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Short Communication

Increased solubilization of excess sludge does not always result in enhanced anaerobic digestion efficiency

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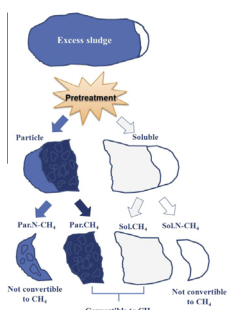
HIGHLIGHTS

- BMP of whole and soluble fraction of pretreated (alkali + ultrasonication) sludge.
- Decrease of CH_4 conversion of soluble fraction with solubilization increase.
- In case of severe pretreatment (pH 13 + Ul 60 min), only 61% was converted to CH_4.
- Soluble but non-biodegradable portion at (pH 13 + Ul 60 min) = 25.8%.
- Increased solubilization does not always result in enhanced AD efficiency.

GRAPHICAL ABSTRACT



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ABSTRACT

"Solubilization (SCOD/TCOD, chemical oxygen demand (COD), %)" is generally used as an indicator for the pretreatment effectiveness in anaerobic digestion (AD) of excess sludge. In the present work, ultrasonic (irradiation 5–60 min) and alkaline pretreatments (pH 9–13) were applied to excess sludge individually and in combination. Biological methane potential tests were carried out using the whole part of pretreated sludge and only the soluble fraction. Although solubilization increased with pretreatment intensity increase, methane production was inconsistent with increased solubilization, due to the lower methane yield of soluble fraction. While the soluble fraction obtained under mild pretreatment condition (pH 9 + ultrasonication 5 min) showed 91% of methane yield, it was only 61% in case of severe pretreatment condition (pH 13 + ultrasonication 60 min). 25.8% of the pretreated sludge at (pH 13 + ultrasonication 60 min) consisted of soluble but non-biodegradable portion. These findings demonstrate that increased solubilization does not always result in enhanced AD efficiency.

1. Introduction

One of the major challenges in wastewater treatment is the management of excess sludge whose treatment cost accounts for up to 60% of the total operating costs of municipal wastewater treatment plants (WWTPs) (Yan et al., 2013). The amount of sludge produced in Korea has continuously increased with the increasing number of WWTPs, recently reaching 3 million tons of sludge cake per year (MOE, 2011). While the sludge is ultimately disposed by ocean dumping, incineration, or land filling, it is important to minimize its production, not only for economical operation of WWTPs but also for global environmental protection. Anaerobic digestion (AD) is the most traditional and widely used excess sludge treatment method, offering advantages of generation of energy-rich gas in the form of methane (CH_4) along with reduction of sludge

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volume. However, its application is often limited by low overall degradation efficiency (20–50%), which is associated with the hydrolysis step of excess sludge (Kuglarz et al., 2013). The major organic fractions of excess sludge are microbial cells, and their cell walls contain glycan strands, which cause resistance to biodegradation. In order to improve digestion efficiency, numerous disintegration methods such as mechanical, chemical and thermal have hence been applied as pretreatment: these methods disrupt cell walls and readily available intracellular organic materials are released (Appels et al., 2008).

Ultrasonication is recognized as the most powerful method to disintegrate sludge cells. Rapid collapse and expansion of microbubbles generated by cavitation give rise to high-temperature and high-pressure gradients in the liquid phase, leading to rupture of cell walls and membranes (Pilli et al., 2011). Cell disintegration of 100% can be achieved at high power levels, but power consumption then becomes a serious economic burden (Weemaes and Verstraete, 1998). Recently, ultrasonicationhas often been combined with alkaline pretreatment (Kim et al., 2010; Li et al., 2010). When ultrasonication was applied to the alkali-pretreated sludge at pH 9–11, a high disintegration degree of sludge was obtained at a low energy level of ultrasonication and a synergistic disintegration effect was observed.

Solubilization, defined as the ratio of soluble organics (usually expressed as "chemical oxygen demand (COD)") to total organics, is generally used as an indicator for the pretreatment effectiveness of excess sludge (Appels et al., 2008; Bougrier et al., 2005). However, according to our findings, increased solubilization does not always result in enhanced AD efficiency, particularly, when a high strength level of pretreatment is applied. Wilson and Novak (2009) reported that temperature higher than 180 °C led to the production of recalcitrant soluble organics or toxic/inhibitory intermediates, thereby reducing the biodegradability of the sludge. According to Park et al. (2012), although combined alkaline and ultrasonic pretreatment increased the initial rate of CH_4 production by increasing solubilization, it had a negligible effect on the ultimate CH_4 yield. Bougrier et al. (2006) found that, in spite of the same solubilization, sludge pretreated by ultrasonication showed higher biodegradability than those pretreated by ozonation and thermal treatment. These findings have following important implications: (1) some part of the soluble fraction generated by pretreatment is not converted to CH_4; (2) some part of particle fraction weakened by pretreatment but not solubilized is converted to biogas; and, finally, (3) increased solubilization cannot be directly linked to improved digestion efficiency.

In the present work, we investigated the relationship between increased solubilization and AD efficiency at various pretreatment conditions. Ultrasonic and alkaline pretreatments were applied to excess sludge individually and in combination. Solubilization at various pretreatment conditions was measured, and biological methane potential (BMP) tests were carried out using not only the whole part of pretreated sludge but also the soluble fraction. The sludge was fractionated into four parts to ascertain the major constituent of total CH_4 produced and attain a deep understanding on the efficiency of pretreatment.

2. Methods

2.1. Excess sludge and inoculum

The excess sludge used in this experiment was taken from the sludge thickener line of Daejeon WWTP. The total COD concentration of excess sludge was 19.6 g/L while 6% of the total sludge were soluble. Total nitrogen (TN) concentration was 1.2 g/L and pH was 7. From the same WWTP, the inoculum, flocculated sludge, for the BMP test was obtained from an anaerobic digester. The pH, alkalinity, and volatile suspended solids (VSS) concentration of the sludge were 7.5, 2.83 g $CaCO_3$/L, and 5.3 g/L, respectively.

2.2. Disintegration methods

Ultrasonic treatment was performed using an ultrasonicator with a frequency of 20 kHz (VCX-750, Sonics and Materials, USA). Ultrasonic treatment (750 W, amplitude = 40%) was applied to 100 mL of excess sludge for 5, 10, 20, 40, and 60 min. In the alkaline pretreatment, a 6 mol/L KOH solution was added to 300 mL of excess sludge in order to reach pH values of 9, 10, 11, 12, and 13. The samples were mixed for 1 h using a magnetic stirrer at 200 rpm. The expected pH levels were achieved instantaneously, but dropped slightly with time. Therefore, the injection of alkaline solution was continued throughout the hour. In the combined pretreatment, alkali-pretreated sludge at pH 9, 11, and 13 for 1 h was ultrasonicated for 5, 20, and 60 min, totally 9 combinations. After pretreatment, the sludge was centrifuged (10,000 rpm for 20 min) and filtered through a 0.45 μm pore size membrane filter to obtain the soluble fraction.

2.3. BMP test and analysis

BMP tests were carried out using a 250 mL serum bottle (effective volume of 100 mL). After placing 69 mL of inoculum, 30 mL of pretreated excess sludge (whole part or soluble part), and 1 mL of concentrated trace element solution into the serum bottle, N_2 gas was purged for 5 min to establish an anaerobic condition. A trace element solution was made according to Kim et al. (2006). The initial pH was set to 7.5 ± 0.1 using 3 mol/L KOH and 3 mol/L HCl. The bottles were incubated at 35 °C using a water bath with agitation at 150 rpm using a shaking incubator. The tests were carried out in triplicate and the results were averaged.

The volume of produced biogas was measured by the displacement of a glass syringe piston, and was converted to a value at standard temperature and pressure (STP, 0 °C and 1 bar) conditions. Biogas at the head space in the reactors was collected using a gas-tight micro syringe, and the biogas composition was analyzed by a gas chromatograph (Gow-Mac Series 580, USA) equipped with a thermal conductivity detector and a stainless steel column. The temperatures of the injector, column, and detector were kept at 50, 80, and 90 °C, respectively. The carrier gas was N_2 and the flow rate was 30 mL/min. The concentrations of COD, TN, alkalinity, and VSS were measured according to standard methods in Part 5220, Part 4500, Part 2320, and Part 2540, respectively (APHA, 2005).

3. Results and discussion

3.1. Ultrasonication

As listed in Table 1, the increase of ultrasonication strength resulted in the increase of both solubilization and CH_4 yield of excess sludge, indicating that the ultrasonication applied in this experimental range was effective for enhancing AD efficiency. As irradiation time increased to 60 min, solubilization and CH_4 yield continuously increased from 6% to 33% and 25.7% to 43.2%, respectively. CH_4 yield was obtained by comparing the actual amount of CH_4 produced to the theoretical maximum (1 g COD = 350 mL CH_4) (Kim et al., 2010).

Solubilization of raw (untreated) sludge was 6% and 75.4% of the soluble fraction was converted to CH_4, which contradicts the general understanding that most of the soluble fraction can be converted to CH_4 by AD. With 5 min of ultrasonication, the CH_4 yield of