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# Preparation and reuse of iron and aluminum oxides activated sewage sludge based coagulants for the post-treatment of up-flow anaerobic sludge blanket reactor effluent



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## ABSTRACT

Present study proposed the iron and aluminum oxides activation approach to produce the sewage sludge derived coagulants (SSCs) through the pyrolysis of sewage sludge (SS). Characterization techniques including X-ray diffraction (XRD), thermogravimetric analysis (TGA), field emission scanning electron microscopy (FESEM) and elemental dispersion X-ray spectroscopy (EDX) were used to investigate the structural change in SSCs. As prepared SSCs were used for the comparative treatment study of up-flow anaerobic sludge blanket (UASB) reactor effluent via coagulation/flocculation process. Response surface methodology was used to optimize the responses namely, chemical oxygen demand (COD) and turbidity removal efficiency influenced by the variables including dose of iron oxide activated SSC, aluminum oxide activated SSC, polymeric composite coagulant ferric aluminum silicate sulfate (PFASS) from the range of 100-500 mg/L and the range of initial pH 7.0-11. Preliminary tests analysis, results of modeling and confirmation tests proved that SSCs exhibited the excellent performance for post-treatment of UASB effluent compared to polymeric coagulant PFASS because activation with pyrolysis enhanced the structures features and metal contents of sludge which favored in coagulation, precipitation, and adsorption. Additionally, sludge itself contains a higher amount of Mg, Ca and Si oxides and/or hydroxides which fairly contributes in the removal process compared to freshly prepared PAFSS. Furthermore, zeta potential study also helpful to understand the bridging of adsorption and charge neutralization during coagulation-flocculation mechanisms and played a crucial role in process optimization via decreased the colloids surface charge. The results of the present study illustrate that iron and aluminum oxides activation is an effective approach for reuse the sewage sludge as coagulants and their reuse for post-UASB reactor effluent treatment as an attractive possibility from waste to resource recovery.

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### 1. Introduction

Sewage sludge, a residue of wastewater treatment has been speedily increasing in worldwide, due to rapid urbanization. It is estimated about 38354 million liters per day (MLD) sewage is

produced in main cities of India, however, only 11786 MLD sewage treatment capacity and 234 sewage water treatment plants serves the Indian population (CGWB, 2011). About 1.0 Mha–1.5 Mha of land area annually irrigate by sewage waters in India (CPCB, 2009). However, more than 80% sewage sludge in India is not correctly disposed off and has developed a cause of secondary pollution to the environment (Tyagi and Lo, 2013; Nair and Ahammed, 2016). Sewage sludge comprises organic contaminants, pathogenic microorganisms and amounts of heavy metals (Liu et al., 2010; Tyagi

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#### and Lo, 2013).

On the basis of sources of generation, sludge can be categories as biological sludge, chemical sludge, and primary sludge (Smith et al., 2009; Tyagi and Lo, 2013). Some of the conventional techniques such as land utilization (landfilled), incineration and cropland application have been reported for the sludge treatment. Nevertheless, these possibilities have progressively been limited because of the severe obligation for environmental protection. Accordingly, the sludge disposal in an environmentally suitable manner is a serious issue. Within this framework, an excessive progress has been made to concerning the reusability of various type of sludge, especially for the swage/industrial wastewater sludge (Smith et al., 2009; Tyagi and Lo, 2013; Antonkiewicz et al., 2016). Recently, Bratina et al. (2016) reported a report on the economic sustainable sludge management of municipal/industrial wastewater sludge and fats, oil and grease (FOG) to fertilizer. Antonkiewicz et al. (2016) reported a 6-year field experiment based study for the application of municipal sewage sludge on energy crops, especially reed canary grass and giant miscanthus are the species of monocotyledonous energy crops. Authors were found that miscanthus and reed canary grass shows the highest heavy metal uptake at the sewage sludge dose of 20 Mg DM/ha and 40 Mg DM/ha.

Sewage sludge pyrolysis has also been used to propose a favorable alternative for sewage sludge management by the generation of porous carbonaceous solid by the conversion of higher contents of organic substances in sewage sludge (Ahmad et al., 2016; Chen and Kuo, 2016). The major advantages of sludge pyrolysis are: (a) destroys the organic compounds, (b) sludge transformed into ash, char, water vapor, pyrolysis, and combination gases, (c) gaseous and/or solid products portion of pyrolysis method are incinerated and used as a source of heating in the pyrolysis process (Tyagi and Lo, 2013).

Sewage sludge based coagulant (SSC) and sewage sludge based adsorbent (SSA) have been used to remove the various contaminants such as heavy metals, phosphates, dyes, phenolic compounds and others organically polluted wastewaters (Smith et al., 2009; Xu et al., 2015). In thermal treatment technologies, sludge using as a low-cost feedstock, which has increased the advantages of sludge by the conversion into value-added products, extracting useful energy to be self-sustaining and export, and reducing waste volume. Particularly, SSC and SSA have superb thermal stability and harmless leaching toxicity (Xu et al., 2015).

Studies have been confirmed that the sludge based coagulant and adsorbent prepared from the chemical sludge pyrolysis had a higher pollutants removal efficiencies and larger surface area compared that prepared from biological activated sludge (Pan et al., 2011; Yang et al., 2016). In comparison of biological sludge, chemical sludge is a common form of sludge, which is formed from the precipitation reaction in presence of chemical coagulants (Smith et al., 2009; Jangkorn et al., 2011; Singh et al., 2016a). The presence of coagulants such as aluminum and iron salts and its residue in chemical sludge might improve the porous structure of the sludge for the development of SSC/SSA, and increase the removal efficiency of the contaminants from the wastewater (Pan et al., 2011; Singh et al., 2014). However, the usefulness of ferric salts activation in chemical sludge pyrolysis has been established in the literature, there is dominative evidence on the use of aluminum and ferric activation in sewage sludge for the preparation of SSC and their reuse for post-treatment of UASB reactor effluent.

The chemical and physical properties of SSC depends on the preparation conditions, like as mass ratio (activator/sludge), pyrolysis temperature, dwell time and heating rate. Between them, mass ratio and pyrolysis temperature are the major factors, highly influence the textural properties of SSC (Smith et al., 2009; Xu et al., 2015). Moreover, iron and aluminum mixed coagulants such as

polymeric aluminum ferric sulfate (PAFS) and poly-ferricaluminum-silicate-sulfate (PFASS) have shown the excessive potential as a coagulants for the removing of contaminants from the wastewater (Sun et al., 2011; Zhu et al., 2012).

The aim of present study is to estimate the practicability of asprepared SSC from sewage sludge by iron oxide activation (Feactivation) and aluminum oxide activation (Al-activation) and their application for post-treatment of UASB reactor effluent by the coagulation-flocculation process. A comparative coagulationflocculation study was performed with as-prepared Fe/Al-activated sewage sludge coagulants and most relevant literature reported PFASS coagulants for determining the optimum treatment condition for post treatment of UASB reactor effluent. The batch experiment was done to optimize the treatment parameters such as types of coagulates, solution pH and dosage of coagulants. Response surface methodology (RSM) was used for optimizing the parameters, the effect of an individual variable along with the interaction effects between different variables. In this study, the effect of Fe-activated SSC (SSC-I) and Al-activated SSC (SSC-II) dose, solution pH, and freshly prepared PFASS doses on the COD and turbidity removals were investigated by using the factorial Box-Behnken design (BBD) methodology to evaluate the optimal treatment conditions. Consequently, the main objective of this study was first prepared the Fe/Al-activated sewage sludge coagulants than examining the practicability of reuse the SSC-I and SSC-II as a coagulants for the post treatment of UASB reactor effluent. Fig. 1 presented the conceptual framework of the present study.

## 2. Materials and methods

#### 2.1. Chemicals and materials

SS as a raw sludge for present study was collected from the two different location: 25 MLD and 30 MLD wastewater treatment plant located at Noida, Uttar Pradesh and Ghansoli, Navi Mumbai Maharashtra, India, respectively, which are treating municipal and small industry wastewater. The Air dry method was used for dried the sewage sludge to a constant weight, and later sludge was grounded into the size less than 100 µm. The UASB reactor treated effluent was collected from municipal wastewater treatment plant located Noida Uttar Pradesh, India. Both sewage sludge and UASB reactor effluent were collected and preserved at 4 °C in environmental engineering laboratory of Indian Institute of Technology, Roorkee, India. Characterization of sludge and wastewater were carried out in accordance with the standard method of wastewater treatment (APHA, 1998). Water glass, aluminum sulfate (Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·18H<sub>2</sub>O), ferric sulfate (Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·7H<sub>2</sub>O), sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>), sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), and concentrated nitric acid (HNO<sub>3</sub>) were obtained from Sigma-Aldrich and Ranbaxy Co., Ltd (Delhi, India), respectively. All chemicals were used in this study are analytical grade and used without further purification.

### 2.2. Preparation of coagulants

Ferric sulfate and aluminum sulfate were mixed into the two different dried sludge at mass ratios (ferric sulfate/dried sludge-I and aluminum sulfate/dried sludge-II) of 0.5 at 750 °C (Yang et al., 2016). Normally, about 20 g sulfate salts mixed sludge were used for pyrolysis in a furnace. The pyrolysis heating rate was kept 20 °C/min under the absence of oxygen atmosphere, and the heating was started from room temperature to the final temperature of 750 °C. The hold temperature at the 750 °C was taken for 2 h. After cooling, the sample was collected from the furnace. The

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