Ammonia Recovery from Source-Separated Hydrolyzed Urine via a Dual-Membrane Distillation In-Series Process

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Abstract

This study presents a dual membrane distillation (MD) in-series process to recover ammonia from source-separated hydrolyzed urine. The first MD stage separates and recovers ammonia from the hydrolyzed urine using an acid solution, while the second MD stage helps dewater the acid solution with captured ammonia and maintain its solution pH. The feed temperature and pH were optimized for operational conditions. The specific ammonia transfer (SAT) value was evaluated to obtain comparative results. While the water fluxes of both the single and dual-MD in-series set up systems were similar, the dual-MD in-series module showed higher SAT values and ammonia fluxes. At pH = 6, the ammonia flux values were minimal, but at pH = 13.2, they significantly increased to over 180 g/m^{2}h for the dual-MD in-series setup, which was approximately twice as much as the single MD setup (about 100 g/m^{2}h). In addition, the ammonium concentration in dual-MD in-series setup rose to over 200 mg/L at pH around 9, which is twice higher than that of a single MD setup. The results demonstrated that during the experiment, the dual-MD in-series configuration could help maintain the acid collector pH around 3 while for a single setup it continuously increased and reached to over 8 after 4 h. This innovative design not only preserves the desired pH range but also minimizes the amount of acid consumed, making it a promising approach in advancing nutrient recovery from human urine.

Keywords: Ammonia recovery; Hydrolyzed urine; Membrane distillation; Nutrients

1. Introduction

The release of nutrients and other urine-based micropollutants is highly detrimental to the environment. Nutrient overabundance in water ignites the eutrophication process, which causes the growth and spread of algae. Wastewater treatment plants (WWTPs) are specially designed to remove nutrients, which exposes a significant cost to the treatment process [1, 2]. Human urine is the primary source of nutrients and pharmaceuticals in wastewater treatment plants [3], which comprises about one percent of domestic wastewater by volume while contributing over 50% of the nitrogen and 60% phosphorous in the sewerage system.

The nutrients in wastewater are harmful to the environment. However, they are also a precious source for plants' growth and are the main components of agricultural fertilizers [1, 4, 5] in the form of nitrogen (N) and phosphorous (P) [3, 6]. Nutrient recovery from source-separated human urine is a contemporary circular economy technique for separately collecting human urine via dry toilets and treating it for concentrated nutrient recovery and direct fertilizer production [7-9]. Human urine is a diluted solution containing urea, creatinine and organic salt and is an enriched source of nitrogen, phosphorous and potassium. Many studies revealed that by maximizing nutrient recovery from human urine, there is a potential to reproduce about 13% of agriculture fertilizer demand with over $14 billion annual potential revenue [6, 10, 11].

Recent studies have focused on different methods like distillation, forward osmosis (FO), reverse osmosis (RO), freeze-thaw, membrane distillation (MD), membrane-based technology, etc., for dewatering of urine for fertilizer production [12-14]. MD is a membrane-based method that uses a low-grade waste energy and a microporous hydrophobic membrane to separate volatile compounds from non-volatile components driven by vapor pressure difference, which makes it a promising technology for nutrient treatment and recovery from human urine by separating nitrogen-based compounds from other parts and producing N, P, and K-based fertilizers separately [15-17]. In the MD system, the gaseous ammonia passes through the microporous MD membrane and is then harvested on the permeate side of the membrane [18]. The harvesting process can be carried out using an acid collector system composed of an acid solution that quickly reacts with ammonia forming nitrogenous-enriched compounds, which in most of cases can be directly used as liquid fertilizer. Although the MD process attracted attention as an efficient method for nutrient recovery, it still suffers from some challenges [18-20]. In the MD process, the permeate is affected by a simultaneous transfer of water and ammonia, which dilutes the permeate and the pH [21, 22].

The pH value in the permeate acid collector is one of the main parameters to control the rate of ammonia capture. Ammonium sulphate is a commercial fertilizer that can be used directly in agriculture. At pH around 3, sulphuric acid molecules have the ability to capture two molecules of ammonia, while at the lower pH range, a part of the reaction is directed towards the formation of NH_4HSO_4, which has lower fertilizer property and takes just one ammonium per sulphate molecule, and decreases the process efficiency [23]. Therefore, the persistence of the permeate pH at stable pH of around 3 is highly desirable. Additionally, higher pH represents a lower concentration of sulphuric acid that can contribute to a drop in capturing agents and, consequently, in ammonia capture. Therefore, designing a setup that could retain the pH at a constant level for a prolonged period is highly essential.

The production of ammonia in the urine solution increases the pH of the solution. Therefore, with the progress of the hydrolysis process and decomposition of urea, more ammonia is produced, which causes an increase in the pH of urea from 4 (fresh urine) to over 9 (hydrolyzed urine). Transferring produced ammonia into the permeate solution consumes available hydrogen ions in the acid collector and increases its pH. This process causes a decrease in the efficiency of fertilizer production and a drop in the rate of ammonia capture and fertilizer production [20].

The utilization of MD for harvesting ammonia from urine samples faces a significant challenge in controlling the permeate pH. Several studies have attempted to address this obstacle by renewing the permeate solution with fresh acid, thereby maintaining the desired pH range, dramatically increasing the acid consumption [24]. Additionally, some experiments have been limited to shorter durations to prevent the pH from increasing over time and halting the process [25]. The control of permeate pH has emerged as a key bottleneck in MD for nutrient recovery from urine. By addressing this challenge, the MD process can take a significant step forward in direct nutrient recovery from human urine.

In this study, a novel dual in-series MD setup was designed and used for preserving the acid collector pH in an optimum value to increase the fertilizer production rate in a competitive in-series reaction, simultaneously enhancing the ammonia-capturing efficiency. This innovative design not only preserves the desired pH range but also minimizes the amount of acid consumed, making it a promising approach for direct nutrient recovery from human urine. The performance of the system in different feed pH and feed temperature was evaluated and compared with a single MD module as a benchmark process. In this study, the waste heat from the feed bath was used to warm up the acid collector to lower the consumed energy. Furthermore, higher feed temperatures were studied to evaluate the effect of feed temperature on the ammonia flux and obtain the optimum operational condition. This research represents a significant step forward in advancing MD-based nutrient recovery technologies for sustainable resource utilization.

2. Experimental

2.1. Materials

A commercial flat sheet polyvinylidene fluoride (PVDF) membrane (0.22 µm pore size, 100 µm thickness), was supplied by Millipore (USA). Sodium hydroxide (NaOH), sulphuric acid (H_2SO_4), and hydrochloric acid (HCl) solutions were sourced from Sigma Aldrich Australia. The hydrolyzed urine was used as feed solution for undertaking the experiments. Hydrolyzed urine was collected from the University of Technology Sydney (UTS) Building 11 basement. In this 15-storey building, the nutrient recovery pilot facility in the basement collects all male human urine from each storey using waterless male urinals. The urine is reserved in tanks to complete the hydrolysis process.

2.2. Membrane distillation tests

The schematic diagrams of both single and dual in-series DCMD setups are shown in Figure 1. The experimental procedure for both setups was identical. The regular single MD setup was named Single MD, and the designed dual-MD module was named dual In-series MD. A commercial PVDF membrane was used for the tests with an effective surface area of 26 cm^2. The hydrolyzed urine as feed is heated up to the desired temperature and fed from a feed tank placed inside a water bath (WiseCircu, Digital Circulated Water Bath) into the DCMD module using a gear pump (Cole-Parmer Instrument, USA). The effect of feed temperature (T_f) on the system performance was studied by setting feed temperatures at 40, 50, 60, and 70 °C. The permeate of the 1st MD module was collected in an acid collector filled with 1 ml/L sulphuric acid solution. The acid collector is heated using a heat exchanger utilizing the waste heat from the feed tank. The permeate of the 2nd MD module was collected in a deionized (DI) water tank. The electric cooler was used to cool the permeate tank. The acid collector temperature was set at 40 °C, and the DI permeate water was set at 20 °C. In the single MD setup, the feed temperature was set at 40, 50, 60, and 70 °C and the permeate acid collector temperature was set at 40 °C.

All flow rates were adjusted to 400 ml/min using a gear pump and measured by a rotameter. The inlet temperatures to the module for both feed and permeates were recorded using a digital thermometer (Vernier Go Temp USB Temperature Thermometer Probe). The permeate is weighed using an electronic balance (ADAM PGL 8001) to evaluate the membrane flux. In addition, each bottle was sampled in regular intervals to assess the ammonia concentration. The pH and conductivity of feed, and 1st and 2nd stage permeates were continuously monitored and recorded using pH meter (Tris-Compatible Flat pH Sensor) and conductivity meter probes (HQ40d Portable Conductivity Meter).

In the single MD configuration (Fig. 1B), hydrolyzed urine as the feed solution is circulated over a commercial PVDF membrane at desired conditions, with the permeate solution (either as DI water or sulphuric acid) collected on the opposite side of the membrane. In the Dual In-series MD setup (Fig. 1A), the hydrolyzed urine is first fed through the first MD module, while the permeate is made of an acid solution. This acid solution then flows through as the feed solution for the second MD module where dewatering happens with DI water as the permeate solution.

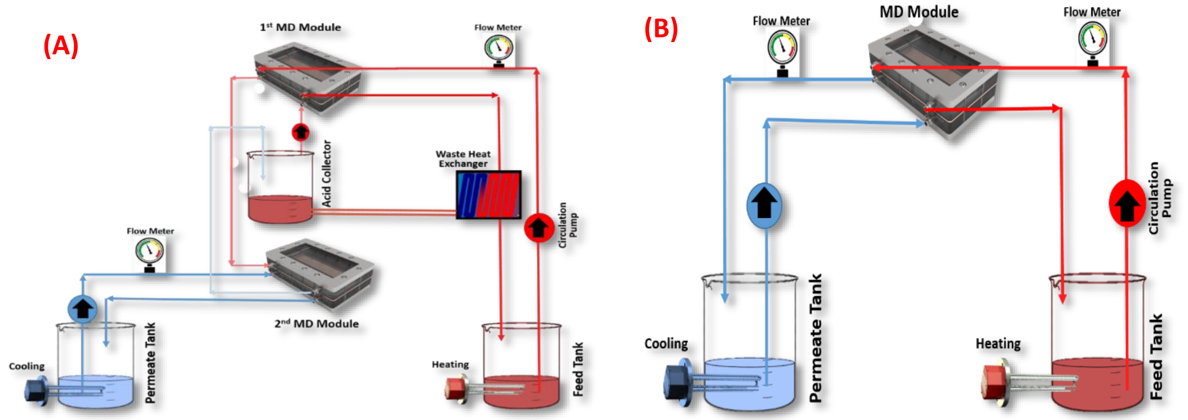

Figure 1. Schematic of (A) a dual, in-series MD module and (B) single MD module for ammonia from hydrolyzed human urine.

The effect of feed pH on the performance of the setups has been studied. For this purpose, the feed pH has been controlled by adding 10 M NaOH solution or 11.3 M HCl solution in the pH range of 6 to over 13, whereas the acid collector and permeate tank pH was initially 3 and 7, respectively. In the last part, the effect of feed flow rate on the membranes was studied. In this step, the circulating feed flow rate was adjusted from 100 to 400 mL/min, and the behaviour of the dual in-series MD module was investigated. The operating condition of the process is shown in Table 1.

Table 1. Experimental parameters and conditions for both single and dual in-series MD setups in the present study.

	Dual in-series MD			Single MD	
	Feed temp (^oC)	***Permeate 1 temp (^oC)***	***Permeate 2 temp (^oC)***	***Feed temp (^oC)***	***Permeate temp (^oC)***
Effect of temperature	40	40	20	40	40
	50	40	20	50	40
	60	40	20	60	40
	70	40	20	70	40
	Feed pH	***Permeate 1 pH***	***Permeate 2 pH***	***Feed pH***	***Permeate pH***
Effect of pH	6	3	7	6	3
	8	3	7	8	3
	9.5	3	7	9.5	3
	10.5	3	7	10.5	3
	12	3	7	12	3
	13.2	3	7	13.2	3
	Feed flow rate (ml/min)	***Permeate 1 flow rate (ml/min)***	***Permeate 1 flow rate (ml/min)***		
Effect of flow rate	100	400	400		
	200	400	400		
	300	400	400		
	400	400	400		

2.3. Characterisation and measurements

Samples were regularly taken from the first permeate tank every 15-minute interval through a sampling valve to measure the change in ammonia concentration. The concentration of ammonia was measured using a spectrophotometer (Spectroquant NOVA 60; Merck KGaA, Darmstadt, Germany). In this method, the samples were diluted using Milli-Q water (20-100 times), and then standardized parts were added to prepare the solution. The additives change the colour of the solution. The prepared sample is placed in the spectrophotometer to compare its colour and transparency with another standard solution to evaluate the ammonia concentration. Human urine is subject to hydrolysis and conversion of its complex compounds to smaller parts. In the hydrolysis process, urea, as the primary nitrogen source, is converted to ammonium and other ions in the following reaction. Excreted urea in urine is decomposed with time to ammonium via enzymatic hydrolysis process as follows [22, 26].

$$CO(NH_2)_2 + 3H_2O \rightarrow 2NH_4^+ + HCO_3^- + OH^- \qquad (1)$$

The produced ammonium (NH_4^+) is in equilibrium with dissolved ammonia:

$$NH_4 + OH^- \leftrightarrow NH_3(aq) + H_2O \qquad (2)$$

The progress of this reaction directly depends on the temperature and pH. The dissolved ammonia is in equilibrium with gaseous ammonia:

$$NH_3(aq) \leftrightarrow NH_3(g) \tag{3}$$

This equation demonstrates the equilibrium between ammonium ions and free ammonia gas, in which the rate of ammonia gas (free ammonia) production directly depends on pH and temperature, as shown in the following equation, where higher free ammonia gas is produced by increasing pH and temperature [27].

$$\mathrm{FA} = [\mathrm{TAN}] \times \frac{10^{\mathrm{pH}}}{e^{\frac{6344}{273+\mathrm{T}}} + 10^{\mathrm{pH}}} \tag{4}$$

In this equation, TAN is Total Ammonia Nitrogen measured by the spectroscopic method, and FA is Free Ammonia gas.

In the MD process, water and ammonia could transfer through the membrane pores, whereas the operational condition determines the passing competition. In this study, water and ammonia flux is calculated to demonstrate the influence of operating conditions on the performance of the process. The flux of membranes is calculated using the following equation:

$$J = \frac{\Delta m}{A \times \Delta t}$$

where J is membrane flux (L/m^2h or LMH), Δm is accumulated weight in the permeate (kg), A is membrane surface area (m^2), and Δt is time (h). The accumulated weight of water is measured using a digital balance and is normalized to take into account the accumulated weight of absorbed ammonia, which is measured after analyzing the permeate samples.

Ammonia flux is an important factor in the nutrient recovery process, as it shows nitrogen transport between wastewater and permeate. Understanding ammonia flux is essential for optimizing the efficiency of nutrient recovery systems and minimizing the environmental impact of waste streams from domestic, agricultural, and industrial processes. The ammonia transmembrane flux is the rate of ammonia transferred through the membrane pores during nutrient recovery via the MD process, which shows the process efficiency. Ammonia flux (J_A, g/m^2h) can be determined by dividing the ammonia capture ratio (CR) over the membrane surface area (A). CR (g/h) is the slope of the linear line fitted on the amount of ammonia captured in the acid collector versus time [27].

$$J_A = \frac{CR}{A} \tag{5}$$

Moreover, the ammonia concentration profile in the acid collector was used to measure the Specific Ammonia Transfer (SAT), which shows the competition of water and ammonia in the MD process:

$$SAT = \frac{Transferred\ Ammonia\ (g)}{Transferred\ Water\ (g)} \tag{6}$$

In this equation, transferred ammonia is the amount of ammoniacal nitrogen transferred through the membrane pores, and transferred water is the weight of accumulated water in the permeate that passes through the pores. The SAT value is a precise ratio to measure the competency of the process for nutrient recovery. Higher SAT is favourable in the MD process, which shows selective transportation of ammonia in the process.

The availability of sulphuric acid in the permeate is a crucial parameter determining the driving force for capturing the ammonia and evaluating the ammonia transfer rate. The ammonia transfer in the presence of sulphuric acid in the permeate causes the following reaction:

$$NH_3 + H_2SO_4 \leftrightarrow NH_4HSO_4\ /\ (NH_4)_2SO_4 \tag{7}$$

In pH<1, mostly NH_4HSO_4 is produced, and with pH rise, the ratio of $(NH_4)_2SO_4$ to NH_4HSO_4 increases. pH around 3 is the optimum point to produce a higher percentage of ammonium sulphate $(NH_4)_2SO_4$ as suitable fertilizer [25].Therefore, the reaction of ammonia and sulphuric acid directly depends on the permeate pH [23].

The mass transfer coefficient was utilized to objectively evaluate and compare the impact of different operational conditions on the efficiency of ammonia removal. The overall mass transfer coefficient (K_{ov}) was determined by analyzing the time-dependent variation in ammonia concentration, employing the following calculation.

$$K_{OV} = \frac{V_o}{A\Delta t}\ln\left(\frac{C_0}{C_t}\right) \tag{8}$$

In the equation, C_0 represents the initial total ammonia concentration, Δt signifies the time, V_o represents the total volume of ammonia solution, and A represents the area per volume, respectively.

Based on the pore size dimension, the movement of ammonia gas through the porous membrane follows the Dusty gas model, which comprises Knudsen and viscous diffusions in a sequential network. The passage of ammonia across the membrane can be described as a resistances-in-series model. The comprehensive mass transfer coefficient, K_{ov}, can be represented as the summation of mass transfer coefficients on the aqueous feed side, membrane, and receiving solution side. Furthermore, ammonia is assumed to be in thermodynamic equilibrium with its vapor at the liquid-membrane interfaces and the reaction between ammonia and sulphuric acid is regarded as instantaneous. Consequently, Henry's law can be applied at these interfaces, and laminar conditions are considered for the flat sheet membrane module. Equation (8) was derived from mass balances of the permeate and the feed solutions, assuming that the acid collector tank is sufficiently large to capture all the ammonia [28-30].

3. Results and discussion

3.1. DCMD performance

Permeate pH is one of the influential parameters affecting the performance of the gas stripping process [25]. The acid solution's effect on ammonia capture has been studied in this work. In the first step, a single MD module was used to treat the hydrolyzed urine, and DI water or sulphuric acid solution was used on the permeate side. The trend of pH change in the permeate is shown in **Figure 2**, and results show that the pH rises rapidly and reaches above 9 in the case of DI water as the permeate. Consumption of hydrogen ions during ammonia harvesting increases the pH over time. However, the rate of the pH increase around pH = 7 is higher, mostly due to the logarithmic effect of hydrogen concentration on pH values. This result complies with the relationship between hydrogen ions concentration and pH [31].

On the other hand, the rate of increase in pH for the acid collector is dramatically lower than DI water in the first hours. The permeate pH in the acid collector reached from 3 to 4 during the first 2 hours, while for the DI water, it rose from 6.5 to over 9. This signifies that the presence of hydrogen ions and cations that absorb ammonia gas decreases the pH rise rate. In addition, the results show that the conductivity of the DI water increases with time and reaches over 1500 µS/cm, which reveals the ammonia transfer from the feed to the permeate. Opposite to normal MD system, wherein a conductivity increase with time is a sign of wetting, in urine recovery, it is a sign of ammonia transfer.

In the acid collector system, the conductivity trend is different, and the acid solution showed high conductivity. By transferring ammonia, the acid-ammonia reaction takes place, and NH_4SO_4 is formed which has lower conductivity than the reactants [25]. Therefore, with the progress of the process, more ammonia was transferred, and the conductivity continuously dropped from 6200 µS/cm of initial conductivity to around 1700 µS/cm. However, the trends depict that after a while, the conductivity gradually rose with time and reached to around 2700 µS/cm. The turning of the trend is mainly attributed to the dilution of the acid solution in which some transferred ammonia is absorbed by water without any reaction in the permeate. Therefore, there is a competition between the reacted ammonia (which decreases the conductivity) and absorbed ammonia (which increases the conductivity), and the first part is more dominant in the first hours of the process.

Several studies [20, 25, 30, 32] have investigated nutrient recovery using an acid collector system, and the results consistently demonstrate that ammonia transfer to the permeate leads to an increase in pH and a decrease in conductivity of the acid solution. The rate of change in pH and conductivity is directly influenced by the rate of ammonia transfer and the reaction between ammonia and the acid solution, with specific conditions observed for each type of acid. The composition of the acid solution and its reaction after ammonia transfer play a crucial role in determining the quality of the produced fertilizer. Notably, sulphuric acid, known for its strong acidity characteristics, exhibits a higher ammonia capture capacity and yields a desirable final product for fertilizer production [20, 25, 30, 32].

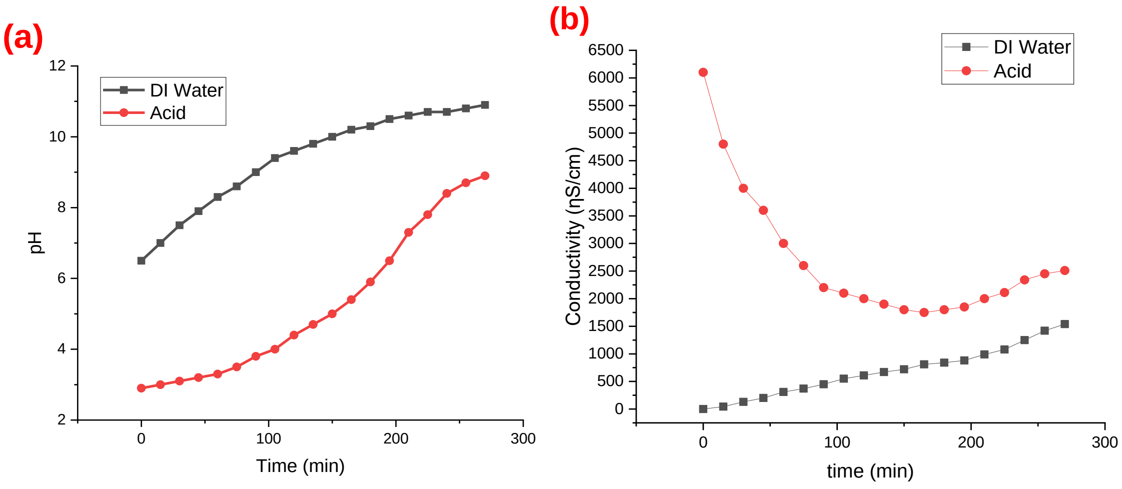

Figure 2. Changes in the pH (a), and conductivity (b) of the permeate with time for a single MD setup using either DI water only or with sulphuric acid in the permeate (Feed temperature = 60 °C, Permeate temperature = 40 °C, feed pH = 12, acid collector pH = 3 and DI water collector pH = 7, Feed and permeate flow rate = 400 ml/hr).

In the next step, the dual in-series MD module was used to study the effect of various parameters on the performance of the designed module. The result of the pH change in the acid collector for both setups is shown in **Figure 3**. The result shows that the pH in the dual in-series MD setup is constant during the test, while the single MD setup showed an increase in pH after the first hour of testing and quickly reached over pH 8 after 5 h of test. The ability of the MD process to remove volatile compounds, dewater the permeate and preserve other parts leads to an increase in the concentration of the sulphuric acid solution, thereby maintaining the pH and compensating a part of the pH increase by ammonia transfer. The single setup MD dealt with pH increase, sourced from ammonia transfer and ion consumption, while the in-series MD showed constant pH in early time and a slight pH decrease with time. The captured ammonia (NH_3) in the acid collector reacts with the hydrogen ions

(H^+) converting into stable ammonium due to low pH. Therefore, only water is transferred to the permeate side in the second module and collected in the tank. The water transfer causes an increase in the concentration of H^+ ions and helps to preserve the first permeate pH as well as increase the concentration of the potentially produced fertilizer.

In addition, membrane fouling is one of the main issues in treating hydrolyzed urine. In the dual in-series setup, the first membrane deals with hydrolyzed urine containing fouling agents, while the second membrane does not involve fouling agent. Therefore, the net permeate flux that entered the first collector decreased with time, while the output flux was almost constant. This negative net flux causes an increase in the H^+ ion concentration in the acid collector and, consequently, drops the pH or helps in maintaining almost constant pH values in the acid collector.

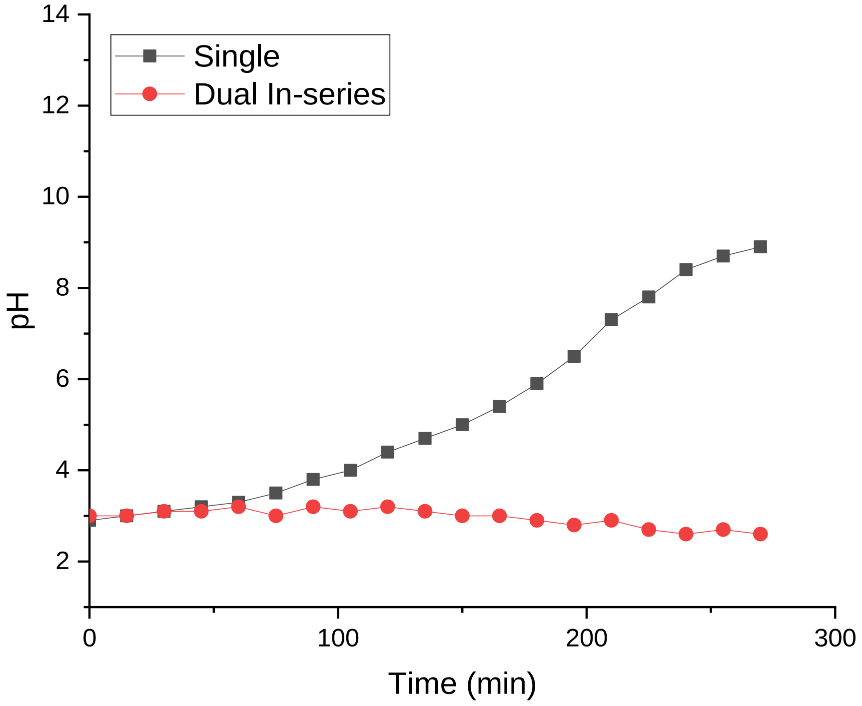

Figure 3. Change in permeate pH (acid collector) with time for both single and dual in-series MD modules (Single MD: Feed temperature = 60 °C, Permeate temperature = 40 °C, Acid collector initial pH = 3, feed pH = 12, feed and permeate flow rate = 400 ml/hr. Dual in-series MD: Feed temperature = 60 °C, Permeate temperature = 40 °C, feed pH = 12, acid collector initial pH = 3, Feed and permeate flow rate = 400 ml/hr, DI water collector pH = 7, DI water collector temperature = 20 °C).

Figure 4 shows the net accumulated water flux of the membranes in a single and in-series MD setups (for 1st MD module as shown in Fig. 1). The results show that the net water flux for both membranes is approximately similar during the test, however, fouling caused a slight decrease in flux over time, and dropped from about 5 LMH to 4.5 LMH

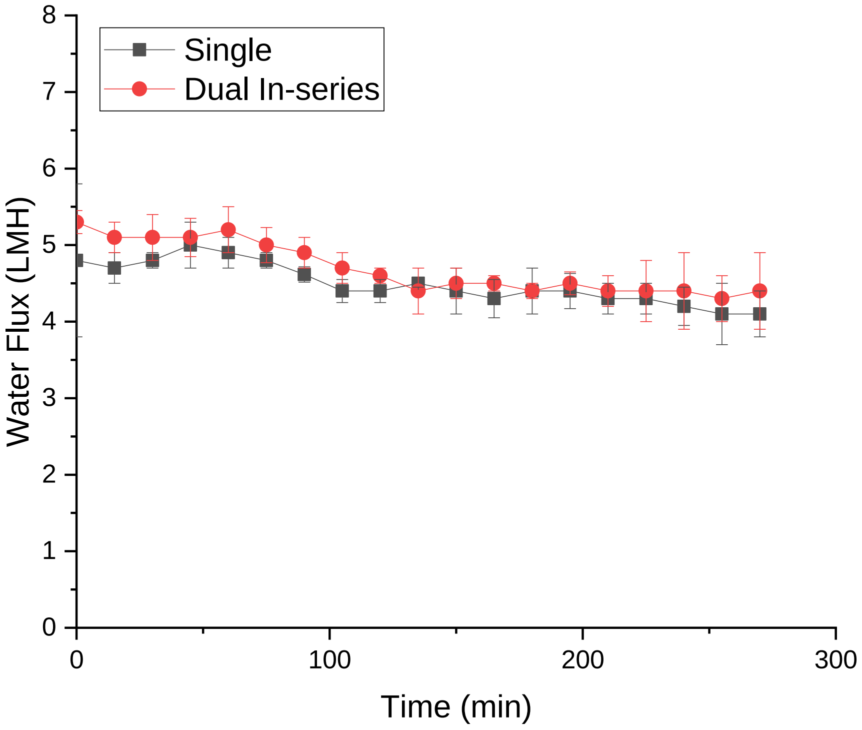

Figure 4. Water flux for both single and dual in-series MD systems (flux of 1st MD Module as shown in Fig. 1) (Single MD: Feed temperature = 60 °C, Permeate temperature = 40 °C, feed pH = 12, acid collector initial pH = 3, feed and permeate flow rate = 400 ml/hr. Dual in-series MD: feed temperature = 60 °C, permeate temperature = 40 °C, DI water collector temperature = 20 °C, feed pH = 12, acid collector initial pH = 3, DI water collector pH = 7, Feed and permeate flow rate = 400 ml/hr).

The change of captured ammonia with time in different pH was used to obtain the captured ratio (see **Figure 5a**). The values of CR in different pH and relevant ammonia flux are measured to calculate the ammonia flux. The results show that the ammonia capture ratio and ammonia flux increased with pH, which complies with the free ammonia correlation (equation 4). A comparison of single and dual in-series MD results shows that the dual in-series MD setup has a higher regression line slope, which derives greater ammonia flux. Moreover, the results show that by increasing the pH over 9.5, the capture ratio significantly increased and reached about 150 mg/h at pH = 10.5 and 480 mg/h at pH>13. The present results demonstrated that the CR of the dual in-series MD setup is over twice the single one used in other studies (40-90 mg/h) [33].

The rise in capture ratio as pH increases, leading to greater ammonia flux, is ascribed to the conversion of more ammonium to ammonia gases. Equation 2 reveals that as pH rises, the concentration of OH^- also increases, which facilitates the consumption of NH_4^+ and the production of more NH_3. The higher levels of accessible ammonia on the membrane surface enhance the potential for ammonia transfer and consequently improve the ammonia flux of the membrane. Nevertheless, the elevation of pH results in greater consumption of alkali, and the addition of alkali is anticipated to be the primary cost factor for ammonia recovery [24]. Therefore, there is a need to strike a balance between achieving high ammonia flux and minimizing the cost of alkali consumption for ammonia recovery. This can be achieved through optimization of operating conditions such as pH and concentration of ammonia in the feed solution.

The ammonia flux also increased from 2.34 g/m^{2}h (pH = 6) to over 29 g/m^{2}h (pH = 10) and 112 g/m^{2}h (pH>13) (see **Figure 5b**). Similar ammonia flux range was achieved in a study by McCartney, however, their initial pH was lower than in our present study [34]. The results show that the ammonia capture in a dual in-series MD setup is about 50% higher than that in a single MD setup. This difference can be attributed as a result of the constant H$^+$ ion concentrations maintained in the acid-collector due to the nature of the in-series MD setup, where, the excess water is continuously removed from the acid collector, and the concentration of ions in the acid collector is constantly maintained high. A higher concentration of available ions increases the reaction rate of transferred ammonia with ions and enhances the ammonia capture rate. Moreover, the ammonia flux could be kept constant during the process and prevent the drop of ammonia flux with time [27, 35]. When comparing the ammonia flux in this study to those of previous studies, it was found that the ammonia flux is strongly influenced by various factors, including the initial concentration of ammonia in the feed, membrane thickness and pore sizes, and the characteristics of the permeate [29, 35]. What sets this study apart from others is that it modifies the process by utilizing a series of MD modules, thus increasing the acid collector's potential to harvest a higher percentage of ammonia. As a result of this modification, there was an improvement in the ammonia flux observed.

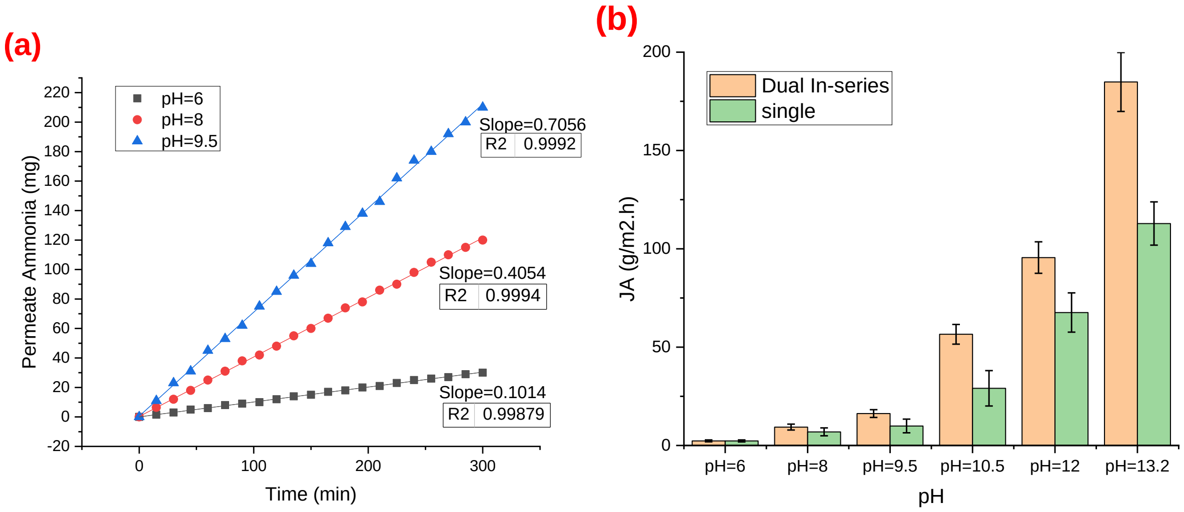

Figure 5. Linear regression of ammonia in the permeate vs time for measurement of ammonia capture ratio for the (a) dual in-series-designed MD setup and (b) ammonia flux for both single and in-series MD setups (Single MD: Feed temperature = 60 °C, Permeate temperature = 40 °C, acid collector initial pH = 3, feed and permeate flow rate = 400 ml/hr. Dual in-series MD: feed temperature = 60 °C, permeate temperature = 40 °C, DI water collector temperature = 20 °C, acid collector initial pH = 3, DI water collector pH = 7, Feed and permeate flow rate = 400 ml/hr).

3.2. Effect of feed temperature

The effect of feed temperature on the system's performance has been studied, and the results are shown in **Figure 6**. The trend indicates that the dual in-series MD obtained higher ammonia concentration and kept its difference over time. Additionally, with an increase in feed temperature, a higher value of ammonia concentration is achieved in a shorter period of time. In other words, higher feed temperatures result in a higher ammonia transfer rate. The increase in the feed temperature obtained higher water and ammonia flux in all setups, which are attributed to the increased driving force of the process. In this study, the permeate temperature was kept constant, while only the feed temperature was varied. Temperature serves as the primary driving force in MD systems, and to ensure a reliable interpretation of the impact of feed temperature on system performance, it is crucial to maintain a constant permeate temperature while varying only the feed temperature. Higher feed temperature and constant permeate temperature (T_p) caused the enhancement in driving force and a greater amount of transfer for both water and ammonia. Other research studies have also investigated the impact of temperature on water flux and ammonia capture, and their findings have consistently shown that capturing ammonia can triple when the temperature rises from 50°C to 75°C [35]. Furthermore, the equilibrium of urea conversion to ammonium ions directly depends on the temperature, in which higher ammonia concentration is available in the feed solution at elevated temperature. A comparison of the single and dual in-series MD setups shows that the water flux and ammonia concentration ratio rise are almost identical, while the direction of SAT is different in elevated temperatures. Although ammonia transfer is higher in elevated temperatures, the water flux is more affected by temperature and causes greater rate of water transfer than ammonia transfer. Therefore, the ratio of transferred ammonia to water dropped with temperature [11, 36]. Other scientific studies have corroborated the decrease in saturation (SAT) value with increasing temperature. For example, Yu et al's research findings revealed that the SAT value dropped to more than one fourth of its initial value when the temperature was raised from 40°C to 70°C [37]. Furthermore, other research studies have also calculated saturation (SAT) values for a basic DCMD setup, which fell within the range of the SAT values obtained in the current study for a single MD setup. However, notably, the SAT value for the dual in-series setup in the current study was found to be significantly higher at 0.05, compared to the values reported in other studies for basic DCMD which was 0.02 [36]. Moreover, the dual in-series MD setup showed a higher drop in SAT with temperature compared to the single setup. This trend is attributed to the decline of ion concentration in the acid collector due to a higher water input and the decrease in the SAT value.

At higher feed temperatures, the feed ammonia concentration decreased more rapidly. This positive effect of increasing feed temperatures on K_{ov} is demonstrated in Figure 7c. As the temperature increases, the rate of ammonia dissociation in urine generally increases. This leads to a higher concentration of ammonium ions in the urine solution. The increase in dissociated ions affects the chemical equilibrium between ammonia and ammonium ions. The mass transfer coefficient, on the other hand, is a measure of how efficiently ammonia molecules are transported across a membrane or interface. It is influenced by various factors, including temperature. Generally, an increase in temperature can increase the concentration of ammonia on the feed side, which enhances the driving force for ammonia transfer across the membrane. On the other hand, temperature enhances the mass transfer coefficient due to the increased kinetic energy of the molecules, resulting in more rapid diffusion or movement of ammonia through the urine solution or across a membrane [8, 38].

Comparing single and dual in-series systems shows that dual in-series systems kept its distance from the single module in all temperatures. The increased NH_3 diffusion in both the bulk solution and membrane pores, attributed to the higher feed temperature, resulted in a higher mass transfer coefficient. Additionally, the feed solution contained more volatile ammonia due to the endothermic nature of ammonium ion dissociation. As a result, the higher mass transfer coefficient facilitated faster ammonia removal from the feed solution, leading to improved separation efficiency in the membrane process. On the other hand, in dual in-series systems, the acid collector with constant pH preserved its capacity for capturing more ammonia and derived higher mass transfer. Specifically, K_{ov} increased from 1.79 to 8.24×10^{-6} m/s as the feed temperature rose from 40 to 70 °C [39-41]. The mass transfer coefficient for a single MD module obtained in this study is within the range of values reported in previous studies [28-30]. However, the coefficient is greatly influenced by the membrane's characteristics, as well as the specifications of the feed and permeate. The notable aspect of this study is that it enhances the coefficient by modifying the acid collector's characteristics and extending the duration of ammonia capturing in the permeate. This results in a higher value of the mass transfer coefficient.

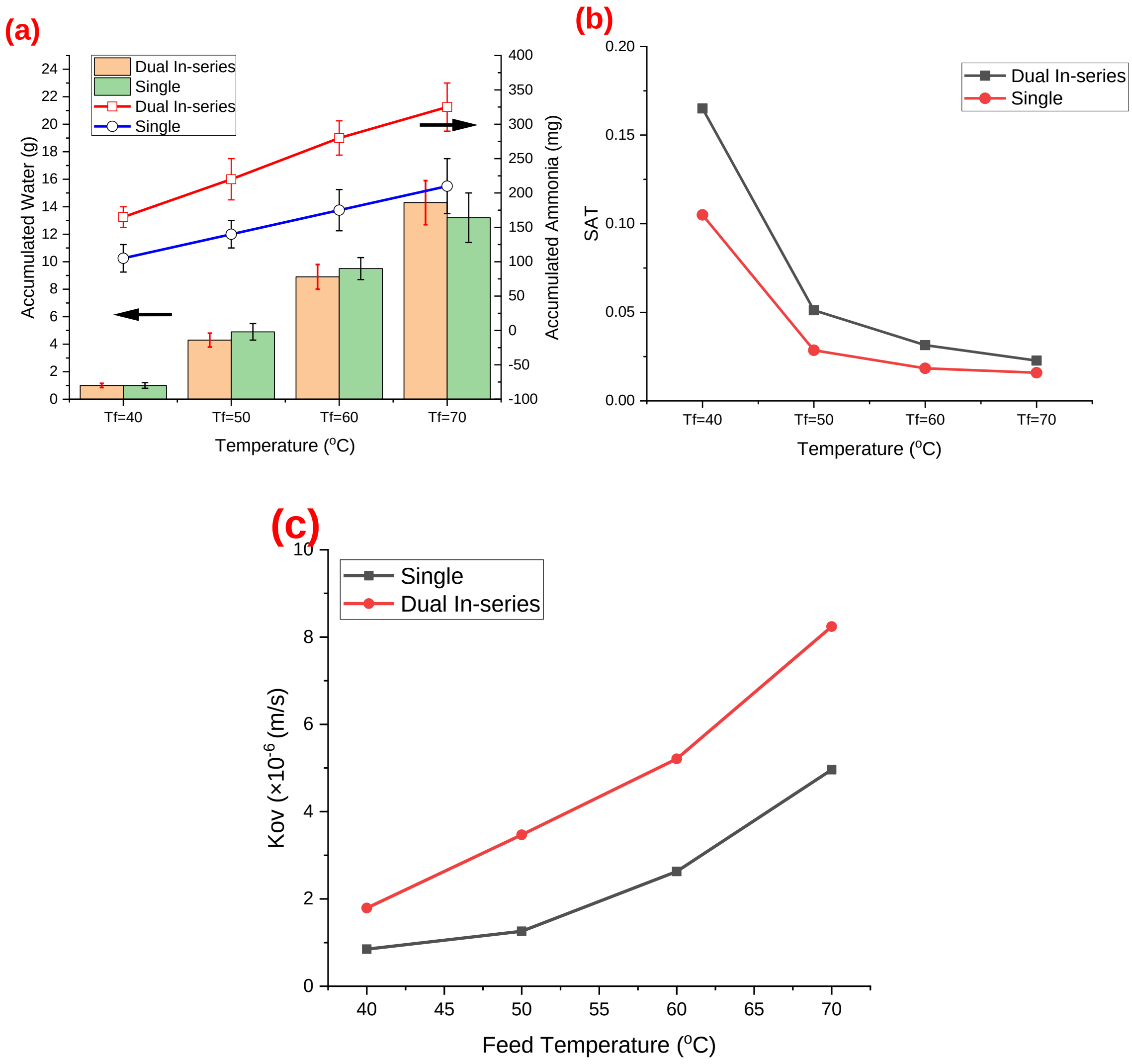

Figure 6. (a) Accumulated water and ammonia measurements in the acid collector for both single and in-series MD setups and, (b) the variation of SAT for both single and in-series setups in different feed temperatures (T_f), (c) the mass transfer coefficient change in different temperatures (Single MD: Permeate temperature = 40 °C, feed pH = 12, acid collector initial pH = 3, feed and permeate flow rate = 400 ml/hr. Dual in-series MD: permeate temperature = 40 °C, DI water collector temperature = 20 °C, feed pH = 12, acid collector initial pH = 3, DI water collector pH = 7, Feed and permeate flow rate = 400 ml/h).

3.3. Effect of pH

The conversion rate of ammonium to ammonia in hydrolyzed urine directly depends on the temperature and pH [27]. Therefore, the change in pH causes a difference in the availability of free ammonium for transferring to the permeate side [42]. **Figure 7** shows the ammonium concentration in the acid collector in both single and dual in-series MD setups. The results demonstrated that pH greatly influences the rate of ammonia capturing. In pH equivalent to acidic or neutral conditions, the ammonium concentration in the acid collector is negligible, and the system's efficiency is very low. By increasing the pH, the ammonium concentration rose dramatically in the feed, which is attributed to the conversion of equilibrium reaction towards forming more ammonium ions. Consequently, more ammonia is produced and increased the driving force of ammonia from the feed side to the acid collector and more ammonia is transferred via the membrane pores. This behaviour caused the increase in mass transfer coefficient, as shown in Figure 7b [18, 36, 42]. These results indicate that running the process at a little higher pH than the natural pH of hydrolyzed urine enhances the performance of the process. However, it is worth mentioning that the change of pH to values greater than 11 needs a higher volume of caustic soda, thus can make additional waste, which is not favorable. Therefore, an optimum pH value could be reached to create a balance between the amount of added basic source and captured ammonium.

From Fig 7a, it shows that approximately no ammonium was captured in pH around 6 in both single and dual in-series MD setups [20]. However, the ammonium concentration in pH = 9.5 rose to over 200 mg/l in the dual in-series MD setup, which is twice higher than the single one. Then after just one grade increase in pH (pH =10.5), the ammonia captured soared to over two times for both single and dual in-series MD setups. These results demonstrate that although an increase in pH causes an increment in the ammonia capture and can reach up to 700 mg/L at pH = 13.2, the high volume of basic source added to the feed decreases its efficiency [30]. In other investigations, it has been observed that there is an increase in ammonia capture with higher pH levels. Yu et al. conducted a study where they found that an increase in pH significantly influenced the transfer of ammonia, primarily due to the rise in free ammonia concentration on the feed side of the membrane under a constant driving force condition [36].

Fig 7b illustrates the relationship between K_{ov} and feed pH values for both single and dual in-series systems. The results show that the dual in-series module has a higher mass transfer coefficient, specially in higher pH, mainly due to higher ammonia capture by time in the dual in-series system. The continuous dewatering of the first module provides the proper conditions for transferring of ammonia to the acid collector. It can be observed that K_{ov} increased significantly from 0.03 to 7.24×10^{-6} m/s as the pH value increased from 6 to 12 for the dual in-series set-sup. However, when the pH value further increased to 13.2, the ammonia transfer coefficient rate drop. This suggests that there may be an upper and lower limit to the effect of pH on the ammonia transfer coefficient [39-41].

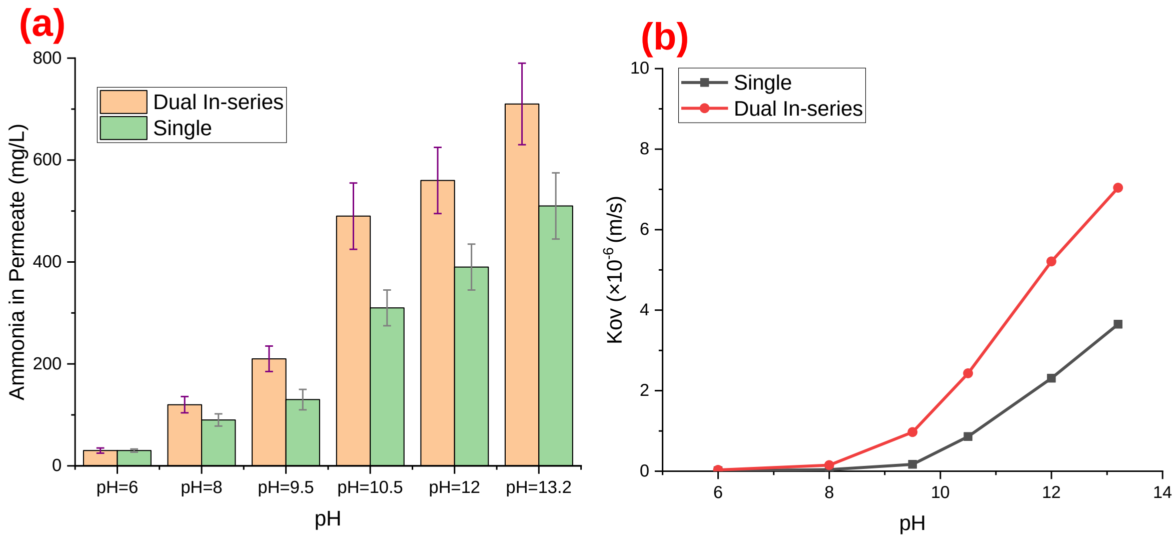

Figure 7. a) Ammonia concentration in the acid collector at various pH values for both single and Dual In-series MD configuration, b) the change in mass transfer coefficient with the pH (Single MD: Feed temperature = 60 °C, Permeate temperature = 40 °C, acid collector initial pH = 3, feed and permeate flow rate = 400 ml/hr. Dual in-series MD: feed temperature = 60 °C, permeate temperature = 40 °C, DI water collector temperature = 20 °C, acid collector initial pH = 3, DI water collector pH = 7, Feed and permeate flow rate = 400 ml/hr).

The advancement of membrane technologies and the advantages of the MD process has drawn attention to its potential use in utilizing human urine resources. Table 2 provides an overview of some studies that have utilized the MD process and compares their outcomes. Our results indicate good ammonia flux and robust ammonia recovery in comparison with others through our dual in-series design. Although the MD process has demonstrated significant success in separating and recovering nutrients from source-separated human urine, further research is necessary to fully comprehend the benefits and drawbacks of this system. Nonetheless, applying the MD process to recover nutrients from source-separated human urine directly presents an appealing solution to mitigate the environmental impact of nutrient waste and meet the demand for fertilizers.

Table 2. Application of the membrane distillation process in nutrient recovery and comparison of their performance with the present study.

Type of Feed	**Module**	**Performance**	**Ref.**
Simulated urine feed solution composed of 250 mM ammonium hydroxide and 250 mM ammonium bicarbonate in DI water	Isothermal membrane distillation	Ammonia Flux: 14 mol/m^{2}h SAT: -3 (mol H_2O/mol NH_3)	[34]
Anaerobic digestion effluent from a large-scale biogas plant using pig manure	Isothermal membrane distillation	Removal efficiency over 98% in 12 h Ammonia flux 8 g/m^{2}h Mass transfer coefficient: 7×10^{-7}	[29]
Source-separated human urine	Membrane Distillation	SAT: 0.01 g-N/g-H_2O	[36]
Digester centrate from Aaby wastewater treatment plant in Aarhus	Vacuum and direct contact membrane distillation	Recovery factor: 80% Ammonia flux: 18 g/m^2.h	[43]

Fresh urine, acidic urine, and hydrolysed urine	Membrane distillation	SAT: 0.001 g-N/g-H_2O Removal factor: 90% Higher ammonia recovery for the hydrolyzed urine Higher fouling of the hydrolzsed urine	[20]
Synthetic urine	Direct contact membrane distillation	Mass transfer coefficient: 8×10^{-5} Ammonia flux: 10 g/m^2.h Ammonia removal efficiency of DCMD, HMC and MDCMD was 52%, 88% and 99.5%, respectively	[30]
Aerobic effluent	Direct contact membrane distillation	Mass transfer coefficient: 1.0×10^{-6} ammonia removal efficiency over 99.95% High fouling of the membrane	[41]
Hydrolyzed urine	Direct contact membrane distillation	31% ammonia rejection Urine concentration about 18 times 97% rejection of P and K	[18]
Source-separated human urine	Direct contact membrane distillation	Removal rate 81.8% Doing hybrid system of nitrificatino and distillation	[44]
Source-separated human urine	Direct contact membrane distillation	SAT= 0.17 (Max) Ammonia flux= 180 g/m^{2}h Mass transfer coefficient = 11×10^{-6}	Present study

3.4. Effect of flow rate

The variation of the ammonia in the acid collector at different feed flow rates is shown in **Fig. 8**. It can be seen that a rise in flow rate increased the ammonia capture. The ammonia concentration increased from 360 mg/L to 490 mg/L when the feed flow rate rose from 100 mL/hr to 400 mL/h. Since dissolved agents require a specific time for transferring from the bulk fluid to the membrane boundary layer, changes in feed velocity affect the amount of the transferred water and ammonia. On the other hand, rises in flow rate enhance the turbulence of the fluid and decrease the mass transfer boundary layer and improve the mass transfer coefficient [30]. Higher mass transfer coefficient yields enhancement in transferring ammonia, which is in gaseous form in the feed flow, and, as shown in Figure 8, derives higher ammonia transfer. Meanwhile, a higher flow rate also decreases the heat transfer boundary layer and affects the driving force for water vapour transfer. However, the results show that ammonia transferring is more affected by flow rate, and a higher flow rate is more desirable. The effect of flowrate on the performance of the MD process was also studied by Xie et al. and results showed the same trend for the ammonia flux and permeate flux which rose about 100% by doubling the flowrate [35].

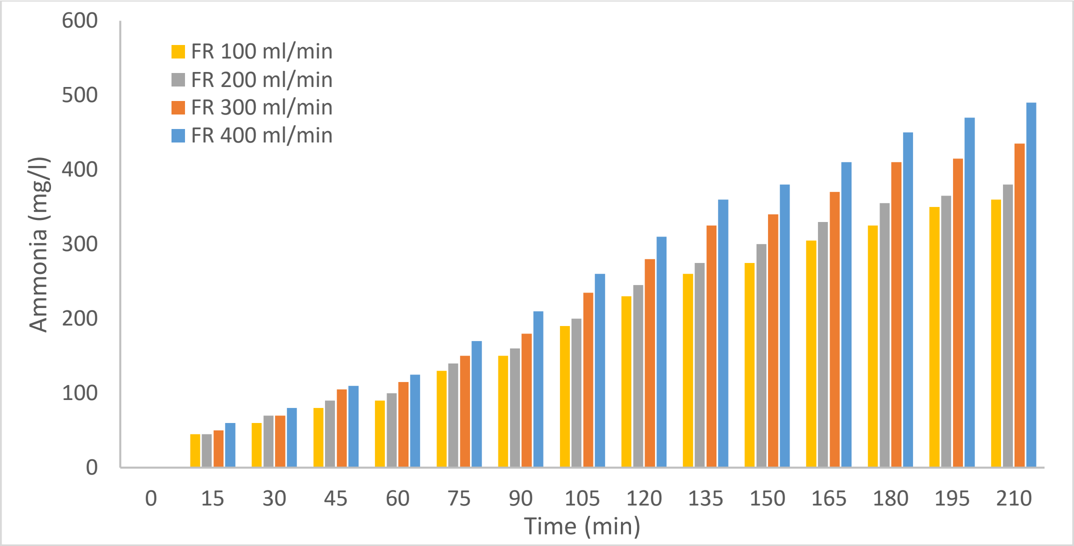

Figure 8. Variation of the ammonia concentration in the acid collector at different feed flow rate in a dual, in-series MD module (Dual in-series MD: feed temperature = 60 ºC, permeate temperature = 40 ºC, DI water collector temperature = 20 ºC, feed pH = 12, acid collector initial pH = 3, DI water collector pH = 7).

4. Conclusion

The harvesting of ammonia from hydrolyzed human urine in an acid collector via a dual, in-series MD process was proven to be technically feasible for nutrient recovery and potential fertilizer production. Controlling the acid collector pH has been observed to be an important parameter to retain the efficiency of the process. In the designed dual in-series MD module setup, the dewatering of the acid collector can provide two simultaneous benefits: it increases the concentration of H^+ ions and, consequently, the permeate pH, and it also increases the concentration of produced fertilizer. The results revealed that the dual in-series-designed MD modules produced higher transfer rate and ammonia capture compared to single MD set up. While the water flux was comparable between the single and dual in-series setups, the ammonia flux in the in-series MD reached over 180 g/m^2h, which was approximately 80% higher than the single setup. Moreover, higher specific ammonia transfer (SAT) values were achieved at normal pH of hydrolyzed urine in the dual in-series setup. The results also demonstrated that at low feed temperatures, the SAT value of the dual in-series setup was significantly higher than the single setup (0.17 for dual and 0.1 for single), and these values tended to converge as the temperature increased (0.025 for dual and 0.02 for single). However, both flux and accumulated ammonia values showed a significant increase with temperature increment. The utilization of a dual in-series MD setup proved to be effective in maintaining a constant acid collector pH during the test, in contrast to the single MD setup where pH continuously rose to over 9, resulting in decreased process efficiency. Furthermore, increasing the feed flow rate from 100 mL/min to 400 mL/min resulted in an increase in ammonia capture from 360 mg/L to 490 mg/L. These findings highlight that modifying the regular setup and designing MD modules specifically to enhance process efficiency is a promising approach for increasing nutrient recovery from urine. Overall, the findings suggest that the dual MD in-series module process has the potential to improve nutrient recovery from human urine, and further research could focus on optimizing the operational conditions for enhanced performance.

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5. References

[1] P. Simha, M. Ganesapillai, Ecological Sanitation and nutrient recovery from human urine: How far have we come? A review, Sustainable Environment Research 27 (2017) 107-116.
[2] S. Guida, L. Van Peteghem, B. Luqmani, M. Sakarika, A. McLeod, E.J. McAdam, B. Jefferson, K. Rabaey, A. Soares, Ammonia recovery from brines originating from a municipal wastewater ion exchange process and valorization of recovered nitrogen into microbial protein, Chem. Eng. J. 427 (2022) 130896.
[3] Á. Robles, D. Aguado, R. Barat, L. Borrás, A. Bouzas, J.B. Giménez, N. Martí, J. Ribes, M.V. Ruano, J. Serralta, New frontiers from removal to recycling of nitrogen and phosphorus from wastewater in the Circular Economy, Bioresour. Technol. 300 (2020) 122673.
[4] T. Yan, Y. Ye, H. Ma, Y. Zhang, W. Guo, B. Du, Q. Wei, D. Wei, H.H. Ngo, A critical review on membrane hybrid system for nutrient recovery from wastewater, Chem. Eng. J. 348 (2018) 143-156.
[5] L. Wooram, A. Seonyoung, C. Yongju, Ammonia harvesting via membrane gas extraction at moderately alkaline pH: A step toward net-profitable nitrogen recovery from domestic wastewater, Chem. Eng. J. 405 (2021) 126662.
[6] A. Patel, A.A. Mungray, A.K. Mungray, Technologies for the recovery of nutrients, water and energy from human urine: A review, Chemosphere 259 (2020) 127372.
[7] J. Ren, D. Hao, J. Jiang, S. Phuntsho, S. Freguia, B.-J. Ni, P. Dai, J. Guan, H.K. Shon, Fertiliser recovery from source-separated urine via membrane bioreactor and heat localized solar evaporation, Water Res 207 (2021) 117810.
[8] A. Almuntashiri, A. Hosseinzadeh, F. Volpin, S.M. Ali, U. Dorji, H. Shon, S. Phuntsho, Removal of pharmaceuticals from nitrified urine, Chemosphere 280 (2021) 130870.
[9] U. Badeti, N.K. Pathak, F. Volpin, U. Dorji, S. Freguia, H.K. Shon, S. Phuntsho, Impact of source-separation of urine on effluent quality, energy consumption and greenhouse gas emissions of a decentralized wastewater treatment plant, Process Saf. Environ. Prot. 150 (2021) 298-304.
[10] T.M. Martin, F. Esculier, F. Levavasseur, S. Houot, Human urine-based fertilizers: A review, Crit. Rev. Environ. Sci. Technol. 52 (2022) 890-936.
[11] Z. Hu, W. Qing, W. Zhang, Dewatering of source-separated human urine for nitrogen and water recovery using air gap membrane distillation (AGMD), AGUFM, 2020, pp. H115-0005.

[12] A.N. Petukhov, A.A. Atlaskin, S.S. Kryuchkov, K.A. Smorodin, D.M. Zarubin, A.N. Petukhova, M.E. Atlaskina, A.V. Nyuchev, A.V. Vorotyntsev, M.M. Trubyanov, I.V. Vorotyntsev, V.M. Vorotynstev, A highly-efficient hybrid technique – Membrane-assisted gas absorption for ammonia recovery after the Haber-Bosch process, Chem. Eng. J. 421 (2021) 127726.

[13] L. He, Y. Wang, T. Zhou, Y. Zhao, Enhanced ammonia resource recovery from wastewater using a novel flat sheet gas-permeable membrane, Chem. Eng. J. 400 (2020) 125338.

[14] M. Asghari, M. Afsari, Effect of Ethylene Oxide Functional Groups in PEBA-CNT Membranes on CO2/CH4 Mixed Gas Separation, Journal of Membrane Science and Research 4 (2018) 34-40.

[15] M. Afsari, H.K. Shon, L.D. Tijing, Janus membranes for membrane distillation: Recent advances and challenges, Adv. Colloid Interface Sci. (2021) 102362.

[16] M. Toriello, M. Afsari, H.K. Shon, L.D. Tijing, Progress on the Fabrication and Application of Electrospun Nanofiber Composites, Membranes 10 (2020) 204.

[17] M. Yao, Y.C. Woo, L.D. Tijing, J.-S. Choi, H.K. Shon, Effects of volatile organic compounds on water recovery from produced water via vacuum membrane distillation, Desalination 440 (2018) 146-155.

[18] K. Xu, D. Qu, M. Zheng, X. Guo, C. Wang, Water reduction and nutrient reconcentration of hydrolyzed urine via direct-contact membrane distillation: ammonia loss and its control, J. Environ. Eng. 145 (2019) 04018144.

[19] F. Volpin, J. Jiang, I. El Saliby, M. Preire, S. Lim, M.A.H. Johir, J. Cho, D.S. Han, S. Phuntsho, H.K. Shon, Sanitation and dewatering of human urine via membrane bioreactor and membrane distillation and its reuse for fertigation, Journal of Cleaner Production 270 (2020) 122390.

[20] H. Yao, Z. Hu, W. Qing, S. Sun, W. Zhang, Water and nutrients recovery from synthetic source-separated human urine using AGMD, Journal of Environmental Chemical Engineering 10 (2022) 107176.

[21] S. Kartohardjono, G.M. Damaiati, C.T. Rama, Effects of absorbents on ammonia removal from wastewater through hollow fiber membrane contactor, Journal of Environmental Science and Technology 8 (2015) 225.

[22] E.E. Licon Bernal, A. Alcaraz, S. Casas, C. Valderrama, J.L. Cortina, Trace ammonium removal by liquid–liquid membrane contactors as water polishing step of water electrolysis for hydrogen production from a wastewater treatment plant effluent, J. Chem. Technol. Biotechnol. 91 (2016) 2983-2993.

[23] C.-L. Lai, S.-h. Chen, R.-M. Liou, Removing aqueous ammonia by membrane contactor process, Desalination and Water Treatment 51 (2013) 5307-5310.

[24] J. Zhang, M. Xie, X. Tong, S. Liu, D. Qu, S. Xiao, Recovery of ammonium nitrogen from human urine by an open-loop hollow fiber membrane contactor, Sep. Purif. Technol. 239 (2020) 116579.

[25] M.M. Damtie, F. Volpin, M. Yao, L.D. Tijing, R.H. Hailemariam, T. Bao, K.-D. Park, H.K. Shon, J.-S. Choi, Ammonia recovery from human urine as liquid fertilizers in hollow fiber membrane contactor: Effects of permeate chemistry, Environmental Engineering Research (2020).

[26] B.-B. Lind, Z. Ban, S. Bydén, Volume reduction and concentration of nutrients in human urine, Ecol. Eng. 16 (2001) 561-566.

[27] M. Fillingham, A.C. VanderZaag, J. Singh, S. Burtt, A. Crolla, C. Kinsley, J.D. MacDonald, Characterizing the performance of gas-permeable membranes as an ammonia recovery strategy from anaerobically digested dairy manure, Membranes 7 (2017) 59.

[28] M. Xiao, Y. Shang, L. Ji, M. Yan, F. Chen, Q. He, S. Yan, Enhancing the Ammonia Selectivity by Using Nanofiber PVDF Composite Membranes Fabricated with Functionalized Carbon Nanotubes, Membranes 12 (2022) 1164.

[29] M. Shi, M. Xiao, L. Feng, T. Tu, Q. He, S. Yan, Water and green ammonia recovery from anaerobic digestion effluent by two-stage membrane distillation, Journal of Water Process Engineering 49 (2022) 102949.

[30] D. Qu, D. Sun, H. Wang, Y. Yun, Experimental study of ammonia removal from water by modified direct contact membrane distillation, Desalination 326 (2013) 135-140.

[31] N. Coote, B. Kirsop, Factors responsible for the decrease in pH during beer fermentations, Journal of the Institute of Brewing 82 (1976) 149-153.

[32] Z. Ding, L. Liu, Z. Li, R. Ma, Z. Yang, Experimental study of ammonia removal from water by membrane distillation (MD): The comparison of three configurations, J. Membr. Sci. 286 (2006) 93-103.

[33] M. Fillingham, A. VanderZaag, J. Singh, S. Burtt, A. Crolla, C. Kinsley, J.D. MacDonald, Characterizing the performance of gas-permeable membranes as an ammonia recovery strategy from anaerobically digested dairy manure, Membranes 7 (2017) 59.

[34] S.N. McCartney, N.A. Williams, C. Boo, X. Chen, N.Y. Yip, Novel isothermal membrane distillation with acidic collector for selective and energy-efficient recovery of ammonia from urine, ACS Sustainable Chemistry & Engineering 8 (2020) 7324-7334.

[35] Z. Xie, T. Duong, M. Hoang, C. Nguyen, B. Bolto, Ammonia removal by sweep gas membrane distillation, Water Res 43 (2009) 1693-1699.

[36] L.L. Tun, D. Jeong, S. Jeong, K. Cho, S. Lee, H. Bae, Dewatering of source-separated human urine for nitrogen recovery by membrane distillation, J. Membr. Sci. 512 (2016) 13-20.

[37] C. Yu, W. Yin, Z. Yu, J. Chen, R. Huang, X. Zhou, Membrane technologies in toilet urine treatment for toilet urine resource utilization: a review, RSC advances 11 (2021) 35525-35535.

[38] A. Almuntashiri, A. Hosseinzadeh, U. Badeti, H. Shon, S. Freguia, U. Dorji, S. Phuntsho, Removal of pharmaceutical compounds from synthetic hydrolysed urine using granular activated carbon: Column study and predictive modelling, Journal of Water Process Engineering 45 (2022) 102480.

[39] A. Hasanoğlu, J. Romero, B. Pérez, A. Plaza, Ammonia removal from wastewater streams through membrane contactors: Experimental and theoretical analysis of operation parameters and configuration, Chem. Eng. J. 160 (2010) 530-537.

[40] M.J. Semmens, D. Foster, E. Cussler, Ammonia removal from water using microporous hollow fibers, J. Membr. Sci. 51 (1990) 127-140.

[41] P. Jacob, P. Phungsai, K. Fukushi, C. Visvanathan, Direct contact membrane distillation for anaerobic effluent treatment, J. Membr. Sci. 475 (2015) 330-339.

[42] Y. Ahn, Y.-H. Hwang, H.-S. Shin, Application of PTFE membrane for ammonia removal in a membrane contactor, Water Sci Technol 63 (2011) 2944-2948.

[43] G. Simoni, B.S. Kirkebæk, C.A. Quist-Jensen, M.L. Christensen, A. Ali, A comparison of vacuum and direct contact membrane distillation for phosphorus and ammonia recovery from wastewater, Journal of Water Process Engineering 44 (2021) 102350.

[44] K.M. Udert, M. Wächter, Complete nutrient recovery from source-separated urine by nitrification and distillation, Water Res 46 (2012) 453-464.