



# Effect of deflocculation on the efficiency of low-energy microwave pretreatment and anaerobic biodegradation of waste activated sludge



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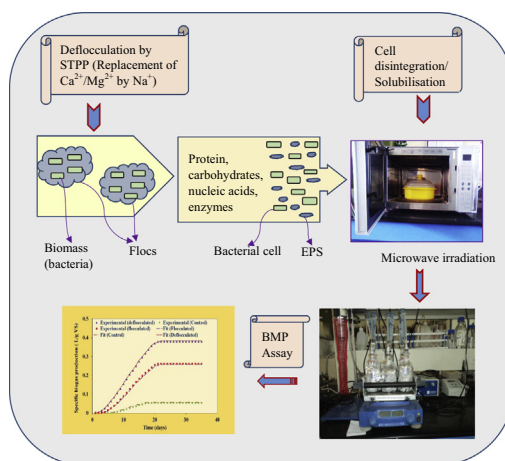
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## HIGHLIGHTS

- Sludge deflocculation was done using sodium tripolyphosphate.
- 0.175 g/g SS dose of STPP disturbed flocs efficiently.
- Floc disruption facilitated increase in sludge disintegration efficiency of MW.
- MW specific energy reduced from 26,000 to 14,000 kJ/kg TS for deflocculated sludge.
- 50.7% of cost reduction in pretreatment can be achieved through deflocculation.

## GRAPHICAL ABSTRACT



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## ABSTRACT

This study focuses on improving the efficiency of the microwave (MW) pretreatment of waste activated sludge (WAS) through deflocculation mediated by sodium tripolyphosphate (STPP), a cationic binding agent. Deflocculated sludge was subjected to MW pretreatment to assess its impact on biomass disintegration. At the optimised energy for MW pretreatment (14,000 kJ/kg TS), the chemical oxygen demand (COD) solubilisation was 28% and 21% and the reduction in suspended solids (SS) was 38% and 26%, respectively, for deflocculated (treated with a cationic binding agent followed by microwaves) and flocculated (treated by microwaves alone) sludge samples. The formation of volatile fatty acids in the deflocculated sludge medium (840 mg/L) was comparatively higher than that in the flocculated sludge (420 mg/L) and the control (62 mg/L). This study indicates that deflocculated sludge is more amenable to hydrolysis. The results of a test of biochemical methane potential also confirmed the greater amenability of deflocculated sludge for anaerobic degradation.

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## 1. Introduction

Rapidly increasing energy demands and their economic and environmental consequences urgently require a solution. Generating energy from waste is a new potential solution to this problem.

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Among waste materials, activated sludge, a bioproduct of aerobic wastewater treatment, can be a better option for generating energy because it has a high organic matter content [1]. However, this organic matter is hardly biodegradable, which limits hydrolysis in anaerobic digestion [2]. Through various pretreatment methods, such as biological [3], mechanical [4], thermal [5] or some combination [6,7], the degree of anaerobic biodegradability of sludge can be enhanced by releasing its intracellular components into a medium through cell disintegration.

Recently, studies have focused on using microwaves (MWs) for the pretreatment of sludge mainly because the desired temperature can be reached more rapidly, and the potential for hazardous emissions is minimised [8]. As a result, MW irradiation increases the rate/extent of anaerobic and aerobic digestion, improves dewaterability, reduces sludge and inactivates faecal coliforms. The application of a microwave pretreatment as a means of improving waste activated sludge (WAS) solubilisation and therefore biogas production [8–10] and pathogen destruction [5] has previously been investigated.

The goal of the present study was to reduce the amount of microwave energy required for sludge pretreatment and energy production. Currently, a variety of combinative pretreatment methods to reduce the microwave energy needed for biogas production have been tested by several investigators. The disadvantages associated with these methods are both high energy consumption and cost as the goal is cell disintegration. It is known that merely increasing cell disintegration does not increase the subsequent energy recovery from the waste activated sludge [11], so if any method focusing on cell disintegration were to be combined with MW pretreatment, it would definitely increase the energy and cost required for pretreatment and not the amount of energy recovered. Therefore, in the present study, an attempt has been made to decrease the microwave energy required for sludge pretreatment through cost effective sludge deflocculation with a cation binding agent. To remove the polymers from the floc structure of the sludge, chemical extraction techniques, such as acids and bases, chelating agents (e.g., EDTA) or cation exchange resins (CER) were used. Cation-binding agents such as sodium tripolyphosphate (STPP), deflocculate the sludge structure by removing bridging ions like  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  [12] and thus releasing the EPS from the flocs. The objectives of the present study were envisaged as follows: (1) deflocculate or remove the EPS from the biosludge using a suitable cation binding agent, STPP, (2) pretreat the deflocculated sludge at different microwave energy levels and (3) investigate the effect of deflocculation on anaerobic fermentation and biodegradability.

## 2. Materials and methods

### 2.1. Sludge samples

Sludge samples were collected from an activated sludge treatment plant located in Kerala, India. Prior to use, the samples were stored at 4 °C. Table 1 depicts the characteristics of the raw sludge.

### 2.2. Experimental conditions for pretreatment

#### 2.2.1. Deflocculation using a cation binding agent

Deflocculation experiments were carried out in 500-mL conical flasks at working volumes of 200 mL. Known doses of STPP were added to the sludge samples, and the solutions were kept in an orbital shaker at 150 rpm. The supernatant was filtered through a 0.45- $\mu\text{m}$  cellulose acetate membrane to retrieve the soluble organics, which were subjected to further analysis to determine total EPS, turbidity, DNA, protease activity and amylase activity.

**Table 1**  
Characteristics of waste activated sludge.

Sl. no	Parameter	Value
1	pH	6.54
2	Soluble COD (mg/L)	125 $\pm$ 5.95
3	Total COD (mg/L)	12,880 $\pm$ 620
4	Total Solids (mg/L)	14,200 $\pm$ 690
5	Volatile Solids (mg/L)	8700 $\pm$ 410
6	Suspended Solids (mg/L)	10,000 $\pm$ 500
7	Soluble proteins (mg/L)	12.9 $\pm$ 0.65
8	Soluble carbohydrates (mg/L)	4.6 $\pm$ 0.225

### 2.2.2. Optimisation of microwave energy

Waste activated sludge disintegration was carried out in a commercial microwave oven (Make IFB, Model-30SC2, 2450 MHz frequency and power – 900 W). A series of experiments were performed on 300-mL volumes of sludge at varying power levels, and the time of contact varied from 30 to 300 s at intervals of 30 s. The temperature ranged from 40 to 96 °C, and the specific energy was computed according to the method detailed by Ahn et al. [13].

### 2.3. Measurements and analysis

The protein, carbohydrate and DNA contents of the sludge samples were estimated by following the methods in [14]. Total solids (TS), volatile solids (VS) and turbidity were measured following the Standard Methods for the Examination of Water and Wastewater [15]. All analyses were duplicated, and the results are given as the mean values. The enzyme activity tests, such as for protease and amylase, were performed according to [16].

### 2.4. Anaerobic fermentation of WAS

Anaerobic fermentation experiments were carried out in 300-mL serum bottles at working volumes of 250 mL, which spanned 3 days. The substrate and inoculum were placed in the bottles at a ratio of 9:1. To nullify the activity of methanogens, the bottles were treated at 102 °C for 30 min, and the contents of the heat-treated bottles were allowed to cool to room temperature before 50 mM of BESA (2-bromoethane sulfonic acid) were added. The bottles were flushed with nitrogen to remove the oxygen in the head space, and the bottles were sealed air tight and placed in a shaker at 120 rpm for 72 h at 35 °C.

### 2.5. Biochemical methane potential test

The biochemical methane potential (BMP) test was used to estimate the methane yield potential of the target substances under mesophilic conditions. The substrates were classified as control (untreated sludge), flocculated and deflocculated sludge samples treated at the optimum energy level. The inoculum was composed of rumen microorganisms, which have a high degradation efficiency and conversion rate [9]. 180 mL of the sample were seeded with 60 mL of the inoculum and fed into a 300 mL serum bottle, and each bottle was filled with inoculum and a substrate at a ratio equal to 3:1 (S:I) according to [17]. The reactors were subsequently purged with nitrogen gas to ensure strict anaerobic conditions, and the reactors were closed with a rubber septum and an aluminium seal to ensure that they were air tight. They were then placed on a shaker at 150–200 rpm, and the biogas was measured by inserting a needle into the septum. The modified Gompertz equation was used to fit the cumulative biogas generation curve and study the kinetics of biogas production. The relationship between anaerobic biodegradability (BD) and BMP is expressed by Eq. (1) [18,19].

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