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RESEARCH ARTICLE

Acidogenic sludge fermentation to recover soluble organics as the carbon source for denitrification in wastewater treatment: Comparison of sludge types

Lin Lin[1], **Ying-yu Li**[1], **Xiao-yan Li** (✉)[1,2]

1 Environmental Engineering Research Centre, Department of Civil Engineering, The University of Hong Kong, Pokfulam, Hong Kong, China
2 Shenzhen Engineering Research Laboratory for Sludge and Food Waste Treatment and Resource Recovery, Graduate School at Shenzhen, Tsinghua University, Shenzhen 518055, China*

HIGHLIGHTS

- CEPS sludge was compared with conventional primary and secondary sludge for the VFAs yield.
- Fe-based CEPS sludge exhibited the highest efficiency of organic recovery.
- Fermented CEPS sludge liquor provided a sufficient carbon source for denitrification.
- 99% of nitrate removal was achieved based on the Fe-CEPS and sludge fermentation.

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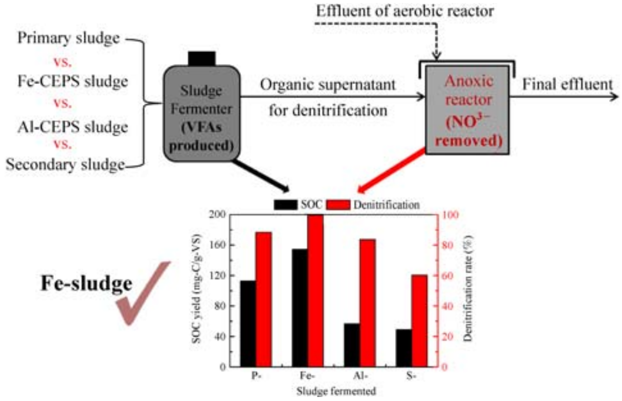
GRAPHIC ABSTRACT



ABSTRACT

For biological nitrogen (N) removal from wastewater, a sufficient organic carbon source is requested for denitrification. However, the organic carbon/nitrogen ratio in municipal wastewater is becoming lower in recent years, which increases the demand for the addition of external organic carbon, e.g. methanol, in wastewater treatment. The volatile fatty acids (VFAs) produced by acidogenic fermentation of sewage sludge can be an attractive alternative for methanol. Chemically enhanced primary sedimentation (CEPS) is an effective process that applies chemical coagulants to enhance the removal of organic pollutants and phosphorus from wastewater by sedimentation. In terms of the chemical and biological characteristics, the CEPS sludge is considerably different from the conventional primary and secondary sludge. In the present study, $FeCl_3$ and PACl (polyaluminum chloride) were used as the coagulants for CEPS treatment of raw sewage. The derived CEPS sludge (Fe-sludge and Al-sludge) was then processed with mesophilic acidogenic fermentation to hydrolyse the solid organics and produce VFAs for organic carbon recovery, and the sludge acidogenesis efficiency was compared with that of the conventional primary sludge and secondary sludge. The results showed that the Fe-sludge exhibited the highest hydrolysis and acidogenesis efficiency, while the Al-sludge and secondary sludge had lower hydrolysis efficiency than that of primary sludge. Utilizing the Fe-sludge fermentation liquid as the carbon source for denitrification, more than 99% of nitrate removal was achieved in the main-stream wastewater treatment without any external carbon addition, instead of 35% obtained from the conventional process of primary sedimentation followed by the oxic/anoxic (O/A) treatment.

1 Introduction

With the fast population growth, urbanization and industrialization across the world, the conventional wastewater treatment technologies are no longer capable of

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✉ Corresponding author
E-mail: xlia@hku.hk

*Special Issue—Bio-based Technologies for Resource Recovery (Responsible Editors: Aijie Wang & David Stuckey) accommodating the increasing wastewater flow and the pollutant load. Particularly, compared with the degradation of organic pollutants, removal of nutrients (nitrogen and phosphorous) is more difficult and costly in wastewater treatment. The situation becomes more challenging in recent years due to the decreasing organic carbon/nutrient ratio in municipal wastewater (Metcalf & Eddy, Inc. 2003). Owing to the lacking of the organic carbon for sufficient N removal, methanol or similar organics are often added to wastewater to provide the external carbon source for biological denitrification, which increase both the cost and fire risk for wastewater treatment facilities (de Barbadillo et al., 2008; Latker et al., 2011).

Meanwhile, the widespread wastewater treatment practice produces an increasing amount of sewage sludge. It is estimated that about 10 million dry tons of sewage sludge are produced every year in the United States (Bandosz and Block, 2006). The high cost of sludge treatment and the environmental risk of sludge disposal give rise to the growing concerns of the human society, especially in large cities (Wei et al., 2003). On the other hand, the organic matter concentrated in the sewage sludge is a valuable carbon source, which should be recovered instead of being wasted with the sludge. Acidogenic fermentation can be an effective method to treat sewage sludge, converting solid organics in sludge into volatile fatty acids (VFAs). Such a sludge treatment not only reduces the amount of sewage sludge but also provides the much needed carbon source for denitrification (Elefsiniotis et al., 2004). However, it is reported that the VFAs conversion of secondary sludge is generally below 10% and cannot meet the denitrification requirement, as the secondary sludge consists predominantly of microbial cells that can be hardly ruptured and degraded (Mao et al., 2004;Burgess and Pletschke, 2008).

Chemically enhanced primary sedimentation (CEPS) is an effective process that applies chemical coagulants to enhance the removal of organic pollutants and phosphorus from wastewater by sedimentation (Wang et al., 2009). CPES can lead to a shift of over 30% of the organic carbon flow from the downstream biological treatment process to the primary sludge, which will not only reduce the pollutant load on the secondary treatment, but also increase the potential of resource recovery from the primary sludge. Currently, CEPS sludge from Stonecutters Island Sewage Treatment Works amounts to around 80% of the total sewage sludge in Hong Kong (EPD, 2016). Such a significant increase of the organic content in CEPS sludge would make sludge fermentation for VFAs technically attractive and economically viable.

Until now, the acidogenic fermentation potential and behavior of CEPS sludge is still unclear, and utilization of the produced VFAs from CEPS sludge for denitrification has not been reported. In the present experimental study, $FeCl_3$ and PACl (polyaluminum chloride) were used as the coagulants for CEPS treatment of raw sewage. The derived CEPS sludge (Fe-sludge and Al-sludge) was then processed with acidogenic fermentation to hydrolyze the organics and produce VFAs for organic carbon recovery, and the results were compared with that from the conventional primary sludge and secondary sludge. In addition, the VFAs-rich supernatants from different sludge were tested for their effectiveness as the supplementary carbon source for biological denitrification.

2 Materials and methods

2.1 Experimental set-up

2.1.1 Sludge preparation

Raw sewage was taken from the Stanley Sewage Treatment Works (SSTW) in Hong Kong. The average sewage quality during the experimental period were as follows: pH 6.82±0.05, suspended solids (SS) 380±25 mg/L, total chemical oxygen demand (TCOD) 415±54 mg/L, soluble chemical oxygen demand (SCOD) 152±10 mg/L, total organic carbon (TOC) 143.5±8.9 mg/L, orthophosphate-phosphorus (PO_4-P) 4.0±0.2 mg/L, and ammonia-nitrogen (NH_4-N) 21.5±0.5 mg/L. The optimum dosages of $FeCl_3$ and PACl for CEPS treatment of fresh wastewater were determined by the laboratory jar-test procedures, to achieve over 70% and 90% removals of TOC and PO_4, respectively. The coagulant was then dosed at the selected dosage (20 mg-Fe/L of $FeCl_3$ or 16 mg-Al/L of PACl) into 60 L of raw sewage in a tank without pH adjustment, followed by rapid mixing at 200 r/min for 1 min, slow stirring at 30 r/min for 15 min, and sedimentation for 1 h. The supernatant was siphoned off, and the settled sludge was retained as CEPS sludge (Fe-sludge and Al-sludge). The simple primary sludge was obtained from wastewater without coagulant addition after the same sedimentation and supernatant discharge procedure. The secondary sludge was collected from the secondary sedimentation tank in SSTW. By dilution or concentration, the total organic concentration in Fe-sludge, Al-sludge, primary and secondary sludge was adjusted to a similar level at 3200±100 mg TOC/L_{sludge}.

2.1.2 Sludge acidogenic fermentation

Glass bottles with a total volume of 550 mL were used for the sludge acidogenic fermentation, each filled with 470 mL of the CEPS sludge and 30 mL seed sludge (8000 mg/L of VS) from a mesophilic sludge fermenter in laboratory, which had been operated for six month with the semi-continuous feed of similar primary sludge. The fermentation bottles were placed in a temperature-controlled air chamber (37±1°C) with magnetic stirring for sludge mixing. At the beginning of the batch experiments, nitrogen gas was sparged to create an anaerobic condition, and pH was not regulated during the fermentation process. The batch fermentation test was conducted in duplicate reactors for 9 days, and the sludge mixture was sampled daily to monitor the VFAs production performance.

2.1.3 Denitrification batch tests

After fermentation, the sludge supernatant was collected by centrifugation at 4000 r/min for 5 min and kept at 4°C for later use. The biological N removal test was performed using the oxic/anoxic (O/A) process as shown in Fig. 1, seeded by the activated sludge taken from SSTW. The primary effluent after simple or enhanced sedimentation was first treated in the aeration tank with activated sludge (1 L) for four hours to achieve nitrification, and the effluent then went to the anoxic tank (0.5 L) for two hours, mixing with the fermented sludge supernatant (1/25 of the influent volume) for denitrification. The tests were operated in parallel for the four types of sludge.

2.2 Analytical methods

The pH of the sludge mixture was measured using a pH meter (Starter 3100, Ohaus, USA). Samples from fermentation and O/A reactors were immediately centrifuged at 8,000 r/min for 10 min to obtain the supernatant for analysis of the soluble parameters. Determinations of the total solids (TS) and volatile solids (VS), COD, BOD, NH_4-N, nitrate (NO_3^-), total nitrogen (TN), PO_4-P and total phosphorus (TP) were in accordance with Standard Methods (APHA, 2005). The organic content in the sludge was measured using a TOC analyzer (Aurora 1030, OI Analytical, USA) for the overall TOC in the mixture and soluble organic carbon (SOC) in the supernatant. The SOC yield per unit of sludge VS was used to indicate the hydrolysis efficiency. VFAs including acetic acid (HAc), propionic acid (HPr), n-butyric acid (n-HBu), iso-butyric acid (iso-HBu), n-valeric acid (n-HVa) and iso-valeric acid (iso-HVa) were quantified by using gas chromatograph (6890A, Agilent, USA) as described previously (Lin et al. 2017a). The experimental data were expressed as the average of triplicate tests, and the analytical errors were within 10%.

3 Results and discussion

3.1 Wastewater treatment by the chemically enhanced primary sedimentation

As shown in Fig. 2, simple sedimentation by gravity only removed 28.6% of TOC and 3.8% of PO_4-P on average from the raw wastewater samples, with 102.4 mg-C/L and 3.9 mg-P/L in the primary effluent. Comparatively, Fe- or Al-based CEPS demonstrated clear superiority in organic and phosphorus removal with removal efficiencies of over 70% and 90%, respectively. After CEPS treatment, TOC and PO_4 in the effluent decreased to below 40 mg-C/L and 0.3 mg-P/L. Besides, PACl showed 9% higher removal efficiency for TOC than $FeCl_3$ This may be attributed to the Al-based polymeric form with higher molecular weights, which increased the intrinsic viscosity and bridging ability for aggregating suspended matters and organic macromolecules during the flocculation process (Wei et al., 2009). However, soluble NH_4-N was not removed by both simple and enhanced sedimentation, for which the subsequent O/A process was applied for biological N removal (Fig. 1). Due to the enhanced organic and

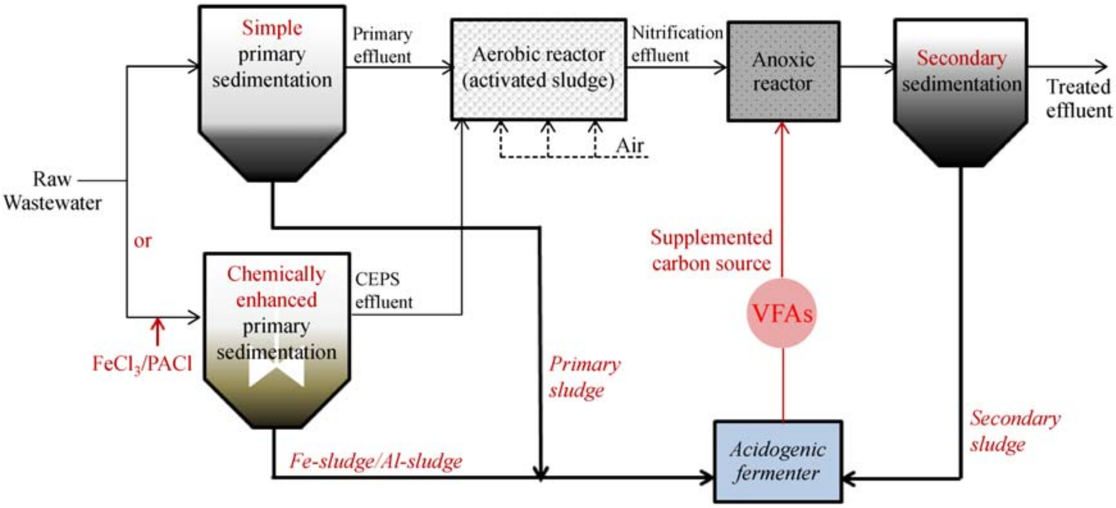

Fig. 1 Schematics of the wastewater treatment by primary sedimentation or CEPS, side-stream sludge fermentation, and the O/A process

phosphorus removals by CEPS, the pollutant load for the downstream biological treatment could be reduced by over 60%, implying half of the energy consumption for aeration can be saved. Meanwhile, instead of being oxidized biologically to greenhouse gas (CO_2), more than 50% of organic carbon in wastewater influent was concentrated in the CEPS sludge, which can be converted to valuable VFAs for beneficial utilization.

3.2 Acidogenic fermentation of the different sludge

3.2.1 Characteristics of the different sludge

Due to the enhanced pollutant removals, the CEPS sludge had a much higher VS concentration than the simple primary sludge (4.5 g/L vs. 1.9 g/L) with the same volume reduction factor (25×) after sedimentation. The concentration of secondary sludge obtained from sewage treatment works was as high as 8.7 g-VS/L. For comparison, dilution or concentration was applied to the four types of sludge to the similar VS (4.6 g/L) or TOC (3200 mg/L) levels, and the resulting characteristics of the sludge samples are summarized in Table 1. Fe-sludge demonstrated a higher BOD/COD value than simple primary sludge (0.44 vs. 0.39), probably attributed to the increased removal of biodegradable organics from wastewater by the CEPS process. However, the BOD/COD ratios of the Al-sludge and secondary sludge were significantly lower at 0.31-0.26, which might bring about difficulties to acidogenic sludge fermentation.

The rate of biotic hydrolysis of particulate organics is partially regulated by the adsorption of hydrolysing bacteria and enzymes onto the solid surface sites (Yu et al., 2008). Therefore, smaller particle sizes would increase the available specific surface area of solid organic matters for microbial cells and enzymes. The particle size distributions of different sludge samples are depicted in Fig. 3. The median particle sizes were 295.7, 61.0, 73.5 and 111.9 μm for the primary sludge, Fe-sludge, Al-sludge and secondary sludge, respectively. As the larger particles are faster to settle, around 56% of primary sludge particles ranged from 200 to 1800 μm. A high and wide peak appeared at 20–200 μm for both Fe-sludge and Al-sludge, amounting to 63%, due to the agglomeration of colloidal

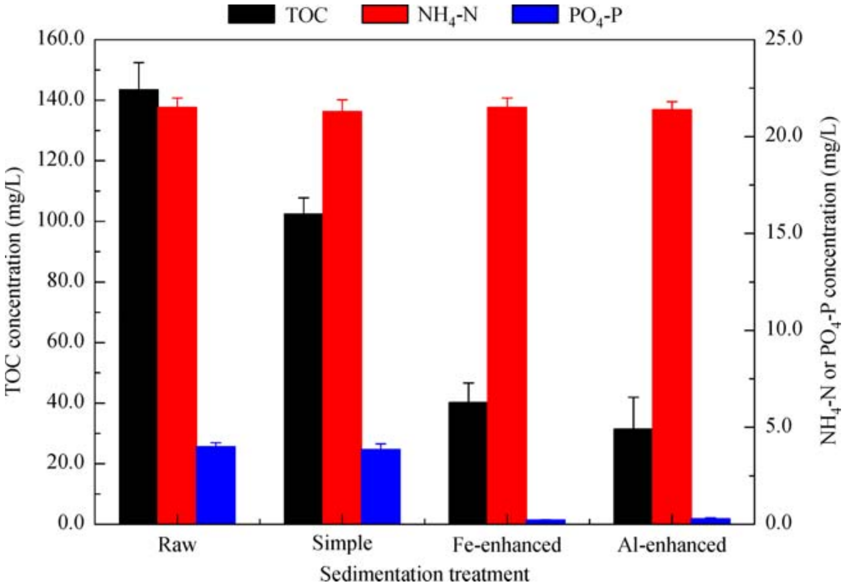

Fig. 2 The pollutant concentrations in the wastewater influent and the effluent after simple and chemically enhanced primary sedimentation

Table 1 Characteristics of the primary sludge, CEPS sludge (Fe-sludge and Al-sludge) and secondary sludge after the concentration adjustment (unit: mg/L, expect BOD/COD)

Index	TS	VS	TOC	TCOD	TBOD	BOD/COD	TN	TP
Primary sludge	4700±120	4680±100	3284±231	9154±120	3576±115	0.39±0.01	167.6±12.1	26.1±2.1
Fe-sludge	6040±230	4640±180	3256±289	9360±200	4076±220	0.44±0.03	203.7±9.5	129.3±5.0
Al-sludge	6280±150	4540±150	3167±156	8904±282	2717±185	0.31±0.02	206.2±10.2	132.6±7.2
Secondary sludge	4567±200	4619±140	3174±117	7785±155	2046±125	0.26±0.02	248.0±15.7	109.8±5.0

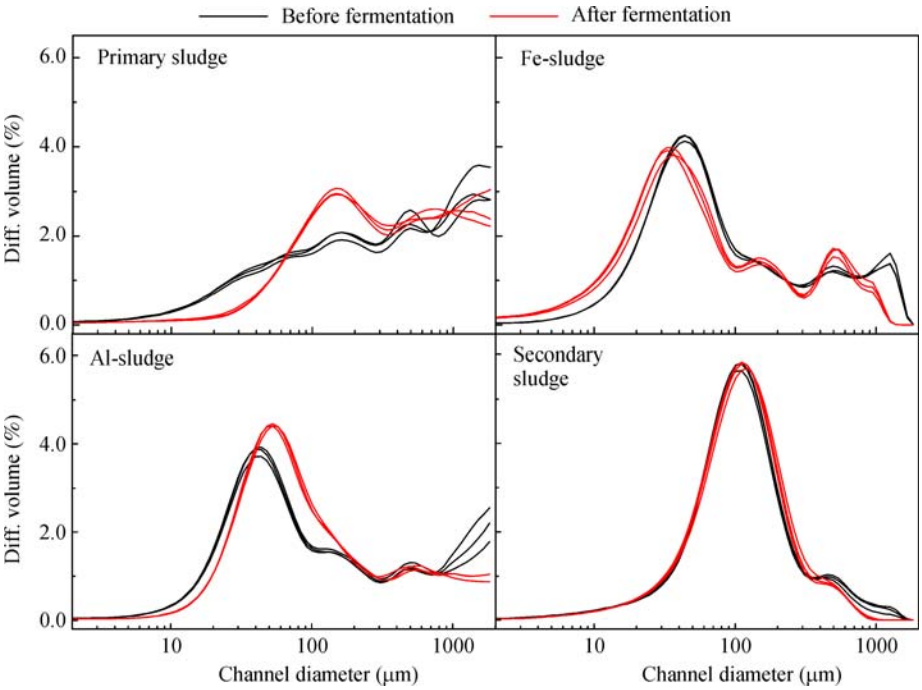

Fig. 3 The particle size distributions of different sludge samples before and after acidogenic fermentation

pollutants. Differently, the particles of secondary sludge appeared to have a more typical normal distribution, with 81% in the range of 50–500 μm.

3.2.2 Organic release and VFA production during sludge fermentation

As shown in Fig. 4, organics kept dissolving into the supernatant during the acidogenic sludge fermentation process. The Fe-sludge exhibited the highest VFA yield at 134.1 mg-C_{VFAs}/g-VS_{fed} within five days, while it took 4 days longer for the primary sludge to reach 112.4 mg-C_{VFAs}/g-VS_{fed}. Compared to the Fe-sludge, Al-sludge was about 61% lower in VFA yield at 52.0 mg-C_{VFAs}/g-VS_{fed}, owing to its apparent difficulty in hydrolysis (55.9 mg-SOC/g-VS_{fed}). It has been reported that coagulants have obvious inhibition on hydrolysis process, due to their "cage" effect on organics in sludge that reduces the organic reactivity and accessibility to enzymes and bacteria (Dentel and Gossett, 1982). Thus, a limited amount of solid organics in Al-sludge was hydrolyzed into the supernatant. In comparison, the reduction of Fe(III) to more soluble Fe (II) would occur rapidly under an anaerobic condition, which led to the dissolution of HO-Fe-P backbones in sludge flocs and the observed organic release (Lin et al., 2017b). As expected, the secondary sludge showed the lowest fermentation result at 34.0 mg-C_{VFAs}/g-VS_{fed} of VFA yield, because microbial cells are not as easily ruptured and degraded as organics in primary sludge (Mao et al., 2004). Previous study also found that the VFA yield was as low as 60 mg-COD/g-VS from the secondary sludge (Zhou et al., 2015).

Figure 3 shows that after fermentation, a new peak ranging 80-200 μm appeared in the particle size distribution of primary sludge, indicating the effective hydrolysis of large particulate organics. However, the secondary sludge and Al-sludge did not demonstrate noticeable reduction of large particles, suggesting their hindered hydrolysis. The VFA/SOC ratio is a measure for the degree of successful acid formation during fermentation, representing the amount of solubilized matter being converted to VFAs (Ucisik and Henze, 2008). The VFA/SOC of Al-sludge and secondary sludge was above 95%, indicating that the acidogenic activity was not hindered. Thus, hydrolysis rather than acidogenesis was the rate-limiting step for fermentation of these sludge samples. The VFAs species of the four sludge showed that HAc and HPr amounted to 40–50% and 20–30% of TVFAs, respectively. It is apparent that the fermented supernatant would provide an appropriate carbon source for the denitrification use (Elefsiniotis et al., 2004).

3.2.3 Nutrients release from different sludge

Although VFAs in the sludge supernatant can be used as the carbon source for biological nitrogen removal (BNR) enhancement, a considerable amount of soluble phosphorus and ammonia would also be released during sludge fermentation, which might deteriorate the effluent quality of the treated wastewater. Figure 5 shows the release of phosphate and ammonia after fermentation of the different sludge. The level of soluble NH_4-N in supernatant was

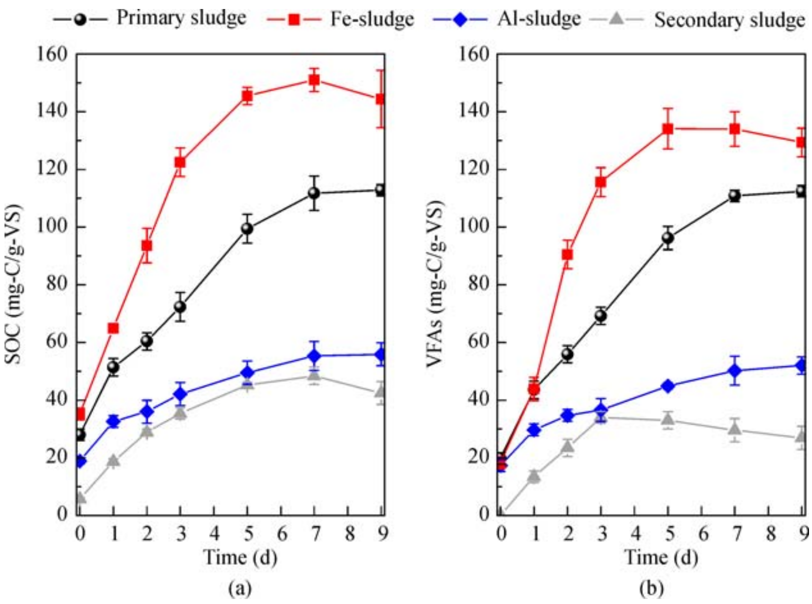

Fig. 4 Performance in (a) hydrolysis and (b) acidogenesis of the different sludge during fermentation

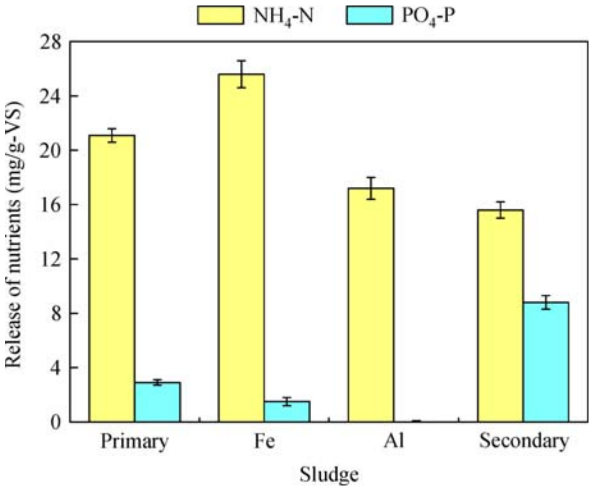

Fig. 5 Release of nutrients from the different sludge after acidogenic fermentation.

positively related to VFAs production, in the order of Fe-sludge (118.8 mg/L)>primary sludge (99.2 mg/L)>Al-sludge (78.0 mg/L)>secondary sludge (72 mg/L). The C_{VFAs}/N_{NH_4} mass ratio can be used to estimate the denitrification potential of the fermented sludge supernatant (Soares et al. 2010), and the C/N ratio should exceed 1 for complete denitrification in practice when using VFAs as the carbon source (Elefsiniotis and Li 2006). The fermentation results showed that the C_{VFAs}/N_{NH_4} ratio was 5.3, 5.2, 3.0 and 2.2 for the primary sludge, Fe-sludge, Al-sludge and secondary sludge, respectively. Thus, the fermented liquor from the primary sludge and Fe-sludge would provide a sufficient amount of carbon source in VFAs for wastewater denitrification.

Around 1.5–2.9 mg-P/g-VS was released from the primary and Fe-sludge, while up to 8.8 mg-P/g-VS was released from the secondary sludge. Similarly, Chen et al. (2007) also found the fermentation of secondary sludge resulted in a significant increase of soluble phosphorus at 8 mg-P/g-VS. Though the VFAs yield of Al-sludge was low, no phosphorus release in the supernatant was detected after fermentation due to the strong bounding between Al and PO_4 in the flocs (Lin et al., 2017b). Soluble phosphorus in the fermented supernatant which is to be returned to the mainstream wastewater flow (Fig. 1) will increase the TP load for the wastewater treatment process. Therefore, Al-sludge fermentation showed an advantage over Fe-sludge in terms of phosphorus retention in the sludge.

3.3 Nitrogen removal performance

For comparison with the process shown in Fig. 1, the control tests were conducted without addition of the fermented sludge liquor. Results in Fig. 6 showed that only 35% of nitrate was removed by simple sedimentation and the biological O/A process. The denitrification efficiency increased to 51%–57% when simple sedimentation was replaced by Fe- or Al-based CEPS, with 12.3 mg-N/L of nitrate in the secondary effluent. However, by adding the fermented sludge supernatant into wastewater at a ratio of 1:25, biological denitrification was greatly improved, leading to a significant increase in nitrate removal, especially for the Fe-sludge case. For example, the denitrification efficiency increased to 88.5%, 99.8%, 83.9% and 60.1% for the primary sludge, Fe-sludge, Al-sludge and secondary sludge cases, respectively. Although around 4.5 mg/L of NH_4-N remained in the secondary effluent for the Fe-based treatment, it can be polished by

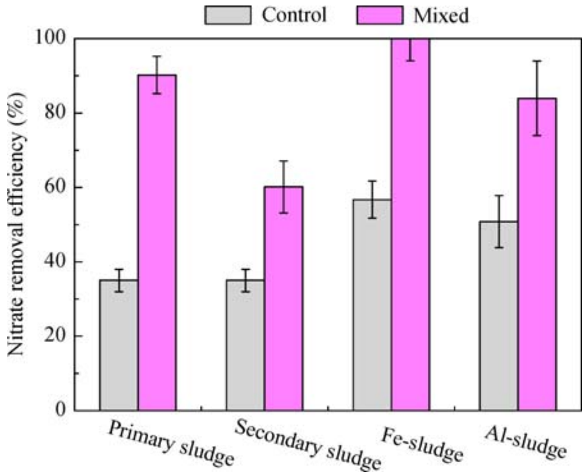

Fig. 6 The nitrate removal efficiency in wastewater treatment without (control) and with (mixed) the addition of the fermented sludge supernatant

further aeration operation. The primary sludge had a similar specific VFAs yield from the sludge, but its organic recovery efficiency from the wastewater influent was hardly significant, owing to its much lower organic removal efficiency than CEPS (Andreasen et al., 1997). On the contrary, 75%–80% of organic in raw sewage can be concentrated by CEPS into sludge for VFAs production. Therefore, the Fe-based CEPS and side-stream sludge fermentation can help greatly improve the energy-efficiency and nutrient removal performance of municipal wastewater treatment.

4 Conclusions

Chemically enhanced primary sedimentation (CEPS) improved the TOC and PO_4-P removal to 70% and 90% from wastewater, which reduced the pollutant load for biological treatment. Acidogenic fermentation was effective to convert solid organics in sludge to VFAs, providing the organic carbon source for denitrification. Compared with the conventional primary sludge and secondary sludge, Fe-sludge exhibited the highest hydrolysis and acidogenesis efficiency, while the hydrolysis efficiency was lower for the Al-sludge. Utilizing the fermented Fe-sludge liquor to provide the carbon source, denitrification could be achieved at an efficiency of over 99%, instead of 35% for the simple primary sedimentation followed by the O/A process without the addition of fermented sludge liquor. This innovative wastewater treatment process is of energy-saving and higher pollutant removal efficiency, while it effectively recovers organic resources from the sewage sludge for beneficial utilization.

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