



# Synthesis, characterization, and secondary sludge dewatering performance of a novel combined silicon–aluminum–iron–starch flocculant



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## HIGHLIGHTS

- Silicon, aluminum, and iron were grafted onto starch chains to synthesize CSiAFS.
- The sludge dewatering performance of CSiAFS was superior to PAC, PAM, and FeCl<sub>3</sub>.
- CSiAFS exhibited a good dewatering efficiency over a wide range of pH (3.0–11.0).
- CSiAFS had a discontinuous surface with channels which helped to sludge dewatering.

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## ABSTRACT

Flocculation is one of the most widely used cost-effective pretreatment method for sludge dewatering, and a novel environmentally friendly and efficient flocculant is highly desired in the sludge dewatering field. In this study, a novel combined silicon–aluminum–ferric–starch was synthesized by grafting silicon, aluminum, and iron onto a starch backbone. The synthesized starch flocculant was characterized using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy, X-ray powder diffraction, and thermogravimetric analysis. The dewatering performance of secondary sludge was evaluated according to the capillary suction time, settling volume percentage, and specific resistance to filtration. The results indicated that the copolymer exhibited: (1) a good dewatering efficiency over a wide pH range of 3.0–11.0, (2) superior sludge dewatering performance compared to those of polyaluminum chloride (PACl), polyacrylamide (PAM), ferric chloride, and (3) a discontinuous surface with many channels or voids that helps to mobilize the impermeable thin layer of secondary sludge during filter pressing. Such a novel copolymer is a promising green flocculant for secondary sludge dewatering applications.

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## 1. Introduction

The sludge produced by a sewage treatment plant often contains a high moisture content of over 90% [1]; such a high moisture content of sludge will waste a large amount of storage space and energy, thus making sludge dewatering inevitable. Flocculation is one of the most widely used cost-effective pretreatment method for sewage sludge dewatering [2–8]. Both inorganic and synthetic organic polymer flocculants exhibited high flocculation efficiency, but some of them often lead to secondary pollution during their use [9–11]. Therefore, it remains necessary to develop environmentally friendly flocculants with high flocculation efficiency.

Starch has the advantages of being inexpensive, renewable, fully bio-degradable and easy to obtain [12]. In recent years, starch derivatives used as flocculants have attracted considerable attention [13–15]. Cationic groups (amine and acrylamide [16–18]) and anionic groups (phosphate, xanthate, and carboxylate [14,19–22]) have been incorporated in starch to obtain high-efficiency flocculants.

Colloids in sludge are usually negatively charged, and thus, cationic flocculants are suitable for charge neutralization and are favorable for flocculation [23]. However, some synthetic cationic polymers could increase the viscosity of the sludge system and reduce the permeability of the filter cake, thus increasing the difficulty of sludge dewatering [24,25]. Therefore, synthesis of a new green cationic starch to promote the filter cake permeability and porosity is highly necessary.

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In our earlier paper, a combined aluminum–ferrous–starch flocculant (CAFS) was prepared with aluminum sulfate, ferrous sulfate and starch, and it exhibited a good flocculation efficiency [26]. Silicon addition was found to increase the net chain size [27], promote the formation of porous structure [28], enhance aggregating efficiency and give better flocculation effects [29]. In this study, a combined flocculant (CSiAFS) was prepared with starch, sodium silicate, aluminum sulfate, and ferric chloride. The structure and morphology of CSiAFS were characterized by Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy, X-ray powder diffraction and thermogravimetric analysis. Also, the dewatering ability of CSiAFS was evaluated by capillary suction time, settling volume percentage and specific resistance to filtration. The aim of this work was to prepare a starch-based flocculant with a high molecular size and bridge-aggregating ability, and to better understand the mechanisms involved in the flocculation dewatering process.

## 2. Materials and methods

### 2.1. Materials

#### 2.1.1. Reagents

Maize starch with a moisture content of 13.4% was purchased from Changchun Dacheng Maize Starch Co., Ltd. PR China. Polyaluminum chloride (PACl) with a basicity (OH/Al) of 1.35 and an  $Al_2O_3$  content of 30% was purchased from Nanning Chemical Industry Co., Ltd, PR China. Aluminum sulfate, sodium hydroxide, and sulfuric acid were purchased from Guangzhou Chemical Reagent Factory PR China, and sodium silicate, ferric chloride, and polyacrylamide (PAM) were purchased from Tianjin Damao Chemical Reagent Factory, PR China. All of these reagents were of analytical

grade, and all of the solutions were prepared by deionized water.

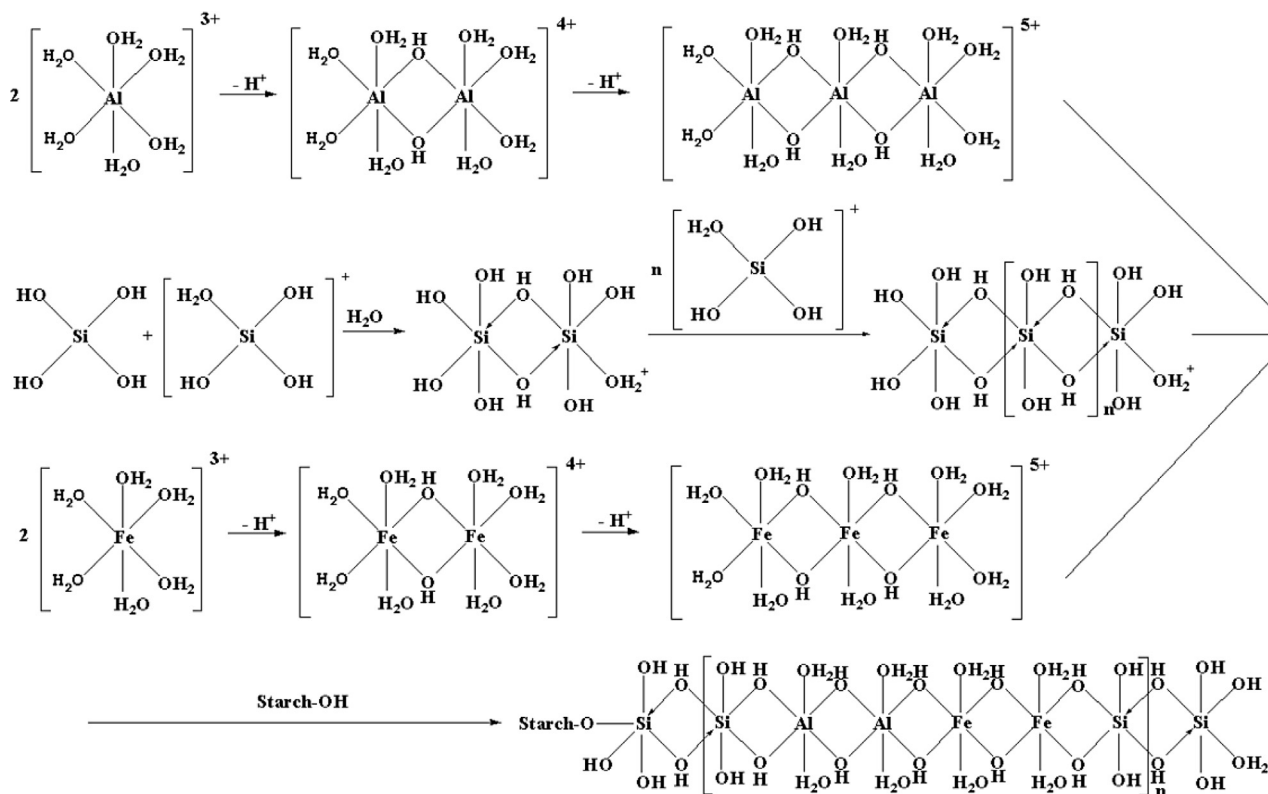
#### 2.1.2. Secondary sludge

The secondary sludge samples were collected from the secondary settling tank of the Guangzhou Lijiao sewage treatment plant, China. The plant treats 500,000 m<sup>3</sup>/d of domestic wastewater using an anaerobic–anoxic–oxic process. The samples were transported to the laboratory within 30 min following sampling; next, the samples underwent gravity thickening, and then, the supernatant was removed. Subsequently, the samples were stored in a refrigerator at 4 °C for less than 2 days. The properties of secondary sludge samples were as follows: moisture content 95.0%, SRF  $98.3 \times 10^{11}$  m/kg, CST 120 s, SV<sub>30</sub> 85.0%, and pH 6.75.

### 2.2. Synthesis of CSiAFS

A modified starch solution was prepared by adding approximately 80 mL of a 0.16 mol/L NaOH to an aqueous slurry containing 5.000 g of maize starch (13.4% moisture content, China) at 50–55 °C with continuous low speed stirring (50 rpm) for 1 h. Meanwhile, 0.3 mol/L of Na<sub>2</sub>SiO<sub>3</sub> was prepared, regulating the pH to 3.0 by using 5 mol/L of H<sub>2</sub>SO<sub>4</sub>, and then, the mixture was stirred at 50 rpm for 1 h. Next, 100 mL of 0.3 mol/L of Na<sub>2</sub>SiO<sub>3</sub>, 80 mL of 1.4 mol/L of Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and 20 mL of 0.9 mol/L of FeCl<sub>3</sub> were added to the modified starch solution, and the pH was adjusted to 3.0 using 5 mol/L of H<sub>2</sub>SO<sub>4</sub>. The mixture was stirred for 4 h in a water bath at 50–55 °C, followed by cooling to room temperature and freeze-drying to produce CSiAFS. The synthetic pathway is shown in Scheme 1.

Single silicate is a weak amphoteric acid, and at strong acid condition (pH 3.0), polymerization occurs between single acid molecules to generate polysilicate. Meanwhile, at low pH



Scheme 1. Synthesis of CSiAFS.

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