy in the slurry phase might have declined, resulting in poor cavitation. In addition, the lack of soluble gas at higher TS level might also have inhibited the formation of cavitation. It is important to point out that the degree of disintegration is also governed by the types of ultrasonic unit used. Thus, each ultrasonic unit must be tested at wide ranges of TS contents and amplitudes before full-scale application.

**Protein release at different specific energy inputs, TS levels and ultrasonic densities**

The protein content increased with increase in specific energy inputs at all TS contents as shown in Figure 3. The protein release increased rapidly up to specific energy input of
10 kWs/gTS at 2% TS content; thereafter it slowed down (Figure 3A). For higher TS contents of 4 and 6%, the protein release, however, slowed down at specific energy input of <5 kWs/gTS (Figure 3B and 3C). The maximum protein releases with respect to control were 73 (at 2% TS content and specific energy input of 10 kWs/gTS), and 40 and 22 mg/gTS (at TS contents of 4 and 6%, respectively, and specific energy input of 5 kWs/gTS). The protein release was significantly reduced at higher TS content. This is most likely due to decreasing cavitation effect at higher TS level. Another notable observation at higher TS levels was the lower ultrasound density even though the sludge was sonicated at the same amplitudes. This apparently suggests that lower power was dissipated to the sludge at higher TS content. One study that examined protein release, reported maximum protein release of c. 83 mg/gTS with 3% TS content at much higher specific energy input of 50 kWs/gTS (Wang et al., 2006), which was nearly a five-fold higher energy input than this study. Thus, the energy input needed to obtain the same degree of sludge disintegration depends on the type of ultrasonic unit employed. In contrast to SCOD release, protein release was not significantly affected by ultrasonic density at all TS levels. This statement contradicts many of the earlier studies, which reported that ultrasonic density is more important for sludge disintegration than the specific energy input (Grönroos et al., 2005; Wang et al., 2006). These studies, however, reported sludge disintegration efficiency in terms of SCOD release and did not examine the protein release.

All earlier studies examined the degree of disintegration by SCOD release. Schmitz et al. (2000) argued that the degree of disintegration in terms of SCOD release (DDCOD) is rather slow in the range of a day and is also expensive due to the need of large numbers of COD sample analyses. The authors therefore proposed protein measurement as an alternative to DDCOD determination. In their study, correlation coefficients ($R^2$) for increase in protein ($\Delta_{\text{Protein}}$), DDCOD$_1$, and DDCOD$_2$ with respect to increase in biogas yield ($\Delta_{\text{biogas}}$) due to sonication were compared to evaluate the reliability of the new sludge disintegration assessment technique. When the sludge samples were collected twice from one plant and once from another plant and sonicated, the authors found that
the combined coefficients for $\Delta_{Protein}/\Delta_{biogas}$ were much higher ($R^2 = 0.97$) than that for $\Delta_{DCOD1}/\Delta_{biogas}$ ($R^2 = 0.54$) and $\Delta_{DCOD2}/\Delta_{biogas}$ ($R^2 = 0.83$) suggesting that irrespective of sources of sludge and times of collection, protein determination was a more reliable technique of assessing the ultrasonic disintegration potential of sludge. More research is needed to correlate the sludge digestibility with the degree of disintegration.

Specific oxygen uptake rate (SOUR) at different specific energy inputs and ultrasonic densities

As shown in Figure 4, the SOUR of sludge (with 2% TS) declined almost exponentially following sonication at specific energy input of 10 kWs/gTS or less. The maximum degree of inactivation [(SOUR$_{control}$-SOUR$_{sonicated}$)/SOUR$_{control}$] was about 65%. The ultrasonic density did not have significant effect on SOUR. Similar trends were also observed for higher TS contents of 4 and 6% (data not shown). In terms of duration of sonication, microbial inactivation of 53 to 69% was achieved during 60 second of sonication, and the SOUR values did not change appreciably during longer sonication (Figure 5). The SCOD, however, continued to increase at longer sonication (data not shown). This apparently suggests that microbes are inactivated well ahead of their...

Figure 4 Specific oxygen uptake rate (SOUR) of TWAS at different specific energy inputs and ultrasonic density (2% TS content)

Figure 5 Effect of TS contents on SOUR at different sonication times (Ultrasonic density: 2.07 – 3.05 W/ml)
Conclusions
This study examined different methods to evaluate WAS disintegration efficiency at various TS contents, specific energy inputs and ultrasonic densities. The results show that the cut diameter ($d_{50}$) for WAS with 2% TS content declined nearly 6.5-fold at an ultrasonic density of 0.67 W/ml. For higher TS contents of 4 and 6%, higher densities of 1.03 and 0.86 W/ml, respectively, were needed to achieve the same degree of particle size reduction. The ultrasound disintegration, measured in terms of SCOD release was found to be strongly dependent on both TS content and ultrasonic density. The SCOD release of about 320 mg SCOD/gTS was obtained at TS content of 2% and specific energy input of 5 kWs/gTS. The protein release, however, was not significantly affected by ultrasonic density. The highest protein release of 73 mg/gTS was obtained at TS content of 2% and specific energy input of 10 kWs/g. Higher TS content did not necessarily improve the sludge disintegration. The degree of inactivation (based on SOUR) was found to decline by as much as 60% at specific energy input of 10 kWs/gTS at 2% TS content. Although this study clearly demonstrated the effectiveness of ultrasound on sludge disruption, the most appropriate method of evaluating sludge disintegration efficiency based on sludge digestibility (aerobic or anaerobic) needs to be examined.

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