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Exploration of ammonia stripping coupled adsorption-membrane filtration process for treating kitchen waste biogas slurry

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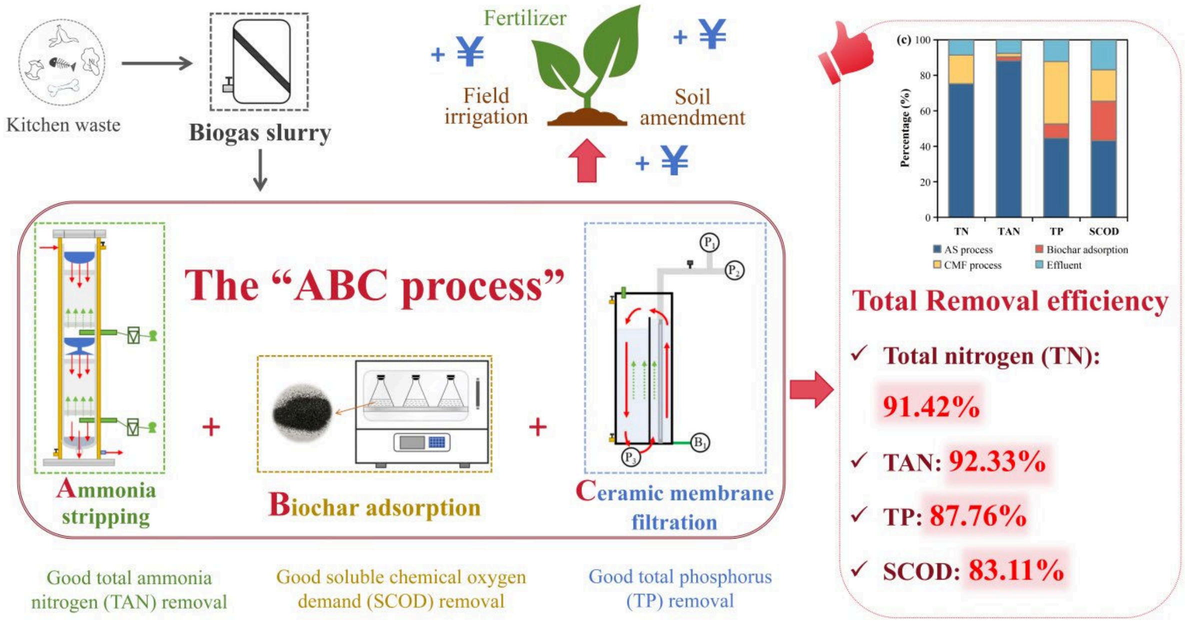
Abstract:

The potential contamination of biogas slurry generated from the anaerobic digestion of kitchen waste (KW) poses a considerable challenge to its safe and effective utilization as a fertilizer. To tackle this problem, a novel process termed “ABC” was developed, integrating ammonia stripping (AS), biochar adsorption, and ceramic membrane filtration (CMF) for comprehensive pollutant mitigation. A stepwise optimization was carried out, comparing batch investigation, the AS process, and the combined AS+CMF process. Results indicated that the AS process possessed the maximum total ammonia nitrogen (TAN) removal of 86.61% at an airflow rate of 40 L/min. The combined AS and CMF process with 0.1 μm membrane was ineffective for TAN removal, while it had best performance for total phosphorus (TP) with removal efficiencies ranged from 86.67% to 88.00%. Under the optimal biochar addition condition of 5 g/L with a particle size of 0.25-0.85 μm, the adsorption pretreatment effectively removed a significant portion of SCOD, prevented nutrient loss, and substantially enhanced pollutant removal efficiency in the subsequent CMF process. The ABC process achieved a significant improved performance of 91.42%, 92.33%, 87.76%, and 83.11% of TN, TAN, TP, and SCOD, respectively, compared to the raw biogas slurry. Additionally, the ABC process demonstrated its cost-effectiveness (8.51 CNY/kg) in producing clean effluent and nutrient-rich concentrate suitable for use as fertilizer. In summary, the “ABC process” proved highly effective in treating the KW biogas slurry, while generating economically valuable by-products.

Keywords: Ammonia stripping, biogas slurry, biochar adsorption, ceramic membrane filtration, kitchen waste



Kitchen waste
Biogas slurry
Fertilizer
+ ¥
Field irrigation
+ ¥
Soil amendment
+ ¥
The “ABC process”
Ammonia stripping
+
Biochar adsorption
+
Ceramic membrane filtration
Good total ammonia nitrogen (TAN) removal
Good soluble chemical oxygen demand (SCOD) removal
Good total phosphorus (TP) removal
(c)
Percentage (%)
TN
TAN
TP
SCOD
AS process
Biochar adsorption
CMF process
Effluent
Total Removal efficiency
✓ Total nitrogen (TN): 91.42%
✓ TAN: 92.33%
✓ TP: 87.76%
✓ SCOD: 83.11%

1. Introduction

Anaerobic digestion (AD) is one of the most widely used technology and has gained increasing attention in waste treatment due to its high efficiency, low cost, and low global warming footprint. The liquid residue after AD, known as biogas slurry, is generated in large quantities and is considered as a promising liquid fertilizer that is readily absorbed by plants. However, biogas slurry may contain high levels of total ammonia nitrogen (TAN, 1500-6000 mg/L) (dos Santos et al., 2020), total phosphorus (TP), and soluble chemical oxygen demand (SCOD) with turbidity and malodor. While these compounds represent valuable nutrients, if discharged untreated, they can lead to environmental issues such as aerosol production, acid rain formation, and eutrophication of water bodies, if discharged untreated (Bezuglova & Klimenko, 2022).

According to the requirement of Ministry of Agriculture and rural of China (2022), such biogas slurry must undergo harmless treatment before used as fertilizer to the field (Jin et al., 2022). Extensive efforts have been made to investigate biological, physical, and chemical techniques for nutrients recovery from organic waste (Chen et al., 2021; Palakodeti et al., 2021). Among them, the ammonia stripping (AS) is an attractive TAN removal technology due to its insensitivity to solids and low energy demand (Değermenci & Yildiz, 2021). Provolo et al. (2017) found that a high TAN removal efficiency of 87% was achieved in biogas slurry of animal manure under pH = 9 and 40 °C conditions. The AS process can not only significantly reduce the content of TAN in biogas slurry, but also effectively produce ammonium fertilizers (Melgaço et al., 2020). Stripping removal efficiency would decrease in the later stage of AS process when ammonia contents are low, and it was not effective in removing contaminants other than TAN (Palakodeti et al., 2022). Moreover, it was reported that high airflow rate can improve ammonia removal efficiency and shorten retention time. However, excessive flow rate may also lead to a decrease in liquid phase temperature, foaming, and evaporation (Zhao et al., 2015). It was reported that the suitable airflow rate of the AS system was related to substrate characteristics and liquid flow rate (Palakodeti et al., 2021). However, the optimization of airflow rate and efficient deep removal of ammonia with low levels in AS process remains poorly investigated. Meanwhile, significant amounts of SCOD and phosphorus persist in the effluent after AS treatment.

To address such issues, AS process was explored to integrate with other technologies, e.g., ceramic membrane filtration (CMF), for the end-of-pipe treatment (ref). The CMF technology is selected because of its simplicity, high efficiency, low contamination, and high P removal potential (Chen et al., 2021). CMF for SBS treatment recovers particles and macromolecules matters, which could obtain clean effluent and nutrient-rich concentrate for fertilizer production at the same time (Yang et al., 2024). Xiao et al. (2021) used the CMF technology to treat biogas slurry of pig manure founding that when the pore size was 0.1 μm, the removal of turbidity and soluble chemical oxygen demand (SCOD) could reach 83.91% and 13.12%, respectively. The primary challenge with CMF is membrane fouling during the treatment process (Li et al., 2023). Raw materials with the high turbidity value, such as biogas slurry, can severely clog the ceramic membrane pores, reducing both efficiency and service life (Alengebawy et al., 2021). Thus, pretreatment of the biogas slurry is essential before applying the CMF process.

Adsorption, e.g., using biochar, could couple with the CMF technology as pretreatment of the

SBS to reduce membrane fouling. Biochar refers to the solid product of biomass undergoing thermochemical conversion in anaerobic or low oxygen environments (Guo et al., 2020). It is an economical and efficient adsorbent with a wide range of raw materials and simple preparation. Due to its stable structure and large surface area, biochar is highly effective in reducing odor and turbidity while also contributing to the removal of various pollutants to a significant extent (Ye et al., 2022). Chen et al. (2017) demonstrated that the addition of biochar from different materials could reduce TAN levels by 9.2%-24.8%. Additionally, biochar can be used as a soil amendment with inorganic nutrients desorption to increase soil nutrients (Yu et al., 2020). Therefore, the combined use of CMF and biochar for SBS treatment can achieve complementary advantages and disadvantages.

At present, the integrated process research of AS and struvite precipitation technology was relatively mature. Antonini et al. (2011) has shown that the AS process combined with struvite precipitation can achieve an 94% TAN removal efficiency on human urine. However, the entire process requires high temperature and pH requirements, requiring a significant amount of energy input (Wu & Vaneeckhaute, 2022). Alengebawy et al. (2021) designed polyaluminum chloride coagulation, biochar adsorption, and ceramic membrane filtration to the biogas slurry ammonia stripping. This combination process achieved a maximum TN removal efficiency of 88%, indicating that the combination of ammonia stripping-adsorption-membrane filtration had good benefits. Nevertheless, integrated approach has been rarely investigated for process optimization, and such methods tailored specifically for KW remain underdeveloped..

Kitchen waste (KW) constitues a significant part of municipal solid waste, and China produces substantial quantities of KW biogas slurry annually through AD (Liu et al., 2023). The AS integrated approach could be optimal for upcycling KW biogas slurry regarding its high organic matter and TAN concent(Zhang et al., 2024). Therefore, in this study, the KW biogas slurry was used as the subject with the treatment of a novel “ABC process” that combines AS process, biochar adsorption, and CMF process. Additionally, two control groups, AS treatment alone and AS combined with CMF treatment, were established to demonstrate the comparative effectiveness of the treatments. To improve the efficiency, the airflow rate, biochar dose, and biochar particle size were systematically optimized, and the removal of total nitrogen (TN), TAN, TP, SCOD and other indicators were analyzed. This study aims to provide a method reference for the combination process in efficient treatment of kitchen biogas slurry.

2 Materials and Methods

2.1 Experimental materials

The raw biogas slurry was collected from a real-scale kitchen waste biogas plant in Jinhua City, Zhejiang Province, China, and stored in 4 °C cold room prior to use. The average values of pH, electrical conductivity (EC), total ammonia nitrogen (TAN), soluble chemical oxygen demand (SCOD), total nitrogen (TN), total phosphorus (TP), and total solid (TS), and volatile solid (VS) of the raw biogas slurry were determined at 8.3, 24.9 mS/cm, 2.9 g/L, 12.6 g/L, 3.2 g/L, 70%, 2.8%, respectively. The corn stover derived biochar was manufactured by a tubular furnace under the heating rate of 10 °C/min wit and keep warm for 1 h after reaching 600 °C with N_2 as a protective gas (0.2 L/min). After the reaction, it was cooled to below 100 ºC and passed through the 1.4 mm sieve. The prepared biochar was dried in a dryer at 105 ºC for 48 h and stored in a sealed plastic bag.

2.2 Experimental operation

The novel "ABC process" included ammonia stripping (AS) + biochar adsorption + ceramic membrane filtration (CMF) treatment (Fig. 1). Before the formal "ABC process", two systems, AS alone (System I) and AS combined with CMF treatment (System II), were designed to determine the optimal airflow rate and the ceramic membrane working effect. In addition, batch experiments were used to explore the optimal dose and particle size of biochar. The specific experimental operation is as follows:

The AS process was conducted in a 7 L ammonia stripping tower utilizing a countercurrent dynamic method for biogas slurry treatment, based on the setup and parameters described by Liu et al. (2022). The tower consisted of two 0.3 m layers of multi-surface hollow plastic balls (25 mm diameter) to enhance ammonia removal efficiency (Fig. 1, System I). The stripped ammonia gas was subsequently passed through 0.5 mol/L NaOH and 1 mol/L H_2SO_4 solutions (150 mL, respectively) before being discharged. The process was maintained at 37 °C, with 40 L/min airflow rate regulated via the air pump and monitored by a flow meter. The pre-stripping for CO_2 removal was performed with liquid and gas inlet speeds of 140 mL/min and 20 L/min, respectively for 8 h. After that, 8.7 g/L $Ca(OH)_2$ was added to adjust the pH to 10.8. During the AS optimization, the gas inlet was switched to air with flow rates of 10, 20, 30, and 40 L/min while keeping the liquid flow constant. The test was conducted for 20 h. Effluent samples of 10 mL were collected every 4 h to monitor pH, EC, TAN, TN, TP, and SCOD.

The CMF operated using cross-flow filtration based on vacuum negative pressure suction (Fig. 1, System II). The setup had a total volume of 7 L and a working volume of 5 L, divided into a liquid storage chamber and a membrane filtration chamber. A microfiltration-grade ceramic membrane plate with a pore size of 0.1 μm and a surface area of 0.1 m^2 was used. The membrane was positioned vertically in the reaction chamber, with two U-shaped aerator pipes placed on both sides. An air pump (B1) provided aeration from the bottom at a rate of 10 L/min. The circulation pump (P3) continuously cycled the biogas slurry from the storage chamber into the filtration chamber. After filtration, the effluent was collected from the outlet. A negative pressure digital pressure transmitter measured the membrane outlet pressure in the range of -100 to 0 Pa. Concentrate was extracted via water pump (P2), and the membrane was cleaned through backwashing using pump (P1). The system operated for 24 h with a 10-min filtration cycle followed by a 2-min pause. The ammonia-stripped biogas slurry (SBS) generated from the System I with different airflow rates was tested. The TAN, TN, TP, and SCOD were measured of the effluent and concentrate every 4 h. Raw and centrifugal biogas slurry as well as partial effluents and concentrates from treatment and control groups were studied of its humic acid, fulvic acid, Cl^-, K^+, and the three-dimensional excitation-emission matrix (3D-EEM) fluorescence spectra. The optimal airflow rate of 40 min/L was determined from the System I and System II, which was used for subsequent batch tests and the complete "ABC process".

Batch experiments were conducted in flasks (100 mL working volume in 150 mL conical flasks) in order to select the appropriate biochar dose and particle size. All flasks were placed under the same conditions (37 °C, 120 rpm). Biochar dose of 5 g/L, 10 g/L and 15 g/L, and particle size groups of < 0.25 mm, 0.25-0.85 mm, and > 0.85 mm (at an addition dose of 5 g/L) were investigated. The dose screening experiment was running for 12 h and samples of 6 mL every 1 h were taken. The particle size screening experiment was running for 6 h and samples of 6 mL every 30 min were taken. All experiments were conducted in duplicate. The pH, EC, TAN, TP, and SCOD were measured after filtering the samples with 0.45 μm filter.

For the "ABC process", the kitchen waste biogas slurry was first treated by the AS process with the airflow rate of 40 min/L and working time of 20 h. Other experimental conditions were the same as those of the System I. After that, the SBS was adsorbed using biochar, and 0.25-0.85 mm biochar with 5 g/L addition was optimum according to batch tests. The adsorption of SBS was carried out in 5 L Erlenmeyer for 120 min at 37 °C and 100 rpm. Then, SBS passed through 0.180 mm and 0.125 mm sieve successively and sent to the CMF device for treatment. The operating conditions of the CMF process were the same as those of System II, and the experiment was conducted for 20 h. 10 mL of the effluent and concentration was taken to measure TAN, TN, TP, and SCOD every 4 h.

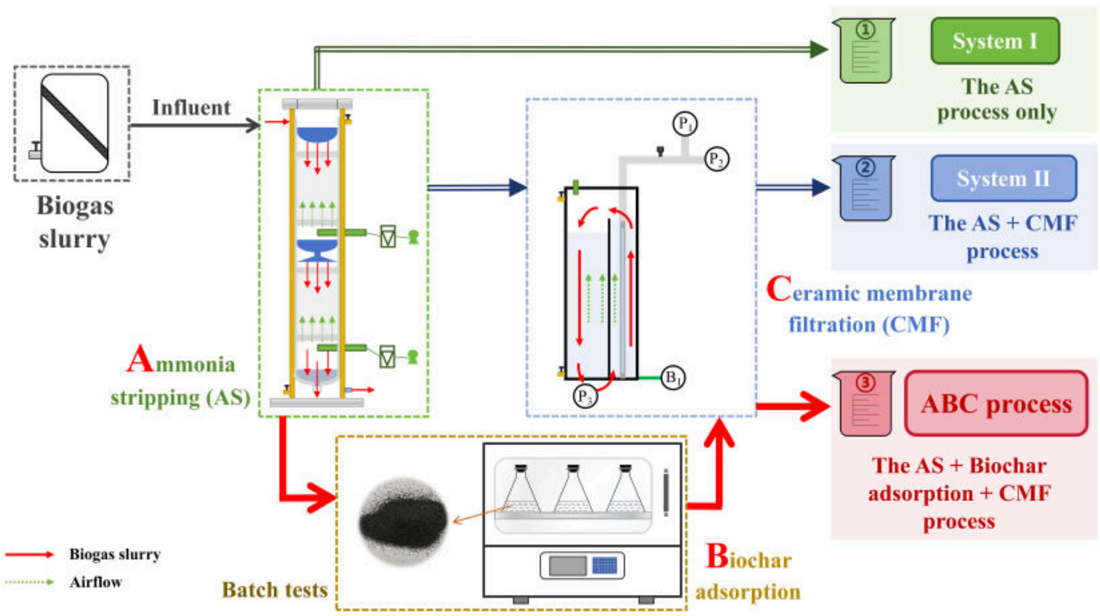


Fig.1. Process flow chart of biogas slurry by ammonia stripping combined with membrane filtration and biochar adsorption

2.3 Preliminary cost analysis

In order to lay a foundation for the large-scale treatment, the cost consumption associated with different ammonia stripping treatment processes in this study were estimated. Formula (1) used for the calculation was adapted from the study of Brasil et al. (2021), as follows:

$$C = C_R + C_P + (C_B) \quad (1)$$

Where C is the total cost of treating per unit volume of biogas slurry (CNY/m^3), C_R is the cost of added reagents (CNY/m^3), C_P is the energy consumption cost of system operation

(CNY/m^3), and C_B is the cost of biochar addition (CNY/kg). The cost of removing unit mass NH_4^+-N is shown in formula (2):

$$C' = \frac{C}{C_t - C_0} \quad (2)$$

Where C' is the total cost of treating per unit mass of NH_4^+-N (CNY/kg), C_0 is the initial concentration of NH_4^+-N (mg/L), and C_t is the concentration of NH_4^+-N (mg/L) after treatment.

The cost of reagents used in the experiment primarily stems from the use of $Ca(OH)_2$ for the pH adjustment and H_2SO_4 and NaOH for the acid absorption process. At the same time, it was easy to bubble after adding $Ca(OH)_2$, and 1 drop of defoaming reagent (0.08 mL) was added per liter of biogas slurry to reduce this phenomenon. Specific parameters related to the reagent cost were shown in Table 1. The energy consumption in the biogas slurry treatment process mainly included the maintenance system temperature (water bath, 1800 W), gas pump A (20 W), gas pump B (14 W) and peristaltic pump (30 W). Relevant process operating conditions were: reaction temperature of 37°C, air flow rate of 20 L/min for 8 h and 40 L/min for 12 h in ammonia stripping process, membrane filtration aeration rate of 10 L/min for 24 h. The electricity fee was calculated according to the average value of 0.4883 CNY/ (kW·h) in Beijing, China.

Table 1 Reagent cost per litre of biogas slurry ammonia stripping treatment

Reagent	Unit-price	Purity/Concentration	Dosage
$Ca(OH)_2$	50 CNY/kg	99%	8.7 g
NaOH	58 CNY/kg	99%	3.0 g
H_2SO_4	38 CNY/L	99%	11.12 mL
Defoaming reagent	32 CNY/L	10%	0.08 mL

2.4 Chemical analytic methods

Sample pH and EC values were measured using an acidometer (FE20, Switzerland) and an conductivity meter, respectively. The water quality indexes, including TN, TAN, TP, SCOD were determined by national standards using the UV-Vis spectrophotometer. TN was measured through alkaline potassium persulfate digestion UV spectrophotometric method (HJ 636-2012). TAN was measured through Nessler's reagent spectrophotometry (HJ 535-2009). TP was measured through ammonium molybdate spectrophotometric method (GB 11893-89). SCOD was measured through fast digestion-spectrophotometric method (HJ/T 399-2007). K^+ and Cl^- was measured by flame atomic absorption spectrophotometry (GB 11904-89) and silver nitrate titration method (GB 11896-89), respectively. Humic acid and fulvic acid were measured by capacity titration method with standards of NY/T 3162-2017 and NY/T 3162-2017, respectively. All drugs used in measure were in guaranteed reagent grade. Dissolved organic matter (DOM) changes were determined using the three-dimensional excitation-emission matrix (3D-EEM) fluorescence analyzer (AquaLog, HORIBA, Japan) with the excitation wavelength (EX) of 250-450 nm and the emission wavelength (EM) of 300-550 nm.

2.5 Data analysis

SPSS 26.0 software was used for one-way analysis of variance (one-way ANOVA) with a p value of 0.05 as the significance level. Microsoft Office Excel 2021 was used for statistical calculation and Origin 2022 was used for mapping.

The removal efficiency of TN, TAN, TP, SCOD was measured by the following formula (3):

$$\eta_A = \frac{C_{A_0} - C_{A_t}}{C_{A_0}} \times 100\% \quad (3)$$

Where η_A is the removal efficiency of substance A (%), C_{A_0} is the initial mass content of substance A (mg/L or g/L), C_{A_t} is the mass content of substance A at time t (mg/L or g/L).

The TN, TAN, TP, SCOD removal per biochar unit mass was measured by the following formula (4):

$$\eta'_A = \frac{C_{A_0} - C_{A_t}}{C_B} \times 100\% \quad (4)$$

Where η'_A is A removal per biochar unit mass (mg/g or g/g), C_{A_0} is the initial mass content of substance A (mg/L or g/L), C_{A_t} is the mass content of substance A at time t (mg/L or g/L), C_B is the amount of biochar added (g/L).

3. Results and Discussion

3.1 Airflow rate and pH optimization of ammonia stripping (AS) process

3.1.1 System I: AS process

pH and flow rate significantly affect the efficiency of AS process. The pH slightly increased to approach 9 after pre-stripping for 8 h (Fig. 2a). AS process is based on gas-liquid mass transfer, driver by the partial pressure difference between the free ammonia in the liquid phase and the ammonia in the stripped gas phase. This pressure difference causes the ammonia to volatilize from the liquid into the gas stream, facilitating its removal from the solution (Palakodeti et al., 2022). Therefore, creating an alkaline environment (10-11) that promotes the conversion of ammonium ions to free ammonia would enhance the removal efficiency (Palakodeti et al., 2021). To ensure an optimal condition for AS, $Ca(OH)_2$ was added after pre-stripping to adjust the pH to 10.8. During the AS treatment, OH^- was continuously consumed, resulting in a pH decrease. In oder to keep a favorable condition for ammonia dissociation (pH greater than 9.26 at 37 °C) (dos Santos et al., 2020), the airflow rates of 10 and 40 L/min, which resulting in pH values of 9.97 and 9.32 at 20 h, respectively, were chosen. Additionally, during the AS process, EC value kept stable.

The removal of total ammonia nitrogen (TAN) increased with the rise of airflow rates (Fig. 2b). At 20 h, the content of TAN decreased from 2540.12-2642.45 mg/L to 595.78-345.63 mg/L. The TAN removal efficiency of the 40 L/min group was 86.61%, which was significantly higher than that of the 10 L/min group and other investigations (up to 81.7%) (Değermenci & Yildiz, 2021). Due to the high solubility of ammonia in liquids, the majority of mass transfer resistance in the AS process occurs on the air-film side (Zangeneh et al., 2021). By increasing the airflow rate, this resistance can be reduced to promote the stripping of ammonia in wastewater (Zhao et al., 2015). Therefore, the most efficient removal was observed in the 40 L/min treatment.

Due to the relatively low remaining concentration of TAN (357 mg/L) after treatment, further increasing airflow rate may not significantly increase the removal efficiency but lead to a big rise of cost. Therefore, the optimal airflow rate of 40 L/min is decided.

The AS process didn't promote any total phosphorus (TP) removal in this study, consistent with findings from other studies that indicate the AS process has a limited effect on P removal (Wu & Vaneeckhaute, 2022). The observed TP removal of up to 23.6% at 20 h was mainly due to the addition of $Ca(OH)_2$, which generated phosphate precipitation in alkali environments. Besides, the increased flow rate also led to an improved soluble chemical oxygen demand (SCOD) removal (Fig. 2d), which reached 23.6% removal with 40 L/min flow rate.

In summary, the results suggested an airflow rate of 40 L/min as optimum for TAN removal, while also yielding relatively lower TP and SCOD levels in the effluent.

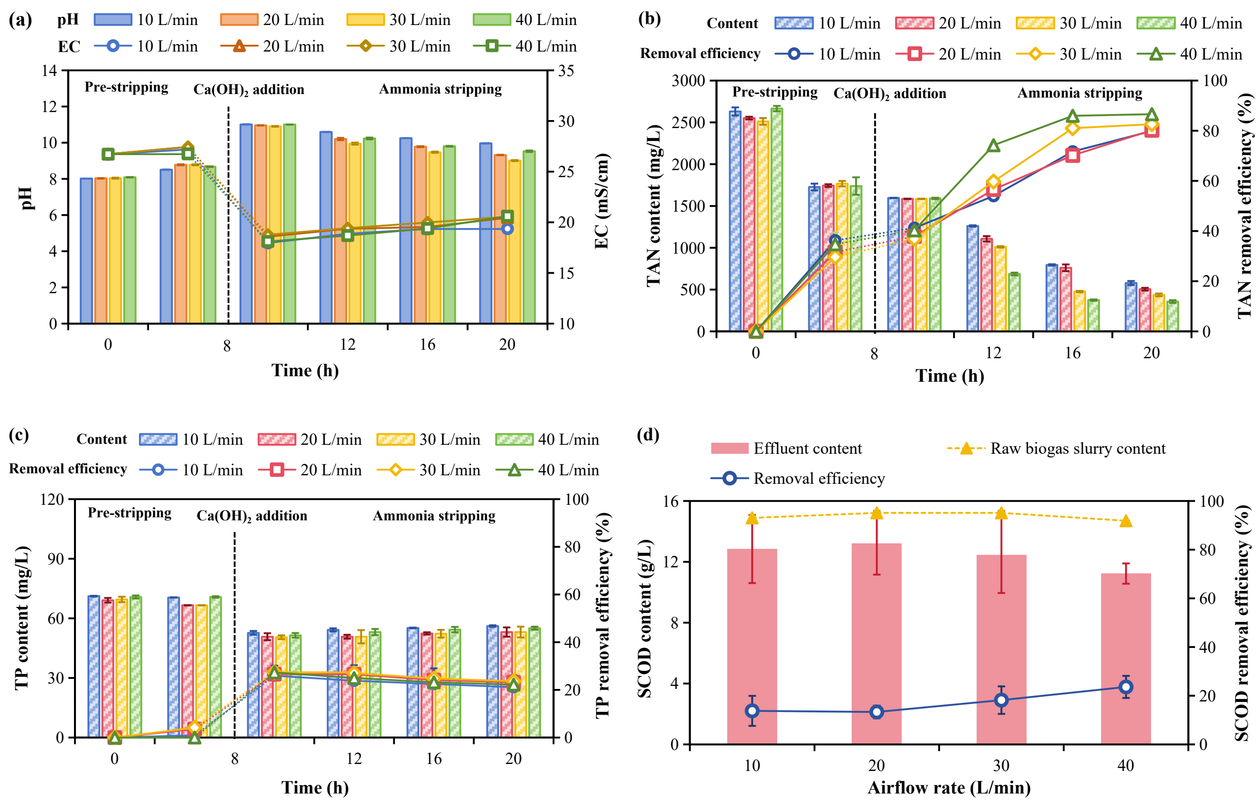


Fig. 2. Variation of pH and EC (a), content and removal efficiency of TAN (b), TP (c) with time; the average content and removal efficiency of SCOD (d) under different airflow rates

* The average data of SCOD were based on the relatively stable performance obtained after 16 h.

3.1.2 System II: AS+ceramic membrane filtration (CMF) process

System II was conducted to further investigate the optimal removal efficiency of the CMF process for treating ammonia-stripped biogas sludge (SBS) with different airflow rates. As shown in Table 2, after AS process, CMF performed efficiently in TP removal (up to 88%). Phosphorus could exist as a colloid form in high pH or high ionic content solutions. Thus, the colloidal phosphorus can retain to the microfiltration ceramic membrane with a pore size range of 0.02-0.2 μm, which significantly contributed to the TP removal (Liu et al., 2024). Meanwhile, due to the limitation of pore size, only organic particles with larger size could be filtered by CMF, leading to a SCOD removal efficiency up to 62.18%. However, the further treatment with

CMF didn't efficiently promote further removal of TAN in the effluent, which is consistent with previous studies, showing a less than 20% TAN removal using micro-filtration membrane (Waeger et al., 2010). This may be due to the pore size of ceramic membrane employed in this study, which was 0.1 μm, which cannot filter out the ionic substances in SBS (Alengebawy et al., 2021). A higher removal efficiency of TN than TAN, up to 61.4-63.5%, was observed. The large molecular organic nitrogen in TN may be efficiently adsorbed onto the microfiltration-grade ceramic membranes, leading to higher TN removal than TAN (Liu et al., 2024). Therefore, a combination with further treatment is needed to remove contaminants thoroughly and protect the ceramic membrane.

Table 2 The average content of TN, TAN, TP and SCOD in membrane influent, effluent and concentrate using SBS under different airflow rates

Parameter	Unit		Airflow rates of SBS (L/min)			
			10	20	30	40
TN	mg/L	Influent	1001.09 ± 0.00a	920.87 ± 33.10b	881.14 ± 4.70b	850.73 ± 11.00b
		Effluent	386.73 ± 19.85a	355.34 ± 125.77a	320.81 ± 8.10a	323.95 ± 5.13a
		Concentrate	1297.04 ± 8.88a	1303.32 ± 0.00a	1158.92 ± 0.00b	1297.04 ± 1.50a
TAN		Influent	578.72 ± 1.50a	504.82 ± 0.00b	436.59 ± 3.60c	340.00 ± 1.50d
		Effluent	322.82 ± 4.50a	224.92 ± 19.69c	247.21 ± 1.59b	248.00 ± 8.01b
		Concentrate	448.58 ± 11.26a	349.03 ± 2.25a	341.92 ± 18.01c	386.49 ± 0.00b
TP		Influent	56.14 ± 0.58a	53.10 ± 0.27b	53.10 ± 0.00b	55.03 ± 0.39a
		Effluent	7.34 ± 0.00a	6.37 ± 0.39b	6.76 ± 0.39b	7.34 ± 0.00a
		Concentrate	92.30 ± 2.18ab	88.83 ± 2.73ab	87.28 ± 3.82b	98.48 ± 5.46a
SCOD	g/L	Influent	12.84 ± 0.08b	13.59 ± 0.04a	13.45 ± 0.09a	11.73 ± 0.04c
		Effluent	5.52 ± 0.21c	5.14 ± 0.32c	6.74 ± 0.33a	6.14 ± 0.35b
		Concentrate	22.21 ± 0.21a	21.68 ± 0.32a	20.78 ± 0.11b	21.98 ± 0.00a

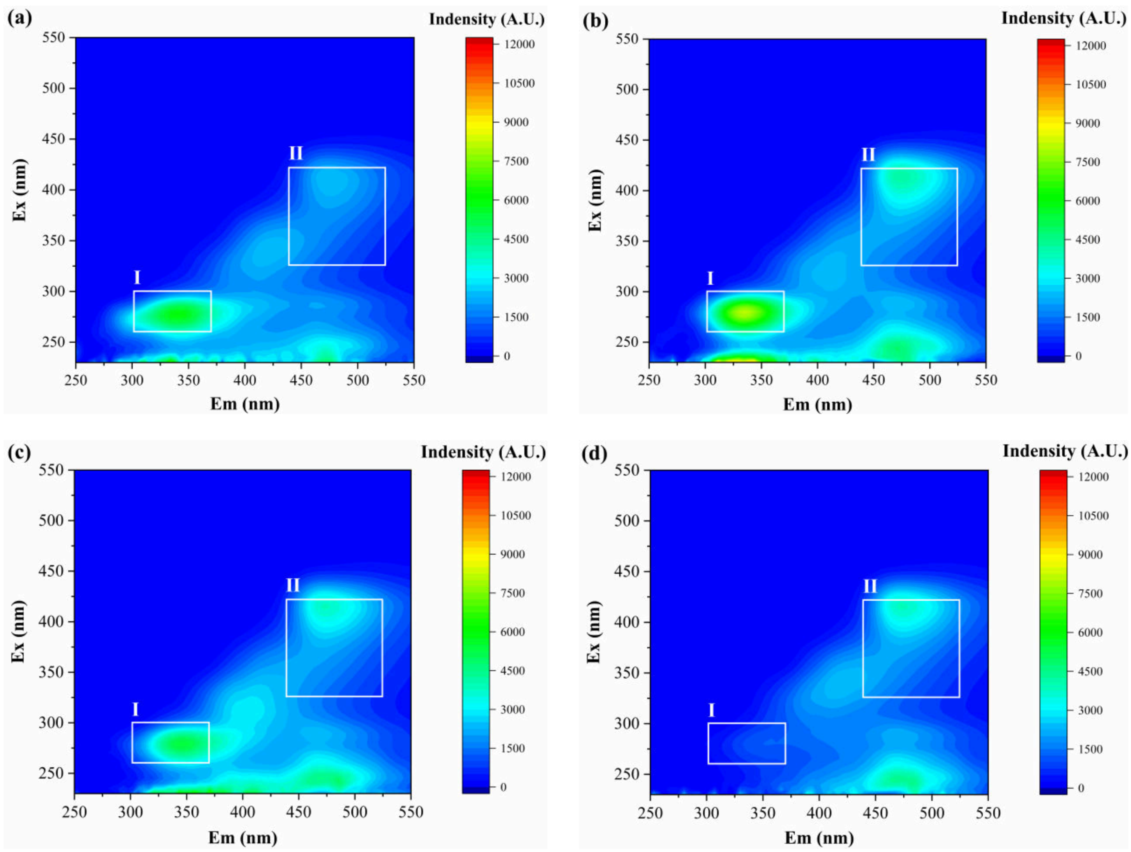
* SBS, stripped biogas slurry.
* The average data were based on the relatively stable performance obtained after 20 h.

Fig. 3 shows two peak at excitation/emission wavelengths (Ex/Em) of 294 nm/335 nm and 276 nm/315 nm in region I, which were reported to be protein-like peaks associated with tyrosine-like substances (Ex/Em = 294 nm/335 nm) and tryptophan-like substances (Ex/Em = 276 nm/315 nm) (Wan et al., 2012). The peaks appearing in region II with Ex/Em = 327 nm/410 nm and Ex/Em = 417 nm/470 nm, were defined as humic substances (Li et al., 2024).

Tyrosine and tryptophan are common amino acids that were frequently found in biogas slurry (He et al., 2023). The decomposition of abundant organic matters in kitchen waste during anaerobic digestion produced tyrosine-like, tryptophan-like substances and humus substances (Xiao et al., 2021). With the treatment of biogas slurry, the intensity of the peaks in the 3D-EEM spectroscopy changed, indicating that the DOM in biogas slurry was altered. Especially compared with the original biogas slurry, the peak in region I of the CMF effluent after 25 h significantly weakened, indicating that the ceramic membrane had a good effect on the treatment of protein-like substances.

Table 2 demonstrates the specific content changes of humic acid, fulvic acid, Cl^- and K^+ during CMF process. Both humic acid and fulvic acid contents in the effluent of CMF process were lower than that in SBS, indicating that microfiltration-grade ceramic membranes could retain some humic substances. Moreover, compared to SBS, the humic acid and fulvic acid contents in the concentrate of CMF process increased by 1.16 and 1.73 times, respectively. Humic substances were widely used not only as fertilizers but also to improve soil quality and regulate crop growth (Bezuglova & Klimenko, 2022). Several studies have been conducted to support the availability of the humus-rich membrane filtration concentrate for agricultural use after processing (Khouni et al., 2020; Suwaileh et al., 2020). Due to its high organic matter and salt content, KW biogas slurry commonly contains high levels of K^+ and Cl^- (Zhang et al., 2024). During the AS process, K^+ and Cl^- content rose due to water evaporation, and slightly declined again after the CMF treatment.

In a word, the CMF technology had the best treatment effect on TP and protein substances, and concentrated humic acid and fulvic acid in the biogas slurry. However, the ceramic membrane had a mediocre effect on SCOD, TN, and TAN, and required coupling with other processes for pre-treatment.



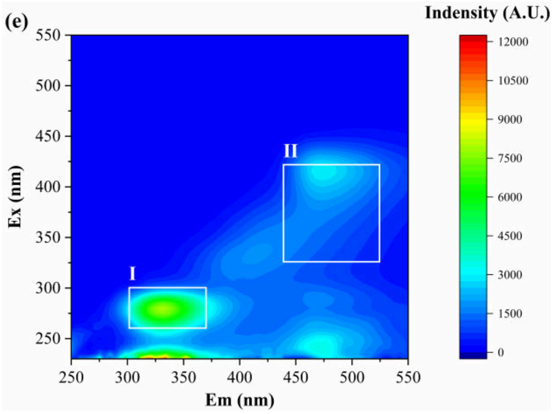


Fig. 3. Excitation emission matrix (EEM) fluorescence spectra of raw biogas slurry (a), SBS (b), effluent after CMF process for 5 h (c) and 24 h (d), concentrate after CMF process (e)

* Ex, excitation wavelengths; Em, emission wavelengths.

* Fractions C1 (Ex/Em = 327 nm / 410 nm) and C3 (Ex/Em = 366 nm / 435 nm) were categorized as humic acid-like fractions; and fractions C2 (Ex/Em = 417 nm / 470 nm), C4 (Ex/Em = 294 nm / 335 nm), and C5 (Ex/Em = 276 nm / 315 nm) belonged to the fulvic acid-like fractions, tyrosine fractions and tryptophan fractions, respectively.

* SBS, stripped biogas slurry; SBS with an airflow rate of 40 L/min was selected for testing in this section.

* CMF, ceramic membrane filtration.

Table 3 The change of humic acid, fulvic acid, Cl^-, and K^+ during the CMF process coupled with ammonia stripping

		Mass fraction (%)		Mass concentration (mg/L)	
		Humic acid	Fulvic acid	Cl^-	K^+
Raw biogas slurry		0.64 ± 0.01	0.11 ± 0.01	4415.31 ± 44.29	1394.00 ± 8.03
Centrifugal biogas slurry		0.94 ± 0.06	0.11 ± 0.01	/	1262.57 ± 73.81
SBS		1.44 ± 0.01	0.15 ± 0.00	5310.85 ± 88.36	1762.57 ± 65.78
Effluent of CMF	5 h	1.39 ± 0.00	0.13 ± 0.04	5217.13 ± 132.54	1601.14 ± 37.10
	24 h	1.39 ± 0.00	0.11 ± 0.00	5217.13 ± 44.18	1629.53 ± 3.06
Concentrate of CMF		1.67 ± 0.00	0.26 ± 0.00	5123.41 ± 265.08	2095.57 ± 55.72

* CMF, ceramic membrane filtration.

* SBS, stripped biogas slurry; SBS with an airflow rate of 40 L/min was selected for testing in this section.

3.2 Biochar dosage and particle size optimization

3.2.1 Biochar dosage effect

Optimizing the biochar dosage is crucial for wastewater treatment, as the removal efficiency typically correlates with the dosage applied (Ye et al., 2022). As shown in Table 4, the addition of varying biochar concentrations didn't significantly impact the pH (a slight decrease from 9.27 to 8.99). pH significantly affects the adsorption of TAN by biochar. Studies indicate a pH range within 7-9 is optimal for TAN adsorption (Zhang et al., 2021). The main removal mechanism was the electrostatic interaction between the oxygen-containing functional groups (-OH, -COOH) on the surface of biochar and NH_4^+-N in TAN (Gu et al., 2023). pH lower than 7 may lead to H^+ competing with NH_4^+ adsorption, while, higher pH than 9 leads to TAN in form of $NH_3 \cdot H_2O$, and thus, also with a release of NH_3 gas into the atmosphere, as well as a reduced electrostatic adsorption (Palakodeti et al., 2021). Therefore, the biogas slurry conditions after the AS process were suitable for the removal of TAN using biochar.

The addition of biochar led to enhanced removal of nitrogen, phosphorus, and SCOD in the biogas slurry, stabilizing after 180 min. Specifically, different dosages of biochar result in similar final TAN levels (269.4-275.6 mg/L, Fig. 4b), however, 5 g/L biochar addition showed the best removal capacity (up to 8.83 mg TAN/g biochar) among all. This is consistent with previous studies (Gu et al., 2023; Su et al., 2022). Biochar mainly remove TAN through surface electrostatic interaction and cation exchange (Ye et al., 2022). However, excessive adsorbent mass could lead to a decrease in the unsaturation of ion exchange sites, resulting in a decline in the number of exchange sites per unit mass (Zhang et al., 2021). Considering efficiency and economy, the appropriate amount of biochar added to remove TAN was 5 g/L in this experiment. TN removal by biochar showed similar trend with TAN (Fig. 4c). When the mass of biochar added was 5 g/L, the highest TN removal per unit mass of 13.17 mg/g was achieved (Fig. 4d). Even with less adsorption capacity than NH_4^+, biochar also adsorbed NO_3^- in SBS. The relatively high pH of SBS generated excessive OH^-,forming electrostatic repulsion on the surface, resulting in a smaller amount of NO_3^- adsorption (Wang et al., 2020). Meanwhile, negative salt ions, such as Cl^- and SO_4^{2-}, could also affect the adsorption of NO_3^- (Hua et al., 2022). In addition to inorganic nitrogen, kitchen waste biogas slurry also contained abundant organic nitrogen, including protein, thiamine, urea (Liu et al., 2023). The mechanisms of biochar adsorption of organic nitrogen are still unclear, however, the pore size of biochar (mostly above 9 nm) indicates a conducive potential of organic nitrogen adsorption (Guo et al., 2020).

Unlike nitrogen, the adsorption effect of biochar on TP in SBS was weak. The the maximum removal capacity of 0.81 mg/g was achieved with 5 mg/L biochar addition (Fig. 4f). Phosphates mainly exist in the form of $H_2PO_4^-$ and HPO_4^{2-} at relatively low and high pH conditions, respectively . Due to the lower adsorption free energy of $H_2PO_4^-$ compared to HPO_4^{2-}, $H_2PO_4^-$ is more easily adsorbed (Chowdhury & Yanful, 2010). The high pH value of SBS in this experiment may lead to HPO_4^{2-} being dominant and not easily removed by biochar. In addition, the competition of OH^- on the surface of the adsorbent was also a reason why phosphate was difficult to adsorb (Zhang et al., 2021). The removal of SCOD was similar to TP, with a maximum removal efficiency of 0.53 mg/g which achieved when the addition of 5 mg/L biochar (Fig. 4h). The main SCOD removal mechanism was based on chemical absorption, and the optimal additive amount of biochar was 5 mg/L (Akay, 2021).

In summary, the mild alkaline effluent of biogas slurry from AS process generated an ideal condition for combining biochar to remove pollutants. Results indicated an optimal addition dosage of 5 mg/L, with significantly improved removal efficiency on TAN and TN.

Table 4 The changes of pH during biochar adsorption in different addition amounts

Time (min)	Biochar addition (mg/L)		
	5	10	15
30	9.27 ± 0.00a	9.27 ± 0.01a	9.27 ± 0.02a
60	9.21 ± 0.02a	9.21 ± 0.01a	9.22 ± 0.01a
90	9.15 ± 0.01a	9.14 ± 0.01a	9.14 ± 0.00a
120	9.10 ± 0.01a	9.09 ± 0.01a	9.09 ± 0.00a
180	9.09 ± 0.00a	9.09 ± 0.01a	9.09 ± 0.00a
360	9.04 ± 0.00a	9.04 ± 0.00a	9.04 ± 0.0a
540	9.01 ± 0.01a	9.02 ± 0.01a	9.16 ± 0.20a
720	8.99 ± 0.03a	8.99 ± 0.02a	8.99 ± 0.02a

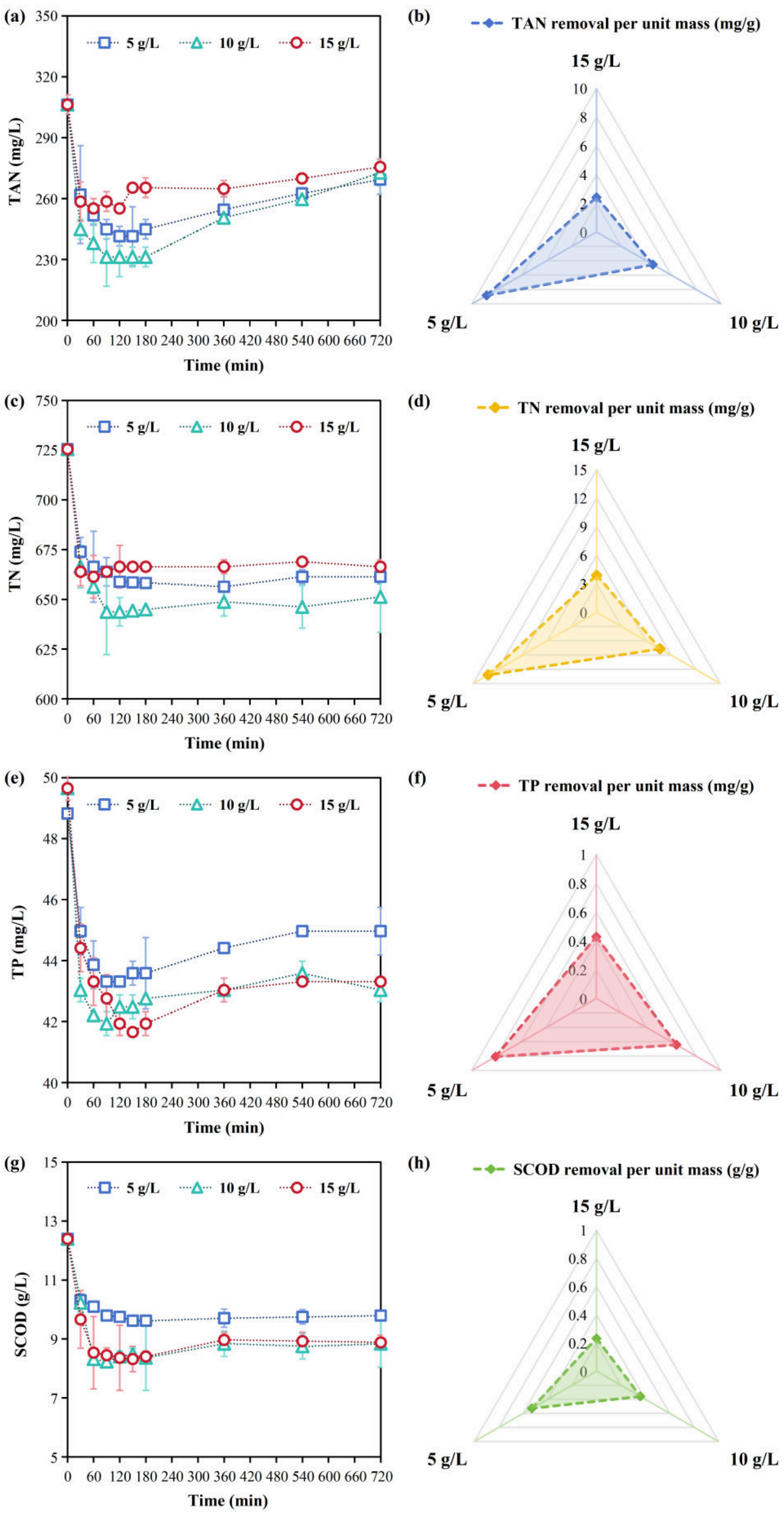


Fig. 4. Changes in content with time and average removal per unit biochar mass after stabilization of TAN (a, b), TN (b, d), TP (e, f) and SCOD (g, h) under addition of different biochar amounts

* The average removal data were based on the relatively stable performance after 180 min.

3.2.2 Optimization of biochar particle sizes

The particle size influences the specific surface area, which in turn significantly determines the adsorption capacity (Wang et al., 2020). In this study, smaller particle sizes only generated a rapid removal of TN and TAN within 30 min. However, the removal efficiency approached similar levels after 180 min (12.56-13.00 mg/g and 12.17-12.39 mg/g, respectively, $p < 0.05$). While smaller particle sizes offer the advantage of a larger specific surface area for adsorption, excessively small particles can increase surface tension and viscosity, potentially reducing adsorption efficiency (Guo et al., 2020). At 180 min, the biochar with particle size between 0.25-0.85 μm generated the highest removal efficiency of TP and SCOD, which were 0.88 mg/g and 0.41 g/g, respectively (Fig. 5f, h). Thus, this investigation identified biochar with a particle size range of 0.25-0.85 μm as the optimal choice.

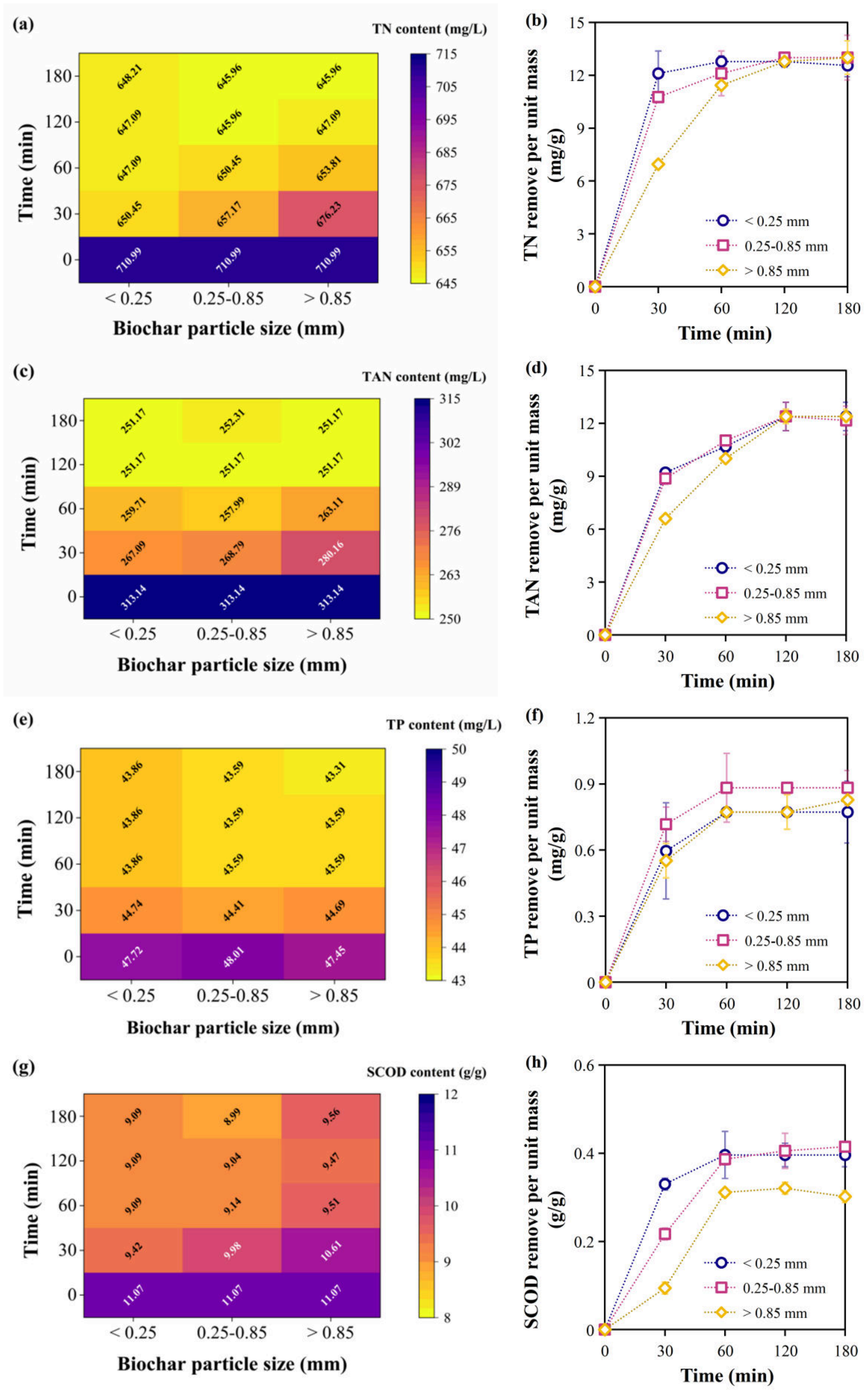


Fig. 5. Changes in content and removal per unit biochar mass with time of TN (a, b), TAN (b, d), TP (e, f) and SCOD (g, h) under addition of different biochar particle sizes

3.3 CMF efficiency of the "ABC process" for biogas slurry treatment

The CMF efficiency in “ABC process” treatment (AS+biochar adsorption+CMF process) demonstrated an overall higher removal efficiency of TN, TAN, TP, and SCOD compared to that in the System II (AS+CMF process without biochar adsorption), achieving 299.55 mg/L, 220.44 mg/L, 7.34 mg/L, and 3.55 g/L, respectively. The addition of biochar process notably improved TAN and TP removal, with final removal efficiency increased to 30.98% and 86.67%, respectively (Fig. 6b, c). For TN and SCOD in the “ABC process”, the initial removal efficiency was low with a gradual increase, ultimately surpassing the System II, reaching 61.7% and 50.5%, respectively. Alengebawy et al. (2021) also showed that the removal of TN, TAN and TP from the CMF process of biogas slurry increased with the rise of biochar addition and reached a maximum at 5 g/L. This may be due to the addition of trace biochar changed the surface property of the ceramic membrane (Xia et al., 2023). Although the SBS after biochar adsorption was filtered prior to the CMF process, a portion of small particle size (<0.125 mm) biochar may still enter the system. The biochar attached to the membrane surface increased the contaminant adsorption sites, surface charge, roughness and hydrophilicity of the CMF system, which improved the efficiency of membrane filtration (Yang et al., 2024). At the same time, the biochar filtered a portion of the suspended and particulate matter, which reduced the fouling layer, slowed down membrane contamination, and increased membrane life (Su et al., 2022).
The flow directions of pollutants in the CMF process of SBS in “ABC process” and System II process was displayed in Table 5. The content of TN, TAN, and SCOD in the effluent and concentrate flowed towards biochar adsorption, with SCOD accounting for the largest proportion. However, the proportion of TP in the effluent and concentrate increased after the addition of biochar. It may be due to the physical processes such as adsorption oscillation of biochar or aeration that disrupt the structure of SBS residual sludge, leading to the release of phosphorus from the solid phase to the liquid phase (Yu et al., 2021). In addition to the effluent and concentrate of the CMF process, a considerable portion of nitrogen, phosphorus and SCOD remained in the sludge to be cleaned by elution or lost by evaporation due to aeration. The addition of biochar reduced the proportion of this part from 11.6%-39.98% to 5.07%-21.43%. The matters adsorbed by biochar can be recovered through pyrolysis, gasification, or acid treatment, reducing resource waste (Yu et al., 2020).

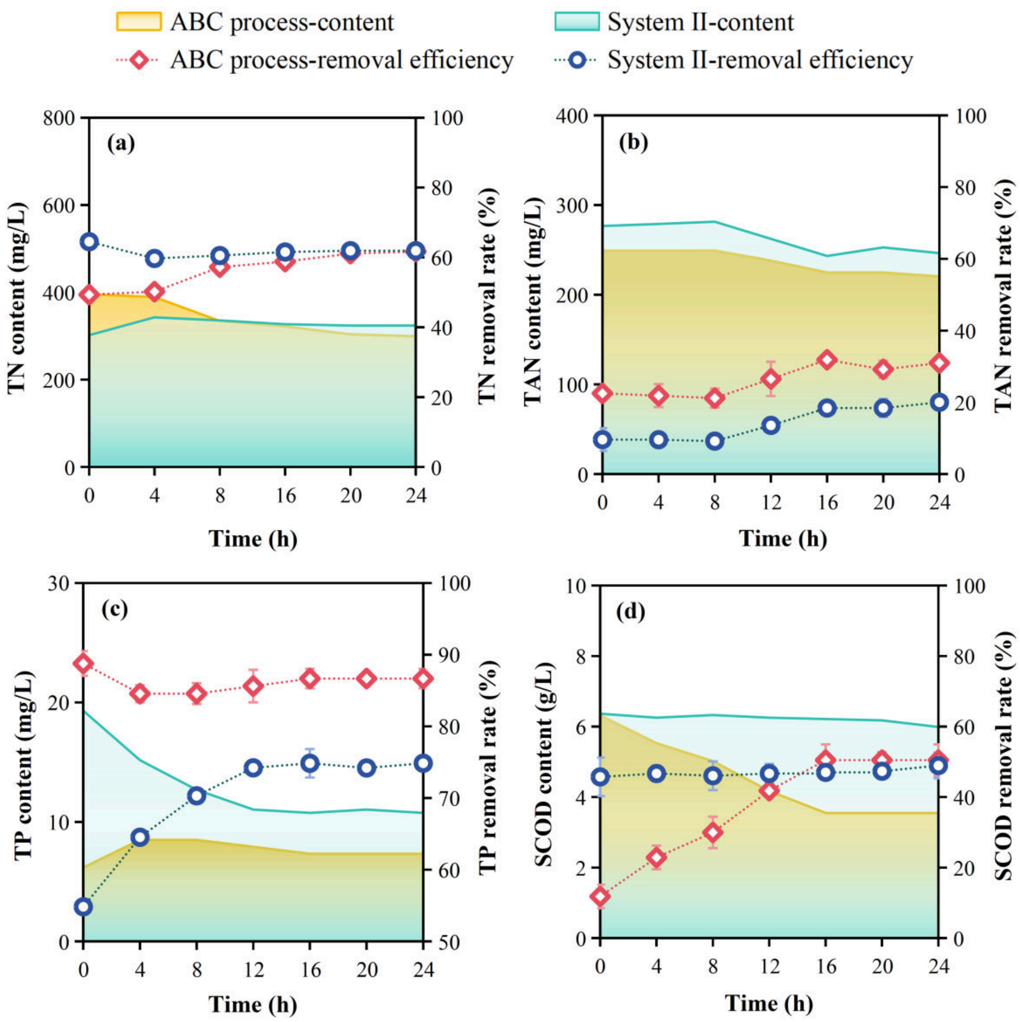


Fig. 6. Variation of TN (a), TAN (b), TP (c), SCOD (d) content and removal efficiency in effluent of the CMF process in the "ABC process" and System II

* TN, total nitrogen; TAN, total ammonia nitrogen; TP, total phosphorus; SCOD, soluble chemical oxygen demand.

* "ABC process", ammonia stripping + biochar adsorption + ceramic membrane filtration process; System II, ammonia stripping + ceramic membrane filtration treatment.

Table 5 The flow directions of TN, TAN, TP and SCOD in effluent of the "ABC process" and System II

		TN	TAN	TP	SCOD
		%			
System II	Effluent	23.07	47.42	8.44	32.18
	Concentrate	45.74	34.10	51.58	56.22
	Sludge + aeration volatilization	31.19	18.48	39.98	11.60
"ABC process"	Effluent	24.41	46.18	14.59	27.69

Concentrate	44.31	29.76	59.33	40.06
Sludge + aeration volatilization	21.43	5.07	11.72	5.50
Biochar adsorption	9.85	18.99	14.36	26.75

* TN, total nitrogen; TAN, total ammonia nitrogen; TP, total phosphorus; SCOD, soluble chemical oxygen demand.

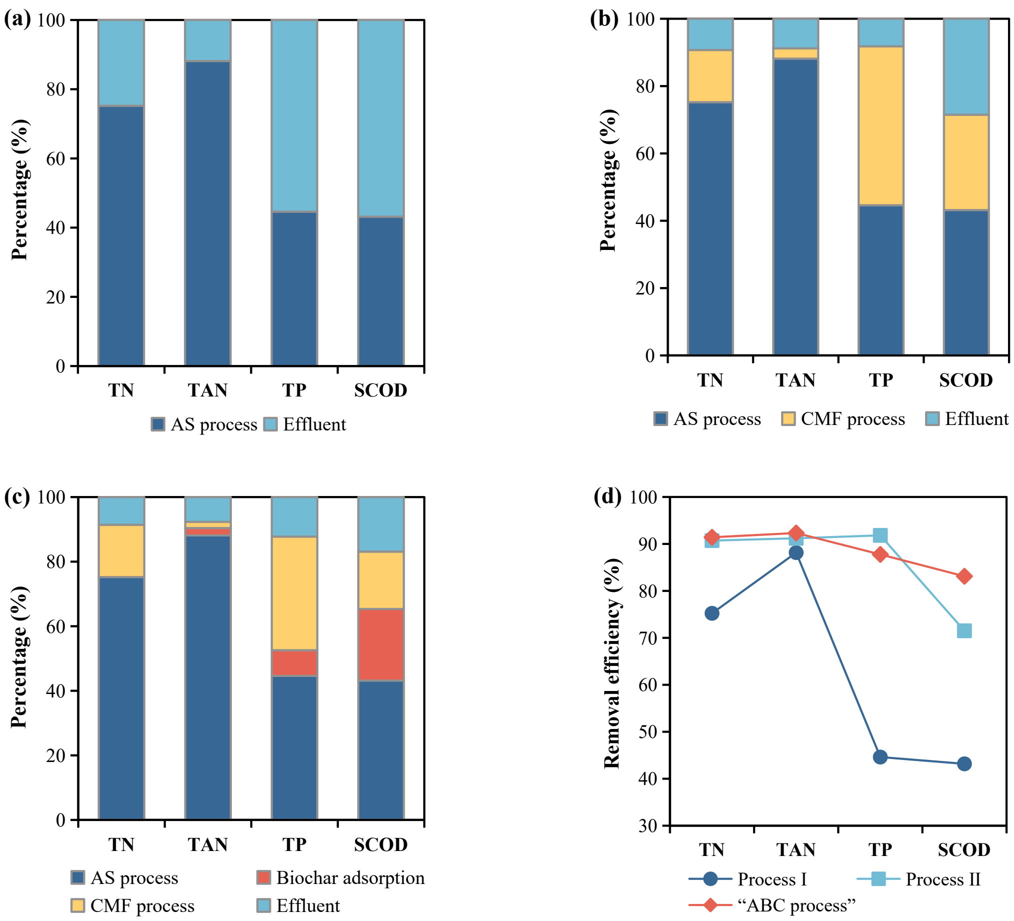
* "ABC process", ammonia stripping + biochar adsorption + ceramic membrane filtration process; System II, ammonia stripping + ceramic membrane filtration treatment.

3.4 Analysis of the whole ammonia stripped biogas slurry treatment system

As shown in Fig. 7, the proposed "ABC process" demonstrated the best overall removal for TN (91.42%), TAN (92.33%), TP (87.76%), and SCOD (83.11%), compared to the two control systems, System I and System II. The efficiency of the "ABC process" was better than many studies (Alengebawy et al., 2021; Antonini et al., 2011; Palakodeti et al., 2021). Among the processes, AS primarily facilitated TAN removal (up to 88.15%), while CMF mainly targeted on TP removal. Biochar adsorption contributed to the removal of all pollutants, with the greatest impact on SCOD.

The removal efficiency of TN and TP in both the "ABC process" and System II were increased, indicating the membrane filtration as the main process for TN and TP removal. The oscillation caused by biochar addition led the dissolution of solid phosphorus in the sludge, resulting in a decrease in the TP removal efficiency of the "ABC process". For SCOD, the removal efficiency in the "ABC process" was maximized due to the addition of biochar, fully realizing nutrient recovery and turning waste into treasure. In total, the system of the "ABC process" had the best kitchen waste biogas slurry treatment effect. The turbidity and odor also decreased during the process. Moreover, the biochar in this experiment was unmodified and the ceramic membrane was only micro-filtration level. In subsequent studies, the process can be further improved or coupled with other processes, such as microalgae treatment, to further treat the effluent to obtain cleaner products and more complete resource recovery.

Since the effluent has different qualities, the cost per unit NH_4^+-N mass was converted and compared (Fig. 7e). With the increase of processing steps, the removal efficiency of NH_4^+-N increased, but the cost per unit NH_4^+-N mass also rose. In particular, the treatment cost of the "ABC process" reached 8.51 CNY/kg, which was lower than a part of studies in wastewater ammonia nitrogen treatment process (Astals et al., 2021; Zhou et al., 2024). At the same time, according to the cost distribution of different parameters, biochar adsorption accounted for the largest proportion, more than 50% of the whole treatment process. Notably, the cost calculation of this study was limited to the laboratory level. In practical application, improvements can be applied to reduce costs, such as the replacement to industrial grade hydrated lime (0.2 CNY/kg). The byproducts obtained by the process can also be reused to generate positive economic benefits and further offset the costs (Fig. 7f). This study found that the combination process recovered most of the nitrogen and phosphorus in the biogas slurry and treated a portion of SCOD. The NH_4^+-N concentration in the effluent was significantly reduced, and the risk of ammonia volatilization and nutrient loss during the returning field process was reduced (Provolo et al., 2017). Due to the decrease in liquid volume after treatment, the concentration of Cl^-, K^+, and large molecular organic compounds such as humic acid increased, which could improve the organic environment of soil (Table 3). At the same time, the ammonium sulfate produced by producing ammonia gas had a good fertilizer effect (Melgaço et al., 2020). Considering the price of 230 USD per ton, based on the data in September 2022), the treatment in each ton of biogas slurry can generate approximately 2.5 USD (about 18 CNY) in economic value from the obtained ammonium sulfate in this study (Xu et al., 2023). Furthermore, biochar after adsorption can also be used as soil amendments improving the soil granular structure. Its carbon fixation capacity could help to achieve the goal of carbon peaking and carbon neutrality (Yu et al., 2020).



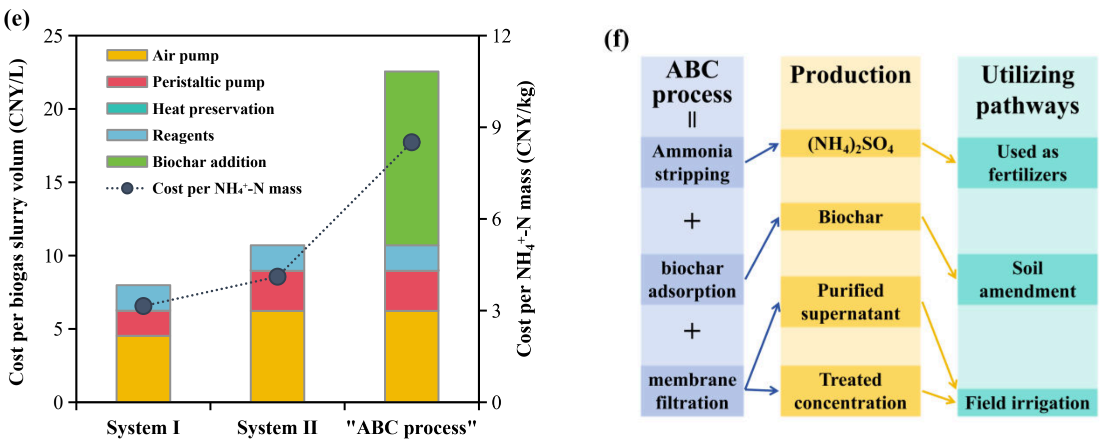


Fig. 7. The proportion of TN, TAN, TP, SCOD removal effect in each step of system I (a), process II (b), the “ABC process” (c); the final removal efficiency of TN, TAN, TP, SCOD from raw slurry biogas in the whole treatment of three systems (d); composition of the primary cost analysis in different processing systems (e); different process products and resource utilization ways (f)

* AS, ammonia stripping; CMF, ceramic membrane filtration.

* TN, total nitrogen; TAN, total ammonia nitrogen; TP, total phosphorus; SCOD, soluble chemical oxygen demand.

* “ABC process”, AS + biochar adsorption + CMF process; System I, AS only; System II, AS + CMF treatment.

4. Conclusion

This study demonstrated that the novel “ABC process” of the ammonia stripping (AS) + biochar adsorption + ceramic membrane filtration (CMF) process had the best kitchen waste biogas slurry treatment effect. As a result, the “ABC process” possessed the relatively best TN, TAN, TP, and SCOD removal efficiency of 91.42%, 92.33%, 87.76%, and 83.11%, respectively. Among them, the AS process mainly removed TAN, and the CMF process had the highest removal efficiency for TP. Biochar can adsorb SCOD in the biogas slurry and changed the surface structure of the ceramic membrane to increase the working efficiency and reduce the membrane contamination. Meanwhile, the turbidity and odors of the digestate were greatly reduced after treatment by the “ABC process”, and clean effluent and highly nutritious concentrate were obtained. The cost analysis at the laboratory level indicated that the operational investment of this coupled system was relatively low. Furthermore, it obtained reusable by-products to protect the environment, generated positive economic benefits, and further offset costs. These findings provided basic data for optimizing the biogas slurry treatment process.

Data availability

Data will be made available on request.

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Exploration of ammonia stripping coupled adsorption-membrane filtration process for treating kitchen waste biogas slurry

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