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Effect of sonically induced deflocculation on the efficiency of ozone mediated partial sludge disintegration for improved production of biogas

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A B S T R A C T

In this study, ultrasonication was used for sludge deflocculation, followed by cell disintegration using ozone. The effect of this phase separated sono-ozone pretreatment is evaluated based on extra polymeric substances release, deoxyribonucleic acid (DNA) in the medium, solubilization of intra cellular components and suspended solids (SS) reduction. Ultrasonically induced deflocculation was optimized at an energy dosage of 76.4 (log 1.88) kJ/kg TS. During cell disintegration (ozone dosage 0.0011 mg O_3/mg SS), chemical oxygen demand solubilization (COD) and SS reduction of sonic mediated ozone pretreated sludge were 25.4% and 17.8% comparatively higher than ozone pretreated sludge, respectively. Further, biogas production potential of control (raw), flocculated (ozone pretreated), and deflocculated (sonic mediated ozone pretreated) sludges were observed to be 0.202, 0.535 and 0.637 L/(g VS), respectively. Thus, the phase separated pretreatment at lower ultrasonic specific energy and low dose ozone proved to enhance the anaerobic biodegradability efficiently.

1. Introduction

Dairy industries are producing wastewater that are rich in organic matter, resulting in the creation of highly odorous and COD containing water [1]. Hence, the disposal of dairy wastewater poses a major problem to the environment. Several approaches have been made to overcome this problem, among which the activated sludge process is the typically used biological treatment [2]. Further, excess sludge treatment and its disposal in the biological wastewater treatment processes account for up to 65% of the total operating costs of the wastewater treatment plant (WWTP) [3]. At present, social and environmental concerns have created the need for more rigid regulations concerning the treatment and disposal of sludge. These have led to a significant increase in the associated costs, which is expected to rise even more. Hence, it is essential to reduce sludge production at the source: in the wastewater treatment plant. This is quite possible with anaerobic digestion (AD). The biodegradation potential of AD is assessed through biochemical methane potential (BMP) assay [4]. AD has been used as an effective method of sludge stabilization, since it reduces nearly 40–50% quantity of sludge along with the production of biogas, thus making the process profitable [5]. However, its application is hindered by hydrolysis of waste activated sludge (WAS), which consists of microbial cells, cell wall and membranes that act as tough barricades and hinder the permeation of hydrolytic enzymes. Hence, various physical, chemical and mechanical pretreatments are implemented to improve the potential of AD [6]. This paper presents an extensive study on phase separated pretreatments, deflocculation using ultrasonication (US) and cell lysis using ozonation, in particular for effective sludge disintegration.

Ultrasonication is a known technique for disrupting sludge floc and lysing microbial cells, thus leading to the organic matter degradation, particle size reduction and inactivation of sludge microorganisms. US pretreatment results in the formation of cavitation bubbles in the aqueous phase [7]. As acoustic cavitation proceeds, high hydrodynamic shear forces that react on the matters are created; this is primarily responsible for the sludge disintegration [8]. Furthermore, it has been evidenced that the disruption of surplus sludge is more effective.

Ozone decomposes itself into radicals and reacts with the soluble and particulate matters, organic or mineral fractions present in the sludge [9]. It has been reported that excess sludge production can be reduced significantly by partial ozonation of the returned sludge in an activated sludge process [10].

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In this study, sonic mediated ozone pretreatment of WAS was executed to enhance the treatment efficiency. Since it requires high energy, the operating parameters had to be optimized in order to acquire cost-efficient results. The flocculated sludge was pretreated with ozone alone, whereas the deflocculated sludge was floc disrupted and then pretreated with ozone. The treatment was carried out for varied ultrasonic specific energy and ozone dosage. Sludge pretreatment could be divided into two phases: deflocculation and cell breakage along with macromolecular compounds release. Here, the first phase of pretreatment was carried out using ultrasonication.

Although combination of ultrasonication and ozonation with different methods has been reported in previous literatures, phase separation using sonication for deflocculation and cell lysis through ozonation has not been investigated so far. Thus, the crucial objectives of the present study is (1) to disrupt the flocs with reduced ultrasonic energy consumption, (2) to examine the effect of deflocculated sludge (Extracellular Polymeric Substance (EPS) detached) on low dose ozone pretreatment and (3) to investigate the pretreatment efficiency on subsequent anaerobic biodegradability.

2. Materials and methods

2.1. Sludge sampling and characterization

Experiments were carried out using dairy WAS collected from Aavin dairy WWTP at Madurai, India. The characteristics of the raw sludge were as follows: pH = 6.9, Total Solids (TS) = 12,560 mg/L, Suspended Solids (SS) = 7000 mg/L, Volatile Solids (VS) = 5600 mg/L, Total Chemical Oxygen Demand (TCOD) = 10,000 mg/L, Soluble Chemical Oxygen Demand (SCOD) = 400 mg/L.

2.2. Deflocculation using sonication

Deflocculation using sonication was carried out with 500 mL sludge without temperature adjustments. Experiments were performed under the conditions of pH 6.8 and SS – 7100 mg/L at room temperature. Sonication was carried out using an ultrasonic homogenizer (Make: Bandelin, Germany; Model: HD 2200) .The equipment was furnished with VS 70 T probe with a fixed operating frequency of 20 kHz, which is considered to be the most effective US frequency for sludge pretreatment [11] and a power supply of 200 W to get active floc disruption. Highest pretreatment yield was observed when the probe was immersed to a depth of 2 cm into the sludge [12]. Experiments were carried out by varying the range of specific energy (SE) from 0 kJ/kg TS to 1200 kJ/kg TS.

2.3. Cell lysis using ozonation

The experimental set up of ozone disintegration of sonically treated sludge is given in Fig 1. Ozone generator produced ozone gas from pure oxygen (Make: Faraday; Model: L10G). The sonically pretreated sludge was then ozonized in batch in an ozone contactor of 500 mL capacity. The working volume of contactor was 250 mL. Ozone was uninterruptedly bubbled into the reactor, which was stirred manually to reduce the foam formation. A micro porous diffuser was also used to distribute the ozone into the sludge sample. The flow rate was maintained at 0.075 L/min, using a flow rate adjuster which had been set just before the inlet into the contactor. The excess ozone after treating the sludge was removed through an outlet at the top and let into the excess ozone absorption trap. Here two traps of potassium iodide (KI) were used to absorb the excess ozone. Each trap was filled with 200 mL of 2% KI solution, to estimate the ozone demand according to Standard Method 2350 E – Ozone Demand [13]. From the KI trap, the ozone gas was let into the atmosphere. Triplicate experiments were carried out by varying the treatment time from 1 min to 15 min. Ozone dose was calculated to be 0.85 mg/min and hence the range of ozone was maintained from 0.0001 mg O_3/mg SS to 0.0018 mg O_3/mg SS.

2.4. Biochemical methane potential (BMP) assay

BMP assay can be used as an indicator for assessing the anaerobic biodegradability of pretreated sludge [14]. The BMP tests were carried out in serum bottles of capacity 300 mL and sealed hermetically. Each reactor was maintained with 3:1 inoculum substrate ratio. The inoculum used for these investigations was the bovine rumen fluid. As bovine rumen fluid seeded in a reactor as inoculums produces noteworthy results on increasing the biogas production rate [15,16]. After the addition of the inoculum and substrates, the reactors were sealed and purged with nitrogen gas. The reactors were then incubated at a temperature of 35 °C in an orbital shaker (Digital IKA KS 130) (220 rpm). The gas was recorded by injecting a needle into the stopper. The biogas volumes produced by treated and untreated samples were compared, from which the enhancement of biodegradability was later evaluated. The anaerobic biodegradability test's performance was determined by fitting the specific biogas production data to the modified Gompertz equation, assuming biogas production as a function of microbial growth [17]. The modified Gompertz equation is given below as Eq. (1):

$$B_t = B^* \exp[-\exp[R_b/B^* \exp(\lambda - t) + 1]] \quad (1)$$

where B_t is the increasing biogas produced (mL) at time (t), B is the biogas production potential (L/(g VS)), R_b is the utmost biogas production rate (L/(g VS d)), and λ is the lag phase (days), which is the least time taken to produce biogas. These parameters were estimated by Non-linear regression through Polymath software (version 6 Shareware Company). A schematic representation and flow diagram of the present study is depicted in Fig 2.

2.5. Analytical parameters

The substantial effect of pretreatment on sludge degradation was evaluated by analyzing the following parameters before and after the treatment: suspended solids (SS), soluble chemical oxygen demand (SCOD), and turbidity as per Standard Methods [18]. Protein and carbohydrate concentrations were determined using the Lowry and Anthrone method, respectively. The Deoxyribonucleic acid was measured by Diphenylamine colorimetric method with *Escherichia coli* DNA as the respective standard [19,20]. Loosely bound EPS (LB-EPS) and tightly bound EPS (TB-EPS) were quantified by a modified heat extraction method [21]. Triplicate experiments were carried out.

2.6. Calculations

The degree of COD solubilization was calculated using the Eq. (2),

$$\text{COD solubilization } (\%) = \left[\frac{\text{SCOD}_p - \text{SCOD}_i}{\text{TCOD}_i - \text{SCOD}_i}\right] * 100 \quad (2)$$

where $SCOD_p$ was SCOD concentration of the sludge after pretreatment (mg/L), $SCOD_i$ was SCOD concentration of the sludge before pretreatment (mg/L), $TCOD_i$ was TCOD concentration of the sludge before pretreatment (mg/L). The economy of a sonication is primarily governed by the energy input needed to attain effective sludge