



# Synergetic effect of combined pretreatment for energy efficient biogas generation



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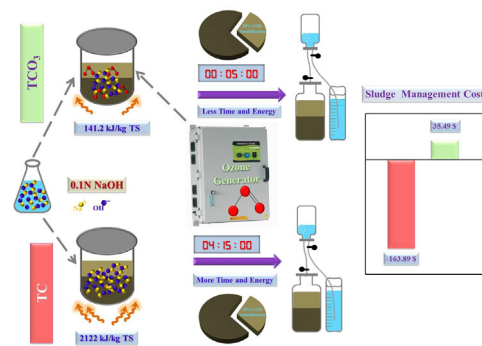
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## HIGHLIGHTS

- Combined  $\text{TCO}_3$  pretreatment improves disintegration of WAB effectually.
- This novel method aid solubilization at lesser specific energy of 141.02 kJ/kg TS.
- COD solubilization of about 30.4% was achieved in 0.0012 mg  $\text{O}_3$ /mg VS ozone dosage.
- Highest methane yield of 0.32 g COD/g COD was observed in  $\text{TCO}_3$  pretreated biosolids.
- Combinative pretreatment offers a net profit of about 35.49 \$/ton of WAB.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Physiochemical disintegration of waste activated biosolids (WAB) through thermochemical (TC) pretreatment requires high energy and cost for efficient energy generation. Therefore in the present study, an attempt has been made to enhance the biodegradability and to minimize the operational cost of TC pretreatment by combining it with ozonation. A higher solubilization of about 30.4% was achieved at lesser energy input of about 141.02 kJ/kg TS and a ozone dosage of about 0.0012 mg  $\text{O}_3$ /mg SS through this combined thermo chemo ozone ( $\text{TCO}_3$ ) pretreatment. The methane production potential (0.32 g COD/g COD) of  $\text{TCO}_3$  pretreatment was comparatively higher than the (0.19 g COD/g COD) TC pretreatment. The energetic analysis and economic assessment of the proposed method of pretreatment can possibly reduces the energy requirement of TC pretreatment with a positive net profit of about 35.49 \$/ton of biosolids.

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## 1. Introduction

Excess amount of waste activated biosolids (WAB) is the unavoidable derivative of biological wastewater treatment (Kavitha et al., 2013). Biosolids are enriched in organic matter

which creates numerous environmental issues without proper treatment (Ak et al., 2013). Anaerobic digestion process is most effective in stabilization of biosolids and reduces the economic constraint of treatment facility through energy recovery in the form of methane (Kavitha et al., 2014a; Gayathri et al., 2015). On the other hand, hydrolysis of WAB is the rate-limiting step in AD process due to prolonged retention time of about 25–30 days (Kavitha et al., 2015, 2016a,b,c). To increase the working efficiency of anaerobic digestion and hydrolysis, pretreatment of biosolids is

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essential. Consequently, numerous sole and combinative pretreatment techniques has been adopted to solubilise or destruct the cell wall of microorganism (sludge lysis) such as thermal, ultrasonic, high pressure homogeniser, chemical, microwave, ozonation and mechanical, thermochemical pretreatments (He and Zhang, 2011; Saha et al., 2011; Ariunbaatar et al., 2014; Pei et al., 2016). Several researchers have proposed the advantageous outcome of the combined pretreatment methods on subsequent anaerobic digestibility. Among those methods, thermochemical pretreatment of biosolids have wide application and known for its efficient disintegration of sludge (Uma et al., 2012). Although it has many advantages, it has some limitations; For instance, the efficient sludge disintegration index for biodegradation process is 30% solubilization (Parvathy et al., 2016). Achieving similar solubilization through thermochemical pretreatment demands high energy input, cost and extended treatment time. This comprehensively limits the overall profitability of the process and resulted in loss of organic matter which in turn limits the methane production in degradability process. In addition, pretreatment of sludge through ozone was one of the potent technologies with the highest disintegration capability (Salsabil et al., 2010). However from an economical point of view, employing ozone for biosolids pretreatment is expensive and this is the major limitation for it to be used in pilot scale plants. Employing higher dosage of ozone also resulted in loss of organics. Therefore to address these issues, in the present study, it was planned to combine the thermochemical pretreatment with ozonation in order to minimize the energy requirement, organic loss and cost of the disintegration process with limited loss of organics. This combinative pretreatment is considered to be novel due to the above mentioned advantages and it has not been documented in literature so far. Therefore, the main objectives of the present study was i) to optimize the pretreatment conditions for effective Thermochemical (TC) disintegration ii) to optimize the operational conditions of combined Thermo chemo ozone (TCO<sub>3</sub>) pretreatment for cost effective performance iii) to investigate the synergistic effect of this combinative pretreatment on effective solubilization in a cost effective manner iv) to evaluate the effectiveness of this combined pretreatment on biodegradability process and methane production v) to assess the economic viability of TCO<sub>3</sub> pretreatment based on energetic analysis and economic assessment.

## 2. Methods and materials

### 2.1. Waste activated biosolids and inoculum

Municipal Waste Activated Biosolids (WAB) sample used in the present study was collected from a secondary sedimentation tank of the local MWWTP at Chennai, India. The physiochemical characteristics of WAB were analyzed and indicated in the (Table 1). Anaerobically Digested Sludge collected from the existing biogas plant of waste water treatment facility was used as inoculum in the anaerobic batch test. The collected inoculum was in semi solids state and it was filtered using 0.1 mm filter mesh or dry cotton cloth to separate the solid and liquid portion. Therefore the separated liquid part is used as inoculum for methane production. The initial characteristics of inoculum was analyzed and the results were indicated in the (Table 1).

### 2.2. Thermochemical (TC) pretreatment

TC pretreatment was conducted with 5 L of WAB. Six different temperatures such as 50, 60, 70, 80, 90 and 100 °C were maintained to pretreat the biosolids. 0.1 N NaOH was used to adjust the pH value of biosolids to 9, 10, 11 and 12 for each pretreatment

**Table 1**  
Initial characteristics of WAB and inoculum.

S. No.	Parameters	Values
<i>Initial characteristics of WAB</i>		
1	pH	7.08
2	Soluble Chemical Oxygen Demand (SCOD)	200 ± 25 mg/L
3	Total Chemical Oxygen Demand (TCOD)	12,500 ± 250 mg/L
4	Suspended Solids (SS)	10000 ± 200 mg/L
5	Volatile Suspended Solids (VSS)	8250 ± 250 mg/L
6	Total Dissolved Solids (TDS)	7750 ± 150 mg/L
7	Total solids (TS)	17850 ± 350 mg/L
8	Soluble protein	120 ± 30 mg/L
9	Soluble carbohydrate	60 ± 15 mg/L
<i>Initial characteristics of inoculum</i>		
1	pH	7.11
2	Soluble Chemical Oxygen Demand (SCOD)	380 ± 125 mg/L
3	Total Chemical Oxygen Demand (TCOD)	14650 ± 350 mg/L
4	Suspended Solids (SS)	11850 ± 450 mg/L
5	Volatile Suspended Solids (VSS)	9550 ± 250 mg/L
6	Total Dissolved Solids (TDS)	7450 ± 225 mg/L
7	Total Solids (TS)	19750 ± 350 mg/L
8	Soluble protein	120 ± 30 mg/L
9	Soluble carbohydrate	60 ± 15 mg/L

temperature. The samples were collected at equal time intervals from 0 to 180 min. COD Solubilization and SS reduction were analyzed to evaluate the effect of pretreatment and to categorize the optimized condition (temperature, pH and pretreatment time).

### 2.3. TCO<sub>3</sub> pretreatment

Combined TCO<sub>3</sub> pretreatment of WAB was performed in cylindrical reactor of 5.5 L of capacity with working volume of 5 L, diameter of 12.5 cm and height of 45 cm (Fig. 1). Reactor is designed with double jacket provision with inlet and outlet port to circulate the hot water. It maintains the required temperature inside the reactor to heat the biosolids. Likewise the inlet and outlet port is designed in the inner reactor to pump the biosolids in and out before and after pretreatment respectively. A commercial available ozone generator (Faraday L10G) is used to generate ozone gas from pure oxygen and the flow of gas was regulated using gas flow rotometer (1LPM). During ozone treatment the O<sub>3</sub> gas was constantly bubbled in the reactor leads to foam formation. To avoid this problem, a magnetic stirrer was used for mixing the substance in a homogeneous state (Zhang et al., 2009). KI traps were provided to collect the excess ozone gas after pretreatment of biosolids. pH probe to record pH drop. TCO<sub>3</sub> pretreatment was done with the optimized pH, Temperature and pretreatment time by varying the ozone dosage from 0.0004 mg O<sub>3</sub>/mg SS to 0.0016 mg O<sub>3</sub>/mg SS based on different flow rates 0.05–0.2 L/min, respectively.

### 2.4. Anaerobic biodegradability assay

Anaerobic biodegradability assay was performed to evaluate the efficiency of TCO<sub>3</sub> pretreatment on methane production. Three reactors (R<sub>1</sub>, R<sub>2</sub> and R<sub>3</sub>) containing control (raw sample), Thermochemically pretreated and combined thermo chemo ozone pretreated samples were involved in this assay. Three reactors were filled with 250 ml of substrate and 750 ml of inoculum. This shows the food to microorganism ratio as 1:3. To study the effect of methane production first order kinetics was considered based on Batstone et al. (2009) using the following Eq. (1):

$$B(t) = B(X_d) * (1 - e^{-k*t}) \quad (1)$$

where B(t) is the cumulative methane yield at digestion time, t days (g COD/g COD added), B(X<sub>d</sub>) is the methane potential of the substrate (fraction of the degradable substrate that can be converted to methane) (g COD/g COD added), and k is the first order disinte-

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