



Effect of deflocculation on photo induced thin layer titanium dioxide disintegration of dairy waste activated sludge for cost and energy efficient methane production

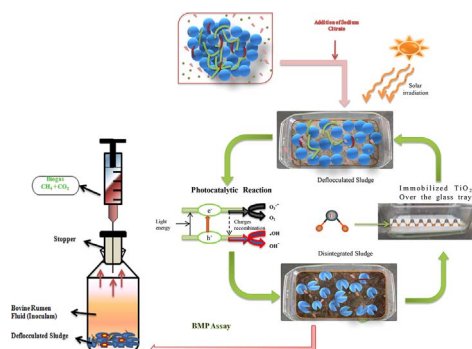


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GRAPHICAL ABSTRACT



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ABSTRACT

In the present study, the deflocculated sludge was disintegrated through thin layer immobilized titanium dioxide (TiO_2) as photocatalyst under solar irradiation. The deflocculation of sludge was carried out by 0.05 g/g SS of sodium citrate aiming to facilitate more surface area for subsequent TiO_2 mediated disintegration. The proposed mode of disintegration was investigated by varying TiO_2 dosage, pH and time. The maximum COD solubilization of 18.4% was obtained in the optimum 0.4 g/L of TiO_2 dosage with 5.5 pH and exposure time of 40 min. Anaerobic assay of disintegrated samples confirms the role of deflocculation as methane yield was found to be higher in deflocculated (235.6 mL/gVS) than the flocculated sludge (146.8 mL/gVS). Moreover, the proposed method (Net cost for control – Net cost for deflocculation) saves sludge management cost of about \$132 with 53.8% of suspended solids (SS) reduction.

1. Introduction

The excess waste activated sludge (WAS) produced from activated sludge process in wastewater treatment plant is considered as the major environmental problem (Xu et al., 2015). Sludge management alone cost around 60% of the total operational cost in WWTP. However, due

to its rich organic content, WAS can be used as a beneficial resource for bioenergy generation. Treatment of sludge through conventional method includes landfill, incineration, aerobic digestion and anaerobic digestion (Kavitha et al., 2014a). Among them, anaerobic digestion (AD) of WAS is of considerable interest owing to its green-energy recovery in the form of biogas, which is documented as cost-effective way

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of treatment method (Li et al., 2013). The hydrolytic and acidogenic phases play intermediary role in the bioconversion of WAS to biogas. Among these phases, hydrolysis limits the biodegradation rate of WAS. Thus, proper disintegration is mandatory prior to AD for accelerating biodegradability of WAS. Various disintegration methods have been reported by researchers in recent years to intensify the hydrolysis process, such as mechanical, chemical, thermal, biological, physiochemical and alkaline (Pilli et al., 2016). Advanced oxidation process using semiconductor catalysts (TiO_2) have acquired more attention for sludge treatment (Arlos et al., 2016). The semiconductor catalyst (TiO_2) has been applied to resolve the various environmental issues, such as hazardous chemicals, presence of organic pollutants, contaminants and pathogens by oxidising them in a non toxic way for the last few decades and is extensively investigated catalysts due to its low cost and non toxicity with high chemical stability (Adams et al., 2013). During solar photocatalytic process, the generated hydroxyl radical disintegrates the organic matters due to photo-induced oxidative reaction under sunlight (Vitor et al., 2011) that promotes the sludge solubilization. The usage of nano-sized TiO_2 in the form of powder for disintegration of WAS has major drawbacks, such as inefficient photo catalysts separation from suspension after disintegration. This necessitates the implementation of recovery step after sludge disintegration. To avoid this issue, many explorations have been carried out on immobilization of photo catalysts onto the static surface, such as glass, cotton and ceramics. There are various methods applicable to immobilize the TiO_2 , such as sol gel (Hanel et al., 2010), dip coating (Lee et al., 2013), combined sol gel dip coating (Blanco et al., 2015) and simple incipient wetness impregnation (SIWI) (Wu and Li, 2011). Among them, SIWI method with TiO_2 /Zeolite catalyst has many advantages such as higher disintegration, easy recovery and reusability (Jan et al., 2015). Hence, it is used in this study.

Extracellular polymeric substances (EPS) are high molecular weight, three dimensional, highly hydrated biological glue and leads to the development of microbial aggregates (Bao et al., 2016; Yang et al., 2016). In order to reduce the organic solid content and to increase the surface area before sludge disintegration, deflocculation process was carried out, which in turn boost up the disintegration process (Kavitha et al., 2014a). To our best knowledge, no previous work has been done on disintegration of sodium citrate mediated deflocculation of sludge using immobilized solar photo catalysts (TiO_2) thin layer by simple incipient wetness impregnation technique, which is a novel approach. The aim and objectives of the study is to 1] to optimize the dosage of sodium citrate for effective removal of EPS with limited cell lysis 2] to immobilize TiO_2 for effective reusability 3] to investigate the efficiency of deflocculation on solids reduction and solubilization 3] to assess the sludge disintegration potential of this proposed method through kinetic analysis 4] to evaluate the efficiency of sodium citrate mediated TiO_2 on biomethane generation. 5] to investigate the feasibility of implementing this novel pretreatment process at large scale.

2. Materials and methods

2.1. Sample collection

Dairy waste activated sludge (WAS) was sampled from secondary clarifier of wastewater treatment plant in Aavin dairy industry, Madurai. The collected sample was stored in laboratory at 4 °C to minimize the bacterial activity before experimentation. The initial characteristics of the waste activated sludge was determined as follows: pH = 6.5, total solids (TS) = 11560 ± 500 mg/L, suspended solids (SS) = 7000 ± 200 mg/L, dissolved solids = 4560 ± 200 mg/L, total oxygen demand (TCOD) = 10500 ± 250 mg/L, soluble chemical oxygen demand = 100 ± 5 mg/L, soluble protein = 4 ± 0.1 mg/L, soluble carbohydrate = 2 ± 0.1 mg/L.

2.2. Deflocculation of WAS using sodium citrate

To determine the effect of sodium citrate dosage on the EPS removal from WAS, an experiment was carried out in series with 10 sets of 250 mL conical flasks containing different dosage of sodium citrate ranges from 0.01 g/g of SS to 0.1 g/g of SS. EPS extraction was carried out by adding different dosage of sodium citrate in each respective conical flask containing 200 mL sludge. All the flasks were kept in the orbital shaker at 150 rpm at ambient room temperature for 1 h. After 1 h, samples were collected from the conical flask and centrifuged at 7500 rpm for 15 min. Then the supernatant removed after centrifugation was filtered and analyzed.

2.3. Preparation of TiO_2 thin layer by simple incipient wetness impregnation method for sludge disintegration

Using simple incipient wetness impregnation technique (Jan et al., 2015), Zeolite/Titanium dioxide composites obtained, which in turn forms the immobilized TiO_2 thin layer on the glass substrate for photocatalytic disintegration. The following procedure has been followed as given below:

2.3.1. Preparation of zeolite

Zeolite act as supporting material as well as a binder for the photocatalyst (TiO_2) coating. Due to its significant role, the sodium form of zeolite (sodium aluminium silicate) type A was used. To prepare this form of zeolite, the zeolite and 0.05 M ammonium nitrate solution needs to be prepared (15 mL/g of Zeolite). The above solution was stirred at 80 °C for 12 h in a shaking water bath. Then the solution was filtered and washed with distilled water. Then the filtrate was dried at 120 °C for 6 h in oven. After 6 h, the oven dried zeolite was calcined at 550 °C for another 6 h in muffle furnace.

2.3.2. Preparation of TiO_2 colloidal suspension

The TiO_2 /Zeolite based composite photocatalysts were primed by simple incipient wetness impregnation method. To the 20 mL distilled water, varying dosage of titanium dioxide ranging from 0.1 g/L to 0.5 g/L was added. The suspension was stirred for 1 h using magnetic stirrer. Then the sodium form of prepared zeolite was added to the titanium dioxide mixture and again stirred for another 1 h using magnetic stirrer. After stirring, the colloidal suspension was subjected to ultrasonication for 10 min under agitation.

2.3.3. Coating of colloidal suspension on glass substrate

In this study, AOP (Advanced Oxidation Process) glass tray was used as the substrate for TiO_2 coating. Before coating TiO_2 , the AOP glass tray was heated at 110 °C in an oven. Then the prepared TiO_2 colloidal suspension was sprayed over the heated glass tray. To prepare the TiO_2 layer, 12 mL of colloidal suspension was used. The coated tray was allowed to dry at ambient room temperature for 12 h.

2.4. Photocatalytic disintegration

Six identical 1000 mL glass trays were used for photo induced catalytic disintegration. Optimum dosage for TiO_2 was predicted by observing the maximum sludge disintegration. For this optimum dosage, thin layer of TiO_2 was prepared by immobilizing it on AOP glass tray. The solar photocatalytic process was carried out with 250 mL deflocculated sludge in the varying immobilized thin layer TiO_2 coated AOP glass tray and kept under solar irradiation for about 1 h. The experiment was carried out during the month of February and March in Tirunelveli under the solar irradiation with UV (Ultra Violet) intensity in the range of about 5.47–5.89 kWh/m³. The samples were collected in various interval of time from 0 to 60 min and the efficiency of disintegration was determined. Immobilized photocatalytic disintegration was optimized using Response surface methodology (RSM). A Central

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