



## Screening pretreatment methods for sludge disintegration to selectively reclaim carbon source from surplus activated sludge



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### H I G H L I G H T S

- Pretreatment methods had different preference in carbon and nutrient release.
- Ultrasound was good for selective carbon source release.
- Homogenization was economic for selective carbon source release.

### A R T I C L E I N F O

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### A B S T R A C T

Four pretreatment methods, including alkaline, chlorine dioxide, ultrasound, and homogenization, were screened for sludge disintegration to selectively release carbon from surplus activated sludge (SAS), while avoiding nutrient (phosphorus and nitrogen) release. The characteristics of these methods in terms of carbon, nitrogen, and phosphorus releases were compared, and the variation between the actual and theoretical values of those releases was evaluated. The results showed that the four pretreatment methods had different preferences in both carbon and nutrient release, indicating a possibility of selective release. Ultrasound had a preference in carbon release while not in nutrient release, and was a good method for selective release of carbon source. But homogenization was chosen for sludge disintegration to obtain carbon source from SAS with a cost consideration. And alkaline and chlorine dioxide pretreatment methods were not advocated for a low selective release of carbon source and a gas emission into environment.

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## 1. Introduction

High concentrations of nutrients in rivers have been related to eutrophication. The leak of phosphorus and nitrogen from wastewater effluent is restricted by framework directive. Although the biological nutrient removal (BNR) process has been successfully applied in nutrient removal from wastewater, the wastewater must have sufficient carbon to favor the metabolism of phosphate accumulating organisms (PAO) and heterotrophic denitrifiers, which serves a primary function in phosphorus and nitrogen

removal [1]. Chemical oxygen demand (COD)/total nitrogen (TN) and Chemical oxygen demand (COD)/total phosphorus (TP) ratios above 6 and 35, respectively, were feasible for the removal of these nutrients [2]. As a carbon source is usually lacking in wastewater, external carbon sources, such as methanol, ethanol, acetic acid, acetate, methane, and industrial effluent rich in soluble carbon, can be supplemented to the wastewater to achieve the essential carbon-to-nutrient ratio.

The disposal of surplus activated sludge (SAS) is of great concern for many wastewater treatment facilities, because it accounts for over half of the total cost of wastewater treatment. An attempt to supplement external carbon sources may further aggravate the yield of SAS and increase the sludge disposal cost. Alternatively, a new carbon source called internal carbon source was produced by SAS. Internal carbons comprised a suitable carbon source for BNR, and the application of internal carbons to BNR to improve nutrient removal was successful [1,3,4]. The reuse of SAS as carbon

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source not only reduces the overall cost of carbon source for phosphorus and nitrogen removal but also addresses the issue of sludge disposal.

Sludge disintegration has been widely examined for improving sewage sludge handling and disposal problems, such as sludge anaerobic digestion [5–9], cryptic growth sludge reduction [10,11], sludge dewatering [12,13], and biohydrogen production [14,15]. A range of mechanical, thermal, chemical, and biological technologies have been investigated. However, few of these technologies have been used for sludge disintegration to obtain carbon sources. Ultrasound and deflaker pretreatments were examined for sludge disintegration to provide carbon source for BNR, resulting in a soluble COD (SCOD) increase up to 48-fold and a maximum volatile fatty acids (VFAs) concentration of 530 mg/L [16]. Hydrothermal reaction was applied to liquefy SAS to obtain a carbon source, the ratio of dissolved organic carbon to total organic carbon was sustained at approximately 65% under most tested conditions, and the readily biodegradable substrate, such as acetic and propionic acid, increased with increasing reaction temperature [17]. The readily biodegradable COD in ozonolysate was substantially increased as ozone dose increased, and the maximum COD of 1588 mg/L was obtained at an ozone dose of 0.2 g O<sub>3</sub>/dry sludge [2]. A combination of alkaline hydrolysis (7.78 mM NaOH) and gamma-ray irradiation (20 kGy) was also used to recover carbon source from SAS. SCOD reached 1900 mg/L at an initial total solid (TS) concentration 16220 mg/L [18]. It is concluded that pretreatment methods evidently affect carbon release significantly.

Sludge disintegration induced a release of a large amount of carbon, measured as SCOD, which contained not only high levels of VFAs (12%) but also a variety of mixed carbon substrates, such as proteins (30%), carbohydrates (13%), and other uncharacterized substances (45%), making it generally useful for a wide range of biomass [16]. In the phosphorus release test, the addition of disintegrated sludge enhanced phosphate (PO<sub>4</sub>-P) release by 14.9 mg/L PO<sub>4</sub>-P versus that of 4.3 mg/L PO<sub>4</sub>-P in the control vessel without carbon addition. In the denitrification test, the nitrate (NO<sub>3</sub>-N) consumption rate was improved after the addition of disintegrated sludge (14.9 mg NO<sub>3</sub>-N/g VSS h) compared with the control vessel (6.9 mg NO<sub>3</sub>-N/g VSS h) [4]. The denitrification rate reached 41.2 mg N-NO<sub>3</sub>/g VSS h [3]. High phosphorus release and denitrification rates were also obtained for disintegrated SAS (0.031 mg PO<sub>4</sub>-P/g VSS min, 0.038 mg NO<sub>3</sub>-N/g VSS min) compared with acetate (0.024 mg PO<sub>4</sub>-P/g VSS min, 0.027 mg NO<sub>3</sub>-N/g VSS min) [1]. Thus, the overall performance of the disintegrated sludge could be satisfactory [19,20]. However, the drawback is the consequently higher addition of phosphorus and nitrogen concentration to the wastewater, which could negatively affect nutrient recycling in the BNR process. Although some researchers cited this problem [1,18,21], no solutions have been suggested for a nutrient removal from the disintegrated sludge. A better solution is to selectively release carbon through an appropriate pretreatment method, and with the maximum carbon release and minimum phosphorus and nitrogen release. This idea is based on our previous work, where we found that the pretreatment methods can significantly affect carbon and nutrient release from SAS and result in different SCOD/TN and SCOD/TP ratios for the disintegrated sludge [11,22,23]. We suppose that a possibility exists that an appropriate pretreatment method can selectively release carbon (indexed by SCOD) while avoiding the release of phosphorus and nitrogen (indexed by TP and TN). In this work, four pretreatment methods, including alkaline, chlorine dioxide, ultrasound, and homogenization are investigated for a preference in SCOD, TN and TP release, with the aim of determining an appropriate method for sludge disintegration to selectively reclaim carbon source from SAS.

## 2. Materials and methods

### 2.1. Pretreatment methods to disintegrate surplus activated sludge

SAS was cultivated by a pilot-scale SBR treating sewage wastewater with COD of 100–500 mg/L (average 157 mg/L), TN of 20–40 mg/L (average 23.8 mg/L), and TP of 2–8 mg/L (average 2.36 mg/L). A portion of the mixed liquor was regularly discharged to maintain a mixed liquor suspended solid (MLSS) concentration of approximately 3500 mg/L in SBR [11]. The discharged mixed liquor was precipitated gravitationally until total solids reached above 30,000 mg/L. The thickened sludge was stored at 4 °C for pretreatment within a week. The sludge samples for disintegration with required concentration were obtained by diluting the thickened sludge with tap water.

For alkaline pretreatment, certain amount of sludge sample with a concentration of 20 g/L was equally divided and were fed into eight beakers, then NaOH of 10 mol/L was added to adjust their initial pH to 8.0, 9.0, 10.0, 11.0, 12.0, 12.5, 13.0 and 13.5, and they were stirred at 150 rpm for 2 h. The chemical pure NaOH was purchased from Kermel Plant (Tianjin, China). For chlorine dioxide pretreatment, 100 mL of sludge sample with a concentration of 15 g/L was subjected to varying doses of chlorine dioxide from 0 to 10 mg chlorine dioxide/g dry sludge in a beaker experience, and chlorine dioxide reacted with sludge sample for 40 min at a stirring rate of 120 rpm. Highly pure chlorine dioxide (95%) was supplied by a chlorine dioxide generator (HYCB-50, China), which used the hypochlorite reaction with hydrochloric acid. For ultrasound pretreatment, the pole of the ultrasonic generator was put into the sludge sample with a concentration of 15 g/L for a reaction time of 6 min, and the ultrasonic intensity varied from 0 to 3 W/mL. The pole ultrasonic generator (SCIENTZ-IIID, China) had a frequency of 20 kHz and an adjustable power of 0–950 W. For homogenization pretreatment, the sludge sample with a concentration of 15 g/L was pumped through the homogenization with one cycle process, and the working pressure varied from 0 to 150 MPa (more frequent blockage happened when the working pressure higher than 150 MPa). The high pressure homogenizer (JN-02HC, Juneng Biology & Technology Co., Ltd, Guangzhou, China) had a working pressure range of 0–210 MPa.

After the disintegration as described above, the sludge samples were filtered immediately by 0.45 μm microporous membrane for a determination of soluble substances that released from those sludge samples, and the filtrates were stored for analysis. For the filtrates, COD was measured according to Method 5220D (closed reflux, titrimetric method), with detecting concentration above 40 mg O<sub>2</sub>/L; TN was measured according to Method 4500-N (persulfate method), with detecting concentration below 2.9 mg N/L; TP measurement was carried out according to Method 4500-P (ascorbic acid method), with detecting concentration in a range of 0.01–6 mg P/L; TS and VS measurements were carried out according to Method 2540G [24]. All of the experiments were conducted at least in duplicate at room temperature (20–27 °C) and the average value was reported for all data.

### 2.2. Carbon and nutrient release

The operation condition of the four pretreatment methods was determined according to SCOD release, which is commonly used as an index to evaluate the degree of sludge disintegration. The optimum conditions with maximum SCOD release are listed in Table 1, which have been reported in our previous research; except for alkaline that will be reported in another study. For a carbon source application of disintegrated sludge, the release of

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