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Enhancing the anaerobic digestion potential of dairy waste activated sludge by two step sono-alkalization pretreatment

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ABSTRACT

High efficiency resource recovery from dairy waste activated sludge (WAS) has been a focus of attention. An investigation into the influence of two step sono-alkalization pretreatment (using different alkaline agents, pH and sonic reaction times) on sludge reduction potential in a semi-continuous anaerobic reactor was performed for the first time in literature. Firstly, effect of sludge pretreatment was evaluated by COD solubilization, suspended solids reduction and biogas production. At optimized condition (4172 kJ/kg TS of supplied energy for NaOH – pH 10), COD solubilization, suspended solids reduction and biogas production was 59%, 46% and 80% higher than control. In order to clearly describe the hydrolysis of waste activated sludge during sono-alkalization pretreatment by a two step process, concentrations of ribonucleic acid (RNA) and bound extracellular polymeric substance (EPS) were also measured. Secondly, semi-continuous process performance was studied in a lab-scale semi-continuous anaerobic reactor (5 L), with 4 L working volume. With three operated SRTs, the SRT of 15 d was found to be most appropriate for economic operation of the reactor. Combining pretreatment with anaerobic digestion led to 58% and 62% of suspended solids and volatile solids reduction, respectively, with an improvement of 83% in biogas production. Thus, two step sono-alkalization pretreatment laid the basis in enhancing the anaerobic digestion potential of dairy WAS.

1. Introduction

The dairy industry, like most other agro-industries, generates more amount of sludge characterized by high biological oxygen demand (BOD) and chemical oxygen demand (COD) due to their high organic content [1]. Increasing industrial effluent loads on existing plants and more stringent government discharge requirements have put considerable pressure on the dairy industry to refine and understand better the design and management of biological wastewater treatment processes [2]. Among several methods, anaerobic digestion is one of the most efficient commonly used stabilization processes in sludge management. In particular, intracellular biopolymers solubilization and conversion to the lower molecular weight compounds of solid degradable organics such as sludge through hydrolysis is a rate limiting step. Therefore, a pretreatment step would render hydrolysis less difficult, thus giving a more efficient process. With the advancements in various sludge pretreatment techniques like thermal, chemical, mechanical and physical and several combinations such as mechanical–chemical [3] and thermal–chemical [4,5], biodegradability of sludge can be enhanced by several orders; however, economical constraints of these technologies have limited their scale-up and lab-to-field implementation. Among these alternate technologies, combination of alkaline and sonication pretreatments are found to be the most effective sludge disintegration methods and this was expansively studied in the present study.

The mechanism of alkaline pretreatment, which is commonly the subject of investigations, is to induce swelling of particulate organics at high pH, making the cellular substances more susceptible to enzymatic action and also it has advantages of simple device, convenient operation and high efficiency [6]. Recently, this has been combined with other pretreatment methods such as thermal [7] and sonication [8,9] pretreatment methods to enhance the solubilization of sludge, before the sludge digestion. Sonication is also a novel approach with high application potential in sludge disintegration for its effectiveness and simple operation. However, since sonication is an energy-intensive process [10], its major disadvantage is its high energy consumption. To lessen its energy consumption, sonication can be applied together with alkaline agents [8]. The simultaneous combination of sono-alkaline pretreatment can take the advantages of both methods and achieve a higher efficiency [11]. Accordingly with the combined method, the reaction time needed can be effectively reduced and the disintegration

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efficiency can be improved distinctly. Although sonication and its combinations with different methods were studied in the literature, sono-alkalization pretreatment using different alkaline agents and various pH has not been investigated so far. Thereby, the main objective of the present work is to provide more insights into the sludge disintegration by this new combined method on both physical and chemical properties to reduce the energy consumption and to examine the influences of the pretreatment on subsequent anaerobic digestion.

2. Materials and methods

2.1. Sludge sampling and characterization

Dairy WAS was sampled from Aavin dairy effluent treatment plant at Tirunelveli, India. Samples were collected and stored at 4 °C. The characteristics of the raw sludge were as follows: total COD (TCOD) was 10,200 mg/L, soluble COD (SCOD) was 200 mg/L, pH was 7.1, total solids (TS) concentration was 13,850 mg/L, suspended solids (SS) concentration was 9900 mg/L, volatile solids (VS) concentration was 7250 mg/L and the dry content of the sludge was 1.3%. In order to minimize random errors, every experiment was triplicated and the results were averaged.

2.2. Sono-alkalization pretreatment studies

Sono-alkalization pretreatment was performed as batch tests for each experimental run with a volume of 500 mL sludge sample. For the WAS studied, various alkaline agents (NaOH, KOH, $Ca(OH)_2$) of various concentrations were added to reach different pH values of 8, 9, 10 and 11. The alkali consumption for boosting the pH form near neutral range to alkaline range (pH 8–11) was in the range of 0.005–0.01 g/g SS in the case of NaOH, 0.006–0.06 g/g SS in the case of KOH and 0.008–0.09 g/g SS in the case of $Ca(OH)_2$. Then, sludge disintegration was carried out using an ultrasonic homogenizer (Make: Bandelin, Germany; Model: HD2200). This apparatus was equipped with a VS 70 T probe with a fixed operating frequency of 20 kHz, which is the most effective ultrasonic frequency for sludge disintegration [12] and a supplied power of 200 W to get an effective sludge disruption. The immersion depth of ultrasonic probe into the sludge was 2 cm, which provided the highest disintegration yield [8]. It was well-known that at low frequencies, the implosions of stable cavitation bubbles create high energy waves and micro-jetting effects together with the inside-bubble pyrolysis phenomenon, which explains the slight reduction in the mass of the dry solids. This complex matrix medium provides release of intracellular materials into the bulk liquid. Besides, Sahinkaya et al. also reported 20–40 kHz as the optimal frequency range to achieve strong mechanical forces. Moreover, the effect of this pretreatment mainly depends on treatment time, and it was evaluated by taking samples at different times (5–30 min) to study the combined effect. During experiments, the sludge temperature was not controlled as it aid disintegration of the WAS [12].

2.3. Biochemical methane potential (BMP) assay

The potential biogas production and biodegradation of pretreated WAS relative to untreated sludge were investigated in triplicate batch mesophilic reactors of 300 mL capacity with BMP assay. Each serum bottle was filled with 100 mL of inoculum and 50 mL of substrate. After adding the substrates and inoculum, the reactors were closed with a rubber septum and an aluminium seal to make them air tight and was subsequently purged with nitrogen gas at the rate of 10 mL per second for 20 min into the reactors to maintain anaerobic conditions. An internal temperature of 35 °C was maintained by incubating the reactors in a temperature controlled mechanical shaker (220 rpm). The inoculums needed to be quite active, with a good adaptability and a low endogenous respiration. For these experiments, the rumen microorganisms of cattle dung were used as an inoculum. The rumen is an exclusive organ of ruminant animals. A major portion of digestion in ruminants is accomplished by the anaerobic microflora of the reticulo rumen [13]. The capability of cellulose degradation that these animals possess is related to the presence of anaerobic microorganisms in its rumen, which decompose the glucose polymer chains to acetate. Methanogenic microorganisms that convert acetate into methane and carbon dioxide also exist in rumen [14]. Moreover, in case of anaerobic biodegradability, the use of a highly active animal inoculum waste will reduce the experimental time significantly, or reduced the amount of inoculum required in full-scale batch digesters, and consequently, the corresponding digester volume [15]. The initial pH was recorded as neutral before the start of BMP assay tests. Batch reactors were operated with a residence time of 50 d. Enhancement of biodegradability was evaluated by comparison of biogas volumes produced by treated and untreated samples. Based on the outcomes of the preliminary investigations, the conditions of the batch anaerobic reactors were selected. The anaerobic biodegradability test's performance was evaluated by fitting the cumulative biogas production data to the modified Gompertz equation. The Gompertz equation describes cumulative biogas production assuming that, biogas production is a function of bacterial growth [16]. The modified Gompertz equation is presented below as Eq. (1):

$$B = P\{1 - \exp[-R_m(t - \lambda)/P]\} \quad (1)$$

wherein, B is the cumulative biogas production (mL), R_m is the maximum biogas production rate, P is the biogas yield potential (mL), and λ is the duration of lag phase, d. Using Matlab 7.0, the unknown parameters were calculated in order to measure the difference between the experimental measurement and the corresponding stimulated value.

2.4. Semi-continuous anaerobic sludge digestion

Two identical laboratory scale round bottom (Borosil make) semi continuous reactors for the batch process were used as anaerobic reactors at mesophilic temperature (35 °C). The total volume of each reactor is 5L; corresponding to 4L working volume (sludge) and 1L headspace volume (biogas). Among the two, one is designated as Control Semi Continuous Anaerobic Reactor (CSCAR) and other is designated as Experimental Semi Continuous Anaerobic Reactor (ESCAR), where anaerobic digestion of pretreated study was carried out. The sludge retention times (SRT) is the average time the activated-sludge solids are in the system. During the total period of 310 d, the performance evaluation of SCAR was carried out at 3 different SRTs such as 20, 15 and 12 d. A control digester fed with untreated sludge was operated at same SRTs. Feeding and withdrawals were carried out each day by peristaltic pumps in a semi continuous mode. Biogas production was measured by water moving in graduated test tubes linked to the reactors.

2.5. Analyses

Right before and after the experiments to determine the effect of pretreatment on sludge digestion, several measurements which include TS, SS, COD and pH were carried out on samples, as per Standard Methods for the Examination of Water and Wastewater [17]. The analyses were performed on both the sludge and the supernatant to identify the total and soluble fractions of the specific component. The EPS was extracted by thermal extraction method as described by Cho et al. [18]. RNA concentration was