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Biochemical methane potential test for pulp and paper mill sludge with different food / microorganisms ratios and its kinetics

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ABSTRACT

The application of anaerobic digestion is mounting worldwide because of its economic and environmental benefits. As a concern, the rate and extent of the anaerobic digestion of pulp and paper mill sludge was tested using the biochemical methane potential (BMP) with different food/microorganisms (F/M) ratio. The study was evaluated at five different F/M ratios (i.e. 0.5, 1.0, 1.5, 2.0, 2.5). In addition to that reaction kinetics and the influence of biodegradability were also evaluated for the biomethane potential of substrates. The highest cumulative methane yield was obtained in F/M ratio 2.0 with 3.4 L followed by F/M ratio 1.5 and 2.5 with 3.3 L and 2.9 L, respectively. The maximum methane production rate 3.66 L CH_4 was perceived as per kinetics. The biomass activity may be affected due to the high substrate concentration during BMP test of lower F/M ratios. On the other hand, F/M ratio greater than 2.0, methane yield was decreased due to increased volatile fatty acid production reduces the reactor pH and inhibits methanogens activity.

1. Introduction

Huge amount of sludge was produced by the pulp and paper industry owing to huge volumes of wastewater production. Hence its sludge contains 35–50% organic matter could be converted into renewable energy in the form of methane. Although anaerobic digestion (AD) has gained increasing attention in late 1980s in the industrial level. But AD system has been widely used in wastewater treatment of various industries and in the municipal sector; AD of mill derived sludge is still in its infancy (Meyer and Edwards, 2014). Bayr and Rintala (2012) reported from the pulp and paper industry that the aerobic activated sludge process are still commonly used for the wastewater treatment that was followed by the primary clarifier, either sedimentation or flotation from which primary sludge was produced.

Based on the raw material utilization, pulp and paper manufacturing process steps, and the subsequent wastewater treatment produce varying characteristics of both primary and secondary sludge from this industry. Thus produced primary sludge consists of wood fibres i.e. cellulose, hemicellulose and lignin and papermaking fillers i.e. kaolin and calcium carbonate, pitch, lignin by-products and ash. While the secondary sludge consists of microbial biomass, cell-decay products and non-biodegradable lignin precipitates (Puhakka et al., 1992). Unlike primary sludge, biosludge was difficult to dewater because of the ubiquitous of microbial biomass. Thus the sludge usually comprises chlorinated organics, pathogens and trace amount of heavy metals (Monte et al., 2009). To date, in pulp and paper mills both primary and secondary sludge were mixed and thickened/dewatering. Then it was generally incinerated, which is often not energetically favourable or landfilled, which is becoming restricted due to emissions and loss of resource (energy) (Bayr and Rintala, 2012).

Kim et al. (2000) depicted that the kraft pulp mill primary sludge was testified to contain 58, 12 and 20 weight percentage of cellulose, hemicellulose and Klason lignin respectively. Thus AD has an advantage such as reduction of the sludge volume by 30–70% (Elliott and Mahmood, 2007), sludge stabilization, sludge disinfection, improved dewatering properties of treated sludge and the production of biogas and its low cost. The energy trapped from the organic matter can be transferred for the production of steam for boiler heating, vehicle fuel or electricity (Espinosa-Solares et al., 2009), which will reduce the dependence of energy in fossil fuels

Abbreviation: AD, anaerobic digestion; BMP, biochemical methane potential; CH_4, methane; SMA, specific methanogenic activity; ETP, effluent treatment plant; F/M, food/microorganisms; MC, Moisture content; NaOH, sodium Hydroxide; PPMS, pulp and paper mill sludge; sCOD, soluble chemical oxygen demand; TKN, total Kjeldahl nitrogen; TS, total solids; VS, volatile solids; VFA, volatile fatty acid.

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and reduce the greenhouse gas emission. Also digestate was the nutrients can be used as fertilizer for arable land or forests. Karlsson et al. (2011) reported that effluent from the anaerobic treatment processes also release nutrients rich slurry product. This nutrient-rich effluent can be re-circulated to both AD and aerobic activated sludge biological process, thereby reducing the need for commercial nitrogen and phosphorus fertilizers to support activated sludge function.

The determination of specific methanogenic activity (SMA) in relation to the cumulative methane production rate can be used to predict the activity of biomass in a reactor and also to assess the capacity of potential loading rate for the anaerobic reactor (Ince et al., 1995). By using the kinetics of known quantity of substrate removed or the methane produced, the activity of the microbial cluster can be measured in an anaerobic digestion process. SMA test for the acetate quantify only the activity of acetoclastic methanogenic but not the hydrogenoclastic methanogens while the propionate spikes quantify both the acetogenic and methanogenic activity. But in general response, cellulose spike represent the combined activity of acidifying, acetogenic and methanogenic bacteria. Generally SMA tests was done by adding the known amount of specific substrate concentration in a confined batch reactor with the addition of nutrients and pH buffer (prevent from inhibition or deficiency) and then incubated in a controlled temperature. Methane production are measured daily and quantified as mL CH_4 g $VS^{-1}d^{-1}$ according to the equation of Buswell's as labeled by Angelidaki and Sanders (2004).

In this decade, AD of organic solid waste has gained increased attention due to the pollution reduction from the environment at the same time producing energy-rich biogas (renewable source), destroying pathogenic organisms and reducing problems associated with the disposal of organic waste. The objective of this study was to evaluate the feasibility of pulp and paper mill sludge as substrates for biogas production through BMP test and also to investigate the rate and extent of the anaerobic digestion of different F/M ratio and its reaction kinetics.

2. Materials and methods

2.1. Substrate and inoculum

The PPMS was collected from a Nagaon paper mill situated at Jagiroad, Assam, India. Nagaon paper mill uses Kraft pulping process for the paper production. Sludge samples were collected from the filter house (belt press) at the primary treatment of effluent treatment plant (ETP) in paper industry. The pulp and paper mill ETP has preliminary and primary treatment tailed by conventional aerobic lagoon unit. After sampling at ETP, it was transported to the laboratory and kept at 4 °C prior to use. Seed sludge (Cow dung) was obtained from a nearby farm in Amingaon, Indian Institute of Technology Guwahati, North Guwahati, India and used as inoculum for the BMP study.

2.2. Anaerobic BMP setup

The rate and extent of AD of PPMS was tested using the BMP assay as described in Owen et al. (1979). The BMP assay is a batch digestion where a substrate being tested for anaerobic degradability is incubated in a sealed bottle with a sample of anaerobic microorganisms in a defined nutrient medium. The volume of gas produced during the incubation is measured and is an indication of the rate of substrate digestion.

By using 1000 mL reagent glass bottles and rubber cork for closing, batch reactor was prepared. For different F/M ratio, 18 reactor were used. Among that 15 reactor were fed with varied quantity of PPMS, considering essential macro and micro nutrients in toting of 100 g of inoculum. Remaining 3 reactor were fed with 100 g of inoculum, and its nutrients used as a control. After that all the reactors were made up to equal volume using deionised water. Finally nitrogen gas was purged to maintain the anaerobic condition in the reactor. Then by means of water displacement method, using aspirator bottle with 1.5N NaOH solution used for measurement of methane produced daily. The reactors were maintained at room temperature approximately 30–38 °C. Essential environmental parameter such as pH, stirring intensity that affects BMP test were maintained (Browne and Murphy, 2013). The experiment was conducted for 45 days with triplicate.

2.3. Parameter analysis

Different parameter such as moisture content (MC), total solids (TS), volatile solids (VS), sulfate (SO_4^{2-}) and soluble chemical oxygen demand (sCOD) were analysed using standard protocols according to APHA. (2005). To measure pH, 10 g of PPMS was taken in a conical flask with 100 mL of deionised water and mixed for 2 h at 150 rpm in a horizontal shaker. Volatile fatty acid (VFA) was analysed using DiLallo and Albertson (1961) pH titration method. At 105 °C oven dried and 0.2 mm sieved powered PPMS was used for the analysis of total Kjeldahl nitrogen (TKN) (Behera, 2006).

2.4. Methanogenic activity test

Methanogenic activity test was performed to decide inoculum activity that was based on VS content. For that 2 g VS content of inoculum material was taken in serum bottles with mineral media i.e. neutralized acetic acid (Jawed and Tare, 1999) was added as the substrate. The organic loading was 1.0 g COD g^{-1} VS_{added}. By using de-aerated water, the bottles were makeup to 500 mL then it was converted to anaerobic condition with the help of nitrogen purging. Then the bottles were connected to aspirator bottles having 1.5N NaOH, to absorb CO_2 and displace NaOH solution to quantify the amount of methane produced from the inoculum in gas collection system (Esposito et al., 2012). SMA is attained by dividing the maximal rate of methane production (M) to the quantity of volatile solids reduction in the anaerobically digested pulp and paper mill sludge in each treatment.

2.5. Kinetic study

The accumulative methane production attained from the BMP as a batch assay was fitted to the modified Gompertz equation as reported by Lay et al. (1996) which defines the accumulative methane production from the different F/M ratio in batch assay was assumed that methane production is a function of bacterial growth. i.e

$$Y = M \cdot \exp\left\{ -\exp\left[\frac{R_m \cdot e}{M}(\lambda - t) + 1\right]\right\}$$

Where, Y represents the cumulative methane production volume (mL) with respect to time t (d), M is the potential CH_4 production (mL CH_4), R_m is the maximum rate of methane production (mL CH_4 d^{-1}), λ is the lag phase time (d) and e is constant equivalent to 2.71. The bio-kinetics variables such as M, Rm and λ can be calculated using MATLAB R2015a by adjusting the pair of experimental data (Y,t) in a non-linear regression method.