



## Performance and evaluation of aerobic granular sludge in oily wastewater treatment

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### ABSTRACT

Oily wastewater treatment through membrane separation is remarkably effective, but the high operation cost and poor resource recovery potential of this method necessitate the application of an integrated physical/chemical–biological oily wastewater treatment. In this study, aerobic granular sludge was applied for oily wastewater treatment from the ultrafiltration (UF) effluent of a two-stage UF–reverse osmosis process. The removal efficiencies of the sludge for oil and chemical oxygen demand reached 94.1% and 85.6%, respectively. The protein concentration of extracellular polymeric substances (EPS) increased to 34.6 mg/g MLSS, and the relatively high protein/polysaccharide ratio was found to be closely related to the formation of aerobic granular sludge. In addition, protein in loosely bound-EPS was converted to tightly bound-EPS (TB-EPS), which indicated that the protein in TB-EPS could be the major factor affecting the granulation process. Separate sludge incineration could be achieved due to the lower heating value of granules was as high as 8.5 MJ/kg. Furthermore, the proposed process could enhance the treatment efficiency of the sludge incinerator and increase the amount of heat energy that could be recycled.

**Keywords:** Aerobic granular sludge; Tightly bound-extracellular polymeric substances; Oily wastewater treatment; Sludge treatment; Heating value

### 1. Introduction

Oily wastewater from oil refinery seriously harms aquatic organisms because the dissolved oxygen in the receiving water significantly decreases with the degradation of organic matter, which consequently disrupts the ecological balance in such environment [1,2]. Membrane separation methods, including ultrafiltration (UF), nanofiltration, reverse osmosis (RO), and forward osmosis, have been widely established for full-scale applications [3–7] of oily wastewater treatment. These methods display satisfactory performance, but their high operation cost,

serious membrane fouling, and short life span limit their applications [8–10]. Membrane separation is not preferred when dealing with emulsified and soluble oil. Therefore, biological or integrated physical/chemical–biological oily wastewater treatment has become a promising technology. Immobilized bacteria and activated sludge exhibit notable removal efficiencies for contaminants from oily wastewater [11,12]. However, the presence of solvents, surfactants, and metal ions renders conventional biological processes unstable and costly [13].

Aerobic granular sludge (AGS) is a promising and innovative technology in domestic sewage treatment, high organic wastewater treatment, and toxic (including heavy

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metal) wastewater treatment [14–16]. Recent investigations on the performance of AGS in lab-scale and full-scale reactions [17] have indicated that this technology can be used in oily wastewater treatment and in emulsified and soluble oil degradation. Several studies focused on the cultivation, granulation, and performance of AGS in oily wastewater treatment [18], whereas only few paid attention to sludge characteristics and oily sludge treatment process. For example, research has shown that microbial forces induced by extracellular polymeric substances (EPS) significantly influence the formation of AGS [19,20]. However, the evaluation of EPS is still depicted by component and trend analyses. Therefore, the application of oily wastewater treatment using AGS still warrants further investigation.

Oily sludge incineration is an important sludge treatment and disposal process that was established in many areas [21] because of its potential threat to the environment. Conventional oily sludge incineration involves high water content and enriched crude oil and mineral particles [22]. The use of chemical conditioners results in low operation capability and high operation cost [23,24]. Hence, the dewatering characteristics of oily AGS and its potential incineration capacity could draw significant attention in further studies.

To date, exhaustive studies concerning oily wastewater treatment by AGS and the sludge treatment process are still sparse. Accordingly, this work provides useful information about the formation of AGS and the contaminant removal efficiency of oily wastewater from UF effluent. To illustrate the influence of EPS on aerobic granulation, the characteristics of EPS were thoroughly analyzed by fluorescence spectroscopy. The possible advantage of sludge treatment based on the comparison between AGS cultivated by oily wastewater and domestic sewage was also examined to determine its feasibility and sustainability in practical and commercial applications.

## 2. Materials and methods

### 2.1. Cultivation of AGS

Compressed air was supplied via a diffuser at the bottom of a sequencing batch airlift reactor (SBAR, working volume of 12.0 L, aspect ratio of 6.0:1) at a flux of 120 L/h. Influent was introduced at the bottom of the reactor by means of a peristaltic pump. Effluent was discharged by a peristaltic pump from the middle part of the reactor with a volumetric exchange ratio of 50%. The operating cycle time was 3 h, including 30 min for static feeding, 140 min for aeration, 5 min for settling, and 5 min for effluent drainage. The operation temperature of the SBAR was controlled at  $25 \pm 1^\circ\text{C}$  in a water bath. Accordingly, reaction pH and solid retention time were adjusted to 7.0 and 30 days, respectively. The experiment was run in triplicate.

The oily wastewater was derived from the UF effluent of a two-stage UF-RO process located in an oil refinery with an oil concentration of 160 mg/L, and the main component was emulsified and soluble oil. The additional components and their concentrations are listed as follows: 60.0 mg/L  $\text{CaCl}_2$ , 42.0 mg/L  $\text{MgSO}_4$ , 60.0 mg/L  $\text{NH}_4\text{Cl}$ , 250.0 mg/L

$\text{NaHCO}_3$ , 58.0 mg/L  $\text{K}_2\text{HPO}_4$ , and 24.0 mg/L  $\text{KH}_2\text{PO}_4$ , which represented a total chemical oxygen demand (COD) concentration of 600 mg/L and oil concentration of 70–80 mg/L. The seed activated sludge was taken from the aerobic tank of Lucun (Wuxi, China) wastewater treatment plant with an anaerobic/anoxic/oxic process.

### 2.2. Conditioning and dewatering of AGS

A sludge dewatering experiment and feasible estimation of sludge incineration were performed to evaluate the potential application of AGS in oily wastewater treatment. The first type of AGS, denoted as GS1, was cultivated by domestic sewage, whereas the second type GS2 was fed with oily wastewater. Quick lime was used as a sludge conditioner, and the dosage of quick lime was expressed as 20% of the original sludge dry solids. After conditioning, the sludge mixture was introduced to a lab-scale plate and frame filter press (Ouyuan XMQ5/500-UB). The dewatering sludge was then evaluated by measuring moisture content and heating value.

### 2.3. Estimation of sludge incineration

On the basis of the elemental composition, higher heating values (HHV) and lower heating values (LHV) were calculated using Eqs. (1) and (2) [25]:

$$R1 = 0.3491C + 0.1783H - 0.1034O - 0.0151N - 0.0211A \quad (1)$$

$$R2 = (R1 - 21.8217H)(1 - W) - 2.442W \quad (2)$$

where  $R1$  and  $R2$  (MJ/kg) are the predicted HHV and LHV, respectively;  $C$ ,  $H$ ,  $N$ , and  $O$  denote the mass percentages of carbon, hydrogen, nitrogen, and oxygen in the sludge sample, respectively; and  $A$  and  $W$  represent the ash and water contents of the sludge sample, respectively.

### 2.4. Analytical methods

COD, sludge moisture content, mixed liquor suspended solids (MLSS), and mixed liquor volatile suspended solids (MLVSS) were measured in accordance with standard methods [26]. Oil concentration was determined using the extraction method described by Petrolei [9]. The sludge volume index (SVI) was determined by employing the settled bed volume after 30 min of settling and the dry biomass weight [27]. The extraction of EPS, including soluble microbial by-product-like (SMP), loosely bound-EPS (LB-EPS), and tightly bound-EPS (TB-EPS) from the AGS sample, was performed in accordance with the protocol described by Adav and Liang [19,28]. The respective total polysaccharides (PS) and total protein (PN) contents in EPS were quantified by Dubois and Lowry methods [29,30]. The characteristics of AGS and EPS were analyzed by fluorescence spectroscopy (Jasco FP-6500). The carbon, hydrogen, nitrogen, and oxygen contents in the AGS were sieved and then measured with an elemental analyzer (Elementar vario Micro select). Meanwhile, the heating value of different AGSs was determined with a calorimeter (Lanbo ZDHW-8A).

### 3. Results and discussion

#### 3.1. Formation of AGS

With an initial MLSS of 4.0 g/L, 6.0 L of activated sludge was inoculated in SBAR. More than 75% activated sludge was washed out and the residual MLSS was only 1.1 g/L in the bioreactor because of the short settling time and the negative influence of toxic substances on microorganisms in oily wastewater (Fig. 1). Then, the biomass gradually recovered along with the operation of SBAR and the adaptability to the toxic substances was enhanced in the following 30 days. In addition, small and yellow granules could be observed. Mature AGS with compact structure and smooth surface dominated in the reactor at day 40, and the formation time was in accordance with that of a previous study [18]. However, the start-up period was slightly longer than that in studies that focused on phenol and pyridine degradation [31,32] because the influent quality of oily wastewater is more complex. During this period, SVI decreased from 122.0 mL/g to 56.2 mL/g and MLSS increased to 8.0 g/L. By contrast, the oily sludge obtained after flocculant coagulation and dissolved air flotation showed poor settleability [24]. After 40 d, SVI and MLSS were relatively stable around 54.1–59.2 mL/g and 4.6 to 8.1 g/L, respectively. Furthermore, no significant disintegration can be observed, which mutually agreed with the aerobic granules that were cultivated by other toxic substances [33].

#### 3.2. EPS characteristics of AGS

Bacteria secrete sticky materials called EPS, which assist cell adhesion and promote the aggregation of other particulate matter during aerobic granulation [34,35]. As shown in Fig. 2, protein production of microorganisms hardly changed in the initial period, but small and yellow granules could still be noticed; in addition, the granules did not prevail in the bioreactor. The protein content remained low mainly because of the sludge wash-out. Then, both of the protein and PS contents increased from day 30, and the protein content significantly improved. This phenomenon increased the PN/PS ratio by 2.3-fold, implying that a high total protein content can enhance and promote the forma-

tion of aerobic granules [36,37]. However, the PN/PS ratio was much lower than that mentioned in salty and oily aerobic granules [18] because high microbial activity and high EPS content can both be obtained under high saline concentrations [38]. In addition, the total protein content in EPS increased from 8.2 mg/g to 34.6 mg/g MLSS along with the operation of the bioreactor. However, the total PS content showed no significant change. The PS concentration varied over the range of 10.4–11.5 mg/g MLSS when small and yellow granules appeared and then slightly increased to 12.6 mg/g MLSS at the end of the start-up phase.

As shown in Fig. 3, the protein contents in TB-EPS and LB-EPS varied. The protein content in LB-EPS initially increased, decreased during the initial stage of SBAR operation, and then gradually improved until the end of the start-up period. However, the protein content in TB-EPS declined in the former phase and then sharply increased finally. Granules have not been formed in the first 20 days because of the influence of oily wastewater. Hereafter, granules can be observed and gradually become mature because of the adaptability to toxic substances and high selection pressure [19]. Meanwhile, the protein content decreased

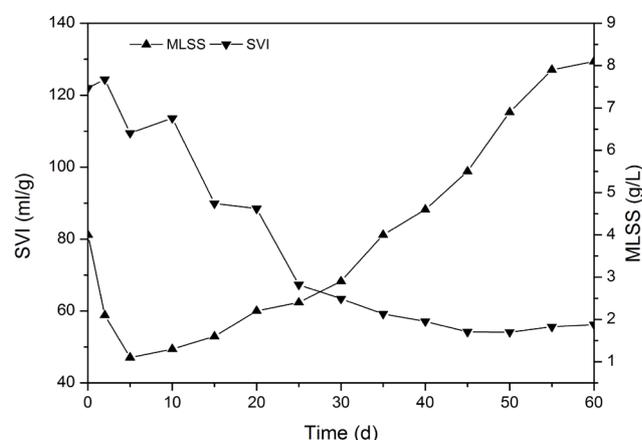


Fig. 1. Variations in MLSS and SVI during the formation of aerobic granular sludge.

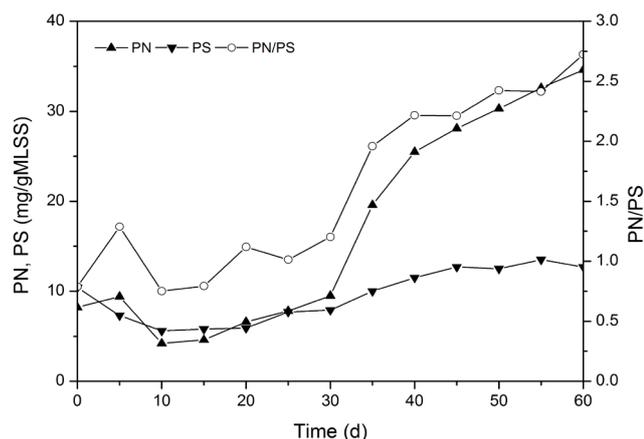


Fig. 2. Variations in EPS and their components during the formation of aerobic granular sludge.

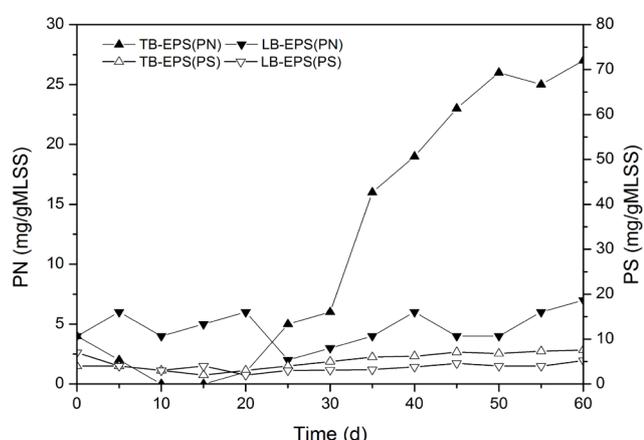


Fig. 3. Variations in LB-EPS and TB-EPS and their components during the formation of aerobic granular sludge.

in LB-EPS but developed rapidly in TB-EPS. This result can be attributed to the protein in LB-EPS that adhered to the surface of the granules and then converted to TB-EPS [19,28,34]. Thus, the protein in TB-EPS could be the major factor influencing the granulation process. In addition, the protein content in TB-EPS further increased and the granules in the bioreactor became dominant after 30 days of operation. Moreover, the PS contents in both LB-EPS and TB-EPS slightly increased. These results suggest that protein substances favor the formation of AGS and that PS substances only exert negligible effects [19,36].

As shown in Fig. 4, SMP maintained a relatively low level of 5.2–6.0 mg/g MLSS because of the toxic substances from the influent at the initial stage of SBAR operation. Then, microbial activity and contaminant removal efficiencies both gradually increased along with the granulation process, and SMP content reached a stationary value of 8.1 mg/g MLSS. In addition, the PS content was much higher than the protein content because the biosynthetic pathway of PS may be faster than that of protein molecules [18,39].

### 3.3. Fluorescence characteristics of EPS

On the basis of the classification scheme [40], the excitation-emission matrix (EEM) characterization of TB-EPS demonstrated two major peaks in the spectrum with excitation-emission (Ex/Em) wavelengths at 220–230/340–350 nm (peak A) and 270–280/340–350 nm (peak B) in Fig. 5. Peaks A and B from TB-EPS were in regions II (aromatic proteins) and regions IV (soluble microbial by-product-like), respectively [19]. With the enhancement of bioactivity and increase of SMP during AGS formation, the fluorescence intensity of Peak B gradually strengthened. Moreover, the granules possibly adapted the oily wastewater and caused considerable oil and COD removal (Fig. 6). Nevertheless, only peak A can be observed from TB-EPS after 20 d (Fig. 5b, 5c, and 5d), which implied that the aromatic protein in TB-EPS significantly affected the aerobic granulation process. The gradual increase in the fluorescence intensity of Peak A (Fig. 5b, 5c, and 5d) and the increased biomass

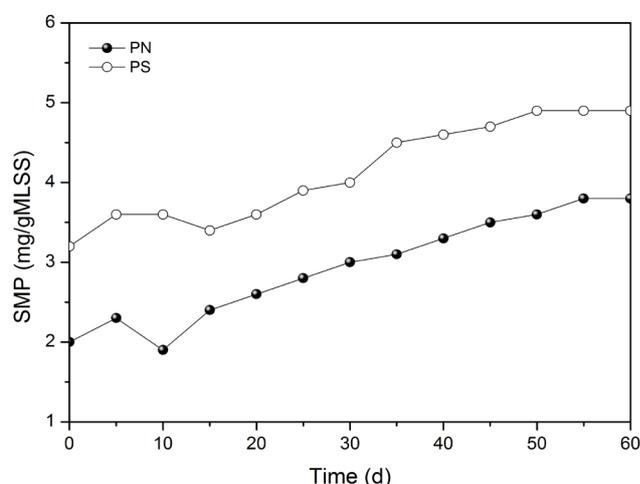


Fig. 4. Variations in SMP and their components during the formation of aerobic granular sludge.

(Fig. 1) during the formation of AGS suggest that only aromatic protein plays an important role in maintaining the stable structure of AGS [41]. Furthermore, AGS would be desirable and feasible for resource recovery or energy recycle because proteins are obviously generated and accumulated in EPS.

### 3.4. Contaminant removal performance of AGS

The oil (mainly emulsified and soluble oil) and COD removal efficiencies of the AGS sample were investigated (Fig. 6a and 6b). As shown in the figures, the initial removal amounts of oil and COD were relatively low, which could be attributed to the negative effects of refractory organics on the microorganisms from the UF effluent and to low biomass retention. Subsequently, the respective oil and COD removal efficiencies gradually increased along with the operation of the bioreactor. This result demonstrates that the microbial activity recovered due to the enhanced degradation of recalcitrant compounds and the strengthened stability of the AGS that was based on the PN/PS ratio (Fig. 2). The effluent oil and COD were 4.5 and 44.1 mg/L, respectively; at the end of the start-up phase, the oil and COD removal efficiencies were as high as 94.1% and 85.6%, respectively. These results are consistent with previous studies on biological treatment and membrane separation [1,9,13]. Consequently, the presence of emulsified and soluble oil exerted no significant effect on contaminant removal efficiencies possibly because of biomass acclimation [18]. In addition, the stable EPS contents could bind with positively charged organic pollutants within the oily wastewater through electrostatic interaction [34,42] and enhance the contaminant removal accordingly.

The performance of oily wastewater by membrane filtration has been widely investigated. Although high contaminant removal could be achieved, more attention should be paid to membrane preparation and membrane fouling. Therefore, a combined physical/chemical-biological technology can be cost effective and environmentally friendly [43]. Furthermore, the use of membrane filtration in oily wastewater treatment is not desirable because of the high energy consumption [44]. The UF-AGS technology studied in this work or even the combined heat and power generation (CHP) that relied on biogas production [45] from the anaerobic digestion of AGS may reduce operation consumption and maintain a considerable contaminant removal capacity.

### 3.5. Sludge incineration potential

Sludge incineration can decrease the risk of heavy metals and produce immense heat [46,47], which can also be recycled for drying the fresh sludge. Therefore, a separate sludge incineration process could be achieved subsequently. As presented in Table 1, the heating value of GS2 was higher than that of GS1 by a factor of 1.3 when quick lime was selected as the conditioner, indicating that GS2 was more suitable for sludge incineration than GS1. To accomplish separate sludge incineration (required heating value: 5 MJ/kg, according to the national standard on the disposal of sludge from municipal wastewater treatment

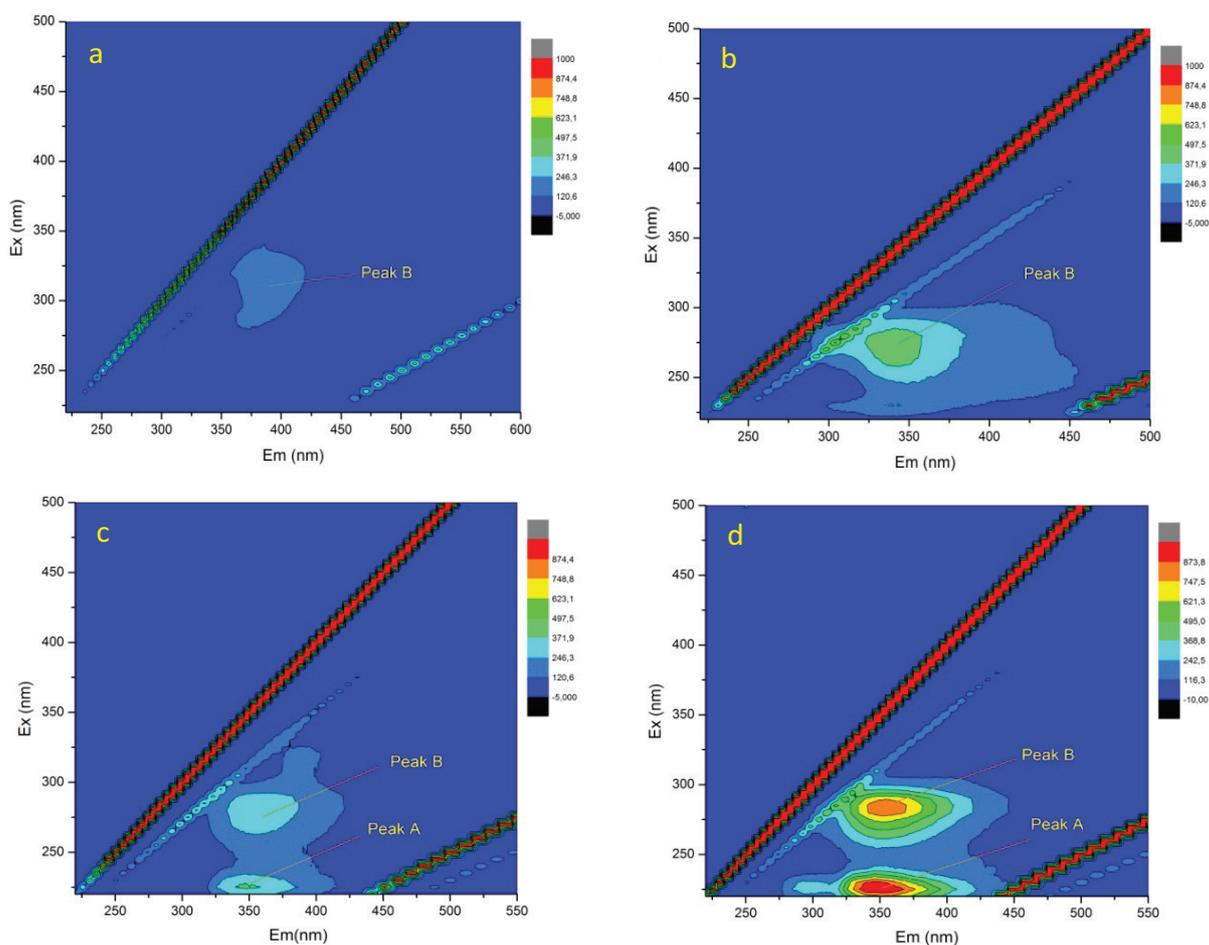


Fig. 5. Excitation–emission matrix (EEM) spectra for extracted TB-EPS from aerobic granular sludge (TOC < 1 mg/L). (a: 0 day; b: 20 days; c: 40 days; d: 60 days).

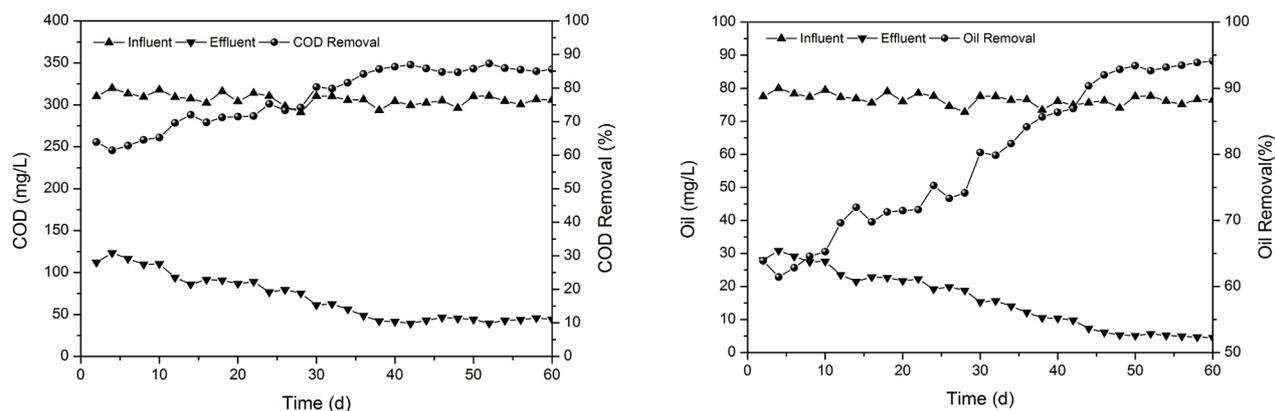


Fig. 6. COD and oil removal efficiencies in oily wastewater treatment.

plant-quality of sludge used in separate incineration), only 3.3 kg of quick lime was needed for GS2 in sludge conditioning, whereas at least 4.9 kg was required for GS1. Thus, the oily AGS was appropriate for incineration.

The elemental analysis and heating value by the two types of AGS were also investigated (Table 2). The carbon percentage of GS2 was higher than that of GS1 because

more organic compounds were accumulated in GS2 from oily wastewater [18,24] and such sludge was inclined to be incinerated. Although the HHVs of GS1 and GS2 were almost the same according to Eq. (1), GS2 was found more suitable for incineration [48] because its LHV of 8.5 MJ/kg was 1.8 times higher than that of GS1 (4.7 MJ/kg) as calculated by Eq. (2). This result indicates that the surplus heat

Table 1  
Characteristics of different aerobic granular sludge after conditioning and dewatering

	Heating value <sup>a</sup> (MJ/kg)	Moisture content <sup>a</sup> (%)	Quick lime dosage <sup>b</sup> (Kg)
GS1 <sup>c</sup>	1.95	74	4.9
GS2 <sup>c</sup>	2.60	72	3.3

a: Quick lime dosage: 20% of sludge dry solids

b: Quick lime dosage: required LHV of 5 MJ/kg

c: Original sludge dry solids: 12 kg

Table 2  
Elements analysis and heating values of different aerobic granular sludge

	C (%)	H (%)	N (%)	Ash (%)	HHV (MJ/kg)	LHV (MJ/kg)
GS1	38.2	5.2	6.8	50.6	18.3	4.7
GS2	42.5	5.5	8.2	49.6	20.4	8.5

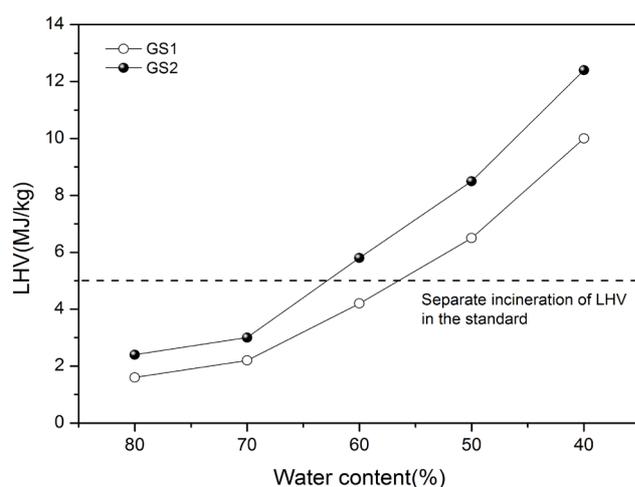


Fig. 7. LHV of aerobic granular sludge under different water contents.

can possibly be stored and reused [25,48]. As illustrated in Fig. 7, the LHV of AGS exceptionally increased along with the decline in water content. The water content of GS2 reached 64.0%, but the relative LHV can still be higher than 5 MJ/kg. However, the water content of GS1 should be below 56.2%, implying that the operation cost of sludge treatment and disposal was lower because the addition of chemical conditioners reduced during sludge dewatering [24]. Moreover, the LHV of GS2 further increased to about 6.9 MJ/kg (Fig. 7) when the water content was decreased to 56.2%. This result means that less GS2 sludge could be obtained, which might enhance the treatment efficiency of the sludge incinerator [49] and increase the amount of heat energy that can be recycled.

#### 4. Conclusion

AGS fed with oily wastewater could be gradually obtained using high biomass retention and good settling property. The presence of emulsified and soluble oil exerted no significant effect on contaminant removal, and the respective oil and COD removal efficiencies were as high as 94.1% and 85.6%, respectively. Relatively high total protein content could enhance and promote the formation of aerobic granules, and the protein in LB-EPS was converted to TB-EPS from the fluorescence spectrogram; hence, the protein in TB-EPS could be the major factor affecting the granulation process. Granules cultivated by oily wastewater were found more suitable for incineration and, thus, surplus heat can possibly be stored and be reused. In full consideration of its characteristics, performance, and sludge treatment potential, AGS might be desirable and feasible for practical application, resource recovery, or energy recycling in oily wastewater treatment.

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