Sewage sludge pretreatment by microwave irradiation combined with activated carbon fibre at alkaline pH for anaerobic digestion

Dedong Sun, Sixiao Guo, Nina Ma, Guowen Wang, Chun Ma, Jun Hao, Mang Xue and Xinxin Zhang

ABSTRACT

This research focuses on the effects of microwave-assisted activated carbon fibre (ACF) (MW-ACF) treatment on sewage sludge at alkaline pH. The disintegration and biodegradability of sewage sludge were studied. It was found that the MW-ACF process at alkaline pH provided a rapid and efficient process to disrupt the microbial cells in the sludge. The results suggested that when irradiated at 800 W MW for 110 s with a dose of 1.0 g ACF/g solid concentration (SS) at pH 10.5, the MW-ACF pretreatment achieved 55% SS disintegration, 23% greater than the value of MW alone (32%). The concentration of total nitrogen, total phosphorus, supernatant soluble chemical oxygen demand, protein, and polysaccharide increased by 60%, 144%, 145%, 74%, and 77%, respectively. An increase in biogas production by 63.7% was achieved after 20 days of anaerobic digestion (AD), compared to the control. The results indicated that the MW-ACF pretreatment process at alkaline pH provides novel sludge management options in disintegration of sewage sludge for further AD.

Key words | activated carbon fibre, batch aerobic digestion, microwave, sewage sludge, sludge lysis

INTRODUCTION

Municipal sewage treatment plants produce large amounts of excess sludge, containing organic bacterial microbes. In China, for example, approximately 35 million tons of dewatered sludge cake (80% moisture content) was produced in 2014. Treatment and disposal of excess sludge from municipal sewage treatment plants can account for up to 50–60% of the total cost of sewage treatment. Anaerobic digestion (AD) as an effective technology for excess sludge treatment has been widely used. Mesophilic AD (MAD) of sewage sludge is an economic approach to reduce the amount of sludge to be disposed. Baier and Schmidheiny (Baier & Schmidheiny 1997) stated that the sewage sludge is mostly composed of microbes and cell walls are physical barriers for digestion. An anaerobic degradation of organics consists of three steps: hydrolysis, acidogenesis, and methanogenesis (Park et al. 2004). In sewage sludge digestion, hydrolysis of the sludge solids is a rate-limiting step. During this process, extracellular enzymes break down and solubilize microbial cells as substrates in subsequent reactions (Ahn et al. 2009). MAD of sewage sludge usually requires long retention times of the order of 20–30 days in conventional digesters (Tyagi & Lo 2013). Extensive research indicates that sludge pretreatment processes could disrupt the extracellular polymeric substances and, therefore, improve the biodegradability of sewage sludge (Tyagi & Lo 2013). Therefore, to increase digestion efficiency, various cell-lysing methods are used in waste activated sludge pretreatment process include ultrasound (Zhang et al. 2007), chemical (Tyagi & Lo 2011), thermal (Morgan-Sagastume et al. 2011) and mechanical disintegration (Kampas et al. 2007) pretreatment methods.

Previous studies have indicated that alkali pretreatment can enhance the biodegradation of solid wastes when combined with thermal hydrolysis (López Torres & Espinosa Lloréns 2008). At present, microwave (MW) irradiation was used extensively as an effective thermal method for sludge solubilization (Tyagi & Lo 2011). Comparing with conventional heating method, the MW irradiation technique has a higher energy efficiency. Studies showed that combined MW–alkali pretreatment might become an efficient method for sludge disintegration (Chang et al. 2011). Studies have reported that MW-induced pyrolysis was possible if the
sewage sludge was mixed with carbon as an effective receptor of MW energy (Menéndez et al. 2002). In this research, activated carbon fibre (ACF) was selected as an optimum MW absorbing material. To the best of our knowledge, no studies have been conducted to evaluate the effectiveness of the MW-ACF pretreatment on sludge disintegration and subsequent aerobic digestion. The objectives of this study were to evaluate the synergetic effect of the combined MW-ACF pretreatment at alkaline pH for sludge solubilization and subsequent aerobic digestion.

Materials and Methods

Materials

Raw sewage sludge was obtained from a local municipal sewage treatment plant, with a design capacity of 60,000 m³/day, in Dalian, China. The volatile solid concentration (VSS) to solid concentration (SS) ratio was 74% in the sludge, indicating that sludge consisted mainly of organic substances. The sludge was kept in storage at concentrations of 2% at 4°C and diluted with distilled water before use.

Commercial activated carbon fibres were purchased from Sutong Carbon Fibre Company, China. The ACF is feltly, 3–4 mm in thickness, and 10–20 μm in diameter.

Pretreatment

Sludge was heated using a MW oven (2,450 MHz, 800 W, KD21B-C, Shantou Huanhai Co., Ltd) with sealed Teflon vessels. The sludge samples were placed in closed Teflon vessels. An ACF dose of 0–1.0 g ACF/g SS was mixed by sewage sludge. The pH value of sludge was adjusted at 7.5, 8.5 and 10.5, respectively, by adding 0.5 mol/L sodium hydroxide (NaOH) before heating by MW. All test samples were heated by MW with power settings of 800 W and the MW heating time was 110 s according to the feasibility study before (Sun et al. 2014). After the treatment, the sludge was cooled to room temperature and the ACF was removed from the Teflon vessel prior to analysis.

Samples analysis

In this study, parameter measurement included pH, SS, VSS, soluble chemical oxygen demand (SCOD), supernatant total nitrogen (TN), total phosphorus (TP), protein and polysaccharide. Parameter measurement was determined according to Standard Methods (APHA et al. 2005). The samples’ pH were measured with a pH meter (PHB-5, Shanghai, Weiye Co., Ltd).

Anaerobic digestion experiment

AD experiments with pretreated sludge and raw sludge were performed. Two batch reactors were the same size with an effective volume of 500 mL each. One batch reactor was fed with pretreated sludge, while the other fed with raw sludge as the control. The volume of methane from the reactor was determined by using a measuring cylinder connected to the reactor (Lin et al. 2009). The AD experiments were run for 20 days, and methane yield was determined during the digestion experiment.

Results and Discussion

Solid phase changes

Although sludge is a poor receptor of MW energy, the solubilization of sludge is possible if the sludge is mixed with an effective receptor of MW energy. The MW energy absorbed by ACF may quickly convert to heat, and MW irradiation provided a rapid temperature increase. Sludge consists mainly of organic bacterial microbes, containing carbohydrates, lipids and proteins. Thermal hydrolysis accelerated the solubilization of organic bacterial microbes, and the release of organic matter from sludge decreased SS concentration. Volatile fatty acid was formed by hydrolysis of organics, so pH value decreased. The SS solubilization of MW-ACF treated sludge is plotted in Figure 1. The SS solubilization ratio for sludge had a clear climbing trend with the increasing of ACF dose. As shown in Figure 1, for only 110 s, the SS...
dissolving ratio increased from 32 to 55% with an increase of ACF dose from 0 g/gSS to 1.0 g/gSS at pH 10.5. The effects of initial pH of sludge on the sludge solubilization were also investigated. From Figure 1, when the ACF dose was 1.0 g/gSS, the sludge solubilization was 27% at pH 7.5 and 47% at pH 8.5, and it increased to 55% as the pH was increased to pH 10.5. Therefore, combined MW-ACF treatment at alkaline pH might act synergistically and enhanced sludge disintegration.

The effect of initial SS concentrations on sludge solubilization was also studied with a dose 1.0 g ACF/gSS at pH 10.5. As shown in Figure 2, the SS solubilization ratio decreased with the increasing of the concentration of sludge. When increasing the initial SS concentrations from 3.2 to 4.3 g/L, 7.4 and 10.2 g/L, it was clear that the SS solubilization ratio decreased obviously from 54 to 49, 35 and 27%. MW heating led fast sludge hydrolysis reaction. Sludge solid content was found to be an influential parameter. When the initial SS concentration was increased, sludge hydrolysis reaction slowed down and the SS solubilization ratio decreased.

**Liquid phase changes**

Combined MW-ACF pretreatment at pH 10.5 can disintegrate the sludge and release extracellular and intracellular biopolymers from the cell structure into the soluble phase. The solubilization degree of the sludge can be estimated from a variety of measurements, including the concentrations of TN, TP, SCOD, protein, and polysaccharide. All of these measurements were greater in the pretreated sludge than in the raw sludge, and this difference increased with ACF dose (Figures 3 and 4).

SCOD increase means that sludge solubilization releases organic matters from sludge into supernatant, which contributes to subsequent AD. A concentration curve of SCOD at different treatment conditions is shown in Figure 3. SCOD concentration benefited from the increase of sludge solubilization and, thus, showed stable increase with ACF dose. Fast sludge solubilization resulted in a significant increase in SCOD concentration. The liquor SCOD concentration at pH 10.5 with 1.0 g ACF/gSS reached above 3,000 mg/L. A high dose of ACF results in an increase of the cost of pretreatment, but AD of sludge can be obviously improved. Likewise, a low dose of ACF means a low capital, but AD of sewage sludge improvement may not be sufficient. Thus, there might exist an optimal ACF dose, and the optimal total cost might exist at an optimal ACF dose considering other relevant costs.

Figure 3 also displays pH changes after the sludge dissolving and pH value of sludge dropped from 10.5 to 8.9. As Figure 3 shows, the final pH of pretreated sludge was
found to be obviously lower than the initial pH, possibly due to dissolution of organic acids generated by complex organic matter in combination with sludge lysis.

The protein and polysaccharide changes represent cell lysis and flocs disintegration that release these organic matters into the supernatant. The release of these organic matters contributes to SCOD increase. The changes of protein, polysaccharide, TN and TP in supernatant are shown in Figure 4. During the course of MW irradiation, there were obvious rises in all these parameters. The supernatant TN, TP, protein, and polysaccharide increased by 43 mg/L, 14 mg/L, 109 mg/L and 70 mg/L, respectively, when the ACF dose was 1.0 g/gSS and the MW irradiation time was 110 s.

Concentrations of polysaccharide showed a relatively linear increase with ACF dose up to the top point (Figure 4), but showed no further increases at higher ACF dose. This increase may be owing to disintegration of cell wall polysaccharides, which are composed of many monosaccharide units connected by glycosidic bonds (Ahn et al. 2009). The combined MW-ACF treatment may break these glycosidic linkages within the polysaccharides at alkaline pH.

With the increase of ACF dose, the concentration of soluble protein first increased and then slowed down. At ACF dose of 0.7 g ACF/gSS, an increase of 96 mg/L of protein was observed. Supernatant protein is bio-available organic matter, which contributes to subsequent AD, and protein can be degraded more effectively by the hydrolytic and acidogenic microbes for protein conversion under an alkaline condition (Liu et al. 2012). However, a further increase in the ACF doses did not result in a further increase in the protein. The MW energy absorbed by ACF may quickly convert to heat, resulting in rapid increase of temperature in ACF body. As temperature increased, the protein stability decreased because some of the chemical bonds that stabilize the protein’s structure may break (Ahn et al. 2009).

Compared to nitrogen, protein and polysaccharide, TP concentration continuously increased even when ACF dose reached 1.0 g ACF/gSS (Figure 4). TP mostly comes from the cell wall of sludge (Zhang et al. 2009). All of these results suggested sludge lysis contributed to the increase of proteins, polysaccharides, SCOD, TN and TP.

**Potential mechanisms**

Such behaviours of supernatant protein, polysaccharide, SCOD, TN, and TP have not been reported before. Menéndez et al. (2002) reported that if just the raw sewage sludge is irradiated using the MW, only drying of the sludge takes place. However, if the sewage sludge is mixed with activated carbon (AC) as an effective MW receptor, high temperatures can be reached in several minutes, so that sludge lysis takes place rather than drying.

MW heating alone cannot generate hydroxyl radical (•OH) because MW has only the energy of 10^-6–10^-3 eV, which is not enough to break chemical bonds (Quan et al. 2007). Quan et al. (2007) reported that AC can generate •OH in MW field. AC is an effective receptor of MW energy. The MW energy absorbed by AC can result in rapid increase of temperature in AC. During MW irradiation, the temperature of the AC surface might be high enough to produce •OH. Booske et al. (1992) supposed that the localized resonant coupling of MW energy in solid materials could concentrate a portion of MW energy into hot spots. The hot spot could contribute to the initiation of •OH generation (Quan et al. 2007). We proposed that •OH disintegrates sludge mainly through the damage of cells. Hydroxyl radical penetrates into the microorganism cells, damages the uniformity of the cell walls, and the intracellular substances are released into supernatant. This process contributes to the rapid initial increase of SCOD, TN, TP, protein, and polysaccharide. Powdered activated carbon and granular activated carbon are the first generation and the second generation of AC, respectively (Zhang & Song 2001). ACF as a third-generation activated carbon is a new type of highly efficient catalysts and, therefore, plays an important role in the generation of •OH under MW irradiation.

The action mechanism of alkali pretreatment is to induce the swelling of the cellular substances at high pH, making particulate organics more susceptible to enzymatic reaction (Chi et al. 2011). Thus, the combination of MW thermal pretreatment with ACF method at alkaline pH can synergistically enhanced the sludge disintegration.

**Methane yield**

Introducing MW-ACF-alkali pretreatment to a digestion system can remarkably improve AD efficiency of sludge. The effects of the pretreatment on the AD, with respect to a control system, were evaluated. In this study, anaerobic digesters were run for 20 days under identical operating conditions. Both digesters operated on a batch basis. In order to keep sludge from setting, both digesters consisted of stirred reactors. Each digester was flushed with pure nitrogen for 4 min to replace the air. The reaction temperature was always maintained at 35 ± 2 °C during AD experiments. The results showed that the cumulative biogas production from the reactor fed with pretreated sludge was greater. As
shown in Figure 5, the percentage increase in methane production showed a relatively linear increase on days 0–5, then showed a slow increase up to the top point. When the system was stable, more than 60% increase of methane yield was attained with the pretreated system because of more soluble organic material contained. Compared with the control system, the cumulative biogas production in pretreated sludge increased by 63.7% at the end of anaerobic reaction. MW-AC pretreatment at alkaline pH should benefit not only sludge disintegration, but also increase the surface area available for enzymatic action, which improves AD performance (Lin et al. 2009).

Figure 6 indicates that the peak value of biogas production rate was attained on day 8 at 92 mL/d in reactor with the pretreated sludge, but the maximum was reached on day 8 at 55 mL/d in reactor with the raw sludge, which indicated that MW-ACF pretreatment at alkaline pH can increase methane yield.

CONCLUSIONS

This paper studied the use of MW-ACF at alkaline pH for disintegrating sewage sludge as a pretreatment of AD. It was seen that a pretreatment with MW-ACF at alkaline pH effectively leads to a disintegration of sewage sludge and achieves 55% SS solubilization. The combined MW-ACF pretreatment at alkaline pH rendered a significant increase in the SCOD, TN, TP, protein and polysaccharide, thus, improving AD performance, measured by an increase of biogas yields up to 63.7% over the raw sludge.

REFERENCES


First received 1 December 2015; accepted in revised form 2 March 2016. Available online 15 March 2016