



# Influence of deflocculation on microwave disintegration and anaerobic biodegradability of waste activated sludge



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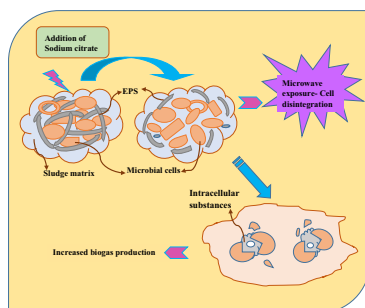
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## HIGHLIGHTS

- Sludge deflocculation by 0.1 g/g SS sodium citrate, enhanced floc disruption.
- Floc disruption facilitated an increase in disintegration efficiency of microwave.
- The irradiation energy of microwave reduced by 1.4 times for deflocculated sludge.
- Cost reduction in pretreatment can be achieved through deflocculation.
- Kinetic parameters demonstrate an efficient sludge degradation rate during hydrolysis.

## GRAPHICAL ABSTRACT



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## ABSTRACT

In the present study, the potential benefits of deflocculation on microwave pretreatment of waste activated sludge were investigated. Deflocculation in the absence of cell lysis was achieved through the removal of extra polymeric substances (EPS) by sodium citrate (0.1 g sodium citrate/g suspended solids), and DNA was used as a marker for monitoring cell lysis. Subsequent microwave pretreatment yielded a chemical oxygen demand (COD) solubilisation of 31% and 21%, suspended solids (SS) reduction of 37% and 22%, for deflocculated and flocculated sludge, respectively, with energy input of 14,000 kJ/kg TS. When microwave pretreated sludge was subjected to anaerobic fermentation, greater accumulation of volatile fatty acid (860 mg/L) was noticed in deflocculated sludge, indicating better hydrolysis. Among the samples subjected to BMP (Biochemical methane potential test), deflocculated microwave pretreated sludge showed better amenability towards anaerobic digestion with high methane production potential of 0.615 L (g VS)<sup>−1</sup>.

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## 1. Introduction

Waste activated sludge treatment and disposal currently complies with an emerging challenge for biological treatment system due to strict regulatory factors. Thus, sludge disposal becomes

complicated, leading to a 50% of additional cost to the total cost (Appels et al., 2013). Taking the waste stabilisation and energy recovery into consideration, attention has gradually been focused on anaerobic digestion (AD), which is considered as one of the sustainable options for the management of sewage sludge (Appels et al., 2008; Duan et al., 2012). However, inadequate hydrolysis of particulate organics present in the sludge limits the efficiency of anaerobic process. Enhancement of biodegradability through anaerobic digestion could be attained by disrupting the chemical

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bonds in the material prone to hydrolysis (Ahn et al., 2009), which is achieved through pretreatment of sludge by various physico-chemical methods. Several pretreatment processes such as ball mill (Rai et al., 2008), ultrasonic (Gayathri et al., 2015), microwave (Kuglarz et al., 2013), sodium hydroxide (Banu et al., 2012), and enzymes (Wawrzynczyk et al., 2007) have been shown to enhance WAS (waste activated sludge) biodegradability by promoting the hydrolysis process.

Past studies have shown an emerging interest in the microwave technique, because the desired temperature can be reached very shortly through microwave heating (Kuglarz et al., 2013). Park et al. (2010) have reported that microwave energy significantly reduces the volume of sludge and removes pathogens (Eskicioglu et al., 2009).

Previous studies have focused on how microwave irradiation and contact time have influenced the solubilisation (Chang et al., 2011), dewaterability (Yu et al., 2010), energy efficiency (Tang et al., 2010), and pathogen destruction (Kuglarz et al., 2013). However, the aim of this study was to enhance the efficiency of MW pretreatment, which was accomplished by the removal of EPS from the sludge matrix. Studies have also revealed that divalent cations ( $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Fe}^{2+}$ ) can bind to negative sites on EPS (such as polysaccharides and proteins) which increase the floc size and strength (Park and Novak, 2007). Also, removal of EPS destabilises the flocs and exposes the biomass to the action of external factors (Merrylin et al., 2013). Therefore, disruption of sludge biomass structure was found to be indispensable. Cation-binding agents deflocculate the sludge floc structure by removing bridging ions like  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  from the flocs, thereby releasing increased amount of proteins, carbohydrates and humics (Wawrzynczyk et al., 2007). Very few reports have addressed on improving the efficiency of microwave energy through floc disruption preceding microwave irradiation. Hence, this paper presents an extensive study on the effect of deflocculation by a cation binding agent, sodium citrate, prior to microwave irradiation and evaluated its impact on matter solubilisation, fermentation and anaerobic biodegradability.

## 2. Methods

### 2.1. Sample collection

Municipal waste-activated sludge was collected from a wastewater treatment plant located in Kerala, India. The sample was stored in a refrigerator at 4 °C. The initial characteristics of the sludge sample were as follows: pH was 6.82; total COD (TCOD) was  $13,500 \pm 300$  mg/L; soluble COD (SCOD) was  $150 \pm 10$  mg/L; total solids (TS) was  $14,400 \pm 300$  mg/L; suspended solids (SS) was  $10,000 \pm 400$  mg/L; volatile solids (VS) was  $8800 \pm 200$  mg/L; soluble proteins was  $15.0 \pm 0.5$  mg/L; and soluble carbohydrates was  $5.2 \pm 0.25$  mg/L.

### 2.2. Experimental design for deflocculation

Dosage optimisation for cation binding agent, sodium citrate, was performed in nine conical flasks of 250 mL capacity containing one litre of sludge, in the range of 0.01–0.2 g/g SS. The contents of the conical flasks were mixed continuously using an orbital shaker for 1 h at 150 rpm. The experiments were carried out in triplicate at a room temperature of 30 °C.

### 2.3. Sludge disintegration using microwaves

A microwave oven (Make IFB, Model-30SC2, 2450 MHz frequency, 900 W power), was used to disintegrate the sludge.

Microwave heating of sludge was carried out with the batch of 300 mL volumes of WAS at varying power levels. The irradiation times varied from 30 to 300 s. at intervals of 30 s., and the temperature ranged from 30 to 96 °C. The specific energy was computed by adopting the method detailed by Yang et al. (2013).

### 2.4. Batch fermentation experiments

Anaerobic fermentation experiments were carried out in 300 mL serum bottles at the working volumes of 250 mL, and the experiments lasted for 3 days. In the bottles, substrate and inoculum were taken at a ratio of 9:1. The bottles were treated at 102 °C for 30 min to nullify the activity of methanogens. Then, the contents were allowed to cool to room temperature before 50 mM (or 9.5 g) BESA (2-bromoethane sulphonic acid) was added. The bottles were purged with nitrogen, and it was sealed air tight and was placed in a shaker at 120 rpm for 72 h at 35 °C.

### 2.5. Anaerobic batch test

The BMP test was conducted in batch reactors at mesophilic conditions for control, flocculated and deflocculated sludge samples, with a retention period of 25 days. The methodology used for BMP test was adopted from the literature (Poornima Devi et al., 2014; Gayathri et al., 2015; Kavitha et al., 2014). The cumulative methane production data of BMP test was simulated using modified Gompertz model (GM) for control sludge and exponential model (EM) and logistic model (LM) were used for both flocculated and deflocculated samples. The modified Gompertz model was adopted following Merrylin et al., 2013. The first-order exponential model (EM) was applied for the first exponential phase of methane production (0–5 days). The logistic model fits the second phase with an initial lag phase, followed by an exponential phase and a final stabilised phase. The logistic model and first-order exponential model were adopted following Rincón et al., 2013. Origin 9.0 software was used to calculate the statistical parameters of the experimental values through non-linear regression for the various stages of methane production, which are summarised in Table 2. Methane production rates of samples were simulated using Gaussian plots, following Rincón et al. (2013), assuming that methane production rates would follow the normal distribution over the digestion period.

### 2.6. Analytical procedure

TCOD, SS, SCOD, and volatile fatty acids (VFA) were evaluated as explained in Standard methods (APHA, 2005). Protein, carbohydrate, and DNA were estimated by adopting the methods described by Yu et al. (2009) and Kavitha et al. (2014). The tests for enzyme activity (protease and amylase) were conducted according to the procedure detailed by Kavitha et al. (2013).

### 2.7. Statistical analysis

Analysis were made for the significant differences between flocculated and deflocculated pretreatment results, in terms of SS reduction, SCOD release, protein and carbohydrate release, by using one-way ANOVA followed by a *t*-test; a level of significance was less than 0.05 (<0.05). Results were analysed individually for each parameter obtained at various specific energy range from 0 to 14,000 kJ/kg TS. The values of the results used for *t*-test were mean of triplicates ( $n = 3$ )  $\pm$  SD (standard deviation).

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