

Ammonia recovery from brines originating from a municipal wastewater ion exchange process and valorization of recovered nitrogen into microbial protein

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Abstract

A hollow fibre membrane contactor (HFMC), and two vacuum thermal stripping processes, a rotary evaporator (VTS) and multi-component system (MVTs) were compared for their ability to recover ammonia (NH₃) from ion exchange (IEX) regeneration brines. The IEX was a 10 m³/day demonstration scale plant fed with secondary municipal wastewater. The 10% potassium chloride regeneration brine was used multiple times leading to ammonium (NH₄⁺-N) saturation (up to 890 mg N/L). When treating the saturated IEX brine, the highest NH₃ mass transfer coefficient for the HFMC, MVTs and VTS were 0.6, 0.7 and 0.1 h⁻¹, respectively, compared to values between 1.7- 3.5 h⁻¹, when treating a synthetic solution. The highest NH₃ recovery was obtained with the HFMC (99.8%) and the ammonium sulphate produced was characterised for impurities, presenting high quality. Concentrated ammonium (NH₄⁺-N) solutions (0.5-3.1 g N/L) were obtained from the MVTs and VTS processes. To further valorise the recovered NH₄⁺-N solution produced from the MVTs process, this was used as a substrate for microbial protein (MP) production. Limited differences were observed for production rate (specific growth rate 0.092-0.40 h⁻¹), protein yield (0.021-0.18 g protein/g acetate-COD_{consumed}) and protein content (0.073-0.87 g protein/g cell dry weight) between recovered and commercial nitrogen (N) sources, indicating that recovered N from IEX can serve as a substrate for MP production. This study demonstrates a comprehensive N management solution for wastewater applications, leading to a range recovered products. These combined technologies can contribute to the local economy, whilst delivering to the ambitious NET-ZERO and circular economy targets.

1. Introduction

Ammonium (NH_4^+) is the inorganic ion form of nitrogen (N) often found in municipal and industrial wastewater. Its concentration can be efficiently reduced by using the ion exchange (IEX) process to achieve effluent concentrations $<0.5 \text{ mg NH}_4^+\text{-N/L}$ [1-3]. However, the implementation of the process at full-scale is still challenging due to capacity of the available ion exchange materials [4], ability to cope with varying ammonia loads [5] and the production of a concentrated brine, the management of which represents a significant environmental and economic challenge [6,7]. In fact, if not effectively managed, the saturated regenerant must be discarded as hazardous waste with an associated high cost of the disposal (typically £65/ton) rendering the process non-economical in the long-term run [8]. For this reason, the conversion of brine waste to a usable resource has recently been investigated [9]. The possibility to recover ammonia (NH_3) from the saturated brine has two main advantages: a) it allows the reuse of the brine multiple times thus decreasing the chemical consumption [10] and b) the high-purity product (ammonia salts or aqueous ammonia) can be valorised into a new end-product (e.g. fertilizers, plastics, textile, cleaning products and microbial protein (MP) targeting feed/food applications), promoting a circular approach of resource utilization [11,12].

In particular, the possibility to recover ammonia (NH_3) from the saturated brine allows for its reuse multiple times thus decreasing the consumption of chemicals [10]. Ammonia recovery is gaining increasing interest and a number of technologies are being tested and evaluated including air stripping, chemical precipitation, absorption, reverse osmosis, electrodialysis, hollow fibre membrane contactor, and membrane distillation, have been recently reviewed by Chen *et al* [13]. The most common technologies rely on the transfer of free ammonia (NH_3) through gas stripping, which can be accomplished by several technologies such as vacuum thermal stripping (VTS) and liquid-liquid membrane contactors (hollow fibre membrane

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contactor, HFMC) [14,15]. These allow for ammonia recovery from concentrated solutions such as IEX brines, to evolve more concentrated ammonia products of higher purity [14,16-18].

Thermal stripping of ammonia is driven by vapour pressure difference, obtained by increasing the temperature of the saturated solution, and reducing the boundary layer thickness at the surface of the solution thus increasing ammonia mass transfer [16]. The boiling temperature can be reduced by applying a vacuum [19] thus reducing energy cost [20]. Vacuum thermal stripping (VTS) can be coupled with distillation to recover the stripped gas [16] where a cold solution is recirculated to cool the vapour (distillate) back into a liquid [21,22]. With this procedure, ammonia gas ($\text{NH}_{3(g)}$) can be recovered as aqueous ammonia ($\text{NH}_{3(aq)}$), which is a valuable chemical widely used in plastics and textile manufacturing, food production and cleaning industry [23]. Alternatively, an acid reaction vessel can be incorporated in the process to recover a stable ammonium salt [24]. For example, by using sulphuric acid, ammonium sulphate ($(\text{NH}_4)_2\text{SO}_4$) has been recovered from raw hydrolysed and struvite-recovered urine (pH 9.2) [19] (boiling point of 65°C and pressure of 270 mbar) as well as from anaerobically digested municipal sludge and dairy manure (pH 9) with a high recovery efficiency of up to 96% [18]. The process is commonly modelled as a first order rate process with a stripping rate coefficient of $0.1\text{-}0.2\text{ h}^{-1}$ [16]. In laboratory scale experiments, vacuum thermal stripping of a saturated ammonia solution has been obtained with a rotary evaporator with a recovery efficiency of around 80% [25]. Rotary evaporators integrate the different components (vertical condenser, heating bath for feed solution, receiving flask for recovered condensate a vacuum pump and controller) in a single piece of equipment [22]. Practical vacuum thermal stripping can also be accomplished with independent stages from evaporation and distillation to provide better control (temperature and pressure) and ease of scale up (multi component vacuum thermal stripping, MVTS). This is important, as it enables the control of selectivity by balancing the flux of water vapour and ammonia.

The highly concentrated regenerant brine obtained in the IEX process is also suitable for treatment with hollow fibre liquid-liquid membrane contactors (HFMC) [26]. In this technology, the high pH (>9) of the brine favours the formation of free ammonia gas ($\text{NH}_{3(g)}$) which diffuses through the microporous hydrophobic membrane, from the shell-side to the lumen-side [27]. Here, a strong acid solution promotes the precipitation of ammonia salts such as ammonium sulphate [27], ammonium nitrate or di-ammonium phosphate [28]. These salts can have application in the fertilizer industry according to their purity level [29]. Previous studies have used the HFMC to recover ammonia from wastewater (pH 11-12, initial concentration of 772 mg $\text{NH}_4^+\text{-N/L}$) [30] as well as from concentrates formed during zeolite regeneration (2.1-2.7 g NH_3/L , pH 11-12) [26] with recovery efficiency higher than 98-99%. By recirculating the saturated brine multiple times around the membrane unit, it is possible to increase $\text{NH}_4^+\text{-N}$ recovery [31] while preventing the dispersion of one phase within another, thanks to the hydrophobic nature of the pores of the membrane [27]. Membrane contactors are relatively simple to use and to make the process rapid and more efficient; individual modules can have a membrane area up to 330 m^2 and a specific surface area of almost 10,000 m^2/m^3 [25] which reduce both foot-print and capital costs [26]. Commonly, sulphuric acid is used to recover ammonium sulphate [27]. However, due to its high solubility in water (75.13 g/100 g H_2O at 20°C), the recovered product can require evaporation of the acid solution and needs accurate control of water transport across the membrane [20].

Thus far, the majority of the products from N recovery technologies are applied in the fertilizer industry [32]. Also synthetically produced reactive N (*i.e.* N produced via Haber-Bosch) is mainly consumed by the fertilizer industry ($\pm 80\%$) [33]. Recently, microbial protein production has been identified and tested as an interesting technology to valorise recovered N [34–39]. Microbial protein (MP) refers to edible protein-rich microbial biomass from cultivation of bacteria, microalgae and fungi, used as food or feed [40]. Commercial examples include QuornTM (fungus *Fusarium venenatum*, Marlow Foods, UK), Spirulina (cyanobacterium *Arthrospira* sp.) and FeedKind[®] (culture dominated by bacterium *Methylococcus capsulatus*,

Calysta, USA). The rising interest in MP production over fertilizers can be justified by its higher economic value (currently estimated at £1,800/ton protein) [41] and resource-efficient production technology [12]. During crop-based protein production almost 70% of the nitrogen is inherently lost due to run-off and volatilisation during land application, giving rise to environmental issues such as eutrophication and greenhouse gas emissions in the form of N_2O [11]. MP-based feed retains up to 80% of the applied N in its end-product, circumventing any uncontrolled release of N in the environment [12]. So far, three strategies have been explored to couple nitrogen recovery with MP production: a) nitrification of urine coupled to photoautotrophic MP production by cyanobacteria or microalgae [37-39], b) electrochemical ammonia recovery from urine coupled to MP production via chemolithoautotrophic bacteria (*i.e.* hydrogen oxidizing bacteria, HOB) [34], and c) electrochemical ammonia recovery from digestate and urine coupled to cultivation of methane oxidizing bacteria (MOB) [36,42]. Even though these strategies constitute a step forward to valorise and optimize recovered N usage, one needs to recognize that cultivation of microalgae suffers from a high land footprint, less digestible biomass compared to other MP types, and technically challenging downstream processing due to poorly concentrated biomass [43,44]. Secondly, both HOB and MOB require the use of explosive gas mixtures of hydrogen and oxygen or methane and oxygen, respectively. Additionally, both hydrogen and methane have low solubility in water, resulting in mass transfer limitations. The use of highly soluble and liquid substrates available for microbes, such as organic acids (recovered or produced from CO_2) can circumvent these issues while enabling resource recovery [45-47].

To date, there have only been a few papers investigating ammonium recovery technologies for the management of saturated brine used for zeolite regeneration in wastewater treatment [6,48,49]. None of these investigated the potential of valorising the recovered N into a new end product. Further, no papers have, as yet, provided a comparison of (M)VTS and HFMC to enable a discussion of their relative suitability and prospects for use with ion exchange systems for the recovery of ammonia from wastewater.

Therefore, the aim of this work was a) to compare trials treating two batches of ion exchange regenerant, one using a HFMC and the other (M)VTS systems to enable a direct comparison to be discussed, and b) to provide a proof of concept to the coupling of N-recovery technologies with MP production. In both HFMC and (M)VTS systems, a combination of synthetic and real brines was treated under conditions previously outlined for each technology. The results are then compared to assess the relative potential of the different options and offer a perspective on future outlook and development. Secondly, aqueous ammonia from the MVTs technology operated with IEX brines and commercial aqueous ammonia were used to grow different microorganisms suitable for MP production. The MP production process was evaluated based on the specific growth rate, lag phase, biomass and protein yields and protein content for microorganisms grown on recovered and commercial N.

2. Materials and methods

2.1. Synthetic solutions

For the experiments using the HFMC, a synthetic ammonia solution was prepared diluting ammonium chloride (NH_4Cl , Fisher Chemicals, Loughborough, UK) in deionized water to obtain a solution with a concentration of 1000 mg $\text{NH}_4^+\text{-N/L}$ and a pH that was adjusted between of 7-11 by adding sodium hydroxide. For the VTS and MVTs trials, the synthetic ammonia solution was prepared by diluting a stock solution (Ammonia 34%, Woburn Chemical Ltd, Milton Keynes, UK) to the required concentration (564 ± 8 mg $\text{NH}_4^+\text{-N/L}$) at a pH of 10. The ammonia concentration and the chemical composition of the synthetic solution were chosen to simulate the real brine used at laboratory and demonstration scale. A similar ratio of free ammonia in the two solutions were obtained by increasing the pH with sodium hydroxide (NaOH pellets, Fisher Chemicals, Loughborough, UK).

2.2. Regenerant brine

For the HFMC experiments, regenerant brine was produced using sodium chloride in deionized water (NaCl, 10% wt., Fisher Chemicals, Loughborough, UK). The brine was used multiple times for the regeneration of an ion exchange (IEX) media (synthetic zeolite Zeolite-N) in a laboratory scale fixed-bed column filled with 50 mL of media. The IEX media was operated with municipal wastewater containing 25-50 mg $\text{NH}_4^+\text{-N/L}$ to a breakthrough of 3 mg $\text{NH}_4^+\text{-N/L}$ at an empty bed contact time (EBCT) of 10 minutes. A single batch of brine (10 bed volumes, BV) was reused 4 times in succession to regenerate the Zeolite-N at 10 minute EBCT up to a concentration of 890 mg $\text{NH}_4^+\text{-N/L}$. After each regeneration cycle, a calcium precipitate (calcium carbonate) was recovered by addition of soda ash from the IEX brine to avoid the build-up of calcium ions in the brine which could interfere with the media regeneration. The pH was increased to 11 with NaOH prior to treating the brine with the HFMC to adjust the fraction of free ammonia.

For the VTS and MVTs experiments, the regenerant brine was produced using potassium chloride in deionized water (KCl 10% wt., Easy Chemicals Ltd, Denbigh, UK). The KCl brine was used multiple times for the regeneration of a column filled with Zeolite-N in a demonstration scale plant (10 m³/day) until a concentration of around 580 mg $\text{NH}_4^+\text{-N/L}$ was reached. The IEX media was operated in municipal wastewater containing 13-20 mg $\text{NH}_4^+\text{-N/L}$ to a breakthrough of 5 mg $\text{NH}_4^+\text{-N/L}$; the media was regenerated 6 consecutive times by passing 10 bed volumes of KCl 10% through the column for 2 hours (13 min EBCT). The pH was adjusted to 10 with NaOH prior to the stripping experiments. A chemical characterization of the KCl brine revealed that the main compounds present in solution were potassium (47.6 g K^+/L), calcium (1.2 g Ca^{2+}/L) and ammonium (0.82 g $\text{NH}_4^+\text{-N}/\text{L}$). The brine also presented magnesium and silicon (45.1 mg Mg^{2+}/L and 7.2 mg Si^+/L) and traces of heavy metals (0.7-37 $\mu\text{g/L}$).

2.3. Ammonia recovery with HFMC

A commercial hollow fibre membrane contractor (HFMC) (Liqui-Cel® 1.7x5.5 MiniModule®, Membrana GmbH, Wuppertal, Germany) was used to strip ammonia from the concentrated solutions by contacting it with an acid stripping solution (sulfuric acid 2% wt., Fisher Chemicals, Loughborough, UK) (Figure 1a). The HFMC comprised 7400 hydrophobic micro-porous propylene membrane fibres with inner and outer diameters of 220 and 300 μm , respectively, a surface area of 0.79 m^2 , a mean pore diameter of 0.03 μm and an effective length of 0.11 m. Flows were induced by two identical centrifugal pumps (Flojet HPR6/8, PS Components, Ontario) and controlled using four needle valves on the inlet and outlet of the HFMC shell and lumen (Platon, Domont, France). Sulfuric acid 2%, was circulated through the fibre lumen at a flow of 40 mL/min while the ammonia-rich solution was fed counter-currently in the HFMC shell at a flow of 900 mL/min. The transmembrane pressure was monitored using four pressure gauges positioned between the HFMC and each needle valve (DPG1000, Omega Engineering Ltd., Manchester, UK). Samples were taken from in-line luer-lock stop-cock valves (Cole-Parmer, USA) using a 5 mL syringe. The ammonium sulphate formed during transfer of NH_3 from used brine to sulfuric acid was recovered by evaporation of the neutralised sulfuric acid solution after the recovery process.

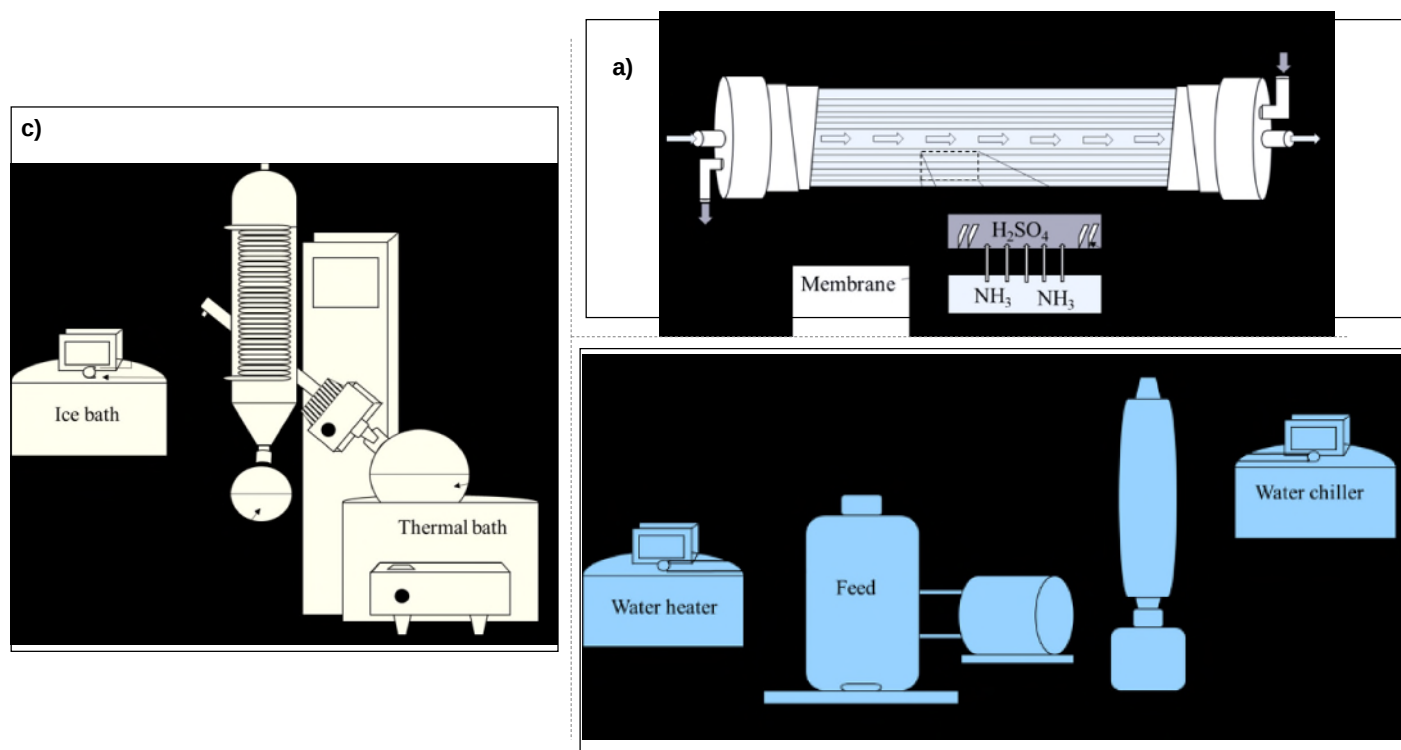


Figure 1. Schematic representation of the hollow fibre membrane contactor (HFMC) used for ammonia recovery as ammonium sulphate (a); of apparatus for vacuum thermal stripping including different components (MVTs) (b) and rotary evaporator used for ammonia recovery as concentrated solution through vacuum thermal stripping (c).

The experiments with the HFMC were performed first with synthetic brine to test the influence of pH and number of recirculations across the membrane on the ammonia recovery. The synthetic brine (1L), at different pH (7-11), was recirculated up to 100 times ($N=100$). For the experiment with saturated IEX brine, the solution (pH=11) was recirculated up to 360 times. The experiments were conducted in duplicate.

2.4. Ammonia recovery with vacuum thermal stripping

Two vacuum thermal stripping systems, with different operating conditions, were investigated in this study. The multiple component vacuum thermal stripping (MVTs) system was tested using separated equipment for each step of the recovery process (Figure 1b). This included: 500 mL evaporating flask for feed solution with heating jacketed, supplied by a recirculating

water heater (MPC-K6, Huber, Offenburg, Germany), and agitated using a magnetic stirrer (SB151, Stuart, Stone, UK) to distribute heat through the boiling solution. The surface area for distillation was approximately $8.0 \times 10^{-3} \text{ m}^2$ and the area-volume ratio was $16 \text{ m}^2/\text{m}^3$. The system also included a vacuum pump (ME-1C, Vacuubrand, Wertheim, Germany) and a vertical condenser (Inland Revenue Condenser, Scilabware Ltd., Stoke-on-Trent, UK) with surface area of $2.5 \times 10^{-2} \text{ m}^2$ supplied with a recirculating water chiller (LT Ecocool 150, Grant Instruments, Cambridge, UK).

The operational set up of the experiments at the MVTs were chosen according to previous studies where the highest recovery was obtained by distillation of the concentrated solution at $65\text{--}70^\circ\text{C}$ and $250\text{--}280 \text{ mbar}$ [16,22]. The operational conditions were fixed and not optimized in this study. Both synthetic solution and IEX brine (500 mL) were stabilized at temperature (T) of 69°C and pressure (P) of $280 \pm 5 \text{ mbar}$ for 30 minutes. The pressure was then decreased to $250 \pm 5 \text{ mbar}$ for 45 minutes while mixing at 920 rpm with $T=69^\circ\text{C}$. Vapour produced during the distillation was drawn through a condenser supplied with 5°C water. At the end of each experiment, the final volume and the ammonia (as $\text{NH}_4^+\text{-N}$) concentration were measured in both the treated and the recovered solution. All experiments were conducted in duplicate.

The vacuum thermal stripping (VTS) system was also tested by using a rotary evaporator (Rotavapor R-200, Buchi Ltd, Oldham, UK) (Figure 1c). This equipment included: a vertical condenser with condensing area of 0.15 m^2 (according to the supplier), an heating bath with a digital display, 1L evaporating flask for feed solution (approximately $23 \times 10^{-3} \text{ m}^2$ area for stripping), 500 mL receiving flask for the recovered solution, a vacuum pump, and a pressure controller with a digital display. Additionally, an ice bath (Grant TC120, Grant Instruments Ltd, Cambridge, UK) was connected to the condenser to cool down the water. For all the experiments, the liquid feed was approximately 1L. The temperature of the synthetic brine was set to 80°C and stabilised for 30 minutes at a vacuum pressure of $300 \pm 5 \text{ mbar}$ reflecting the optimal temperature previously reported for the treatment of concentrated ammonia solution using a rotary evaporator [25]. The pressure was then decreased to $250 \pm 5 \text{ mbar}$ [16] using

the pressure controller monitors and the ammonia was recovered for 45 minutes, while mixing the feed solution at 200 rpm. During this experiment, the temperature of the water pumped into the condenser was kept between 3-7°C with a circulating ice bath thus allowing for a difference in T of 73-77°C between the thermal bath and the condenser. The same operational conditions were used for the treatment of the IEX brine. All experiments were conducted in duplicate.

2.5. Microbial protein production

Cupriavidus necator LMG 1199 was purchased from the Belgian coordinated collection of microorganisms (BCCM/LMG, Ghent, Belgium), *Methylobacterium extorquens* DSM 1338, *Corynebacterium glutamicum* DSM 20300 and *Wickerhamomyces anomalus* DSM 6766 were obtained from the German Collection of Microorganisms and Cell Cultures (DSMZ, Braunschweig, Germany), while *Yarrowia lipolytica* ATCC 20362 and *Komagataella phaffii* ATCC 76273 originated from the American type culture collection (ATCC, Virginia, United States). All selected species are either generally regarded as safe (GRAS) [50], have a history with MP production [51–54] or are associated with wine making [55].

In order to determine specific growth rate, lag phase, biomass and protein yields and protein content of the MP originating from the different cultures, 96 well plate experiments were carried out. The incubation was performed at 28°C, in a plate reader (Infinite M200 Pro, Tecan, Switzerland) with orbital shaking (180 rpm), measuring optical density (OD) at 600 nm every 15 minutes. Adapted ammonium mineral salts (AMS) medium containing acetic acid (800 mg acetate/L, preventing potential toxicity [56]), aqueous ammonia (120 mg NH₄⁺/L) and vitamins was used for the cultivation (Table SI 1). The final working volume in the wells was 0.20 mL, after 10 % v/v inoculation. All experiments were performed in ten biological replicates.

2.6. Data analysis

2.6.1. Ammonia recovery

The stripping process is based on the transfer of ammonia from the liquid phase to the gas phase [16] following Fick's first law of diffusion through liquid/gas phases as expressed by the Lewis-Whittman model Equation 1 [20]:

$$\frac{dC}{dt} = -K_L a(C - C_s) \quad \text{Equation 1}$$

where dC/dt is the rate of ammonia mass transfer (mg/L), K_L is the overall liquid-phase mass transfer coefficient (m/h), a is the area-volume ratio of the liquid (1/m), C is the concentration of free ammonia in liquid (mg/L) and C_s is the saturation concentration of free ammonia in liquid (mg/L) [20,57]. Considering the ammonia concentration at time $t=0$ (C_0 , mg/L) and at time t (C_t , mg/L), Equation 1 can be rewritten as in Equation 2 [16,20,31].

$$t = -\frac{1}{K_L a} \ln\left(\frac{C_t}{C_0}\right) \quad \text{Equation 2}$$

which shows that a decrease in $K_L a$ (h^{-1}) leads to an increase in stripping time required to obtain a desired final ammonia concentration. Equation 2 was adapted to the VTS, MVTS and HFMC according to the parameters known during the experiments. For the HFMC, K_L can be calculated considering $a = A/V$ where A is the surface membrane area (m^2) and V is the volume brine solution (m^3). However, $K_L a$ was chosen as a factor to compare the ammonia mass transfer between the three technologies. Additionally, Equations 3-8 [35,58] were used for the VTS and MVTS:

$$\Delta T = T_{\text{thermal bath}} - T_{\text{condenser}} \quad \text{Equation 3}$$

$$R_{\text{NH}_3} = \left(\frac{C_0 V_0 - C_t V_t}{C_0 V_0} \right) \times 100\% \quad \text{Equation 4}$$

$$J_{\text{H}_2\text{O}} = \frac{\Delta m_{\text{H}_2\text{O}}}{\Delta t} \quad \text{Equation 5}$$

$$C_{\text{losst}} V_{\text{loss},t} = C_0 V_0 - C_t V_t - C_{\text{rec},t} V_{\text{rec},t} \quad \text{Equation 6}$$

$$J_{NH_3 \text{ loss}} = \frac{C_0 V_0 - C_t V_t - C_{rec,t} V_{rec,t}}{\Delta t} \quad \text{Equation 7}$$

$$J_{NH_3 \text{ rec}} = \frac{C_0 V_0 - C_t V_t - C_{loss,t} V_{loss,t}}{\Delta t} \quad \text{Equation 8}$$

where ΔT is the difference in temperature between the thermal bath and the condenser ($^{\circ}\text{C}$); R_{NH_3} is the ammonia stripping efficiency (%), J_{H_2O} is the water flux recovered (g/h), Δm_{H_2O} (g) is the mass of water transferred from the brine to the condensate during the period Δt (h), $J_{NH_3 \text{ loss}}$ and $J_{NH_3 \text{ rec}}$ are the rate of ammonia loss and recovered (g/h), V_0 , V_t are the volumes of the brine at time $t=0$ and time t (L) respectively, $C_{rec,t}$ and $V_{rec,t}$ are the concentration (mg/L) and volume (L) of the recovered solution at time t , $C_{loss,t}$ and $V_{loss,t}$ are the concentration (mg/L) and the volume (L) of ammonia loss at time t , C_t and C_0 are the same as in Equation 2.

2.6.2. Microbial protein production

The calculation of growth rate (μ) and lag time (λ) was performed as described in Candry *et al.* [59]. In short, first the OD of each well was corrected with the average of the un-inoculated samples, subsequently the corrected OD was log-transformed (according to commonly used population growth equations) [60]. Secondly, the nls.lm optimisation algorithm from the minpack.lm package in R was used to fit the Richards equation (Equation SI 1). This fit resulted in a μ , λ , carrying capacity (A) and v (a shape factor with no biological meaning) for each well. The calculations of biomass and protein yield are described in (Equation SI 2 - 5).

2.7. Chemical analysis

The ammonia concentration in the brine was measured with the Smartchem200 (AMS Alliance, France). The IEX brine and the recovered ammonia solution were analysed for heavy metals with the inductively coupled plasma mass spectrometry (ICP-MS, PerkinElmer NexION 450D). The chemical characterization of the product recovered from the IEX brine with the membrane was analysed with a scanning electron microscopy (SEM, Tescan Vega 3, large chamber, Kohoutovice, Czech Republic) including energy-dispersive X-ray

spectroscopy (EDX). The pH was monitored using a pH meter (Jenway 3510 pH and conductivity meter, Camlab, UK).

The OD of the microorganisms was converted to cell dry weight (CDW) using calibration curves. Biomass concentration (in g CDW/L) was quantified as total suspended solids (TSS) according to Standard Methods. Total protein was analysed in triplicate through the Pierce™ BCA Protein Assay Kit (Thermo Scientific™) following the manufacturers protocol. For acetate and ammonia analysis a homogenized sample of the ten biological replicates of each condition was filtered (0.20 µm PVDF filters, Chromafil®). Acetate concentration was determined using an ion chromatograph (930 Compact IC Flex; Metrohm, Switzerland), equipped with a Metrosep organic acids 250/7.8 column, a Metrosep organic acids guard column/4.6 and an 850 IC conductivity detector (Metrohm, Switzerland). Ammonium concentration was determined spectrophotometrically by the Berthelot reaction [61], at 700 nm (Infinite M200 pro, Tecan, Switzerland).

2.8. Statistical analysis

First the normality of the distributions of growth rate and lag time of each condition was verified using QQ-plots and the Shapiro-Wilk test. An independent sample t-test was performed to investigate if the average growth rate and lag time were significantly different, for the organisms that displayed a normal distribution. The homogeneity of the variances was verified with the Levene's test. For all conditions that displayed a non-normal distribution, the Wilcoxon signed rank test was applied. P-values below 0.05 were statistically significant. All analyses were conducted in R (v3.6.1).

3. Results

3.1. Ammonia recovery with HFMC

3.1.1. Effect of the pH and number of recirculations of synthetic brine in HFMC

The capability of HFMC to transfer ammonia from the synthetic brine was examined as a function of the brine pH, after 100 recirculations across the membrane (Figure 2a) (more details on NH_3 speciation with pH in SI). The K_{La} improved with increasing pH with the highest value of 1.5 h^{-1} at pH=11 which decreased to 0.7 h^{-1} at pH=10. These corresponded to ammonia recovery efficiencies, after 100 recirculation, of 95 and 72%, respectively. At lower pH levels, of 7-9, there were reduced recovery efficiencies of 15-36% corresponding to K_{La} values of $0.1\text{-}0.2 \text{ h}^{-1}$. The results confirm the importance of pH as it controls the percentage of ammonia as free ammonia available to be stripped [3]. Further investigation into the impact of the number of recirculations revealed pseudo stable removal levels occurred after only 27 cycles with recovery efficiencies of 68 and 94% at pH 10 and 11, respectively (Figure 2b). This corresponded to a 4 second residence time per pass for the brine flow and 30 minutes of operation overall. Extending the number of cycles did not significantly affect the ammonia recovery efficiency which were 77 and 95%, after 216 cycles. The corresponding mass transfer coefficients decreased as a function of the number of cycles reflecting the decrease in transfer gradients as the ammonia was removed from the solution. To illustrate, the K_{La} decreased from 3.5 h^{-1} after 13 cycles down to 0.37 h^{-1} after 215 cycles when operated at pH 10 (Figure 2c).

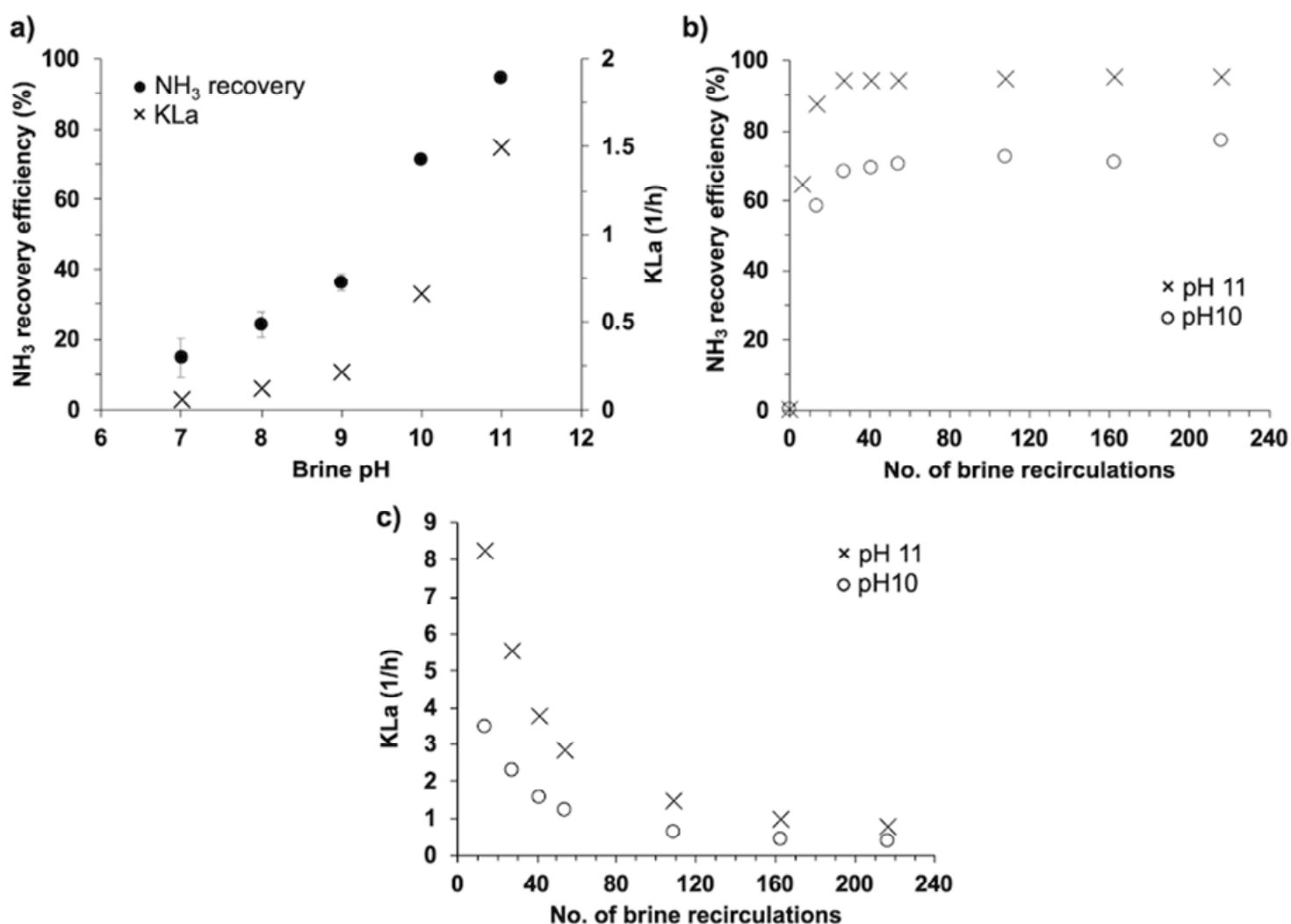


Figure 2. Effect of (a) pH of the synthetic brine ($C_{in} = 1000 \text{ mg NH}_4^+\text{-N/L}$, 100 recirculation), and (b) number of recirculations of the synthetic brine (pH = 10 and 11) across membrane on NH_3 recovery efficiency and (c) on mass transfer coefficient (K_{La} , h^{-1}).

In comparison, operation at pH 11 resulted in a decrease of K_{La} from 8.2 h^{-1} to 0.76 h^{-1} for the same cycle numbers. Overall, this demonstrated the significance in a unit change in pH resulting in a doubling of the mass transfer coefficient. Furthermore, wetting has been recognised as important factor is gas separation membrane contactors and its role in this system deserves further investigation.

3.1.2. Ammonia recovery efficiency from IEX brine through HFMC

Treatment of the IEX brine (NaCl 10%, pH 11) resulted in a significantly different result (Figure 3). The ammonia recovery efficiency increased from 68.8% after 108 cycles up to 99.8% (complete recovery) after 360 cycles corresponding to a total operating time of 6.7 hours. The initial ammonia concentration of 890 mg $\text{NH}_4^+\text{-N/L}$ was reduced down to 132 mg $\text{NH}_4^+\text{-N/L}$ after 165 cycles and 25 mg $\text{NH}_4^+\text{-N/L}$ after 250 cycles. The mass transfer coefficients (K_La) were substantially lower than observed in the synthetic experiments and ranged between 0.09 and 0.58 h^{-1} (Figure 3a).

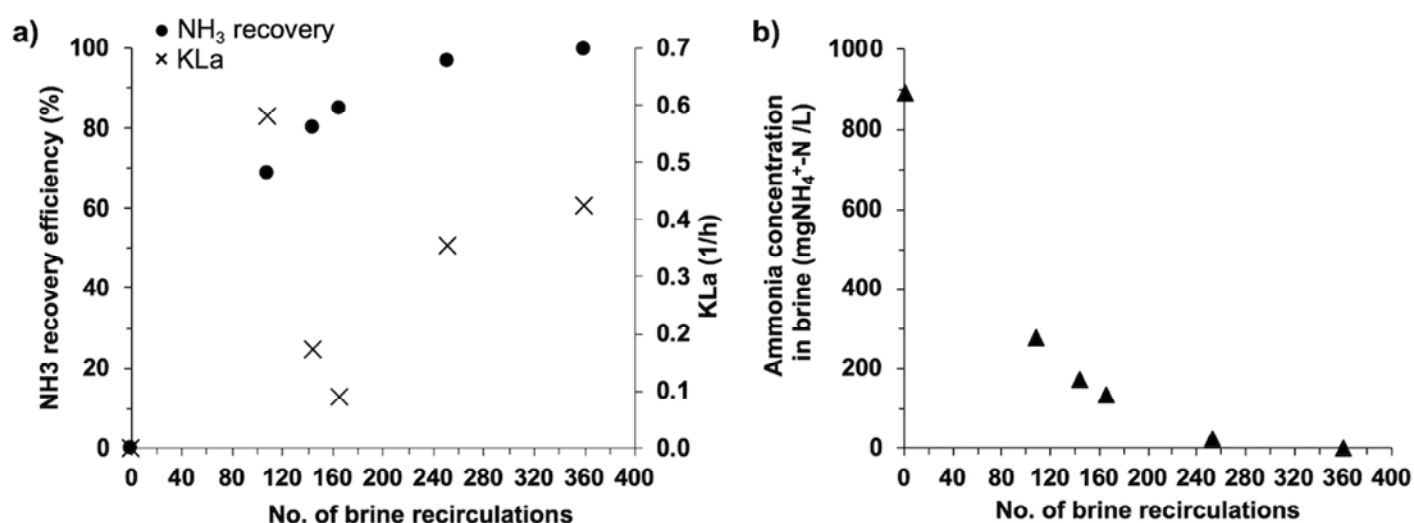


Figure 3. Impact of brine recirculations across the HFMC upon recovery of ammonia and overall mass transfer coefficient (K_La) (a) and ammonia (as $\text{NH}_4^+\text{-N}$) concentration in the treated brine (b).

These levels were lower than the minimum observed before at 0.76 h^{-1} with synthetic solution (Figure 2c) thus indicating that other ions present in the real regenerant brine have an impact on the ammonia recovery compared to the mono-component synthetic solution.

3.1.3. Ammonium sulphate recovered from HFMC

$\text{NH}_4\text{-N}$ was selectively removed from the brine as volatile free ammonia (NH_3) through the gas-filled membrane pores; forming ammonium sulphate upon instantaneous reaction at the interface with sulfuric acid. Some crystals were observed to temporarily accumulate in the membrane surface but rapidly re-dissolving, and no other fouling or biofouling was observed in the tests completed. The dissolved ammonium sulphate that accumulated within the acid phase following contact with the used brine within HFMC was recovered as a solid product by evaporation of the acid phase (Figure 4).

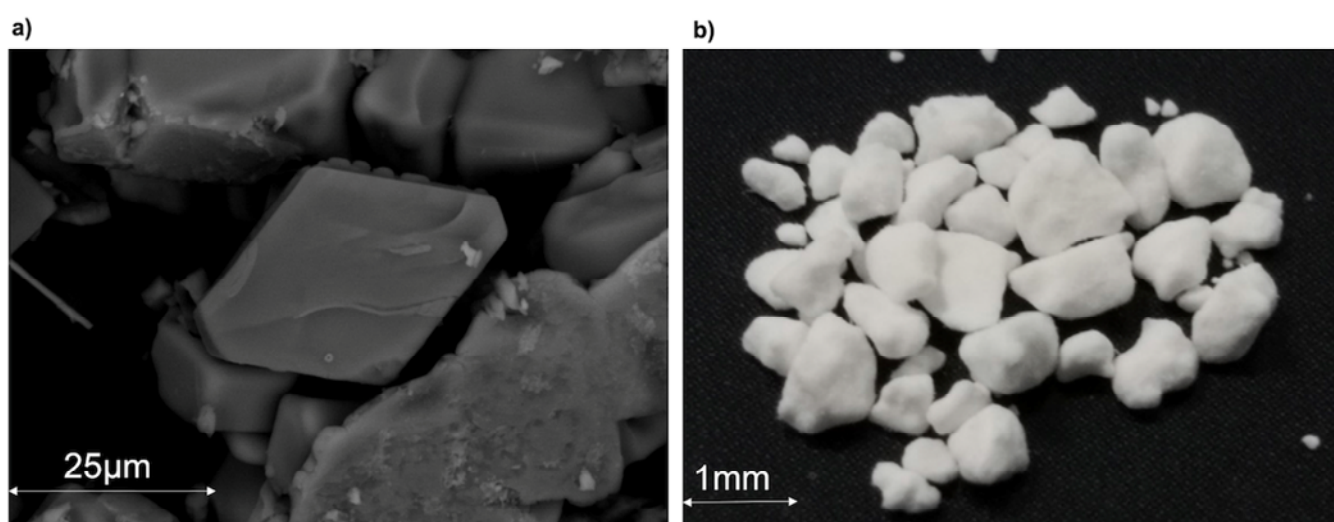


Figure 4. SEM image of recovered crystalline product following dehydration from solution (a) and dried ammonium sulphate recovered from ion exchange (IEX) brine (NaCl 10%) (b).

The identity of the product was confirmed by EDX analysis and comparison of the pattern with literature data revealed a good purity product. Only elements comprising ammonium sulphate (atomic %: N=25.5%, O=55.6% and S=18.8%) were detected and in proportions close to that expected from the chemical formula of ammonium sulphate (atomic %: N=28.6%, O=57.1% and S=14.2%).

3.2. Ammonia recovery from synthetic and real brines with vacuum thermal stripping

Figure 5a compares the results of the MVTs in terms of rate of ammonia loss ($J_{\text{NH}_3 \text{ loss}}$) and recovered ($J_{\text{NH}_3 \text{ rec}}$), and the flux of recovered water ($J_{\text{H}_2\text{O}}$) for synthetic and IEX brine. Ammonia recoveries of $74 \pm 0.9\%$ and $44 \pm 1.6\%$ were obtained when working with synthetic and IEX brine, respectively. These corresponded to recovery fluxes of 225 and 156 mg/h for synthetic and IEX brine, respectively. The corresponding mass transfer coefficients were $1.7 \pm 0.05 \text{ h}^{-1}$ and $0.7 \pm 0.04 \text{ h}^{-1}$ when treating the synthetic and IEX brines, respectively, representing a reduction in transfer coefficient of 58% when moving to the more complex fluids associated with IEX brines. In addition, higher ammonia loss ($J_{\text{NH}_3 \text{ loss}} = 57 \text{ mg/h}$) was obtained when treating the synthetic brine solution compared to the ammonia loss obtained from IEX brine ($J_{\text{NH}_3 \text{ loss}} = 15 \text{ mg/h}$). In contrast, similar water fluxes of 50-51 g/h were obtained (Figure 5a) with a lower ammonia concentration in the recovered solution with the IEX brine ($3.1 \pm 0.3 \text{ g NH}_4^+ \text{-N/L}$) compared to that when treating the synthetic solution ($3.3 \pm 0.2 \text{ g NH}_4^+ \text{-N/L}$). When the ammonia was recovered by VTS, by treating the synthetic solution with a rotary evaporator, a $J_{\text{NH}_3 \text{ rec}}$ of $598 \pm 12 \text{ mg/h}$ was measured (Figure 5b) which is substantially higher than seen with the MVTs system (Figure 5a). Additionally, no ammonia loss was measured but the $J_{\text{H}_2\text{O}}$ was also much higher at $160 \pm 11 \text{ mg/h}$. The recovered ammonia solution had a concentration of $2.5 \pm 0.12 \text{ g NH}_4^+ \text{-N/L}$ which corresponded to around 80% of recovery efficiency with a K_{La} of 1.96 h^{-1} . A much greater impact was also observed with the VTS system when comparing synthetic and IEX brines. The equivalent data when using the IEX brine was a recovery efficiency of 19%, a $J_{\text{NH}_3 \text{ rec}}$ of $110 \pm 7 \text{ mg/h}$, $J_{\text{NH}_3 \text{ loss}}$ was $24 \pm 4 \text{ mg/h}$ and K_{La} was 0.11 h^{-1} .

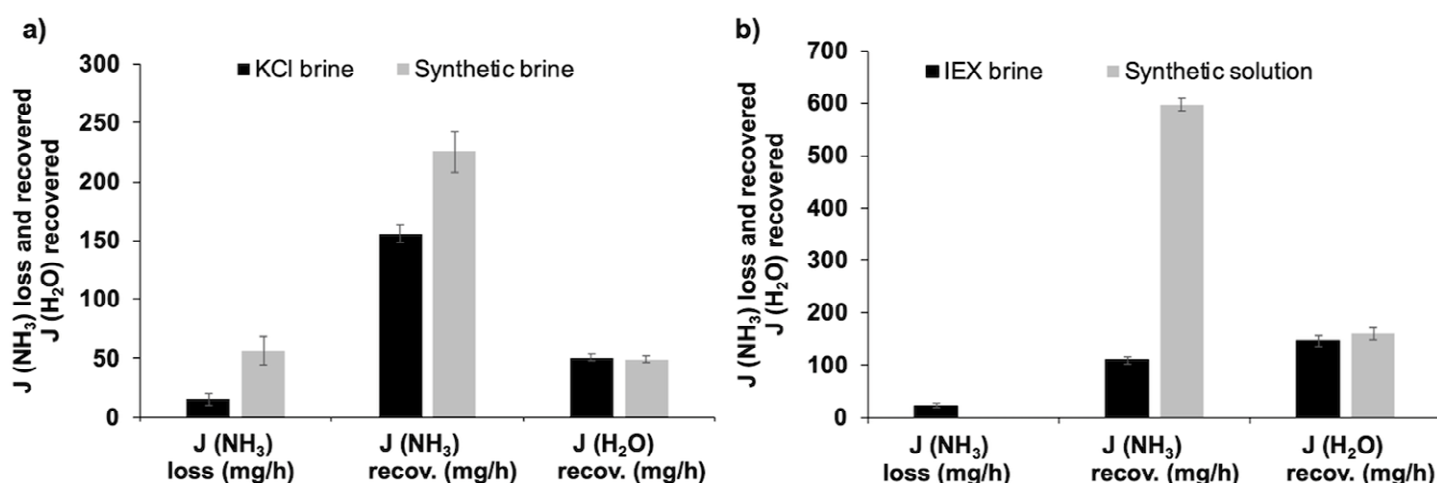


Figure 5. Ammonia recovered (mg/h), ammonia loss (mg/h) and water recovered (g/h) at the MVTS treating IEX brine (KCl, $C_{in} = 580 \text{ mg NH}_4^+\text{-N/L}$, pH=10) and synthetic solution (NH_4Cl , $C_{in} = 564 \pm 8 \text{ mg NH}_4^+\text{-N/L}$, pH=10) (a) and at the VTS treating IEX brine (KCl, $C_{in} = 580 \text{ mg NH}_4^+\text{-N/L}$, pH=10) and synthetic solution (NH_4Cl , $C_{in} = 564 \pm 8 \text{ mg NH}_4^+\text{-N/L}$, pH=10) (b).

This represents a reduction in mass transfer coefficient of 94% indicating the VTS systems was much more impacted when treating the more complex IEX brine solution. The $J_{\text{H}_2\text{O}}$ was $147 \pm 10 \text{ g/h}$ and a lower concentration in the recovered ammonia was obtained ($0.51 \pm 0.03 \text{ g NH}_4^+\text{-N/L}$).

3.3. Chemical characterization and purity of ammonia solution recovered from VTS and MVTS from IEX brine

The chemical characterization of the recovered ammonia solution obtained by treating IEX brine from the demonstration scale treating real wastewater, with VTS and MVTS systems revealed a good quality product (Table 1). The MVTS ammonia solution presented a lower concentration of heavy metals ($0.1\text{-}30 \text{ }\mu\text{g/L}$) and higher concentrations of calcium (55 mg/L), silicon (16.3 mg/L) and potassium (1.2 mg/L). In comparison, the ammonia solution recovered by the VTS systems contained heavy metals in the range of $0.2\text{-}108 \text{ }\mu\text{g/L}$ with traces of potassium and silicon ($0.3\text{-}0.5 \text{ mg/L}$, respectively) and 4 mg/L of calcium. In particular, much

higher levels of copper, magnesium and zinc were carried over into the recovered solution when using the VTS system as this had higher J_{H_2O} compared to MVTS.

Table 1. Chemical characterization of ammonia solution recovered with MVTS and VTS.

	Calcium	Potassium	Silicon	Copper	Magnesium	Zinc	Aluminium	Arsenic
	mg/L	mg/L	mg/L	µg/L	µg/L	µg/L	µg/L	µg/L
MVTS	55.3	1.2	16.3	30.3	14.5	4.4	17.1	2.1
VTS	4.0	0.3	0.5	108.0	92.6	35.0	19.3	1.1

4. Microbial protein production from recovered ammonia

The majority of the tested microbial strains had comparable specific growth rates (ranging from 0.092-0.40 h⁻¹) and lag time (ranging from 0.72-24 h⁻¹) for both N-sources (Figure SI 1). *M. extorquens* was the only organism where the growth performance was significantly different (growth rate: $p=1.0E-09$; lag time: $p=1.3E-08$) between the tested N-sources. It had a 1.7 times higher specific growth rate using recovered N (0.19 ± 0.022 h⁻¹) compared to commercial N (0.11 ± 0.0082 h⁻¹) and 1.2 times longer lag time (recovered N: 24 ± 0.84 h vs commercial N: 20 ± 1.0 h) (Figure SI 2; Table SI 4). From all tested strains *K. phaffii* displayed the highest specific growth rate (recovered N: 0.092 ± 0.0094 h⁻¹ and commercial N: 0.092 ± 0.013 h⁻¹). The protein yield on acetate was equal or higher (1.0-2.6 times) on recovered N compared to commercial N (Figure 6a). The yeast *K. phaffii* and bacterium *M. extorquens* had the largest difference (1.6 and 2.6 times, respectively) in favour of recovered N (*K. phaffii*: 0.060 vs 0.097 g protein/g acetate-COD_{consumed} and *M. extroquens*: 0.038 vs 0.10 g protein/g acetate-COD_{consumed}). A similar trend was observed for the protein content, where growth on recovered N resulted in an approximately equal (0.92 times) and up to 1.8 times higher protein content (0.08-0.87 g protein/g CDW) in comparison to commercial N (0.07-0.75 g protein/g CDW) (Figure 6b).

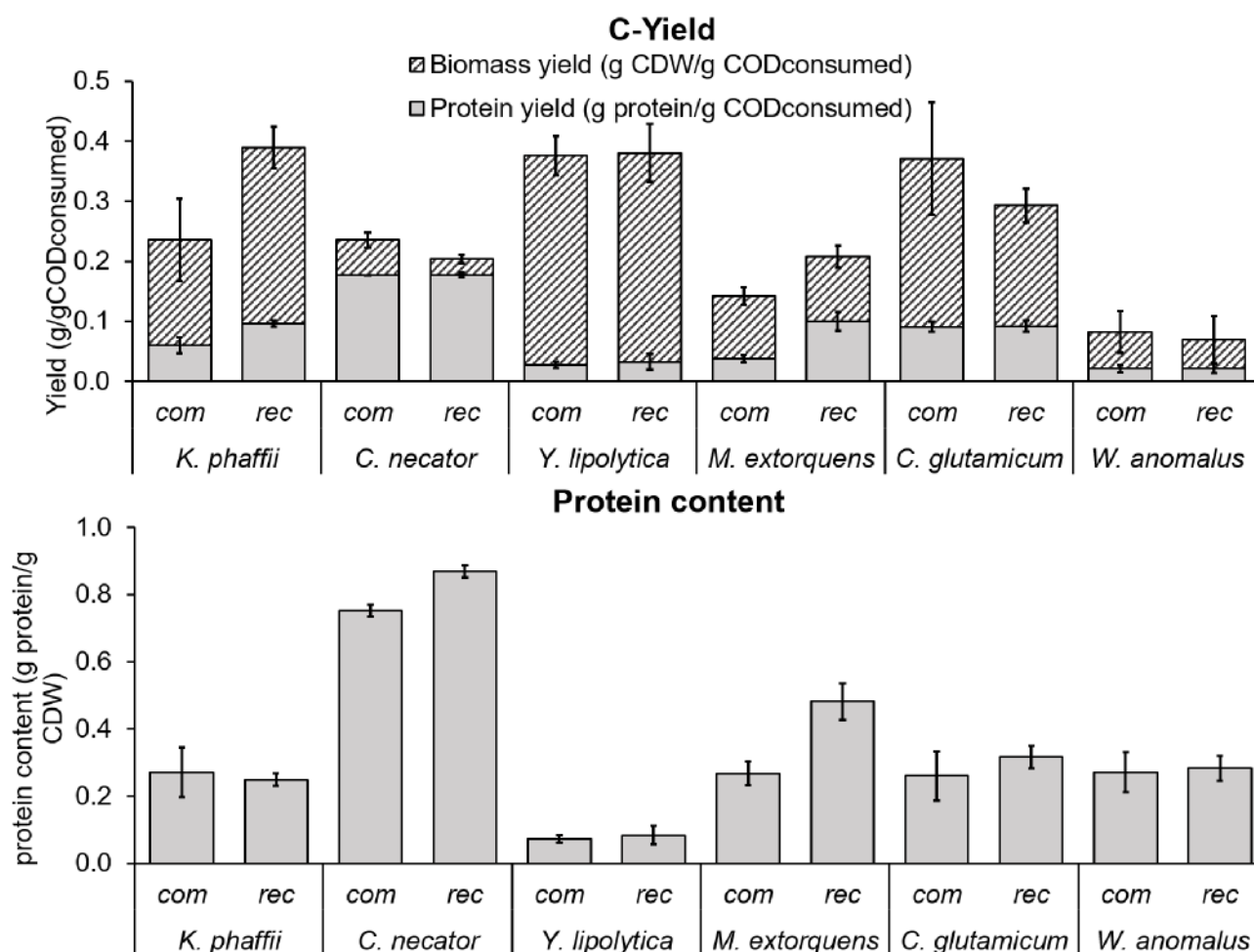


Figure 6. Biomass and protein yield on consumed COD for each tested organism (n=10) (a) and protein content of each tested organism (n=10) (b). Error bars indicate the standard deviation.

C. necator showed the highest protein content (recovered N: 0.87 ± 0.017 g protein/ g CDW; commercial N: 0.75 ± 0.017 g protein/g CDW) and yield (recovered N: 0.18 ± 0.0095 g protein/g acetate-COD_{consumed}; commercial N: 0.18 ± 0.0039 g protein/g CDW) from all tested strains, regardless of the N-source. Finally, all organisms displayed a similar yield on N for both N-sources (ranging from 0.60-3.0 g protein/g N_{consumed} on recovered N and from 0.60-2.7 g protein/g N_{consumed}) (Figure SI 3).

5. Discussion

5.1. Ammonia recovery

In this study, HFMC, MVTS and VTS were assessed for their relative suitability to recover ammonia from synthetic solutions and IEX brines used as part of an ion exchange process for ammonia removal and recovery (Table 2). The most direct comparison can be made through the mass transfer analysis with K_La values at pH 10 during the synthetic trials with values of 3.5 h^{-1} , 1.7 h^{-1} and 1.96 h^{-1} for the HFMC, MVTS and VTS respectively. Raising the pH to 11 increased the K_La of the HFMC to 8.2 h^{-1} , representing an enhancement factor of 2.3, emphasising the importance of pH on the stripping rate due to the existing differences in free ammonia fractions. To illustrate, the percentage of the ammonia that is present as free ammonia at equilibrium at pH 10 and 11 is 80% and 98% respectively, assuming 20°C [25].

Table 2. Overall comparison of the ammonia recovery systems tested.

	HFMC	MVTS	VTS
Recovered product	Ammonium sulphate	3.1 g N/L	0.5 g/L
K_La	0.6 h^{-1}	0.7 h^{-1}	0.1 h^{-1}
% N recovered	>98%	44%	19%

The equivalent transfer coefficients when treating IEX brines were 0.6 h^{-1} , 0.7 h^{-1} and 0.1 h^{-1} for HFMC, MVTS and VTS, respectively (Table 2). The value obtained with VTS agreed with the mass transfer coefficient reported in literature ($0.1\text{--}0.2\text{ h}^{-1}$ [16]) for the ammonium recovery from anaerobic digestate. The HFMC trials were conducted at pH 11 whereas the (M)VTS trials were conducted at pH 10. Adjusting the HFMC based on the enhancement factor observed during the synthetic trials would reduce the K_La to 0.26 h^{-1} . Accordingly, the reduction in K_La was similar for the HFMC and the MVTS systems at 60% and 59% respectively. In

contrast, a much greater reduction was observed for the integrated VTS system where the K_{La} was reduced by 95%. The reduction in mass transfer observed when treating IEX brines with HFMC and (M)VTS is attributed to the impact of the significant ion concentrations. For instance, the IEX brine used during the (M)VTS trials was rich in magnesium and silicon (45.1 mg Mg^{2+}/L and 7.2 mg Si^{+}/L) as well as containing traces of heavy metals (0.7-37 $\mu g/L$). Metal cations are known to lead to the formation of metal amine complexes [16,20] which exhibit much lower volatilities and hence decrease the stripping performance [62]. This idea is supported by the presence of higher levels of copper, magnesium and zinc in the recovered ammonia solution from the VTS system which suffered the greater reduction in K_{La} . The impact of this is to increase the time required to transfer the available ammonia (Equation 2). In the case of the HFMC system, this resulted in an increase in the number of recirculations required to recovery 100% of the ammonia from 27 ($t=0.5$ h) in the synthetic solution to 360 ($t=6.7$ h) in the IEX brine. The equivalent in the case of the (M)VTS systems is a reduction in ammonia recovery for a fixed time period. In the case of the MVTs system, this represented a change from 74% to 44% compared to the VTS system where the change was from 80% to 19% when working with synthetic and real brine, respectively. Additionally, the difference in K_{La} between the synthetic and real solutions could be attributed to the higher solids concentration and viscosities of the latter [63]. These values were not calculated for the real brines in this work. However, it could be hypothesised that the multiple reuse of the brine as regenerant solution for zeolites, used to treat wastewater, could have increased its viscosity due to remaining solids on the media surface [64]. A future more in-depth analysis could investigate the impact of the increased viscosity of the brine on the mass transfer coefficients through the calculation of the alpha factor as reported in Bajón Fernández *et al.* [63].

The overall effectiveness of (M)VTS systems depends on the operating point, controlled by the vacuum pressure, the temperature of the IEX brine, that in turn shifts the boiling point. Other conditions that are of relevance are the cooling solution in the condenser [20] and the mixing of the brine [22]. The comparison of the MVTs and VTS provides an assessment of

this, with the MVTs being more controllable. This was observed in relation to a higher ammonia recovery flux ($J_{\text{NH}_3 \text{ rec}} = 156 \text{ mg/h}$) and lower ammonia loss rate ($J_{\text{NH}_3 \text{ loss}} = 15 \text{ mg/h}$) compared to the values observed for the VTS system ($J_{\text{NH}_3 \text{ rec}} = 110 \text{ mg/h}$, $J_{\text{NH}_3 \text{ loss}} = 25 \text{ mg/h}$). Key differences are due to a higher contribution of latent heat, resulting in reduced ammonia losses and increasing mass transfer of the system. The importance of these conditions relates to more consistent and gradual heat distribution avoiding an abrupt onset of evaporation in a process called *bumping*. The impact of bumping is greater carryover of water as evidenced by the respective water flux rates of 51 g/h (MVTs) and 147 g/h (VTS).

All three processes resulted in the recovery of ammonia either in a liquid form or as a salt. In the case of the HFMC, this can be taken ultimately to the production of a high purity solid (Figure 4). However, due to its high solubility (75.13 g/100g H₂O [20]), the recovery of the solid product required evaporation of the acid phase and the recovery process requires the minimization of both transport of water and heat from a stripper to the acid solution [20]. As an alternative, the production of liquid fertilizer using HFMC has been investigated [26,29,64]. For instance, Licon Bernal *et al.* studied the possibility to recover liquid fertilizer (ammonium nitrate, NH₄NO₃, and diammonium phosphate, (NH₄)₂HPO₄) from ion exchange brine regenerant (1-3 g N/L) using a HFMC with an ammonia recovery >98% [28]. Similarly, Sancho *et al.* recovered ammonium nitrate and di-ammonium phosphate (2-5% wt N) from a solution of NaOH (2-3 g N/L) used to regenerate Clinoptilolite [26]. In fact, reported trials with recovered ammonia from anaerobic digestion effluent were able to generate a solution with an N content of 5-10% wt, albeit from elevated initial ammonia concentrations in the solution of 1.7-4.0 g/L [64]. In the current case, the (M)VTS systems generated ammonia solutions with a maximum concentration of 3.1 g/L with MVTs and 0.5 g/L with VTS. Ammonia solutions have many applications such as plastics, textiles, cleaning industries and feed or food (MP), as previously shown [23].

5.2. Microbial protein production

This paper aimed to provide a proof of concept concerning the use of recovered ammonia solutions for the production of MP. All microorganisms except for *M. extorquens* showed insignificant differences in growth rate and lag time, compared with commercial N dosing, indicating that the recovered N is a suitable substrate for the production of MP. A similar observation was made by Khoshnevisan *et al.*, who used electrochemically recovered N from digestate to grow MOB [36]. All these organisms had similar protein yields on COD, but the biomass yield on COD with recovered N was lower compared to commercial N, resulting in biomass that was richer in protein. Other studies that directly compared MP production from recovered N with commercial N, also reported small differences in biomass yields. Both Khoshnevisan *et al.* and Christiaens *et al.* reported a small increase in biomass yield for the organisms grown on recovered N [34,36]. Christiaens *et al.* attributed the difference to the reduced salinity, due to dissolving the recovered NH_3 directly into the medium rather than supplementing it as commercial NH_4Cl . From all the microorganisms tested in this study, *C. necator* showed the most promising results. *C. necator* reached the highest protein yield on COD and the highest protein content in the biomass (0.87 and 0.75 g protein/g CDW for recovered and commercial N, respectively). When comparing with fishmeal (1800 £/ton) as a valuable end product the biomass of *C. necator* surpasses the nutritional characteristics of typical fishmeal (0.70 g protein/g) [65]. Based on the extensive characterization of the recovered N-source, it appears likely that the resulting biomass end product is suitable towards feed. Arsenic is the only component contained in the recovered N stream that might induce a health risk when it ends up in the biomass and consumed as food. Assuming all arsenic contained in the medium ($0.21 \mu\text{g As/L}_{\text{medium}}$) is incorporated in the biomass, the final product would contain 1.2 mg/kg (calculated for *C. necator*), which is well below the maximum level of 2 mg/kg feed set by the European Commission (Table SI 3) [66].

5.3. Application in wastewater treatment plants

Both HFMC and MVTS systems were shown to be effective for the recovery of ammonia from IEX brines. Both systems have a number of advantages and disadvantages that require

consideration for further development. In the case of the HFMC, a major disadvantage is related to the possibility of fouling of the pores of the membrane caused by particulates (i.e. solids) in the brine solution which could limit the mass transfer and result in possible breakage of the fibres [9]. Accordingly, if the brine presents high concentration of solids, Wäeger-Baumann and Fuchs suggested a pre-treatment step with micro- or ultra- filtration to ensure long lifespan of the membrane [67]. Nevertheless, the membranes have a finite lifespan and would eventually need to be replaced thus increasing the cost of the treatment. According to Darestani *et al.* [9] the cost of the technology is also impacted by the type of solution that is been treated with the HFMC as this will define the number of modules required for the treatment and, therefore, the capital cost. Moreover, the scale-up of the technology should also consider costs connected to the recovered product, i.e. volatility of the fertiliser, production of sodium hydroxide to obtain the required pH as well as transport costs [9].

The equivalent challenge for the (M)VTs system is the energy requirements for heating the brine and driving the vacuum system. Compared to thermal stripping, the vacuum can reduce the energy cost as a result of lower sensible heating requirement [20], however, according to Tao *et al.* [19], the energy requirements for vacuum stripping still account for 87.2% of the total cost of the technology. The heat requirement can be effectively meet through the use of waste heat dissipated from thermal hydrolysis coolers (in the case of advanced anaerobic digestion) and un-utilised waste heat from combined heat and power production, although this is yet to be demonstrated. Recent estimates have indicated this could equate to as much as 40% of the energy produced from biogas on wastewater sludge processing sites and so looks promising [25].

A direct comparison of the total costs requirements of the ammonia recovery from saturated IEX brine through HFMC and (M)VTs has not been performed in this work. However, future work should focus on this to better estimate the viability of the technologies at bigger scale. Overall, considering the ammonia recovery efficiency obtained in this work and the technology readiness level, the HFMC is suggested as the preferred method. However, it is important to

consider that other challenges also remain which can interfere with the scale-up of HFMC for ammonia recovery such as the analysis of viable commercialisation pathway. To increase the market value of the recovered product, alternative acids should be tested to investigate the possibility of recovering different ammonium salt (solids or liquid) with higher market values (such as NH_4NO_3 and $(\text{NH}_4)_2(\text{HPO}_4)$ solutions) for potential use as liquid fertilisers.

6. Conclusions

In this study it was demonstrated that the ammonia stripping efficiency depends on the ratio of free ammonia present in solution and hence pH, i.e., operation below pH 10 (80% free ammonia) stripping was ineffective and it was maximised at pH 11 (98% free ammonia). Among the processes tested there was a significant reduction in transfer coefficient when using real IEX brines (0.6 h^{-1} , 0.7 h^{-1} and 0.1 h^{-1} for HFMC, MVTS and VTS) compared to synthetic solution (3.5 h^{-1} , 1.7 h^{-1} and 1.96 h^{-1} for HFMC, MVTS and VTS) due to the presence of metal ions, which form metal amine complexes. This led to an increase in the required time to recover the available ammonia. Furthermore, the effectiveness of vacuum thermal stripping system required good mixing and temperature control to enable gradual thermal gradients, avoiding bumping in the fluid which led to high levels of water carry over. The better control of the MVTS resulted in higher ammonia recovery flux ($J_{\text{NH}_3 \text{ rec}} = 156 \text{ mg/h}$) and lower ammonia loss rate ($J_{\text{NH}_3 \text{ loss}} = 15 \text{ mg/h}$) compared to the values observed for the VTS system ($J_{\text{NH}_3 \text{ rec}} = 110 \text{ mg/h}$, $J_{\text{NH}_3 \text{ loss}} = 25 \text{ mg/h}$). The HFMC was selected as preferred method for the treatment of saturated IEX brine. However future studies are needed to investigate the recovery of different 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 fertilizers. When valorising the recover nutrient, it was observed similar microbial growth parameters using aqueous ammonia recovered from MVTS and commercially purchased aqueous ammonia. The former was a highly suitable substrate for MP production. Growth of *C. necator* on recovered N yielded 0.18

g protein/g COD_{consumed} and a high-quality biomass with a protein content of 0.87 g protein/g CDW.

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