STUDY TO EVALUATE THE EFFECT OF ULTRA-SONICATION PRIOR TO MICROWAVE DISINTEGRATION ON ANAEROBIC DIGESTION OF DAIRY SLUDGE

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Abstract
The aim of this work is to study the effect of ultra-sonic pretreatment effeminacy prior to microwave (MW) irradiation on dairy sludge characterization and biogas yield. Pretreatment experiments were performed as batch process with 250 mL of sludge placed in ultra-sonication and followed by MW irradiation. Results revealed in higher trend of soluble chemical oxygen demand (SCOD) and dissolved organic carbon (DOC), both, are considered as index for evaluating the efficiency of sludge solubilization. However, further investigation is needed to determine the potential enhancement of methane production after this combined process.

1. Introduction

Unlike conventional heating, heating via Microwave irradiation is delivered directly into the material through a molecular interaction with electromagnetic field and converted into thermal energy. Therefore, emerging research has focused on the effect of MW irradiation on sludge dewaterability, solubility and biogas production. Microwave irradiation is expected to increase the surface area of the biomass and decrease the polymerization and crystallinity of the cellulose and therefore, improve the enzymatic degradation of the organic matter and consequently the digestion itself (Sapci, 2013). The effect of microwave occurs as a result of the interaction between the electrical field with dipolar molecules such as water, proteins, fats and other organic complexes. Hence, changing dipole orientation in the polarized side chains of the cell membrane macromolecules, and results in a breakage of hydrogen bonds and subsequently leading to the disintegration of floc matrix and changes the protein structures of the microorganisms (Appels et al., 2013). Therefore, MW irradiation is expected to enhance the solubility of proteins and volatile fatty acids, while reduce the solubility of sugars (Qiao et al., 2008). Kavitha et al. (2016) evaluated the effect of MW irradiation on COD solubilization and SS reduction and reported a significant enhancement of 22% and 17%, respectively. Other studies have evaluated the effect of MW treatment as a function of temperature such as Toreci et al. (2009) which reported high degrees of sludge solubilization after microwave irradiation when sludge temperature exceeds 175 °C. On the other hand, the study of Eskicioglu et al., (2007) investigated the effect of MW irradiation on waste activated sludge (WAS) in low temperatures (50–96) °C and claimed an enhancement of biogas production up to 16%. Other studies such as Apples et al., (2013) has reported an enhancement of biogas production up 50% in low temperatures by energy input of 336 (KJ/Kg). An interesting findings of the latter study stated that while MW irradiation significantly enhanced the solubilization of the organic materials to the aqueous phase; it did not affect the degradation of the major organic component at temperatures below 80 °C. Therefore, microwave irradiation is expected to increase the conversion of organic into easily accessible compounds (Kovács et al., 2018). Destruction of microorganisms occurs generally due the thermal effects of microwave exposure, however, several studies have investigated whether such irradiation has athermal effect (Tyagi & Lo, 2013).

The prime effect of MW irradiation leverages dielectric parameters such as temperature, radiation time, and penetration depth (Kavitha et al., 2016). The major disadvantage of MW pretreatment is the energy consumption during the pretreatment process. However, the high energy requirement of microwave can be downsized by combining with other disintegration methods to disrupt the flocs such as ultra-sonication (Pilli et al., 2011). Sludge exposure to ultra-sonication is expected to cause higher rate of Extracellular Polymeric Substances (EPS) release. As a result, disrupting the flocs through the release of EPS will enhance the disintegration efficiency. Hence. the main objective of the present study was to disrupt the flocs with ultra-sonication, then evaluate its disintegrattion through subsequent microwave pretreatment.

2. Experimental

2.1 Ultrasonic pretreatment

Ultrasonic pretreatment was carried out using Hielscher UP200S ultrasonic homogenizer (Germany) with operating frequency of 50-60 kHz, rated voltage of 200-240 V, and rated current of 2 A. 250 mL of sludge were placed in a glass beaker without temperature adjustment and an ultrasonic probe was submerged in the sludge to a depth of 2 cm. The effect of this pretreatment, which mainly depends on the treatment time, was evaluated by taking samples at different times (10 s, 20 s, 30 s, 40 s, 50 s, 1 min, 2 min, 3 min) to study the effect of sludge disintegration. Specific energy was considered as a main variable parameter for evaluation of disintegration performance of the sludge. It is determined by using ultrasonic power (P), ultrasonic time (t), sample volume (V) according to the following Eq. (1):

$$E=P(W)\times t(s)/V(L) \quad (1)$$

2.2 Microwave pretreatment

Pretreatment experiment was performed as batch process with 250 mL of sludge in a microwave oven (2450 MHz frequency). The experiments were carried out in Polytetrafluoroethylene (PTFE) vessels for effective microwave diffusion and a cover was employed to evade sludge losses caused by hot spot formation during the disintegration process.

2.3 Analytical methods

COD, SCOD, Volatile Fatty Acids (VFA) were estimated as per standard methods (APHA, 2005).

2.4 Statistical analysis

Statistical analysis was performed to determine differences between each parameter on the investigated characteristics. First-way ANOVA was performed in Excel 2016 at 95% confidence level; when a significant difference was detected post hoc pairwise multiple comparisons were calculated.

3. Results and discussion

3.1 COD solubilization and SCOD release

Ultra sonication pretreatment was done to improve the bioavailability of sludge particulate material by increasing the rate of Extracellular Polymeric Substances (EPS) release. As the ultra-sonic treatment increased from 10 s to 3 min, an increase in COD solubilization was observed. COD solubilization increased by 21.87% to the value of 2500 mg/L after 3 min (data not shown). COD solubilization increased rapidly with the increase in sonic treatment time up to 1 min, after that, the increment in COD solubilization was in a slower rate. The rapid disintegration obtained at the beginning of the study was attributed to the availability of biomass for the action of cavitational forces. In the period between 1-3 min, and even though

cavitational effect remains the same, it releases less amount of SCOD. This attributed to the depletion of easily disintegradable organics and the presence of more recalcitrant organics after the initial period of 1 min treatment (Gayathri et al., 2015).
The behaviour of COD solubilization and SCOD release after microwave pretreatment followed a simple role; when higher specific energy was performed, the increment of COD solubilization to the aqueous phase occurs in higher rates. This increase can be explained by the hydrolysis of the large organic molecules, the lysis of the cell walls and the disintegration of sludge floc, which was intensified by the applied microwave irradiation. Interestingly, ultrasonic pretreatment resulted in higher SCOD initial values, and higher trend of SCOD release when higher MW intensities and/or longer contact times were applied.
Soluble COD of the raw sludge was found to be about 2005 mg/L and it went up to 7040 mg/L prior to the boiling point that occurred at specific energy of 1250 KJ/L. However, for the utlrasound followed by MW; the initial value of SCOD started at 2360 mg/L and raised to 8000 mg/L prior to the boiling point preforming same MW energy; suggesting higher SCOD release caused by the ultra-sonication. The increase of SCOD concentration after microwave pretreatment of dairy sludge is attributed to the transfer of organic substances from non-soluble materials into soluble materials. Nonetheless, the intracellular organic materials may have been released into the medium as a result of cell wall disintegration, indicating that; the effect of microwave irradiation was significant in disintegrating sludge floc.

Table 1 SCOD, DOC and Volatile fatty acids concentrationsn for dairy sludge after MW irradiation and ultra-sonic pretreatment at 700 KJ/L input energy. Statistical differences are indicated by different superscript letters.

Parameter	Raw sludge	MW irradiation	Ultrasonic+MW
SCOD	2005 ± 100^{a}	7042 ± 340^{b}	8000 ± 160^{c}
DOC	2000 ± 200^{a}	7321 ± 520^{a}	8918 ± 211^{b}
Volatile Fatty Acids (VFA)			
Acetic acid (ppm)	370 ± 21^{a}	790 ± 43^{b}	810 ± 16^{b}
Propionic acid (ppm)	5 ± 0.1^{a}	23.0 ± 0.1^{b}	29.3 ± 0.1^{c}
Butyric acid (ppm)	61 ± 7.8^{a}	306 ± 6.8^{b}	308 ± 1.8^{b}
Butyric acid (ppm)	32 ± 4.2^{a}	50 ± 8.3^{b}	91 ± 5.1^{c}
Caproic acid (ppm)	14 ± 0.8^{a}	44 ± 5.4^{b}	33 ± 9^{b}

Conclusion

This study explored the upshot of ultra-sonication as a pretreatment an addition of microwave pretreatment for waste activated dairy sludge on the basis of COD solubilization. The effective floc disruption was achieved at a specific energy input 52.8 kJ/L. This novel pretreatment process reduces the energy consumption significantly with enhanced COD and DOC solubilization. However, a follow up biodegradability studies are needed to determine the potential methane production relatively to MW irradiation alone. Conclusively, this novel process may possibly be entitled as a plausible pretreatment process.

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