



Ammonia recovery from ion exchange brine via hollow fibre membrane contactors: Operational insights and product formation

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ABSTRACT

Ammonia removal from municipal and industrial wastewater streams is critical to ensure environmental protection. Ion exchange (IEX) processes can selectively capture $\text{NH}_4^+\text{-N}$ from wastewater, but this involves a chemical regeneration step with brine (4 % NaOH) that if not suitably managed, can lead to high chemical costs and significant environmental impacts. To achieve efficient brine management, $\text{NH}_4^+\text{-N}$ must be recovered so the regenerant can be reused over and over. This study evaluated the feasibility of hollow fibre membrane contactors (HFMC) for $\text{NH}_4^+\text{-N}$ recovery from IEX brine to enable its reuse. The nitrogen rich brine with 1190 mg $\text{NH}_4^+\text{-N/L}$, collected from a pilot-scale IEX, was processed in two HFMC modules in series, with sulfuric acid used as the stripping solution. The HFMC operational parameters were investigated and optimised leading to 99.98 % $\text{NH}_4^+\text{-N}$ recovery efficiency. Water transport was influenced by the brine ammonia concentration: at 2000 mg $\text{NH}_4^+\text{-N/L}$, 20 % water moved to the stripping side, whereas at 200 mg $\text{NH}_4^+\text{-N/L}$, a 12 % decrease in stripping solution volume occurred. The highest mass transfer coefficient was 6.1 l/h with synthetic brine, increasing to 9.38 l/h with real wastewater brine, indicating enhanced efficiency with wastewater-derived solutions. The recovered ammonium sulphate salt closely matched standard composition and was free from organics, pathogens, and most inorganics and metals, making it suitable for various industrial applications. Overall, this two-step process concentrated $\text{NH}_4^+\text{-N}$ from 42 mg/L in secondary wastewater to 32 g $\text{NH}_4^+\text{-N/L}$ in under 10 h. The findings demonstrate HFMC feasibility for ammonia recovery from municipal wastewater, offering a sustainable, efficient, and economic solution without sludge production or greenhouse gas emissions, supporting net-zero targets and a zero liquid discharge strategy.

1. Introduction

Ammonia ($\text{NH}_4^+\text{-N}$) is an essential nutrient in fertiliser production that has a global market that is increasing around 1.5 % per year [1]. Ammonia production for fertilisers entails 1.5–2.5 % of the global energy consumption via Haber-Bosch process [1]. The fertilised crops are then processed into food products and consumed by animals and humans. Subsequently, ammonia and nitrogen-rich organics end up in industrial and municipal wastewater treatment plants. Once there, ammonia is not recovered but lost to the atmosphere as N_2 through the application of biological processes such as nitrification and denitrification. These processes are responsible for 14–26 % of the overall carbon footprint of wastewater treatment plants (WWTPs), as nitrification/denitrification produce N_2O , which is a powerful greenhouse gas and its production must be controlled in WWTPs to reach the ambitious net-zero targets [1–3]. For comparison, other unit operations such as

primary clarification and sludge handling generally contribute less than 10 % of the total carbon footprint of WWTPs [4]. Additionally, even conversion of 1 % of influent nitrogen to N_2O can increase total plant GHG emissions by ~30 % [5], highlighting the critical need to mitigate these emissions. Adding to these pressures, ammonia discharge standards are becoming more restrictive and many WWTPs will be required to meet <1 mg $\text{NH}_4^+\text{-N/L}$ limits, including small treatment plants that incorporate passive technologies, and will need a complete overhaul [6,7]. Therefore, developing an effective removal and recovery technologies that combines economic, environmental, and energy considerations is crucial to support a sustainable nitrogen cycle. A life cycle assessment conducted by Guida et al. [6] comparing ion exchange (IEX) with the conventional nitrification-denitrification process showed that IEX with brine reuse and ammonia recovery via membrane stripping reduced energy demand by 25 %, global warming potential by 66 %, and marine eutrophication potential by 62 %. Similarly, Guida et al. [6]

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found that coupling the IEX process with an anaerobic membrane bioreactor (AnMBR) reduced direct greenhouse gas emissions by 82 % compared to the biological nutrient removal process.

Despite its potential advantages, such as simple operation, high removal efficiency, and fast kinetics, especially for ammonia removal and recovery, IEX processes are not commonly used in WWTPs [8]. One of the main reasons is high chemical use of the process, as IEX media needs to be regenerated with brine solutions, such as NaOH or KCl. The feasibility of the process depends on the frequency of the regeneration step and ability to reuse the brine, to avoid the costs of its disposal as hazardous waste [8–10]. The use of the brine and its management have previously been highlighted as the key barriers for further implementation and wide use of the IEX process and it has been estimated to contribute up to 80 % of the total operating cost [11]. The ability to reuse brine multiple times in the IEX process is important to reduce chemical costs but also to minimise the environmental impact associated with the disposal of saturated brine solutions, supporting zero liquid discharge [12]. To achieve this efficient brine management and re-use, $\text{NH}_4^+\text{-N}$ must be recovered from the brine. Several processes have been proposed to recover $\text{NH}_4^+\text{-N}$ from brines, such as air stripping, steam stripping, and magnesium ammonium phosphate precipitation [12,13]. Among those membrane-based nitrogen recovery systems have gained attraction as effective alternatives to conventional nitrogen removal methods, particularly in ammonia-rich wastewater streams particularly ion exchange brines. Hollow fibre membrane contactors (HFMC) provide a promising route for ammonia recovery due to their high selectivity, low energy consumption, and modular design [14].

Recent research has extensively explored the use of hollow fibre membrane contactors (HFMC) for ammonia recovery from various wastewater sources. The integration of hybrid systems, such as HFMC coupled with zeolite adsorption, has been proposed as an effective approach for ammonium sulphate production from municipal wastewater [15–17]. Similarly, HFMC applications in anaerobic digestate treatment have demonstrated feasibility for nitrogen recovery from agricultural and municipal waste streams [18–20]. Membrane material and structural modifications were reported to improve ammonia recovery performance focusing on the use of novel membrane coatings [21–25]. The key focus is still on operational modifications to increase process efficiency [26]. Counter-current flow configurations have been shown to increase mass transfer coefficients, leading to improved recovery efficiency [27]. Different receiving solutions have also been compared, demonstrating that sulfuric acid (H_2SO_4) generally provides higher ammonia absorption efficiency than phosphoric acid (H_3PO_4) [10,28], while organic solvents (kerosene based) and non-acidic stripping solutions have been introduced as alternative absorbents, reducing water transport compared to conventional acid-base systems [29,30].

Previous studies have also demonstrated ammonia recovery from real municipal wastewater using ion exchange regeneration coupled with HFMCs. For example, Sheikh et al. [31] used a pilot-scale zeolite ion exchange system treating municipal effluent to generate regenerant brine, which was processed in HFMC units with phosphoric acid (H_3PO_4) as the stripping solution to recover ammonium phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$). Cifuentes-Cabezas et al. [32] investigated sludge centrate treatment using polypropylene HFMCs, testing multiple stripping solutions including H_2SO_4 , H_3PO_4 , and HNO_3 . Chen et al. [63] introduced a high-pH, recirculating batch process for regenerating NH_4^+ -saturated cation exchange resins, recovering ammonia through evaporation, sublimation, and deposition to yield NH_4Cl crystals. Park et al. [33] focused on boundary-layer speciation effects using synthetic feeds under controlled conditions, while Guida et al. [6] compared different ammonia recovery processes from KCl brine at lab scale, including the use of recovered ammonia for protein production. Noriega-Hevia et al. [26] developed a pH slope-based control system to optimise HFMC cycles under laboratory conditions, focusing on feed replacement timing. Schwiebert et al. [64] evaluated fouling mitigation with settling and ultrafiltration pretreatment for anaerobic digester centrate, sustaining over 90 % recovery

over ten cycles. Unlike these studies, the present work focuses on the direct ammonia recovery from pilot-scale IEX brine without additional pretreatment, optimising operational parameters to achieve almost complete recovery while characterising water transport, mass transfer coefficients, and product purity to support full-scale implementation.

However, several challenges hinder the widespread adoption of this technology, including membrane wetting, fouling, and the need for chemical additives to maintain pH stability during operation [34–36]. Membrane wetting significantly impacts mass transfer coefficients by increasing membrane resistance for ammonia transport which is related to the water transport during operation [37]. Water transport stands as a major limitation of membrane contactor technology that dilutes the receiving solution and reduces the economic value of the overall system [38]. While some studies attribute ammonia transport across the membrane to vapor pressure differences [31,36], most research explains the process as being driven by concentration gradients [39,40]. A comprehensive understanding of both ammonia and water transport mechanisms in ammonia-selective membranes is crucial for optimising operational parameters and improving overall process efficiency. Therefore, focusing on high ammonia selectivity, low water flux, and strong resistance to acidic and alkaline conditions is crucial for enhancing system performance and long-term stability [41].

This study aims to optimise operational parameters for ammonia recovery from NaOH brine produced by a pilot-scale IEX system treating secondary effluent from a full-scale WWTP. It investigates mass transfer rate and flux using synthetic brine, focusing on the effects of ammonia and acid concentrations. pH control and water transport were examined to minimise dilution and maintain stability. A comparison between synthetic and wastewater brines was completed to understand efficiency across matrices. Finally, ammonium sulphate characterisation and purity were explored to understand its reuse as a fertiliser. This manuscript offers a streamlined, net-zero-aligned recovery pathway, explains the scientific mechanisms of ammonia and water transfer, and emphasises the scalability of the technology.

2. Material and methods

2.1. Ion exchange process and brine solution

The brine tested in this study was produced in a pilot scale ion exchange process fed with secondary municipal wastewater from a full-scale wastewater treatment plant (Cranfield University, UK). Pilot scale ion exchangers (by MionTec GmbH, Germany) were operated in downflow at a flow rate of $1 \text{ m}^3/\text{day}$ and 10 min empty bed contact time (EBCT). The internal diameter and the height of the ammonia removal column was 220 mm and 1566 mm respectively. 27.78 L (30.124 kg) Zeolite-N (Nanochem Pty Ltd., Australia) filled in the column which was equipped with a nozzle in each of the end plates (0.2–2 mm) to drain the liquid from the bottom and an air vent to discard the air or gas from the top. The columns were operated until the saturation of the media. Following that, regeneration period was initiated with 10 bed volumes of brine solution, i.e., 278 L 4 % NaOH in up-flow operation for 2 h. The regenerant solution was kept in a separate tank ready to be processed by hollow fibre membrane contactors (HFMC). The actual pH of the regenerant solution was already high around 12–13 because of using 4 % NaOH to regenerate the zeolite media, therefore no pH adjustment was required prior to the experiments. Ammonia in the brine reached up to 1190 mg/L in the ion exchange unit after consecutive adsorption and regeneration cycles. The brine characterisation can be found in Table 1.

2.2. Hollow fibre membrane contactors experimental setup

Two commercial hollow fibre membrane contactors (HFMCs) (Liqui-Cel® 1.7 × 5.5 MiniModule®, Membrana GmbH, Wuppertal, Germany) in series (Fig. 1). The membrane system utilised in this work comprises two membranes connected in series. By the implementation of a series

Table 1

Characterisation of the brine obtained after treating wastewater by demonstration scale IEX plant.

Parameter	IEX Brine
pH	12.65 ± 0.6
Temperature (°C)	21.6 ± 0.8
TSS (mg/L)	Not detected
Turbidity (NTU)	0.3 ± 0.1
Ammonia (NH ₄ -N) (mg/L)	1190 ± 5.1
Calcium (mg/L)	55 ± 4.9
Magnesium (mg/L)	45.1 ± 3.3
Sodium (mg/L)	4760 ± 105.3
Silicon (mg/L)	7.2 ± 2.1
Phosphate (mg/L)	0.7 ± 0.1
Heavy metals (µg/L)	0.1–30
Conductivity (µS/cm)	2600 ± 154

configuration, it was intended to enhance ammonia recovery efficiency, thereby maximising product recovery potential. The HFMC comprised 7400 hydrophobic micro-porous propylene membrane fibres, with inner and outer diameters of 220 and 300 µm, respectively. The surface area was 0.56 m², the mean pore diameter was 0.03 µm and the effective length was 0.11 m. Sulfuric acid (H₂SO₄) solutions with a concentration of 0.1–1 M (Sigma Aldrich, UK) were used for stripping of ammonia. Concentrated NaOH (30 %) solution (Sigma Aldrich, UK) were used to raise the pH of the synthetically produced brine solutions.

The HFMCs were connected in series, and the sulfuric acid stripped the NH₄⁺-N simply by circulating the two liquids counter-currently. All experiments were conducted at fixed, optimised flow rates selected through preliminary pre-tests to ensure stable operation. The effect of flow rate was not systematically investigated as an independent variable in this study. The brine solution was pumped through the lumen side of the membranes at a flow rate of 100 mL/min (6 L/h), while sulfuric acid solution was pumped through the shell side at a flow rate of 20 mL/min (1.2 L/h) with the use peristaltic pumps (Watson-Marlow, UK) (Fig. 1).

The experiments were operated in a circulation configuration with a wastewater volume of 1 L to shorten the experimental time. The volume of the acid solution was 0.5 L. The effectiveness of the H₂SO₄ was reported previously by many authors, hence the experiments were performed with only sulfuric acid. The transport of ammonia is governed by a dual mechanism involving both concentration and partial pressure gradients (Fig. 1). At high pH conditions (>9), the chemical equilibrium shifts in favour of ammonia gas form, which is volatile and able to permeate through the microporous hydrophobic membrane. The concentration gradient drives ammonia gas molecules from the bulk feed solution toward the membrane interface. At high pH values (pH ≈ 12), the chemical equilibrium in the feed solution shifts toward the formation of ammonia gas, increasing both the dissolved ammonia and the vapor

pressure of ammonia. The concentration gradient facilitates the movement of ammonia from the bulk feed toward the membrane interface. Once at the membrane surface, ammonia volatilises and diffuses through the microporous hydrophobic membrane driven by the partial pressure difference between the feed and the acid stripping solution, where it is rapidly converted into ammonium salts (e.g., (NH₄)₂SO₄), maintaining a near-zero partial pressure on the lumen side [10,42–44].

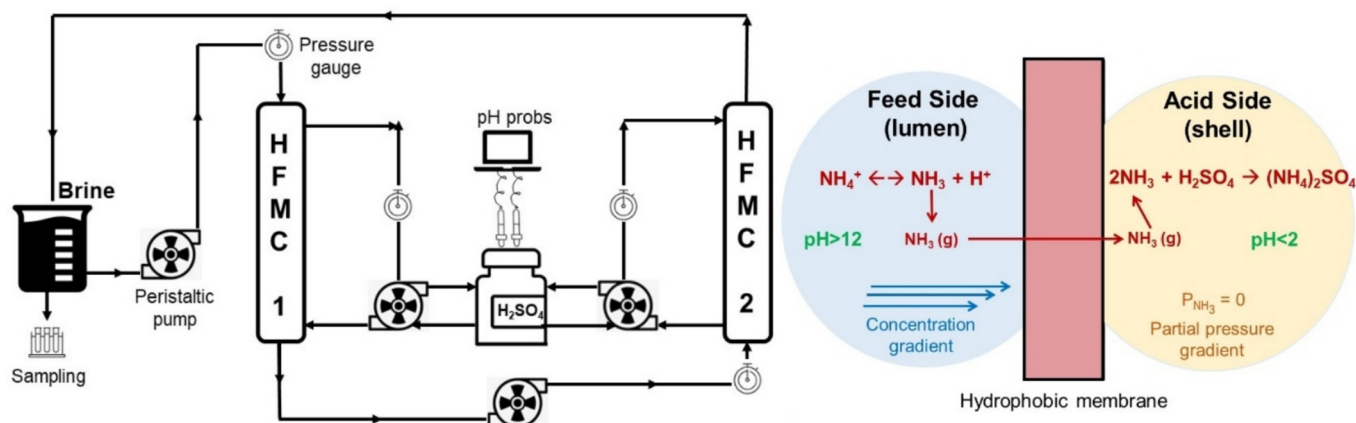
2.3. Ammonia recovery from synthetic solutions

The mass transfer coefficient of ammonia was compared with the real brine and synthetic brine solutions containing the same level of ammonia to verify whether the change of transfer rate was due to any difference of ammonia ion/ammonia gas equilibrium, ammonia loss and pH were also monitored. Synthetic ammonia solutions were prepared with NH₄Cl (Fisher Scientific, UK) to be used in the experiments on the effect of initial ammonia concentrations (200, 400, 600, 800, 1200 and 1400 mg NH₄⁺-N/L) in the feed side and different acid concentrations in the stripping side (0.1, 0.2, 0.4, 0.6 M). The change of ammonia gas flux (J_{NH_3}) and mass transfer coefficients (Km) monitored along 90 min of experiment. In these experiments, the molar ratio between ammonia in the feed and acid in the stripping solution was intentionally varied by design, as acid concentration and ammonia concentration were independently controlled to evaluate their individual effects on mass transfer performance.

Ammonia recovery performance was also investigated at pH 10,11 and 12. Following the pH optimisation, the pH fluctuations were monitored to be able to determine the acid consumption of the system with an initial ammonia concentration of 2000 mg NH₄⁺-N/L. Experiments were conducted over 580 min using the same 1 M H₂SO₄ stripping solution, which was not replaced during the entire run. To maintain effective stripping conditions, pH adjustments were performed at 300 and 430 min to counteract the observed pH increase in the stripping solution. Meanwhile, the feed solution was replaced with a fresh one as needed to sustain continuous ammonia transfer to the acid side. The effect of water transport was monitored at two different ammonia concentrations 200 mg/L and 2000 mg/L representing low and high initial concentration. The H₂SO₄ concentration of the stripping solution was 1 M for both conditions. Experiments with synthetic and real brines were carried out under identical conditions, with matched initial ammonia concentrations, the same module configuration, and constant flow rates to ensure direct comparability.

2.4. Ammonia and water transport kinetics

The water flux through the membrane is controlled by the water partial pressure difference between the feed and the stripping streams

**Fig. 1.** Schematic view of experimental set-up and ammonia recovery mechanisms.

and by the membrane permeability of water vapor. The volume of water transported through the membrane (V_w) was directly measured based on the change in stripping solution volume during the experiment. The water flux (J_w) was then calculated using Eq. (1) by normalising this volume to membrane area and experimental time. The ammonia flux (J_{NH_3}) across the membrane is governed by the difference in partial pressure of ammonia between the feed and stripping streams, as well as the overall ammonia mass transfer coefficient as calculated by Eq. (2).

$$J_w = \frac{V_w}{A_m \Delta t} \quad (1)$$

$$J_{NH_3} = \frac{m_{NH_3}}{A_m \Delta t} \quad (2)$$

where V_w is the water transported through the membrane pores (L), m_{NH_3} is the amount of ammonia (g) transferred from feed side to the stripping side, A_m is the membrane area (m^2), and t is the experimental time (h). Mass transfer coefficient was calculated from the following equation represents an apparent mass transfer coefficient derived from batch-mode first-order kinetics (Eq. (3));

$$\ln \frac{C_0}{C_t} = \frac{K_m A}{V} t \quad (3)$$

where C_0 and C_t are the ammonia concentration in the feed solution at time 0 and t (s) respectively, A is the membrane area (m^2) and V is the feed solution volume (m^3). Thus, the mass transfer coefficient (K_m) was calculated from the slope of the line drawn with the above equation. The percentage of N removal in the feed side was calculated by the following Eq. (4):

$$N \text{ removal} = \frac{C_0 - C_f}{C_0} \times 100 \quad (4)$$

where C_f is the final ammonia concentration in the feed side. The concentration factor of ammonia (CF) was calculated by Eq. (5).

$$CF = \frac{C_s}{C_f} \quad (5)$$

where C_s is the concentration of ammonia in the stripping side and C_f is the concentration of ammonia in the feed side.

2.5. Recovery of ammonium sulphate solid product

Fourteen batch experiments were completed with ion exchange brine solutions, passing fresh solution through the HFMC in each cycle, without replacing the stripping solution. Above >99 % ammonia removal was targeted with only 0.2–2 mg NH_4^+ -N/L residual concentration at the end of each cycle. To produce supersaturated ammonia solution on the stripping side, the volume of the sulfuric acid solution was decreased to 0.2 L. The experiments were continued until the acid solution reached its saturation and the recovery efficiency of ammonia started to decrease. Air drying allowed evaporation of neutralised sulfuric acid solution, with the generation of ammonium sulphate crystals as a solid recovered product that were further characterised by biological and chemical analysis.

2.6. Analysis

Ammonia concentrations in the samples taken from the adsorption and regeneration tests were analysed by ion chromatograph (Thermo-fisher Scientific Dionex ICS-900). pH, temperature and conductivity were monitored by (Jenway 3510 pH and conductivity meter, Camlab, UK). COD were analysed using Spectroquant® Cell Test kit (Merk Millipore). In the HFMC experiments, NH_4^+ -N was analysed by Spectroquant® Cell Test kit (Merk Millipore) and ammonia probe (Hach Lange ISENH4). The chemical characterisation of the recovered product

ammonium sulphate was analysed with a scanning electron microscopy (SEM, Tescan Vega 3, large chamber, Kohoutovice, Czech Republic) including energy-dispersive X-ray spectroscopy (EDX).

3. Results and discussion

3.1. Optimisation of ammonia mass transfer rate and flux in the HFMC system

To establish the optimal conditions to recover ammonia from the brine, different ammonia and the sulfuric acid concentrations were investigated. Fig. 2 shows the changes in ammonia flux as well as the mass transfer coefficients obtained when the HFMC was operated with different initial ammonia and acid concentrations for a period of 90 min. Ammonia recovery efficiencies were between 99.60 and 99.98 % at all the ammonia concentrations tested (Fig. S1). During the initial 10 min, the flux increased with higher initial ammonia concentrations, which can be attributed to a larger concentration gradient across the membrane fibres at the start of the process and the enhanced driving force for diffusion (Fig. 2). In the tests with initial ammonia concentration from 200 to 1400 mg NH_4^+ -N/L, the flux gradually increased from 0.23 to 1.65 g/ m^2 ·h, respectively, after 90 min of operation. Correspondingly, the residual concentrations on the brine/feed side ranged from 0.1 to 2 mg NH_4^+ -N/L, indicating an almost complete recovery. Residual ammonia concentrations, recovery efficiencies and flux values for each condition were all presented in Table S2.

The mass transfer rates depended on several parameters such as initial ammonia concentration (defining the concentration gradient), the solution volumes to be treated, membrane area, membrane morphology (which controls effective diffusion paths and interfacial area), and the length of operation [34]. The mass transfer coefficient was the highest at 200 mg NH_4^+ -N/L initial concentration, at 6.1 1/h, but lower values were obtained for higher initial ammonia concentrations (Fig. 2A). For 1000, 1200 and 1400 mg NH_4^+ -N/L ammonia, mass transfer rate was stable at around 4.3 1/h. This trend is further influenced by acid-base equilibria in the feed solution, as maintaining high pH shifts the NH_4^+ / NH_3 balance toward free ammonia, increasing its partial pressure and facilitating transport across the hydrophobic membrane. At lower initial ammonia concentrations, the concentration gradient and stripping acid capacity remain high throughout the test, maintaining a strong driving force. At higher ammonia concentrations, the stripping acid approaches saturation, reducing the effective driving force and leading to lower apparent K_m values. Higher ammonia mass transfer coefficient, at lower ammonia concentrations, have been previously suggested by other researchers too, that indicated the mass transfer coefficient slightly changes over the increase of ammonia load of the system [30]. As the effectiveness of the H_2SO_4 was reported previously by many authors, the experiments were performed with only sulfuric acid at a concentration of 0.1, 0.2, 0.4 and 0.6 M [10,42–44]. The ammonia recovery efficiency ranged from 97.0 to 99.9 % with 0.1–0.6 M H_2SO_4 solution, which was significantly higher than the recovery efficiency using conventional air stripping process with values between 80 and 98.7 % [45]. Red data points in Fig. 2 represent K_m values calculated at the end of each 90-minute batch experiment using Eq. (3). The observed decrease in apparent K_m at higher initial ammonia concentrations is attributed to stripping solution saturation, which reduces the effective driving force for ammonia transfer. The mass transfer coefficients varied from 2.36 to 4.18 1/h as the acid concentration increased from 0.1 to 0.6 M.

The highest recovery efficiency (99.96 %) and the corresponding mass transfer coefficient (4.18 1/h) were achieved at an acid concentration of 0.6 M. This was attributed to the fact that mass transfer was controlled solely by the free acid concentration in the stripping side, making it the primary driving force for ammonia extraction from the feed solution [17]. Consequently, the mass transfer coefficient increased with higher acid concentrations in the system. Although a 97.0 %

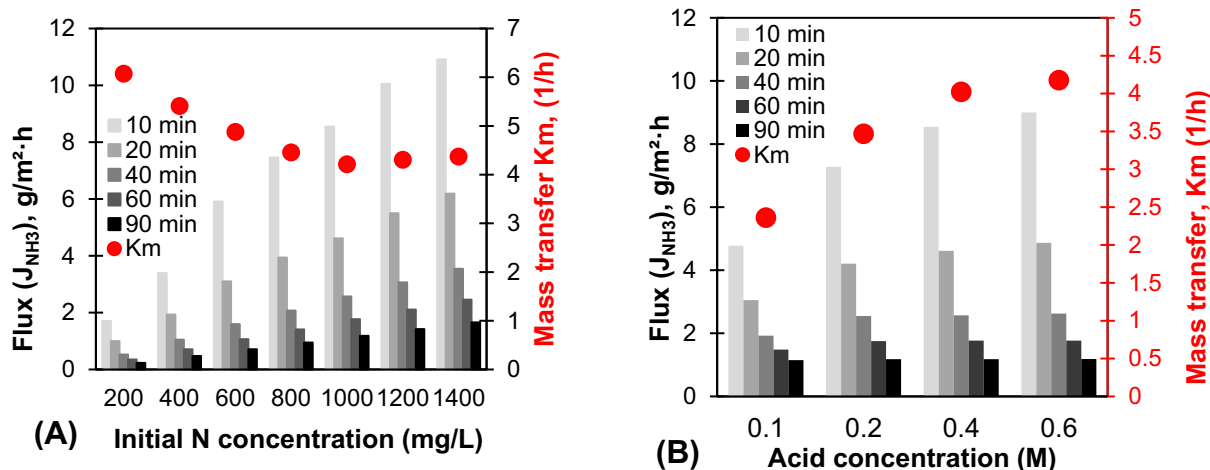


Fig. 2. Change of ammonia flux and mass transfer coefficients along 90 min of operation. A: Effect of different initial ammonia concentrations (200, 400, 600, 800, 1200 and 1400 mg $\text{NH}_4^+\text{-N/L}$) in the feed side with 1 M H_2SO_4 concentration in the acid side, B: Effect of different acid concentrations (0.1, 0.2, 0.4, 0.6 M) in the stripping side with 1000 mg $\text{NH}_4^+\text{-N/L}$ in the feed side.

recovery was still attainable at 0.1 M H_2SO_4 , the transfer kinetics were slower, leading to a lower amount of ammonia being transferred to the acid side. As a result, the residual ammonia concentration in the feed compartment remained at 551 mg/L, showing a 42 % increase relative to the lower acid concentration. Additionally, the gap between the trend lines for 0.1 M and 0.2 M widened as H_2SO_4 was consumed in the later stages of the experiment (Fig. S2). The molar ratio between acid in the stripping solution and ammonia in the feed solution in these experiments corresponded to approximately 28 at 200 mg/L ammonia concentration, 14.3 at 400 mg/L, 9.3 at 600 mg/L, 7 at 800 mg/L, 5.7 at 1000 mg/L, 4.7 at 1200 mg/L, and 4 at 1400 mg/L. For tests with 1000 mg/L ammonia concentration, the acid/ammonia ratio was 1.5 for 0.1 M acid, 2.8 for 0.2 M, 5.7 for 0.4 M, and 8.5 for 0.6 M. Fig. 3 shows that the change in mass transfer coefficient slows at the point where the two curves overlap, indicating an acid/ammonia ratio in the 7–8 range. However, the increase in the mass transfer coefficient when raising the acid concentration from 0.1 to 0.4 M was nearly 85 % lower than the increase observed when increasing it from 0.4 to 0.6 M.

In the first 10 min of the HFMC operation, the ammonia transmembrane flux was observed to be 4.78 g/m²·h at an acid concentration of 0.1 M. As the concentration of acid increased so did the ammonia

transmembrane, reaching 9 g/m²·h at 0.6 M acid concentration (Fig. 2B). Toward the end of the experiment, the flux values decreased for each acid concentration, stabilised at around 1.17 g/m²·h and complete recovery was achieved with a slight difference among tested acid concentrations. This was due to the decrease of the available ammonia on the feed side, which led to a decreased driving force for ammonia transfer to the acid side that resulted in slower flux [46].

Fig. 3 compares mass transfer coefficients under varying acid and ammonia concentrations. The HFMC system achieved a mass transfer rate of 6.073 1/h at an initial ammonia concentration of 200 mg $\text{NH}_4^+\text{-N/L}$. With 0.4 M acid, increasing ammonia concentration from 200 to 1000 mg $\text{NH}_4^+\text{-N/L}$ decreased the coefficient to 4.2 1/h. At 1000 mg $\text{NH}_4^+\text{-N/L}$ ammonia, the coefficient rose from 2.36 1/h at 0.1 M acid to 4.2 1/h at 0.4 M. As demonstrated in Fig. 3, increasing sulfuric acid concentration enhances ammonia transfer kinetics due to greater acid availability. However, as the acid concentration increased to 0.6 M the mass transfer coefficient change was quite minimal and recorded as 4.17 1/h. Increasing sulfuric acid concentration enhances ammonia capture kinetics due to greater acid availability, but also slightly raises solution viscosity, which can limit mass transfer. At our tested levels (0.4–0.6 M, ~4–6 wt%), the viscosity is expected to be closer to pure water (~1.0 mPa·s) [62], suggesting minimal impact on mass transfer in this range. Therefore, the efficiency of the transfer mechanism depends on the concentrations of both acid and ammonia across the membrane, influencing recovery efficiency throughout the process. It should be noted that the concentration of the acid will rely on the operational configuration and the type of acid used for stripping. Simply by raising the concentration does not guarantee a more effective transfer of ammonia; instead, there exists an optimum concentration value where the maximum stripping becomes feasible [16].

It is evident that the driving force of ammonia is based on the difference of partial pressure or concentration difference of both sides of the membrane [47]. Maintaining a sufficient ammonia partial pressure gradient is essential to facilitate continuous diffusion and to limit ammonia loss via volatilisation, thereby maximising recovery efficiency. Several studies have demonstrated that the mass transfer rate is highly dependent on the ammonia concentration difference between the feed and stripping solutions [20,48,49]. Research has demonstrated that acid recirculation and the addition of concentrated acid improve ammonia removal efficiency by sustaining a strong concentration gradient between the feed and stripping solutions. However, the process becomes diffusion-limited rather than concentration-driven beyond a specific acid concentration threshold [48]. Experimental data showed that varying the stripping solution concentration from an $\text{NH}_4\text{:SO}_4$ molar

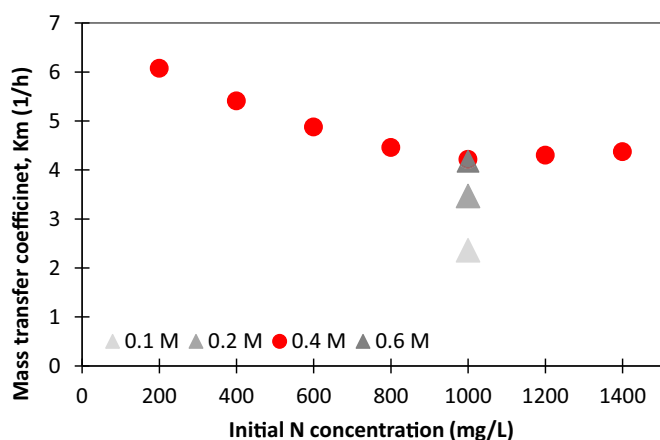


Fig. 3. Comparison of ammonia mass transfer coefficients at varying acid and NH_4Cl concentrations at the end of 90 min batch tests. The data include results obtained with H_2SO_4 concentrations of 0.1, 0.2, 0.4, and 0.6 M at a fixed initial NH_4Cl concentration of 1000 mg/L, as well as results for varying NH_4Cl concentrations (200, 400, 600, 800, 1000, 1200, and 1400 mg/L) at a constant H_2SO_4 concentration of 0.4 M.

ratio of 0 to 1.5 significantly affected the overall mass transfer coefficient, but beyond this ratio, no further enhancement was observed. This suggests that while maintaining a sufficient acid concentration difference is necessary to sustain ammonia removal, excessive acid concentrations introduce diffusion resistance that limits mass transfer rather than enhancing it.

3.2. Control of pH fluctuations

Controlling pH on both sides of the membrane is crucial for the continuity of the ammonia transfer process [32]. Fig. 4 shows the pH change of the system along four cycles with initial ammonia concentration of 2000 mg $\text{NH}_4^+\text{-N/L}$ and the sulfuric acid concentration of 0.1 M. Since the initial ammonia concentration was 2000 mg/L, 1 M acid was selected to maintain the optimal acid/ammonia ratio (7–8) with a minimum acid dosage. During the first cycle, the system's pH remained stable, with no significant fluctuations observed, indicating that the system had the required amount of acid in the water media to enable the ammonia transport. As ammonia was being transferred to the stripping side, the pH tended to rise due to the decrease in acidity and the formation of ammonium sulphate. At the end of the second cycle (after 220 min), the pH of the acid solution was 2, and it increased to 3 at 260 min and to 7.8 at the end of 300 min. This pH increase could potentially reach a highly alkaline level. As such, the free acid concentration of the stripping solution became insufficient to promote ammonia transfer through the membrane which is also related to the partial pressure of ammonia gas [16]. Hence to provide excess amount of H^+ on the stripping side the pH of the stripping solution was maintained in between 1 and 3 by an addition of concentrated H_2SO_4 solution and the pH variation was continuously monitored throughout the experiments. Increasing the pH of the feed solution enable the production of free ammonia and enhance the ammonia transfer from feed side to the stripping side [20].

During the first cycle, the ammonia transfer flux was initially 10.37 $\text{g/m}^2\text{-h}$ but declined to 1.8 $\text{g/m}^2\text{-h}$ as the pH increased to 7.8 after 5 h of experiment. The ammonia removal efficiency was 99.9 % for the first two cycles. In the subsequent cycles, this efficiency progressively decreased to 98.15 % and 94.45 %, respectively. Additionally, due to the declining mass transfer coefficient in each cycle, the experiment duration had to be extended by 40 min in both the third and fourth cycles. At the end of the first hour of each cycle, the residual ammonia concentrations recorded in the system were 6.4 mg/L after the first cycle, 18.1 mg/L in the second, 409 mg/L in the third, and 1310 mg/L in the fourth cycle. To restore the transfer efficiency, H_2SO_4 was added, reducing the pH back to approximately 2 (Fig. 4). Despite this adjustment, an additional acid dose was required during the fourth cycle to sustain ammonia transfer (Fig. 4). This acid demand emerged around 50 min into the cycle. This was approximately 30 min earlier than in the third cycle, indicating an accelerated pH shift. In the fourth cycle, the ammonia flux further declined to 1.2 $\text{g/m}^2\text{-h}$, suggesting a progressive reduction in transfer efficiency. Over two cycles, a total of 4000 mg $\text{NH}_4^+\text{-N/L}$ was

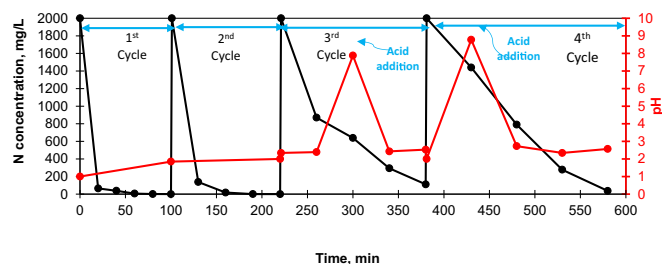


Fig. 4. pH fluctuations over four cycles and acid consumption in the system - batch experiments with an initial ammonia concentration of 2000 mg $\text{NH}_4^+\text{-N/L}$ and acid concentration of 1 M H_2SO_4 , feed solution was renewed at each batch while stripping solution was unchanged.

successfully transferred to the stripping side within 3.67 h, without the need for further pH adjustments.

Although it has been proved that elevated pH levels in the brine contribute to enhance the recovery efficiencies, it is essential to consider the economic aspects of the process when dealing with practical conditions such as chemical consumption for pH adjustments and membrane durability due to high alkaline and acidic conditions [20,50]. Huang et al. [8] shows that regenerant management is a critical cost driver: disposal of spent regenerant without recovery incurs ~£65/ton, making frequent regeneration economically unsustainable. The regenerant cleanup and nutrient recovery via HFMC enables multiple reuse cycles, reducing operating costs by 50 % in IEX N-columns. This underscores that economic feasibility in real-world conditions depends on effective regenerant reuse and product recovery strategies.

3.3. Impact of varying ammonia concentrations of water transport effect

Fig. 5 shows the compared ammonia and water flux changes at 200 and 2000 mg $\text{NH}_4^+\text{-N/L}$ initial ammonia concentrations. At the beginning of the experiment the ammonia flux at an initial concentration of 200 mg $\text{NH}_4^+\text{-N/L}$ was almost half of the flux that was achieved at 2000 mg $\text{NH}_4^+\text{-N/L}$ initial concentration and valued as 1.7 and 3.6 $\text{g/m}^2\text{-h}$ respectively (Fig. 5A). The reason for that, high ammonia concentrations resulted in higher driving force from feed to the stripping side. The flux kept decreasing for both experiments which was inevitable because of the decrease in ammonia concentration in the feed side. With an initial ammonia concentration of 200 mg $\text{NH}_4^+\text{-N/L}$, the ammonia flux decreased from 1.7 to 0.18 $\text{g/m}^2\text{-h}$ after 150 min of the experiment. However, when the initial ammonia concentration was 2000 mg $\text{NH}_4^+\text{-N/L}$, the flux decrease was gradually ended at a level of 1.2 $\text{g/m}^2\text{-h}$ due to the higher concentration gradient, and it stabilised after around 350–400 min (Fig. 5B). On the other hand, both the direction and the trend of the water flux developed diversely from that of the ammonia flux. The water flux for low initial ammonia concentration kept increasing, primarily driven by the vapor pressure difference between the feed and acid sides, as the feed solution became more diluted. It was around 0.1 $\text{L/m}^2\text{-h}$ at the end of 10 min and kept decreasing to 0.03 $\text{L/m}^2\text{-h}$ at 20 min (Fig. 5A). After that point the water flux started to increase and reached 0.12 $\text{L/m}^2\text{-h}$ level until the complete ammonia recovery achieved (Fig. 5A). These findings are consistent with Reig et al. [13] and Sheikh et al. [47], who showed that at low feed ammonia concentrations, the resulting vapor pressure difference favours water transport from the stripping solution to the feed.

At higher initial concentration water flux was 0.12 $\text{L/m}^2\text{-h}$ and stable the first 100 min of the experiment and kept decreasing to a level of 0.02 $\text{L/m}^2\text{-h}$ toward the end of the experiment and slightly increased to 0.04 $\text{L/m}^2\text{-h}$ at the last 60 min (Fig. 5B). The observed increase after 400 min was caused by water vapor transport from the feed side to the stripping side, which diluted the ammonium sulphate concentration in the stripping solution [10,13]. The increase in water transport was also influenced by the extended experimental duration, which led to higher ammonia transfer and ultimately caused water to move from the feed side to the acid side [31].

Ultimately, around 12 % of the stripping solution was transferred to the feed side at 200 mg $\text{NH}_4^+\text{-N/L}$ initial concentration and around 20 % was transferred from the feed side to the stripping side at 2000 mg $\text{NH}_4^+\text{-N/L}$ initial concentration. The direction of water flux is governed by osmotic pressure differences between the feed and stripping solutions. At lower initial ammonia concentrations, the higher osmolality of the acid stripping solution promotes water transfer out of the stripping side. At higher ammonia loadings, the feed solution's osmolality increases, reversing the gradient and driving water into the stripping side. This behaviour is consistent with established findings that water transport in membrane contactors is primarily driven by osmotic pressure gradients [19,30,40]. As such, the water level in the stripping side keeps decreasing for the lower initial ammonia concentration while it was

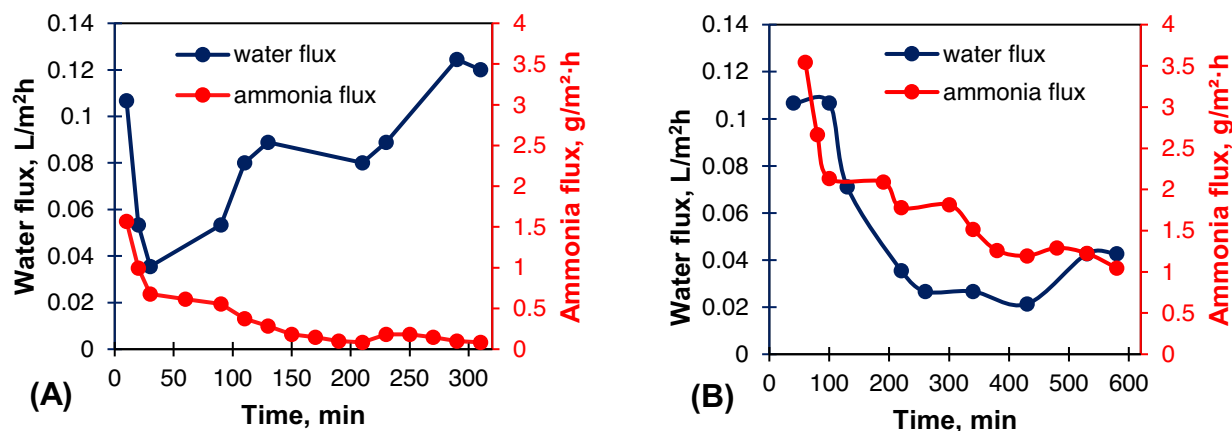


Fig. 5. Change of ammonia and water flux for low and high initial ammonia concentrations A: 200 mg/L (water transfer from acid to feed side) and B: 2000 mg/L (water transfer from feed to acid side) initial ammonia concentration.

increasing at higher initial ammonia concentration. The accompanying water transport also increases the acid consumption for stripping, lower the concentration factor of ammonia, and has adverse effects on the product formation process [30]. Hence considering the production of ammonium sulphate crystallisation, and the concentration factor of ammonia, it is favourable to maintain lower initial concentrations of ammonia that would end up with a more effective recovery process. Lower initial ammonia concentrations are also favourable as the length of the transfer period would decrease and complete recovery could be managed in a very short period of 20 to 60 min for the present case.

3.4. Comparison of ammonia recovery from synthetic and wastewater brine solutions

Experiments were performed with both synthetic and real wastewater brine solutions in order to compare the effectiveness of the HFMC recovery process. There was very little difference in recovery efficiencies between synthetic and wastewater brine, with values ranging from 99.7 to 99.9 %, respectively. However, the mass transfer coefficient for the wastewater brine was 9.38 1/h, which was almost 1.6 times higher than that achieved with synthetic brine (5.84 1/h) (Fig. 3 and Fig. S7). This was attributed to the presence of other inorganic constituents in the wastewater. Real brine solutions exhibit greater chemical stability and buffering capacity compared to synthetic solutions, helping maintain a more consistent $\text{NH}_3/\text{NH}_4^+$ equilibrium even under operational changes and ultimately facilitated faster and higher ammonia transfer. Similar trends were reported in previous studies, where the presence of inorganic ions in wastewater was found to enhance buffering capacity and promote higher ammonia mass transfer efficiencies [20,51].

In a study of [33] it was reported that higher mass transfer rate was achievable with strongly buffered pH conditions reaching up to 78.9 %. This finding highlights the critical role of chemical speciation in the boundary layer, indicating the importance for the overall mass transfer rate of ammonia particularly under specific operating conditions. The study specifically examined a membrane contactor system, demonstrating that under typical operating conditions – such as pH levels ranging from 8 to 11 and controlled feed flow rates – buffering effects significantly enhanced ammonia recovery efficiency.

The mass transfer coefficients obtained in this study are higher compared to the values reported in literature using a membrane contactor similar to the one used in the present study (Fig. 3 and Fig. S7). Liu & Wang, [52] reported higher values of ammonia ($5.3\text{--}8.9 \times 10^{-6}$ m/s) mass transfer coefficients that might be due to the PVDF membranes used in the study which are characterised by high transfer efficiencies [20,50–55]. Park et al., [33] investigated ammonia recovery using flat-sheet PVDF membranes and synthetic solutions of 100 mg/L TAN under controlled pH conditions. They demonstrated that at lower pH and in

buffered solutions, an indirect pathway involving ammonium deprotonation in the boundary layer could contribute up to ~79 % of the total ammonia transfer. In contrast, our system treated real ion exchange brine with initial ammonia concentrations between 200 and 1400 mg $\text{NH}_4^+\text{-N/L}$, operating at naturally high pH (~12–13) without additional buffering. Under these alkaline conditions, the ammonia ion–ammonia gas equilibrium heavily favours free NH_3 , minimising the role of indirect deprotonation pathways. This simplifies operational control and supports high ammonia fluxes and mass transfer coefficients (4.3–6.1 1/h), demonstrating the robustness and practicality of the HFMC process for treating real brines from full-scale ion exchange systems. The higher mass transfer rates achieved in the present study was also based on flow dynamics of the system, i.e., the brine solution was flowed through the lumen side while the acidic stripping solution was flowed through outside of the membrane fibres. This set-up allows more contact time for the ammonia molecules to transfer through the membrane. As the ammonia passes to the stripping side it can be easily transport from the membrane surface allowing more molecules to pass through the membrane fibres [46]. Additionally, the formation of ammonium sulphate on the outer surface of the membrane fibre, rather than the inner surface, prevented pore blockages that might be caused by ammonium sulphate crystallisation inside the fibre [50]. There are different views reported in literature on the impact of flow rate, some studies describe that an increase in flow rate of the brine or stripping solutions could favour the process performance as higher flow velocity generated turbulence that results in reduced thickness of the boundary layer. However, on contrary some reports revealed that there were no obvious effects of an increase in the mass flow and in the flow rate [7,10,48,56,57].

3.5. Ammonium sulphate recovery from wastewater brine and its characterisation

Fig. 6A shows the ammonia concentration and pH variations throughout the 14 batch experiments completed with ion exchange brine solutions, passing fresh solution through the HFMC in each cycle, without replacing the stripping solution and Fig. 6B shows the cumulative ammonia concentration in the acid side with respective recovery efficiencies.

The ammonia recovery efficiencies ranged in between 99.60 and 99.99 % throughout the 14 cycles which is also indicating limited membrane wetting. In the 2nd to 7th runs, pH fluctuations from 0.6 to 1.2 affected the recovery rates of ammonia (Fig. 6A). Then it stabilised between 8th and 13th cycles and started to decrease at 14th run because the water started to transport from feed to the acid side which (Fig. 6A). This was verified by monitoring changes in water volume on both sides of the membrane, which ranged between 1 and 5 mL and corresponded to the point at which the acid solution started to dilute. Hence, the

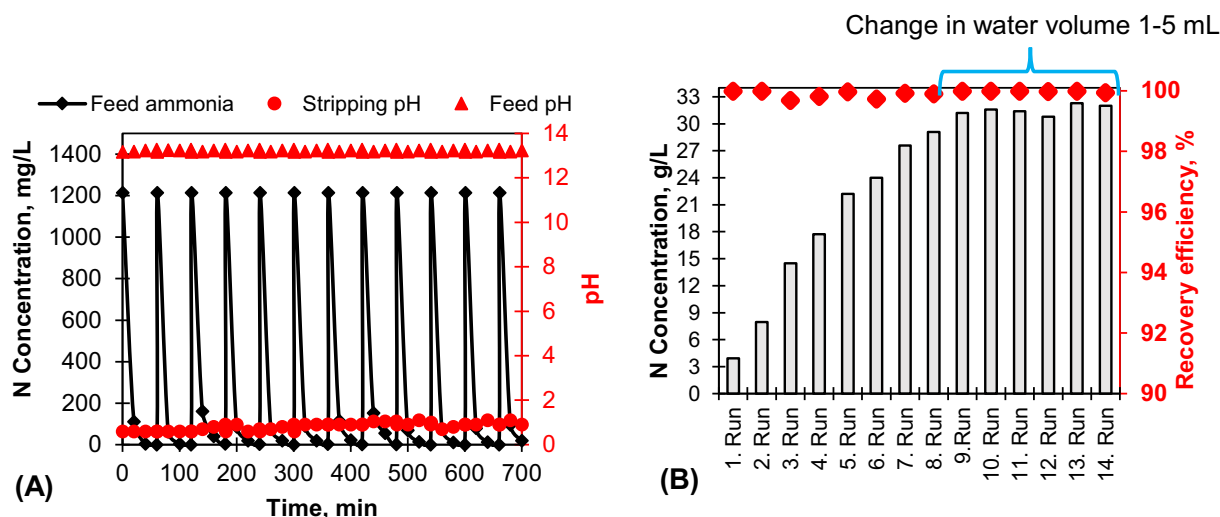


Fig. 6. Performance of ammonia recovery from wastewater brine along 14 batch tests A: change of ammonia concentration in the feed side and change of pH change for both feed and stripping sides, B: Cumulative ammonia concentration in the stripping side and ammonia recovery efficiencies.

experiment was stopped after that point as it was considered that the system reached its capacity. In the last cycle, the ammonia concentration could only be reduced to 19 mgNH₄⁺-N/L despite the addition of excess H₂SO₄.

Ammonia loss as a gas was inevitable considering the length of the experiment. However, it was minimised by keeping the reaction kinetics faster, preventing the water transport to the acid side and maintaining the required pH (around 1 for acid around 12 for feed) for both solutions. After 9.6 h of operation the ammonia concentration in the acid side reached 32.3 gNH₄⁺-N/L, which was 27 times concentrated, compared with brine solution which had 1200 mg NH₄⁺-N/L. It is also worth mentioning that no pressure drops, clogging on the membranes, or changes in flow rate were observed throughout the recovery experiments. This was likely due to the acid solution flowing on the outer space of the membrane modules, causing crystallisation to occur on the shell side rather than inside the membrane fibres. For the same reason, no membrane wetting was observed throughout the experiments. To evaluate potential membrane wetting, the modules were visually inspected after operation for signs of liquid breakthrough, fouling, or irreversible wetting. Consistent with our previous findings [12], only temporary crystal accumulation was observed, which was easily removed by rinsing without evidence of permanent membrane wetting or fouling. This operational validation suggests that the hydrophobic membrane maintained its wetting resistance under the tested conditions. This was confirmed by examining the stripping solution for any organic or inorganic intrusion, which was assessed through COD measurements and ion chromatography analysis.

After recovering the ammonia sulphate solution after the 14th cycle, this was evaporated to dryness to form a crystalline salt. The elemental analysis of the recovered salt and the proportions were found to be close to the chemical formula of the standard ammonium sulphate (Fig. S6.) The recovered product was free from organics, pathogens inorganics and metals, such as TOC, mercury, arsenic, calcium, copper, iron, magnesium, phosphorus and *Total coliforms* and *Escherichia coli* (all below detection limit). Some metals were detected at very low concentrations, cadmium 0.00002 mg/kg, chromium 0.0008 mg/kg, nickel 0.05 g/kg, zinc 0.15 g/kg) (Table S1). It is evident that all values remain below regulatory thresholds, confirming that the recovered product is suitable for land application. According to US EPA 40 CFR Part 503, the ceiling concentrations for key pollutants in land-applied materials are 75 mg/kg for arsenic, 85 mg/kg for cadmium, 4300 mg/kg for copper, 840 mg/kg for lead, 57 mg/kg for mercury, 420 mg/kg for nickel and 7500 mg/kg for zinc [58].

As such, the purity of the recovered product enables its use in many by-products in the chemical sector such as flame-resistant products, wood preservatives and aid in protein purification [59]. Nevertheless, many factors such as the structural properties of the membranes and the module, ammonia and water transport and feed/acid volume ratios, concentrations and flow rates can impact quality of the recovered product, [47] so there is an opportunity to optimise the HFMC to delivered and match specific product purity requirements.

Fig. 7 shows the mass balance of ammonia for the overall process based on the experimental data obtained. The concentration of ammonia in the wastewater before the ion exchange unit was 42 mg NH₄⁺-N/L and 1 m³ was processed each day. The ammonia removal efficiency in the ion exchange process was 96 %, and effluent presented in average 1.6 mg NH₄⁺-N/L. Ammonia was recovered from the brine after 9 adsorption cycles in the IEX, resulting in 9 m³ of wastewater treated. The volume of brine solution used for regeneration was 278 L, and by using the same solution throughout the experiments, the mass of ammonia in the brine was calculated as 303 g. The operation of HFMC with brine, resulted in an ammonia recovery of 99.6 %, resulting in the production of 72 g (NH₄)₂SO₄/m³ of wastewater treated, (i.e. 1.4 kg (NH₄)₂SO₄/m³ for the 9 m³ of wastewater treated in the IEX). As the effluent of HFMC had only 60 mg NH₄⁺-N, the NaOH brine streams can be re-used for regeneration of the ammonia rich zeolite media which can cut the chemical requirements in a less energy intensive way. The combination of the IEX with the HFMC allowed removal and concentration of the ammonia from an initial concentration of 42 mg/L in the secondary wastewater, to then 1190 mg/L in the brine (28 fold increase in concentration) and then to 32 g NH₄⁺-N/L as ammonia sulphate (27 fold increase in concentration) - The combined processes successfully concentrated ammonia 761 times in about 10 h of processing.

In comparison to our previous work [12], where ammonia was concentrated up to 2–6 g of ammonia gas from KCl IEX brine, and subsequently valorised through microbial protein production, the present study significantly improves both the recovery pathway and operational performance. In the previous study, the overall ammonia removal efficiency in the IEX process was 96 % with mass transfer coefficients ranging between 0.09 and 0.58 1/h, primarily due to limited process control and single-module configuration but most importantly KCl was the brine investigated [12]. In contrast, the present system achieved a maximum mass transfer coefficient of 9.38 1/h using real wastewater-derived NaOH brine – more than 16 times higher than previous values – while consistently maintaining a recovery efficiency of 99.98 %.

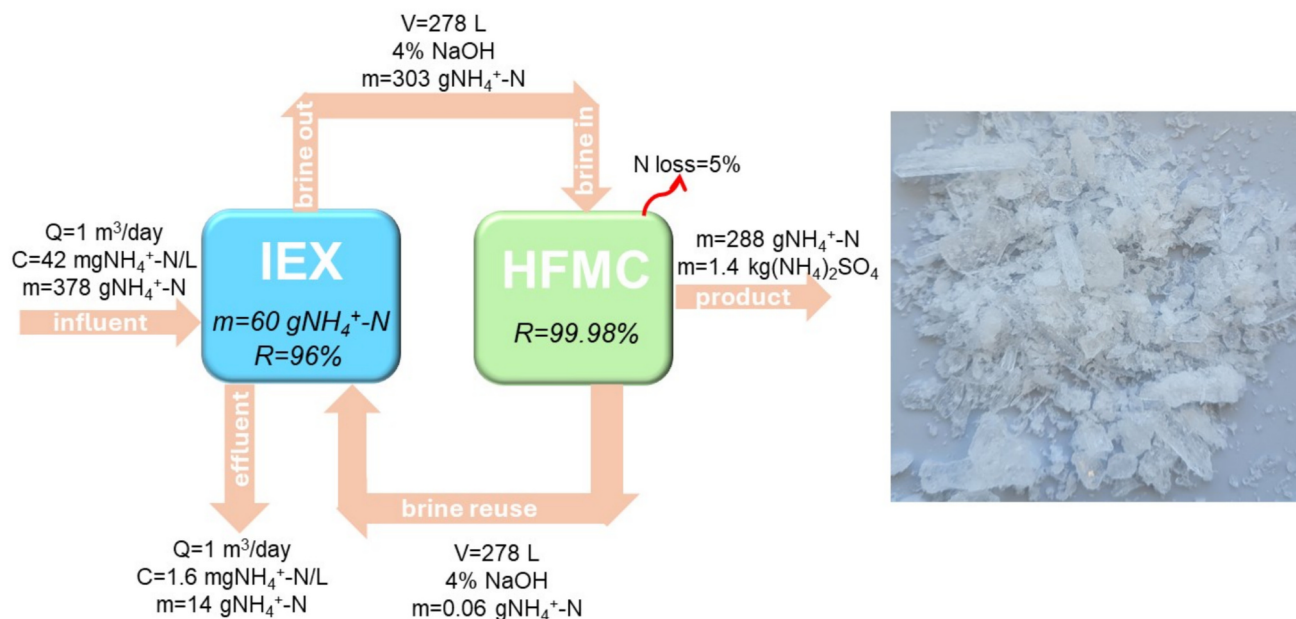


Fig. 7. Ammonia mass balance to the combined IEX and HFMC system (Q: represents flow rate, C: represents concentration, m: represents mass and R: represents efficiency of the process).

To the best of our knowledge considering the suggested operational conditions the present work offers, complete ammonia recovery was obtained, in a very short period of time that was not reported before. Sheikh et al., [31] demonstrated the influence of membrane configuration and material on overall performance and reported that under high initial NH_3 concentrations, the asymmetric PMP-LLMC achieved 90 % ammonia removal over 26 h, whereas the symmetric PP-LLMC module achieved 78 % removal after 20 h. The present study demonstrated high ammonia mass transfer coefficient of up to 9.38 1/h. And water and ammonia transport were optimised, achieving almost complete recovery in 10 h, and the recovered ammonium sulphate had high-purity and suitable for industrial reuse. The produced ammonium salts were at high quality in appreciation to the hydrophobic nature of the membranes allowing only ammonia gas transporting through the membrane pores. However, future investigations are required to explore the recovery of other ammonium salt (solids or liquid) with high market values (such as NH_4NO_3 and $(\text{NH}_4)_2\text{HPO}_4$ solutions) for potential use as liquid fertilisers.

The results also demonstrated that more effective outcomes were achievable when processing brine solutions collected after the regeneration of ion exchange media treated with real wastewater highlighting the potential for brine reuse. Considering the chemical requirements of the ion exchange application, reuse of the brine solutions become critical in order to decrease the cost and environmental impact during the disposal of the saturated brine solutions. The economic feasibility of the ion exchange process could be maintained by the recovery of nutrients as a valuable product that can be used by fertiliser industries and exhausted resin is considered to be safe for final disposal with a minimum waste volume.

Furthermore, HFMC systems are not limited to laboratory-scale concepts but are already commercially available and have been demonstrated at industrial scale for gas-liquid separations [60,61]. These commercial modules typically achieve operational lifespans of several thousand hours (up to 5000–7000 h) depending on maintenance protocols. The modules are designed to be chemically robust, allowing for straightforward cleaning and maintenance. Long-term operation does introduce risks such as biofouling and shell-side scaling, but these have been shown to be manageable via scheduled chemical cleaning cycles. Houlker et al. [60] demonstrated effective cleaning protocols

restoring near-original performance, and recommended duty-standby module configurations to ensure 100 % asset availability during proactive maintenance. Recent work from Cifuentes-Cabezas et al., [32], has shown that implementing a tiered cleaning protocol (preventive, moderate, and severe) can effectively manage fouling and prolong membrane lifespan. Preventive cleaning after every two tests and moderate cleaning every six tests allowed stable operation without significant loss of performance. Such approaches highlight the importance of planned maintenance schedules to ensure reliable long-term operation of HFMC systems.

4. Conclusions

The results of this study confirm the feasibility of hollow fibre membrane contactors (HFMCs) for ammonia recovery from ion exchange brine, achieving a high recovery efficiency of 99.98 %. Water transport effects were highly dependent on ammonia concentrations in the brine, with 20 % water loss to the stripping side at 2000 mg $\text{NH}_4^+\text{-N/L}$, while at 200 mg $\text{NH}_4^+\text{-N/L}$, water transport was reversed, leading to a 12 % decrease in the stripping solution volume. The highest mass transfer coefficient achieved in synthetic brine was 6.1 1/h, while it increased to 9.38 1/h in real wastewater brine, demonstrating improved process efficiency with wastewater derived solutions. Water transport was found to be favourable at lower ammonia concentrations, as water flowed from the stripping side to the feed side, helping to reduce the water content of the ammonium sulphate solution. The combination of the IEX with the HFMC allowed removal and concentration of the ammonia from an initial concentration of 42 mg/L in secondary wastewater to 32 g $\text{NH}_4^+\text{-N/L}$ as ammonia sulphate that is 761-fold increase in concentration in about 10 h processing time. The system maintained high stability during the tests, with no significant pressure drops, clogging, or changes in flow rate during operation. Additionally, membrane wetting was not observed, as confirmed by the absence of organic or inorganic intrusion in the stripping solution. The recovered product presented very high purity, indicating it is suitable for a wide range of uses from chemical industry to protein production. Overall, the findings validate HFMC technology as a highly efficient and scalable ammonia recovery method, offering a sustainable alternative to conventional nitrogen removal processes by minimising chemical waste,

maximising nutrient reuse while addressing Net-Zero targets.

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CRediT authorship contribution statement

Hacer Sakar: Writing – review & editing, Writing – original draft, Resources, Methodology, Formal analysis, Data curation, Conceptualization. **Ana Soares:** Writing – review & editing, Visualization, Validation, Resources, Project administration, Methodology, Funding acquisition, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.cej.2025.167872>.

Data availability

Data will be made available on request.

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