Simultaneous removal of ammonia, P and COD from anaerobically digested piggery wastewater using an integrated process of chemical precipitation and air stripping

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ABSTRACT

The paper presented an efficient integrated physicochemical process, which consists of chemical precipitation and air stripping, for the simultaneous removal of NH_3-N, total P and COD from anaerobically digested piggery wastewater. In the integrated process, $Ca(OH)_2$ was used as the precipitant for NH_4^+, PO_4^{3-} and organic phosphorous compounds, and as the pH adjuster for the air stripping of residual ammonia. The possibility of the suggested process and the related mechanisms were first investigated through a series of equilibrium tests. Laboratory scale tests were carried out to validate the application possibility of the integrated process using a new-patented water sparged aerocyclone reactor (WSA). The WSA could be effectively used for the simultaneous removal of NH_3-N, total P and COD. 3 g/L of $Ca(OH)_2$ is a proper dosage for the simultaneous removal. The simultaneous removal of NH_3-N, total P and COD in the WSA reactor could be easily optimized by selecting a proper air inlet velocity and a proper jet velocity of the liquid phase. In all the cases, the removal efficiencies of the NH_3-N, total P and COD were over 91%, 99.2% and 52% for NH_3-N, total P and COD, respectively. The formed precipitates in the process could be easily settled down from the suspension system. Therefore, the integrated process provided an efficient alternative for the simultaneous removal of NH_3-N, total P and COD from the wastewater.

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1. Introduction

Pig farms with hundreds to several thousands of animals are in operation in many countries without adequate systems for waste treatment and disposal [1]. A large amount of piggery waste is discharged from the cages every day. This waste is a mixture of feces, urine and food wastage [2]. Piggery waste is characterized by a high content of organic matter and pathogenic microorganisms. The disposal of piggery waste without adequate treatment can cause a drastic effect on the environmental and human health [3].

According to the most common characteristics of this waste, anaerobic digestion could be considered as one of the most promising treatment alternatives, which has several advantages such as destruction of pathogenic and parasitic organisms, production of methane, low biomass production, better process stability and lower treatment cost [3,4]. It was reported that the separate anaerobic digestion of liquid and solid fractions of piggery waste is more effective than the digestion of the whole waste [5]. In practice, many large scale local pig farms in Chongqing area are now collecting the liquid and solid fractions of piggery waste separately in pig cages to minimize the amount of piggery waste. This collection mode is a water-saving process and is beneficial to later treatment. The solid fraction is directly transported to an anaerobic digester for fermentation to make organic fertilizer. The liquid fraction, a mixture of pig urine, manure leachate and washing wastewater, flows into an anaerobic digester after passing through a simple screen mesh. Practice illustrates that anaerobic digestion can greatly reduce the COD of piggery wastewater [1]. Practical operation of anaerobic digestion in many local pig farms in Chongqing area can make the COD of piggery wastewater to be reduced to the concentration lower than 500 mg/L. But the anaerobically digested liquor usually still contains more than 160 mg/L of NH_3-N and more than 30 mg/L of total P. The national discharge standard of pollutants for livestock and poultry breeding stipulates that the COD, NH_3-N and total P must be lower than 400 mg/L, 80 mg/L and 8.0 mg/L, respectively [6].

The ammonia in water is one of the major environmental pollutants, which may cause the increase of chlorine consumption and oxygen demand, toxicity to fish, eutrofication and diseases like ethaemoglobinaemia, hypertension and stomach cancer, as well as other serious environmental hazards [7]. So, the anaerobically digested liquor of piggery wastewater needs to be further treated to make its COD, especially NH_3-N and total P to be decreased to be lower than the values stipulated by the national standards.

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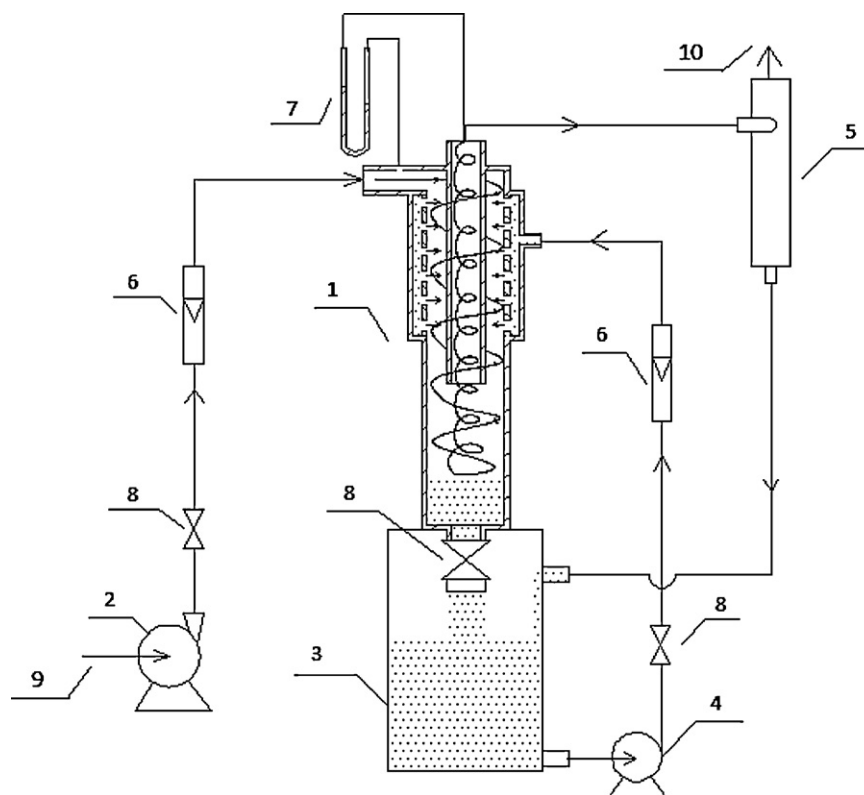
Fig. 1. The flow diagram of the experimental setup and the WSA configuration. (1) Water-sparged aerocyclone; (2) air pump; (3) water tank; (4) circulating pump; (5) gas–liquid separator; (6) rotameters; (7) U-type manometers; (8) valves; (9) air inlet; (10) air outlet.

The further removal of NH_3-N and total P from anaerobically digested liquor can be conducted using air stripping [8–12] and struvite precipitation [13–16]. Compared with the usually used biological processes [17–25], the physicochemical method cannot only remove ammonia and P, but also recover them as valuable products such as NH_3 and $MgNH_4PO_3$, which can be used as fertilizers. In addition, these processes are a time and energy-saving process.

Similar with the struvite precipitation, calcium ions can be also used as a precipitant to form $CaNH_4PO_4 \cdot 4H_2O$ [26]. The aim of this work is to present an efficient integrated physicochemical process, which consists of chemical precipitation and air stripping, for the simultaneous removal of NH_3-N, total P and COD from anaerobically digested piggery wastewater. In the process, cheap $Ca(OH)_2$ was used as the precipitant for NH_4^+ and PO_4^{3-} and meanwhile can be used as a cheaper pH adjuster for the air stripping of ammonia in comparison with the usually used NaOH. In many air stripping processes, NaOH is used as pH adjuster to avoid the serious scaling problem in stripping equipments. Compared with the usually used air stripping equipments like tanks and packed towers, the water-sparged aerocyclone reactor (WSA) is characterized by good mass transfer performance and self-clean function, and is suitable for the air stripping of wastewater system with suspended particles [27]. Therefore, the WSA was chosen as both a mixer and an air stripper to validate the large scale application possibility of the suggested simultaneous removal process. The effects of process parameters, including $Ca(OH)_2$ dosage, air inlet velocity (U_g) and jet velocity of liquid phase (U_l), on the simultaneous removal of NH_3-N, total P and COD were investigated for the optimization of operation conditions. In addition, the mechanisms for the removal of NH_3-N, total P and COD in the integrated process were tentatively elucidated through a series of equilibrium tests.

2. Material and methods

2.1. The anaerobically digested piggery wastewater

The anaerobically digested liquor of piggery wastewater used in this experiment was taken from the effluent of the largest local pig farm in Chongqing city, China. The pig farm is located in the Rongchang County, the modern animal husbandry area of China, about 100 km northwest of Chongqing city. The liquid and solid fractions of piggery waste are separately collected in the pig farm. The liquid fraction (a mixture of urine, leachate of manure and washing water) flows into an anaerobic digester after passing through a simple plastic screen. The effluent generally contains COD 150–500 mg/L, more than 160 mg/L of NH_3-N and more than 30 mg/L of total P with pH 7.3–8.0.

2.2. Equilibrium removal of N, P and COD

First the simultaneous precipitation of N, P and adsorption of COD in the digested liquor should be investigated. $Ca(OH)_2$ was chosen as the precipitant, pH adjuster and adsorbent because of its lower price. The experimental procedure is briefly outlined as follows. A prescribed dosage of $Ca(OH)_2$ powder was added into a 250 ml Erlenmeyer flask with 100 ml of the digested liquor, and then the flask was covered to prevent ammonia escape. Likewise, a series of the flasks with different dosages of $Ca(OH)_2$ powder was prepared. The flasks were then placed in a shaker and shaken at 150 rpm for 24 h at 25 °C to make the chemical precipitation reaction and adsorption reach an equilibrium. After that the solid–liquid system was filtered to get a supernatant sample, which was then used for the determination of NH_3-N, total P and COD. Each experiment was repeated to get experimental data with an error of less

than 5%, and the averaged value was used. The removal efficiencies for NH_3-N, total P and COD could be calculated by the differences between initial and equilibrium concentrations divided by their respective initial concentration.

2.3. Simultaneous removal of N, P and COD

The simultaneous removal of N, P and COD from the anaerobically digested liquor was conducted in the new WSA. The experimental setup, the WSA configuration and its working principle can be found in our recent work [21], as shown in Fig. 1. For every run, 12 L of the digested liquor was poured into the water tank in the experimental setup and then added different dosages of $Ca(OH)_2$ powder under proper stirring to from a suspension with a pH higher than 11. Then the air was pumped into the aerocyclone at a prescribed flow rate. When the pressure reading reached a steady state, the circulation pump at a certain flow rate pumped the suspension in the tank into the WSA. During circulation, the concentrations of NH_3-N, total P and COD in the suspension were continuously decreased because of the chemical precipitation reaction and air stripping of residual ammonia. The suspension samples were taken out from the water tank and centrifuged to get supernatants for the determination of NH_3-N, total P and COD. All the experiments were carried out at ambient temperature (28–30 °C). Each experiment was repeated to get experimental data with an error of less than 5%, and the averaged value was used.

The effects of major process parameters on the simultaneous removal kinetics of NH_3-N, total P and COD removal were investigated, including $Ca(OH)_2$ powder dosage, air inlet velocity and jet velocity of the suspension.

2.4. Analytical methods

Ammonia concentration was measured using the Nessler's reagent ($HgCl_2$–KI–KOH) spectrophotometry at 420 nm according to the Standards of the People's Republic of China (GB7479-87). Total P concentration was measured using the ammonium molybdate spectrophotometric method (GB11893-89). Chemical oxygen demand (COD) was analyzed using the standard potassium dichromate oxidation method (GB11914-89). Concentration of suspended substance (SS) was determined using the standard gravimetric method (GB11901-89).

3. Results and discussion

3.1. Equilibrium removal of NH_3-N, total P and COD and mechanisms

The equilibrium tests help to evaluate the simultaneous removal efficiency of N, P and COD caused by the chemical precipitation reaction and adsorption. The experimental data can provide very important information for the understanding of the process mechanism. The results of the equilibrium tests are summarized in Table 1.

The anaerobically digested liquor used in this experiment contained 120 mg/L NH_3-N, 20 mg/L total P, 198 mg/L COD, 13 mg/L PO_4^{3-}-P and 47 mg/L suspended solid (SS) with pH 7.89. The P in the form of inorganic phosphate (PO_4^{3-}-P) amounted to about 66.8% of the total P and the residual 33.2% should be present in the form of organic compounds. The molar concentration of PO_4^{3-}-P in the liquor is about 0.42 mmol/L, which full precipitation needs 0.42 mmol/L NH_3-N (5.88 mg/L NH_3-N) and 0.42 mmol/L calcium ions (31 mg/L $Ca(OH)_2$) based on the formula of the product $CaNH_4PO_4 \cdot 4H_2O$. Thus, if the product formula is true, dissolving a suitable dosage of $Ca(OH)_2$ in the liquor would result in a simultaneous precipitation of NH_3-N and PO_4^{3-}-P. When the dosage of $Ca(OH)_2$ exceeds about 31 mg/L, theoretically, the removed amounts of NH_3-N and PO_4^{3-}-P should reach the maximized 6 mg/L NH_3-N and 13 mg/L PO_4^{3-}-P, respectively. In this case, the maximum removal efficiencies for NH_3-N and PO_4^{3-}-P should be about 5% and 66.8%, respectively, and the ratio of the removed N/P should be equal to 1 theoretically.

Now it could be seen from Table 1 that when the $Ca(OH)_2$ dosage increased to 0.04 g/L, the removal efficiencies for NH_3-N and total P increased to about 5% and 63% (lower than the theoretical value of 66.8%), respectively, and that the ratios of the removed N/P were in the range of 1.1–1.2, slightly larger than 1. The COD removal in this low concentration of $Ca(OH)_2$ dosage illustrated that the organic phosphorous compounds reacted with Ca^{2+} ions to form precipitate. This could explain why the removal efficiency of P is less than the predicted value, 66.8%, when the $Ca(OH)_2$ dosage was over the theoretic value of 31 mg/L (such as 0.04 g/L). In fact, the removal efficiency of P did not reach the predicted value until the $Ca(OH)_2$ dosage increased to about 0.06 g/L. It was observed, at the same time, that the removal efficiencies of total P and COD reached about 99% and 50%, respectively, when the $Ca(OH)_2$ dosage was increased to 1.5 g/L. Because 1.5 g/L of $Ca(OH)_2$ dosage is just lower than $Ca(OH)_2$ solubility (the solubility is 0.165–0.116 g per 100 g

Table 1
The results of the equilibrium tests for the physicochemical removal of NH_3-N, total P and COD.

$Ca(OH)_2$ dosage (g/L)	Removal efficiency (%)[a]			The averaged molar ratio of removed N/P
	NH_3-N	Total P	COD	
0.02	3.8–4.2	50.5–51.8	8.1–9.0	1.1
0.04	5.1–5.3	63.2–63.7	11.2–12.2	1.2
0.06	5.5–5.8	66.9–67.0	16.7–20.58	1.2
0.08	5.2–5.6	74.8–74.9	22.2–23.4	1.0
0.10	8.9–9.2	90.8–91.3	27.8–28.6	1.3
1.00	9.8–10.3	98.2–98.3	48.2–50.0	1.1
1.50	12.5–12.9	99.2–99.4	50.0–51.8	1.4
2.00	13.1–13.5	98.0–98.1	51.9–53.6	1.5
2.50	13.8–14.1	98.5–98.5	53.6–55.4	1.6
3.00	13.9–14.3	99.2–99.2	55.4–57.1	1.6
3.50	14.1–14.5	98.0–99.0	57.1–58.9	1.7
4.00	14.3–14.7	98.7–99.3	56.8–59.8	1.7
5.00	14.7–15.1	98.5–98.6	58.9–60.7	1.7
6.00	14.9–15.3	97.3–98.0	60.7–62.5	1.8
7.00	15.4–16.1	98.3–99.7	62.5–64.3	1.9
8.00	16.1–16.2	99.6–99.7	59.4–62.5	1.9
9.00	15.9–16.1	99.4–99.7	62.5–64.3	1.9

[a] The initial concentrations of NH_3-N, total P and COD in the anaerobically digested liquor are 120 mg/L, 20 mg/L and 198 mg/L, respectively. The removal efficiency was calculated by the difference between initial and equilibrium concentration divided by the initial concentration.

of pure water from 20 to 60 °C), no Ca(OH)$_2$ powder was present in the liquor and thus adsorption phenomenon should not exist. So, the major mechanism for the removal of the total P should not only include the precipitation reaction for the formation of CaNH$_4$PO$_4$·4H$_2$O, but also include the precipitation reaction taking place between the organic phosphorous compounds and calcium ions.

As shown in Table 1, when the Ca(OH)$_2$ dosage was over 1.5 g/L, the removal efficiencies of NH$_3$-N and COD all increased slowly with increasing the dosage. The continuous slight COD removal is not proportional to the increase of Ca(OH)$_2$ dosage, and thus may be caused by the adsorption of some polar organic compounds on the hydrophilic surface of Ca(OH)$_2$ particles in the liquor through hydrogen bonds. The other residual organic compounds may be non-polar or very weakly polar compounds and they could not be adsorbed by the polar surface of Ca(OH)$_2$ particles. Therefore, it seems that the major mechanism for the COD removal is the precipitation reaction of the organic phosphorous compounds with calcium ions.

Compared with the theoretic removal efficiency of 5%, the higher removal efficiency of NH$_3$-N in the case of higher Ca(OH)$_2$ dosage (>0.1 g/L), may be caused by the mass transfer of molecular ammonia from liquid phase to gas phase. When the liquor was saturated by Ca(OH)$_2$, its pH value reached higher than 11. The gas–liquid distribution ratio of ammonia is the function of pH and temperature. Higher pH results in a higher gas–liquid phase distribution ratio of ammonia, which accelerate ammonia removal from water [27]. As a result, higher Ca(OH)$_2$ dosage led to a higher NH$_3$-N removal efficiency and higher removed N/P ratios. In order to remove and recover the molecular ammonia from the liquor, air stripping could be adopted in large scale production [27].

Therefore, the whole NH$_3$-N, Total P and more than 50% COD could be removed by the integrated process with simultaneous chemical precipitation and air stripping. The air stripped NH$_3$-N can be recovered as a valuable product using other absorption process. The formed precipitate, which mainly consists of CaNH$_4$PO$_4$·4H$_2$O and residual Ca(OH)$_2$, could be separated out and returned to the wastewater treatment process as the precipitant for NH$_4^+$ and PO$_4^{3-}$. When the residual Ca(OH)$_2$ is completely consumed, the composition of the precipitate is almost CaNH$_4$PO$_4$·4H$_2$O, which can be used as a slowly releasing fertilizer.

Although higher Ca(OH)$_2$ dosage led to a slightly higher COD removal, higher dosage would increase separation cost and the consumption of lime. Choosing a proper dosage was recommended for further larger scale investigation.

3.2. Effect of Ca(OH)$_2$ dosage on the simultaneous removal of NH$_3$-N, total P and COD in the WSA reactor

The equilibrium test results, as shown in Table 1, indicated that the total P could be easily removed using a proper Ca(OH)$_2$ dosage (such as 1.5 g/L) by the chemical precipitation reactions, along with more than 5% of NH$_3$-N removal. But higher Ca(OH)$_2$ dosage led to higher COD removal and is beneficial to the air stripping of ammonia. In addition, the dynamic process in the WSA may be different from the equilibrium tests. So, the effects of Ca(OH)$_2$ dosage on the simultaneous removal of NH$_3$-N, total P and COD in the WSA reactor were first investigated to check the equilibrium test results and the variation caused by the dynamic process. The results are shown in Fig. 2.

It could be seen that when the Ca(OH)$_2$ dosage was over 2 g/L, the increase of Ca(OH)$_2$ dosage had little effect on the simultaneous removal of NH$_3$-N, total P and COD. This is basically in accordance with the equilibrium test results. The concentrations of NH$_3$-N, total P and COD simultaneously decreased rapidly with increasing stripping time in the WSA and decreased to the concentration

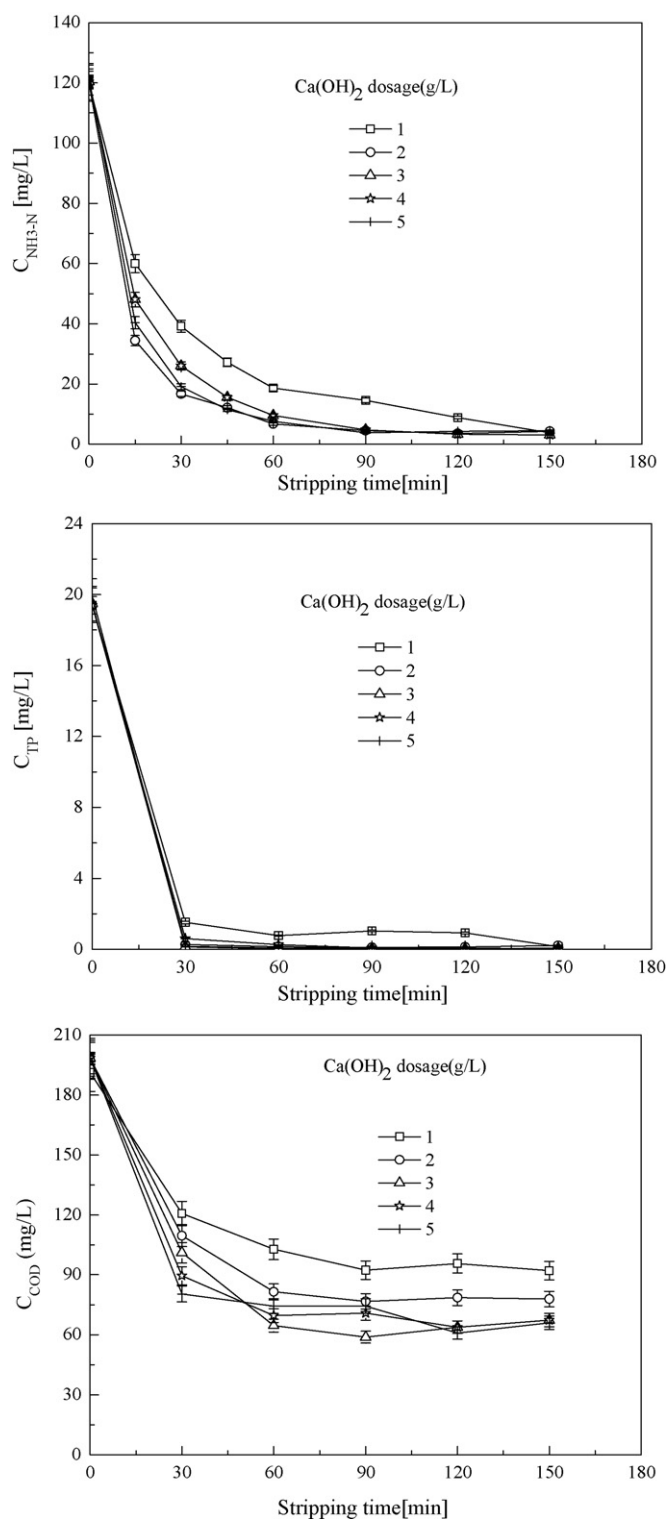Fig. 2. The effect of Ca(OH)$_2$ dosage on NH$_3$-N, total P and COD removal. V_l = 12 L, U_l = 0.37 m/s, U_g = 4.81 m/s, temperature: 28–30 °C.

lower than 7 mg/L, 0.2 mg/L and 90 mg/L, respectively, when the operation time was 60 min. At this time, the corresponding removal efficiencies are more than 94%, 99% and 52% for NH$_3$-N, total P and COD, respectively. The removal efficiencies for the total P and COD are close to that obtained in the equilibrium tests. The obviously high removal efficiency for NH$_3$-N arose from the strong air stripping in the WSA. In fact, the total P and COD concentration almost simultaneously decreased rapidly within the first 30 min and then

slowly decreased to approach their respective minimum value when the operation time was 60 min, as shown in Fig. 2, further reflecting that the chemical precipitation of the organic phosphorous compounds with calcium ions is really the major mechanism for COD removal. This experiment validated the possibility of the simultaneous removal of NH_3-N, total P and COD from the anaerobically digested liquor and exhibited good process efficiency for NH_3-N and total P.

3.3. Effect of air inlet velocity on the simultaneous removal of NH_3-N, total P and COD

Air inlet velocity is a very important process parameter in the WSA. The momentum transfer of the air in the WSA will affect all the physicochemical processes in the simultaneous removal of NH_3-N, total P and COD. The effects of air inlet velocity on the simultaneous removal of NH_3-N, total P and COD are shown in Fig. 3.

It could be seen from Fig. 3 that the higher air inlet velocity facilitated the acceleration of air stripping of ammonia and that the process efficiency of the air stripping could not be obviously enhanced when the air inlet velocity was over 14.42 m/s. The NH_3-N concentration decreased rapidly to the value lower than 10 mg/L within 60 min of operation time when the air inlet velocity was higher than 14.42 m/s, resulting in an NH_3-N removal efficiency more than 92%.

The COD removal rate slowed down with increasing the air inlet velocity within the first 60 min and later reached an almost stable value, as shown in Fig. 3. Meanwhile, the total P and COD concentrations all decreased to their respective minimum value when the operation time was 60 min, under different air inlet velocities. It appears that the higher air inlet velocity would affect the precipitation reaction of the organic phosphorous compounds with Ca^{2+} ions in the WSA. Higher air inlet velocity would result in a higher degree of dispersion of the suspension system, forming finer drops in the air field in the WSA. This probably affected the full mixing of the liquid and solid phases in the drops, making the chemical precipitation reaction slow down. About 14 m/s appears to be a compromised air inlet velocity for COD and NH_3-N removal rates.

A noticeable change of the total P removal rate was not observed with increasing the air inlet velocity. In the case of all the air inlet velocities, the concentration of the total P decreased to that lower than 0.2 mg/L within 60 min, which amounts to a removal efficiency more than 99% and is in accordance with the equilibrium test results. The fact that the removal of the total P and COD was almost not affected by the air inlet velocity after 60 min of operation time further validated the chemical precipitation mechanisms for total P and COD removal.

3.4. Effect of jet velocity of the suspension on the simultaneous removal of NH_3-N, total P and COD

Similarly, the jet velocity of liquid phase is another important process parameter. It deals with the mixing of the suspension and thus may affect the chemical precipitation reactions. Fig. 4 shows the effect of jet velocity of the suspension on the simultaneous removal of NH_3-N, total P and COD.

Increasing the jet velocity did not cause an obvious increase in the removal rate of these three pollutants, but is beneficial for their removal during the operation of air stripping, as shown in Fig. 4. This, again, illustrated a negligible effect of the jet velocity on the mass transfer rate of ammonia from liquid phase to gas phase in the WSA, where the mass transfer process is governed by air flow field [27].

Similar with the observation mentioned above, the concentrations of total P and COD all decreased sharply within the first 30 min
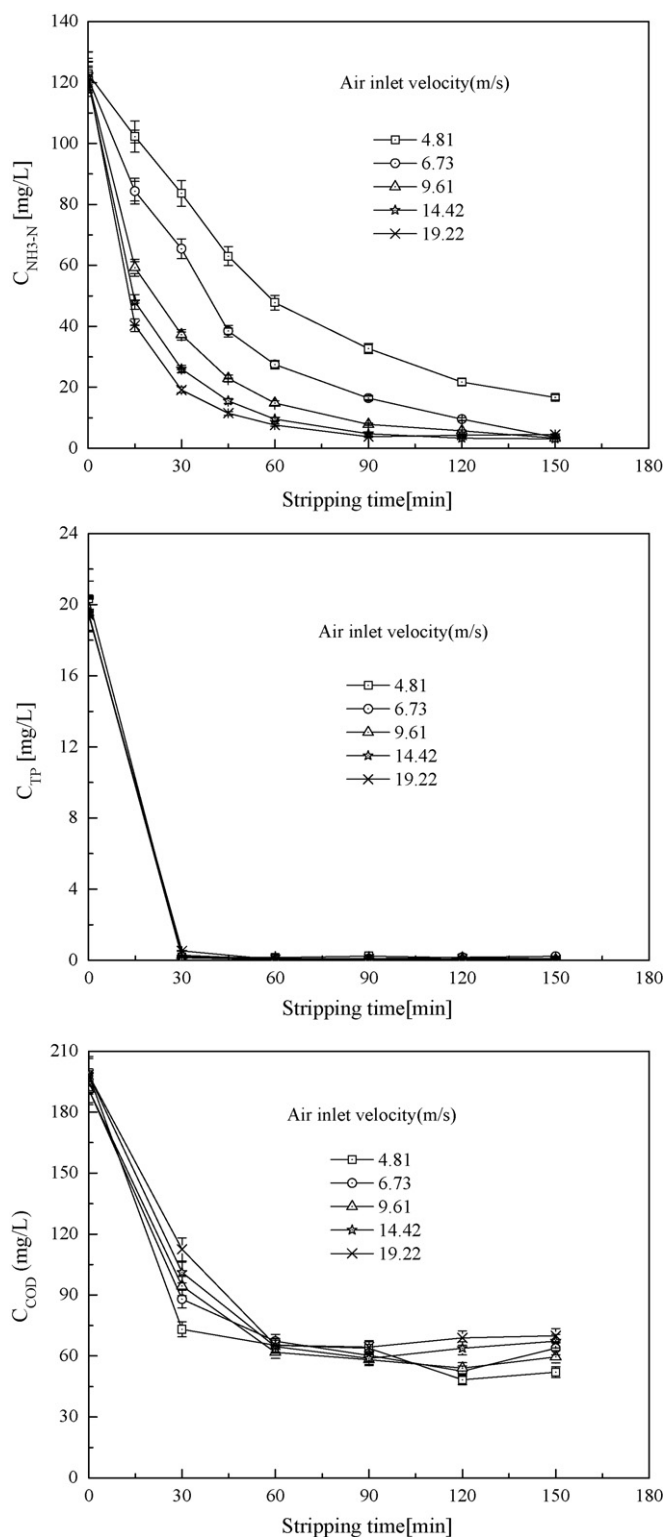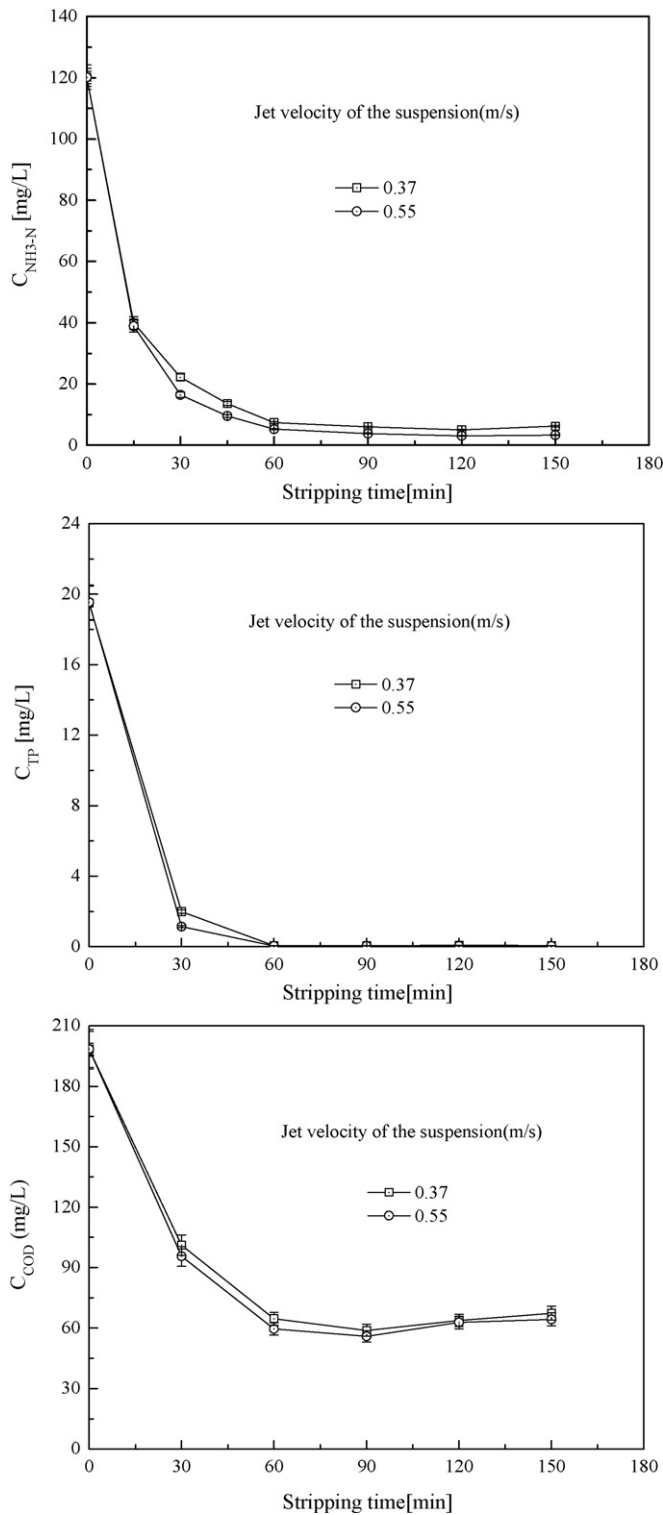

Fig. 3. The effect of air inlet velocity on NH_3-N, total P and COD removal. $V_l = 12$ L, $U_l = 0.37$ m/s, $Ca(OH)_2$ dosage = 3 g/L, temperature: 28–30 °C.
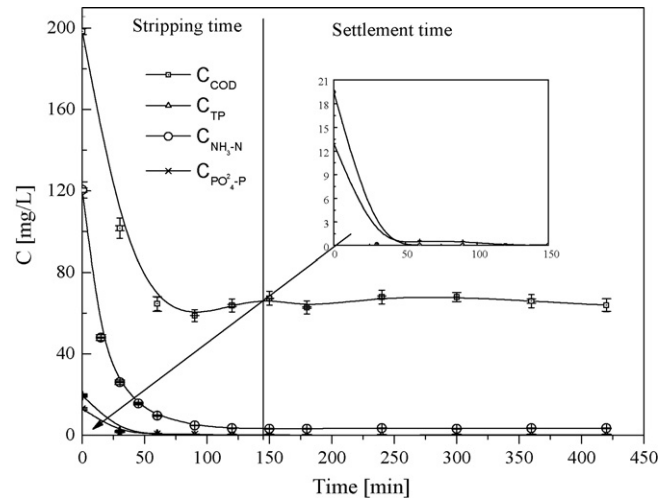
and then decreased to their respective minimum value when the operation time was 60 min, under different jet velocities. This again indicated the chemical precipitation mechanism for P and COD removal.

Fig. 5. The effect of stripping time and settlement time on NH_3-N, total P and COD, PO_4^{3-} removal. V_l = 12 L, U_g = 4.81 m/s, U_l = 0.37 m/s, $Ca(OH)_2$ dosage = 3 g/L, temperature: 28–30 °C.

when 3 g/L $Ca(OH)_2$ dosage was used, so the system had a better separation performance.

From the equilibrium removal tests, it is considered that a quiet environment may be beneficial to the slight COD removal increase. So, the effect of a sedimentation process on the simultaneous removal of NH_3-N, total P and COD was further investigated after the air stripping and the result is shown in Fig. 5. It can be seen that the sedimentation process subsequent to the air stripping did not cause an obvious improvement in the COD removal. This also illustrated that the adsorption is not the major mechanism for the COD removal. The chemical precipitation reactions and air stripping could be simultaneously carried out in the WSA, resulting in a satisfactory removal of NH_3-N, total P and COD from the anaerobically digested piggery wastewater.

4. Conclusions

Anaerobically digested piggery wastewater usually contains higher concentrations of NH_3-N, total P and COD than the discharge standards stipulated by governments. This work presented an integrated process, which consists of chemical precipitation and air stripping, to simultaneously remove NH_3-N, total P and COD from the anaerobically digested liquor. The possibility of the process and related mechanisms were first investigated through a series of equilibrium tests. Laboratory scale tests were carried out to validate the large scale application possibility of the integrated process using a patented water sparged aerocyclone reactor. The following points could be concluded.

(1) The integrated physicochemical process provided an alternative for the simultaneous removal of NH_3-N, total P and COD. They can be simultaneously removed by the air stripping of ammonia, the chemical precipitation reaction of $CaNH_4PO_4 \cdot 4H_2O$ and the precipitation of organic phosphorous compounds with calcium ions. The formed precipitate has better settling performance and can be easily separated.
(2) The integrated process could be conducted in air stripping equipment without any packing in it like the WSA, in which the physicochemical process occurring in the gas–liquid–solid multiphase system can be operated very well.
(3) The WSA could be effectively used for the simultaneous removal of NH_3-N, total P and COD. 3 g/L of $Ca(OH)_2$ is a proper dosage for the simultaneous removal. A higher air inlet velocity is bene-

Fig. 4. The effect of jet velocity of the liquid phase on NH_3-N, total P and COD removal. V_l = 12 L, U_g = 4.81 m/s, $Ca(OH)_2$ dosage = 3 g/L, temperature: 28–30 °C.

3.5. Effect of sedimentation on the simultaneous removal of NH_3-N, total P and COD

The reaction system was fully mixed in the WSA. As an industrial process, the solid–liquid separation is very important for operation. The settling velocity (SV) of the formed precipitate was measured in a settling cup and the change of the supernatant height with settling time was recorded. The averaged SV was 2.0–2.1 cm/min

ficial to the removal rate of NH_3-N. The removal process of total P and COD could be completed within 60 min of operation time under all experimental conditions. In all the cases, the removal efficiencies of the NH_3-N, total P and COD were over 91%, 99% and 52% for NH_3-N, total P and COD, respectively.

Acknowledgement

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