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Simultaneous recovery of ammonium and phosphorus via the integration of electro dialysis with struvite reactor

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ABSTRACT

To eliminate the waste to discharge or backflow to the headstream of the wastewater treatment plant, recovering ammonia and phosphate (N&P) salts from Sidestreams of anaerobic digesting excess sludge was investigated. Electrodialysis (ED) technology was used mainly for desalting and concentrating wastewater. An integration of struvite reactor and ammonia stripping was established to recover N&P from the concentrated wastewater. During single ED experiments, removal ratio of ammonia and phosphate salts were in the ranges of 95.8–100% and 86.1–94.4%, respectively. During the integration of ED and struvite reactor, the concentrated N&P salts were effectively used to form the struvite while the desalination ratio was kept at a high level. After the generation of struvite, a small amount of ammonia salt was still remained in the concentrated N&P solution. The introduction of gas stripping could result in an effective extraction and recovery of ammonia. X-ray diffraction (XRD) analysis and scanning electron microscopy (SEM) were conducted to characterize the obtained precipitates. The diffractograms peaks of the precipitates were well correlated with the struvite pattern, confirming the formation of struvite from the wastes.

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1. Introduction

Eutrophication is a slow natural ageing process which has been greatly accelerated by human intervention in the natural biogeochemical cycling of nutrients [1], and now has become one of the serious ecological and environmental problems. Nowadays, eutrophication phenomenon is mainly ascribed to the increased nitrogen and phosphorus (N&P) input, specifically, the increasing discharge of plant nutrients (mainly N&P) from industrialization, agricultural modernization, and urbanization [2]. So some sewage treatment plants have been constructed at the right moment, in order to take away these nutrients from wastewater as a rescue measure before its discharging into the surface water. Activated sludge process is a common, effective and preferred method employed to treat wastewater containing N&P, where the biodegradable organics can be used as the growing substance for organisms. However, excess sludge, as a secondary solid waste, is one of the most serious challenges in this process, and it must be disposed of in a safe and cost-effective way [3,4].

The N&P resources in wastewater and surface water are increasing, while the reservation resources of them are being depleted, especially for phosphorus. So, it is very necessary to remove and recover them from wastewater or surface water. After making a general survey of activated sludge process, interests of recovering nitrogen and phosphorus focus on the excess sludge developed based on some additional reforming operation and newly introduced technologies [5,6]. Anaerobic digestion process can achieve the degradation of organic matters and the formation of biogas, and it has been widely used to treat the excess sludge. Sidestreams, or “return liquors”, typically have a very high concentration of ammonia and phosphorus because they are released in the anaerobic digestion process: (i) Ammonia is produced by the biological degradation of the nitrogenous matter, such as bacteria, proteins and urea [7,8]; (ii) Phosphorus is produced by re-solution of the part incorporated into bacterial biomass in the enhanced biological phosphorus removal (EBPR) process [7]. It is unwise to return the solution rich in ammonia and phosphorus back to the sewage treatment plant, because the containing nutrients will be aggregated in the newly produced excess sludge rather than through decontamination.

In order to recover and reuse the nutrients in the solution, struvite formation–precipitation is considered as a promising technology. Struvite (*Magnesium ammonium phosphate*) was firstly

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prepared by de Fourcroy in 1789 and its use as a fertilizer was firstly suggested in 1857 by Murray [9]. Struvite production has deserved lots of efforts, from diverse waste streams rich in ammonia and/or phosphorus, investigations on the forming conditions and even pilot productions have been conducted [7,10–13]. Among these studies, Sidestreams are a preferred choice to conduct struvite production as the source solution. Struvite reactor is generally operated under continuous influent and effluent streams, so the unutilized ammonia and phosphorus will exist in the effluent all along. Direct discharge will bring pressure on the subsequent sewage treatment and result in a low utilization ratio. So there need some improvements on the struvite production and/or reuse the effluent ammonia and phosphorus. As pointed out by Zhang et al. [14], improvements on the utilization ratio need concentrate the phosphate in the effluent and recycle back into the struvite reactor.

Electrodialysis technology has some characteristic advantages in concentration process over the conventional chemical engineering processes, such as adsorption and pressure driven membrane filtration. Electrodialysis (ED), which is similar to a process of dialysis but that the ions are accelerated by an applied electric field, can realize recovering ionic species from aqueous solutions without chemical consumption or waste generation [15]. A novel selectrodialysis was used to improve the availability of the phosphate in the effluent of struvite reactor [14]. Also, electrodialysis and membrane electrolysis were coupled to regenerate the nitric acid and ammonia from the wastewater containing ammonia nitrate, solving the problem posed by a salt laden effluent [16]. These feasible practical investigations on single component had been proven to be effective, but there also need some systematic comprehensive studies on a system containing two or more components.

The main interests of this study focused on the simultaneous recovery of N&P salts from Sidestreams of anaerobic digestion by coupling ED and struvite reactor. ED was mainly used to obtain a concentrated N&P salts solution. The concentrated solution was fed into the struvite reactor to precipitate N&P salts as struvite by recycling. Gas stripping was introduced for further recovering the excess part of ammonia in the solution.

2. Methods and materials

2.1. Apparatus and operation of ED and struvite reactor

A laboratory-scale electrodialysis setup was used in this study, and it was composed of electrode compartments, concentrated compartments and diluted compartments. Compartments were separated by cation/anion exchange membranes, silicone gaskets and plastic partition nets. In addition, the specific parameters are

summarized in Table 1. The membranes used in Electrodialysis included cation exchange membranes (JCM-II-07) and anion exchange membrane (JAM-II-07). The membranes were commercially obtained from a Chinese company and their main properties are listed in Table 2.

In this study, three operating modes were conducted and their operation specifications were related as follows:

Part 1: Individual ED operation. Fig. 1(a) shows the flowchart of individual ED operation. The diluted tank was operated in a continuous operating mode, while the concentrated one was only circulated between membrane stack and the concentrated tank. The initial volume of solution in diluted tank is 2.0 L, and that of concentrated tank is 0.4 L. At the beginning of 20 min, ED was operated without feeding and bleeding. After 20 min, feed was pumped into diluted tank continuously by peristaltic pump, and here three different feed flow rates (20, 30 and 40 mL/min) were investigated. Experiments were conducted under a constant operating voltage of 62.0 V. The current variation range is 0–2.0 A, and when the practical value was beyond this range ability, the voltage would have a diminution. During the experiments, the solution volume in diluted tank was kept constant by continuous discharging overflowing. The experiment under each condition lasted for no less than 3 hydraulic retention times (HRTs) for diluted tank, in order to investigate the operational stability of this continuous process.

Part 2: Integration of ED and struvite reactor. Fig. 1(b) shows the flowchart of the integration. The ED operation was the same as Part 1. After the individual ED operating for 20 min, both the pump 5-1# and the struvite reactor began to run. To be specific, pump 5-2# began to circulate solution between concentrated tank and struvite reactor, and the flow rate was controlled to be 10 mL/min. A self-made lab scale struvite reactor (12) was used to precipitate struvite, as shown in Fig. 1. The working volume of the struvite reactor is 2 L and a slowly stirring was made by mechanical stirrer. Clear liquid flows back to the concentrated tank from side settling tube by overflowing (marked with ★ in Fig. 1(b)). This struvite reactor was also equipped with a pH controlling system (15 in Fig. 1(b)) and a peristaltic pump (5-3#) to regulate the feeding rate of magnesium chloride. The pH in the struvite reactor

Table 2
The main characteristics of membranes used in the experiments.

Item	Thickness (mm)	IEC (meq g ⁻¹)	Area resistance (Ω cm ²)	Water content (%)	Transport number (%)
JCM-II-07 ^a	0.16–0.23	1.8–2.2	2.0–5.0	33–40	95–99
JAM-II-07 ^b	0.16–0.23	1.8–2.0	5.0–9.0	24–28	90–95

^a Cation exchange membrane.

^b Anion exchange membrane.

Table 1
Parameters of the electrodialysis.

Items	Parameters
DC power supply	N5772A, Agilent Technologies, Co., Ltd.
Tanks	Breakers of various capacities
Submersible pumps	AP1000, Guangdong Zhenhua Electrical Appliance Co., Ltd., China
Peristaltic pump	Baoding Longer Precision Pump Co., Ltd., China
Configuration	⊕ CACA (CA) _n . . . ⊖ [*]
Units	11
Gaskets	0.3 mm (Plastic)
Partition nets	1.0 mm (Silicone)
Electrode material	Titanium coated with ruthenium.
Electrode rinsing solution	Na ₂ SO ₄ (500 mL, 0.3 mol/L) ^b
Effective membrane area	80 cm ²

^a C—cation exchange membrane; A—anion exchange membrane.

^b The two electrode chambers were operated in a series.

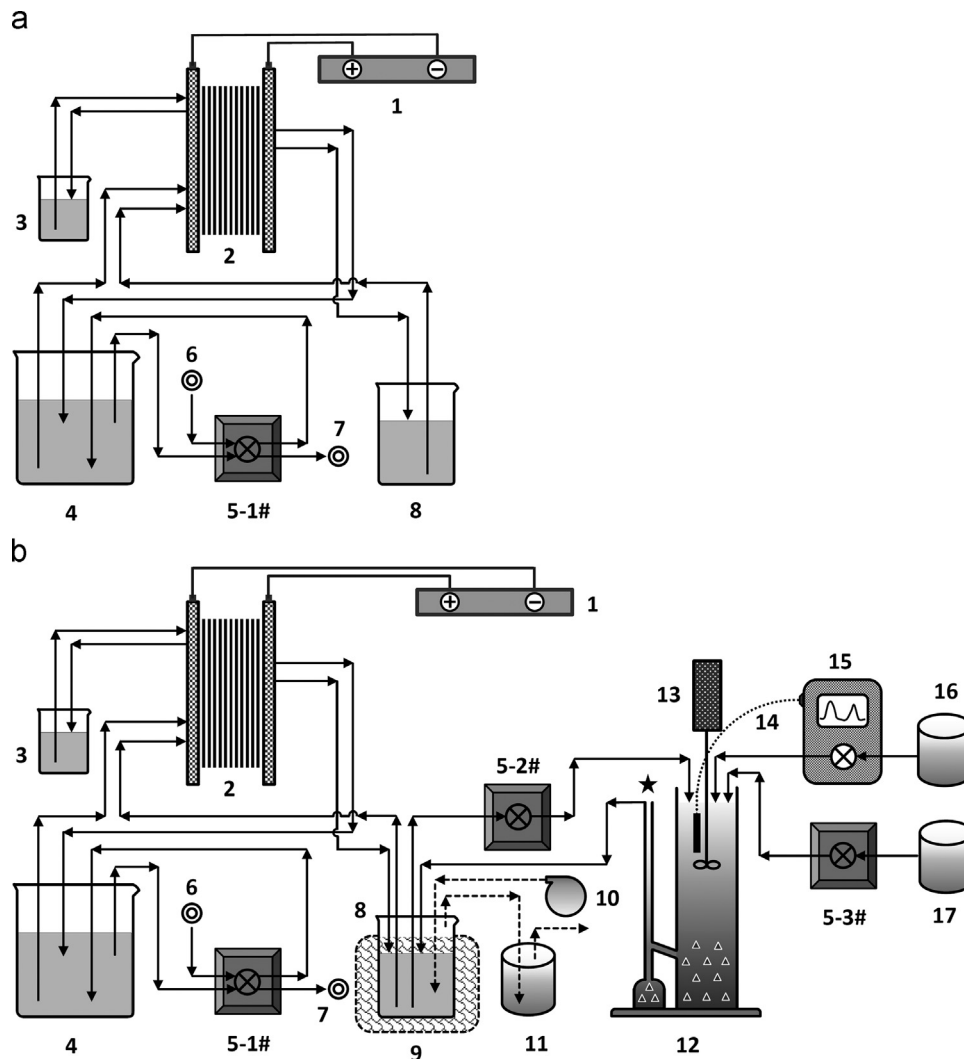


Fig. 1. (a) Flowchart of the individual ED operation. (1) Direct current power supply. (2) ED stack. (3) Electrode chamber (the anode and cathode chambers were operated in series). (4) Diluted solution tank (*Feed tank*). (5) Peristaltic pump. (6) Feeding point of Sidestreams solution. (7) Discharging point. (8) Concentrated solution tank. (b) Flowchart of the integrated operation of electro dialysis and struvite reactor. The apparatus of (1)–(8) are same with that in (a). (9) Jacket heating. (10) Blower. (11) Nitric acid solution. (12) Struvite reactor. (13) Mechanical stirrer. (14) pH electrode. (15) Control host. (16) NaOH solution of 10 M. (17) Solution of magnesium chloride. ★, Side settling tube.

was monitored by a pH electrode (InPro 3030/225, Mettler-Toledo, Switzerland) and maintained at 9.0 ± 0.1 through a one-way controller which filled with 10 mol/L NaOH (16 in Fig. 1) automatically. Magnesium chloride of two concentrations (0.5 and 1.0 mol/L) were used, the flow rate of pump (5-3#) was constant at 200 $\mu\text{L}/\text{min}$. This integrated operation lasted for no less than 3 HRTs for struvite reactor in order to investigate the operational stability. In this part, some apparatus (9, 10 and 11 in Fig. 1(b)) were not involved.

Part 3: Integration of ED, struvite reactor and gas stripping of ammonia. In this part, on the base of the operation described in Part 2, gas stripping for recovering ammonia from concentrated tank was added. It also began to work from the time point, 20 min referred in Part 1 and Part 2. Jacket heating, blower and nitric acid solution (9, 10 and 11 shown in Fig. 1(b)) were equipped. Gas flow was about 100 L/h and the temperature was set at 40 °C. Nitric acid solution here was only used for fixation and quantitative determination of the ammonia.

2.2. Wastewater preparation and analysis

Sidestreams of anaerobic digestion were synthetically simulated according to literature 7 and it contained 200 mg P/L (sum of PO_4^{3-} ,

HPO_4^{2-} and H_2PO_4^-) and 600 mg N/L (NH_4^+ -N) approximately. It was prepared by sodium phosphate tribasic dodecahydrate ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$), disodium hydrogen phosphate dodecahydrate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$), sodium dihydrogen phosphate dehydrate ($\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$) and ammonia chloride (NH_4Cl). Each of the three salts accounted for one-third of the P-concentration through calculating. All the chemicals used in this study were of analytical reagent grade, and deionized water was used throughout. The simulated solution feed into the diluted and concentrated tanks of ED as the initial solution.

Samples were collected with a predetermined time interval and analyzed for the ammonium salt, phosphate and magnesium ion. The ammonium salts and phosphates were measured in accordance with the APHA standard methods. Magnesium ion was determined by Optical Emission Spectrometer (Perkin Elmer, USA).

The removal ratio (R_i) of the salts from the feeding solution was calculated as follows:

$$R_i = \frac{C_{\text{Feed},i} - \bar{C}_{\text{Effluent},i}}{C_{\text{Feed},i}} \times 100\%, \quad i = \text{ammonia salt or phosphate}$$

where $C_{\text{Feed},i}$ is the component i concentration in feed, $\bar{C}_{\text{Effluent},i}$ is the average concentration for component i in the effluent from diluted tank.

2.3. Characterization and analyses of the precipitations

The precipitates from struvite reactor were drying for 48 h at 40 °C. X-ray diffraction was performed for crystal characterization of the precipitates. Images analyses were taken under a scanning electron microscopy (XT30 ES-TMP PHILIP).

3. Results and discussions

3.1. Effect of feed flow rate on the ED performance

Fig. 2(a) and (b) shows the variation of N&P salts concentrations in diluted and concentrated tanks. Overlapping changing trends during the initial 20 min appear for both the two salts. The reason is that the initial 20 min is a static ED process without feeding and bleeding as described in Section 2.1 (Part 1). And then, solution rich in N&P salts was pumped into the diluted tank continuously under different feed flow rates, which will result in the different variations. A steady state could be obtained for the two salts in diluted tank, and the higher the flow rate is, the higher the effluent concentration will be. There is no difficulty to understand, with the increase of higher flow rate, the amounts of salts pumped into the ED unit went beyond the ED processing capacity, and the mean retention time of the newly pumped solution was shortened.

Fig. 2(c) shows the variations of current and voltage across ED stack. An obvious change begins from the point 20 min, and the higher feed flow rate resulted in a higher current. We can know that, from Fig. 2(a) and (b), the ammonia salt or phosphate concentrations both in diluted and concentrated tank appeared

higher under a higher feed flow rate. This higher concentration could decrease the ED membrane stack resistance, and also an increase in the current was found as shown in Fig. 2(c). The ED processing capacity has a lot to do with the current which provides the direct driven force. So a more concentrated solution will be obtained at a higher flow rate, and in return, it causes the increasing current. In a word, this is a process of mutual promotion.

During the ED experiments, results of the solutions volume in concentrated tank and removal ratios of N&P salts are shown in Fig. 2(d). Compared with the initial volume, 0.4 L, there was a multi-fold increasing. This could be the main reason for the subdued increasing of N&P salts in concentrated tank, and even they went into a steady state in the end as shown in Fig. 2(a) and (b). However, the amounts of salts were enriched into the concentrated tank still had an increasing tendency as the feeding rate increased. Removal ratios of the two salts were calculated, and the removal ratios of ammonia salt and phosphate were in the range of 95.8–100% and 86.1–94.4%, respectively. As introduced in Section 2.2, the initial concentration of ammonia salt contained in wastewater is nearly six times higher than that of phosphate, but the results show that the removal ratio of NH_4^+-N is better. In these ED experiments, ammonium ions transferred into concentrated compartment across the cation exchange membrane while phosphates ions transported through the anion exchange membrane. Without regarding to the differences between the two kinds of membranes, what made the different removal ratio is possibly the ion size. The ion size compared to the channel diameter in membrane body has a major impact on the ion transport [17]. An ion M^{z+} may associate 'n' number of water molecules in aqueous systems and it can be described as M^{z+}

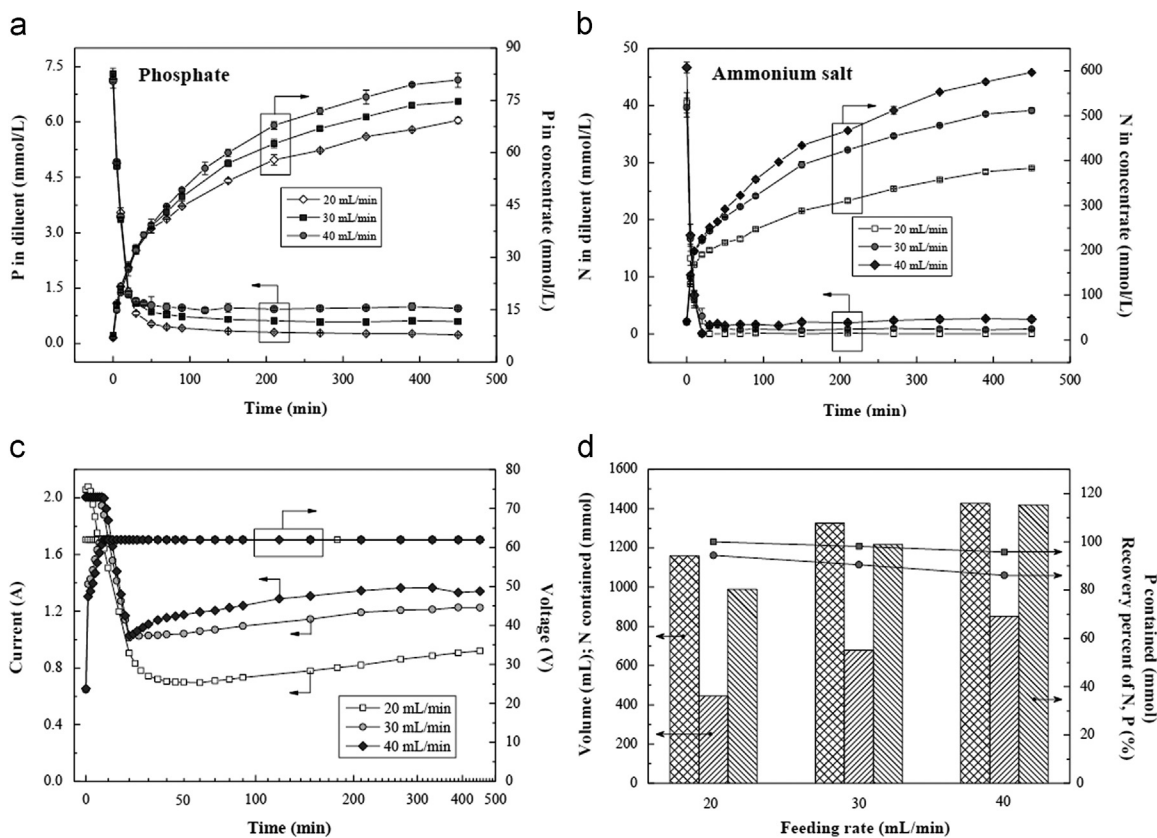


Fig. 2. Effect of feed flow rate on the performance of individual continuous ED. (a) Variations of phosphate in concentrated and diluted tanks of ED, (b) variations of ammonia salt in concentrated and diluted tanks of ED, (c) Variations of current and voltage across ED, (d) \square , the final volume of solution in concentrated tank; \square , the final total ammonia salt quantity contained in concentrated tank; \square , the final total phosphate quantity contained in concentrated tank; \blacksquare , the recovery ratio of ammonia salt; \bullet , the recovery ratio of phosphate.

Table 3
Apparent dynamic hydration radii of ions.

Ion	NH_4^+	Na^+	PO_4^{3-}	HPO_4^{2-}	H_2PO_4^-	Cl^-
Hydrated radius (nm)	0.148–0.161 [19,20]	0.098–0.117 [19–21]	0.238 [22,23]	0.230 [24]	0.238 [24]	0.167 [25]

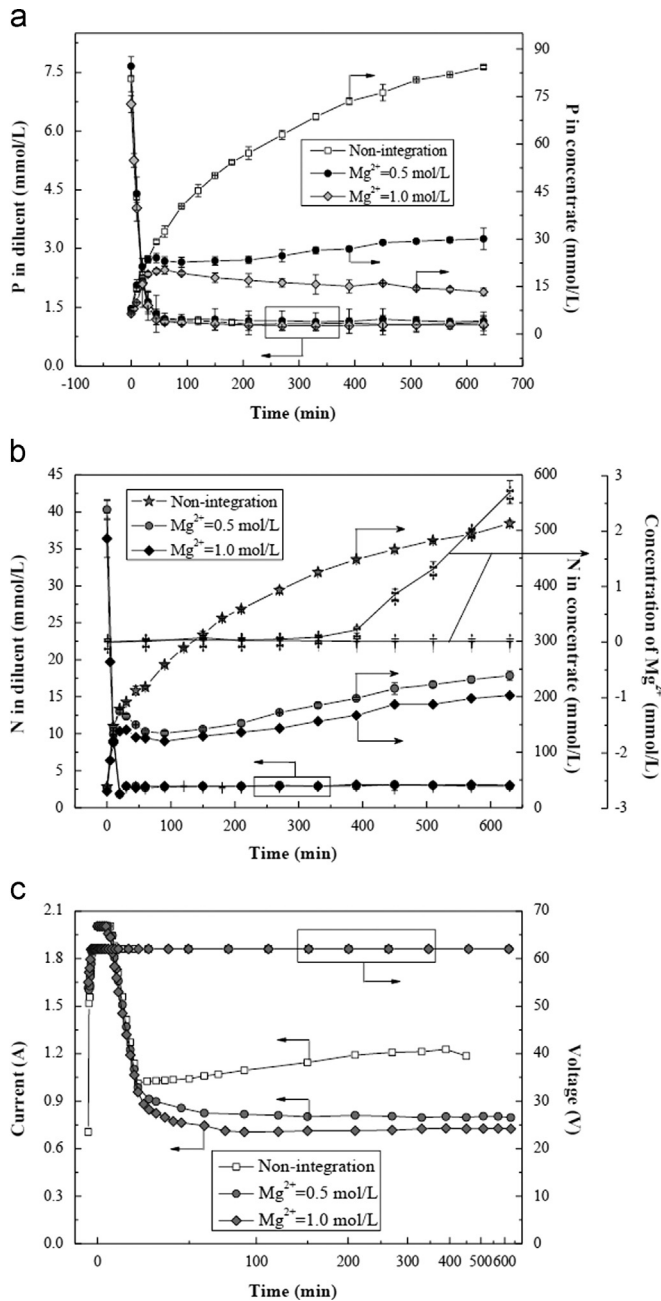


Fig. 3. Performance of integrated operation of ED and struvite reactor. (a) phosphate in ED unit; (b) ammonia salt in ED unit and Mg^{2+} in the effluent of struvite reactor; (c) variation of the current and voltage during ED operation.

$(\text{H}_2\text{O})_n$, so the apparent ionic size is the hydrated radius rather than the ionic radius of M^{z+} [18]. The hydrated radii of the four ions appeared in the solution are listed in Table 3. It can be seen that the gap between NH_4^+ and Na^+ is smaller than that of phosphate ions and Cl^- . So the transport of Cl^- is easier than that of phosphate ions, and this would result in a temporal hysteresis in the transporting of phosphate ions.

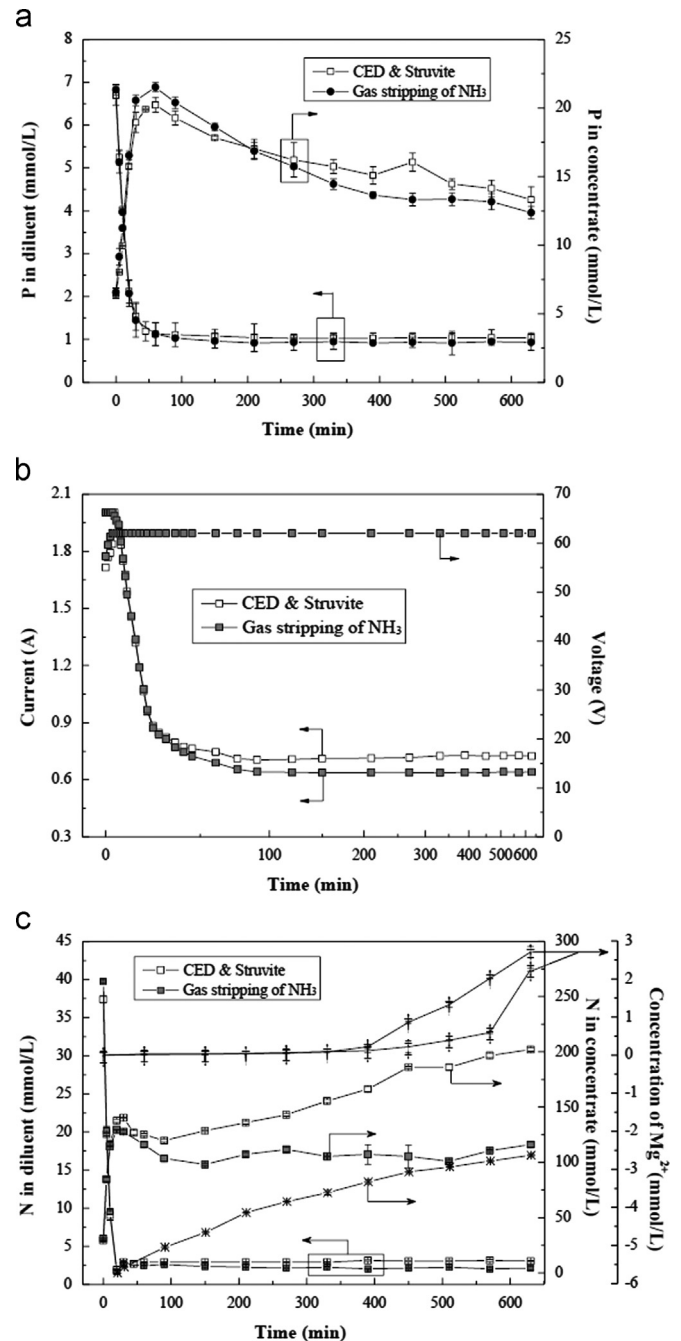


Fig. 4. Performance of integrated operation of three units: ED, struvite reactor and gas stripping of NH_3 . (a) phosphate in ED unit; (c) variations of the current and voltage during ED operation; (b) ammonia salt in ED unit, Mg^{2+} in the effluent of struvite reactor and NH_4^+-N in the absorption liquid (1 mol/L HNO_3). †, operating condition is integration of "ED & Struvite"; ‡, operating condition is integration of "ED & Struvite & gas stripping"; *, NH_4^+-N concentration in the absorption liquid.

3.2. Integration of ED and struvite reactor

In this part, we conducted an integrated operation of ED and struvite reactor. The investigated factor is the concentration of the

additional solution contained magnesium salt. As seen from Fig. 3(a) and (b), phosphate and ammonia salts concentration in diluted tank is nearly not changed compared with non-integration. However, both the two salts in concentrated compartment had obvious drops and a higher concentration of Mg^{2+} resulted in a bigger drop. In this integration, the concentrated solution contained N&P was circulated into the struvite reactor and back to it again. Ammonia salt and phosphate enriched in wastewater would combine with Mg^{2+} to generate the struvite. So a higher concentration of Mg^{2+} will lead to a higher consumption of the two salts. Under the condition of 0.5 mol Mg^{2+} /L, there is hardly no existing Mg^{2+} in the effluent of struvite reactor. This is a sign that struvite formation is mainly controlled by the pumped Mg^{2+} . While under the higher concentration, e.g., 1.0 mol Mg^{2+} /L, it begun to increase in the later stage and the N&P was the governing factor for the struvite reacting.

Fig. 3(c) illustrates the effect of Mg^{2+} on current and voltage during ED experiments. Compared with the non-integration, the ammonia salt and phosphate were shifted out of the ED unit which then led to an increased resistance. So, under the operation model of constant voltage, the current would decrease. While the electric field across the membrane stack depends on the current, the performance of ED becomes poor under a low current. This is also the reason for the larger drop from non-integration under the condition of 0.5 mol Mg^{2+} /L.

3.3. Integration of ED, struvite reactor and gas stripping of ammonia

In Section 3.2, an in-situ utilization of the concentrated N&P solution for forming struvite was conducted. During the generation process of struvite, the consumption ratio of ammonia salt (in terms of N) and phosphate (in terms of P) is 1:1. And there will remain a quantity of ammonia salt after the recirculating of concentrated N&P solution between concentrated tank and struvite reactor. So, some measures should be taken to inhibit its willful accumulation and enrichment. During this part, gas stripping was introduced to draw NH_3 from the concentrated ammonia salt solution.

Fig. 4(a) shows a comparison of phosphate concentration in ED unit between the experiments with and without ammonia stripping. It can be seen that there is little change and the later concentration in the concentrated tank was slightly reduced. It may be caused by the decrease in current as shown in Fig. 4(b), and then the ED processing capacity was decreased. For ammonia salt in the concentrated tank, i.e. the major investigated factor, there is an obvious decrease and the ammonia content in absorption liquid is on the rise. This can prove that the additional ammonia was effectively taken out from the concentrated N&P solution and gathered. For the Mg^{2+} existed in struvite reactor, both of them had a rise in the later time.

In order to confirm the presence of struvite, X-ray diffraction (XRD) analysis and scanning electron microscopy (SEM) were conducted for characterizing the obtained precipitates. Comparing the diffractograms peaks of the precipitates and struvite pattern in Fig. 5, there exists a good correlation. This is a sign that struvite was produced in the reactor. Because the system composition is relatively simple, the sediments appeared as a pure solid without adhering some other precipitates. But the struvite crystals have different shapes and it may be caused by the different concentration proportions between P, N and Mg^{2+} .

4. Conclusions

An experimental study was conducted on the recovery of ammonia salt and phosphate (N&P) by ED integrated with struvite

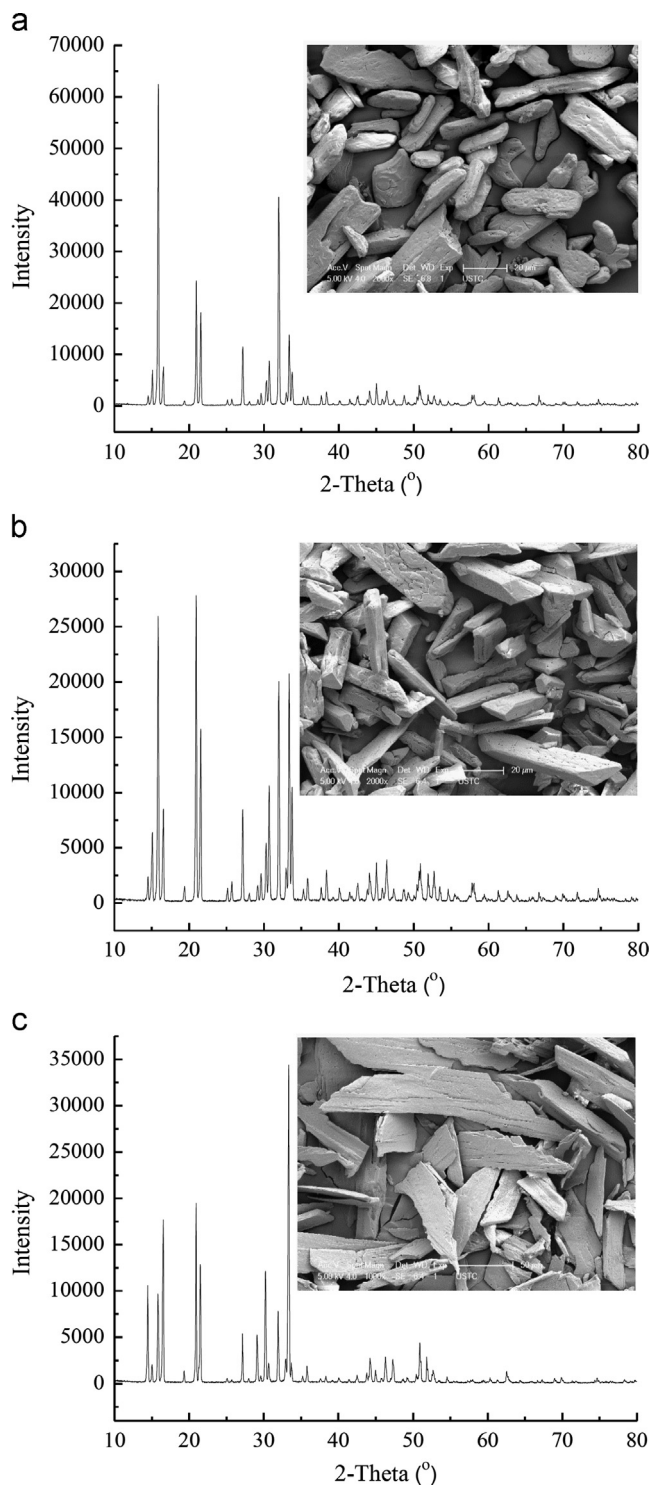


Fig. 5. XRD patterns and SEM images of the precipitates produced in the struvite reactor. Operating conditions are: (a) integration of "ED & struvite reactor" with additional Mg^{2+} solution of 0.5 mol/L; (b) integration of "ED & struvite reactor" with additional Mg^{2+} solution of 1.0 mol/L; (c) integration of "ED & struvite reactor & gas stripping" with additional Mg^{2+} solution of 1.0 mol/L.

reactor and ammonia stripping. Removal ratio about 95.8–100% for ammonia salt and 86.1–94.4% for phosphate could be obtained, and N&P were gathered in concentrated tank. Under the integration of the above three operation units, N&P were recycled effectively and the redundant ammonia salt was individually recovered. Comparing the diffractograms peaks of the obtained precipitates and struvite pattern, there existed a good correlation.

In this study, some meaningful conclusions can be drawn:

- (1) The N&P, contained in the Sidestreams of anaerobic digestion, can be avoided to discharge or backflow to the headstream of the wastewater treatment plant.
- (2) The recirculation of concentrated N&P solution between concentrated tank and struvite reactor could be avoided to generate a struvite reactor effluent, which generally still consists of an amount of N&P.

On the base of struvite formation, part of unreacted ammonia salt can be recovered individually by gas stripping operation.

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