# FULL NITROGEN RECOVERY AND POTABLE WATER PRODUCTION FROM HUMAN URINE BY MEMBRANE DISTILLATION

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## Abstract

Human urine offers some interesting possibilities for ammonia and potable water recovery. Membrane distillation holds possible advantages over existing urine treatment technologies, specifically regarding ammonia recovery. It was shown that up to 95 m% of all ammonia present in hydrolyzed urine could be recovered by increasing the urine pH to 10.5 or higher within a period of 2 hours, with a maximal separation factor of up to 16. The possibility of potable water production was investigated in human urine by assessing the permeate water quality, maximum recovery and mid-term process stability. It was shown that at least 75% of the available water could be recovered from non-hydrolyzed human urine without process failure. As such, membrane distillation is a viable alternative for existing urine treatment.

## Introduction

The invention of the Haber-Bosch process in the middle of the 20$^{th}$ century has had an enormous impact on human society, ranging from increased yields in agriculture and population growth to eutrophication and increased $CO_2$ emissions. In 2008, nitrogen fixation through the Haber-Bosch process was responsible for up to 1-2% of the worldwide energy consumption while producing up to 130 million tonnes of ammonia fertilizer (Canfield et al., 2010). Even though the environmental issues and disadvantages of investing enormous amounts of energy in nitrogen fixation are hard to ignore, the incentive for nitrogen recycling is not driven by depletion, as nitrogen gas is naturally abundant in the air. In fact, the combination of Haber-Bosch and 'recycling' nitrogen by oxidizing it in wastewater treatment plants (WWTP) brings the total energy tag up to 90 MJ $kg^{-1}_N$, as such that nitrogen recovery through e.g. struvite (102 MJ $kg^{-1}_N$) or stripping (90 MJ $kg^{-1}_N$) cannot compete (Maurer et al., 2003).

The road to increasing energy efficiency for the anthropogenic part of the nitrogen cycle therefore leads to a challenge: either the activation energy for the Haber-Bosch process is decreased (Kitani et al., 2012), or either the road leads to innovative nitrogen recycling treatment, using less resources, less exergy and less treatment steps.

Human urine is the major source of nitrogen in domestic wastewater. On its own, it adds more than 80% of total nitrogen, 50 % of total phosphorus and 70% of total potassium in 1% of the total volume. Diluting this urine (step 1) and treating it in WWTP's (step 2) add complexity to efficient nutrient recovery. The future of human nutrient recovery is source separation, as Larsen and Gujer already stipulated in 1996. However, source-separated human urine cannot be used directly as a fertilizer, due to the likely presence of pathogens (Heinonen-Tanski and van Wijk-Sijbesma, 2005) and/or pharmaceuticals (Winker et al., 2008a, Winker et al., 2008b).

A thorough review of the treatment processes for source-separated human urine by Maurer et al. in 2006 compared different techniques towards various criteria (hygienization, volume reduction, stabilization, P-recovery, N-recovery, MP elimination, nutrient-MP-elimination, nutrient elimination, solidification and need for pre- or post-treatment). To the best of our knowledge, no single-step treatment is able to satisfy all these criteria. Many technologies focus on nutrient recovery through struvite precipitation (Ronteltap et al., 2010, Antonini et al., 2011, Ganrot et al., 2007, Etter et al., 2011), ion exchange (O'Neal and Boyer, 2013), adsorption (Lind et al., 2000), distillation and nitrification (Udert and Wächter, 2012) and ammonia stripping (Antonini et al., 2011, Başakçılardan-Kabakcı et al., 2007). Although they may offer interesting perspectives in developed countries, they also require electricity and/or expensive equipment, preventing their use in cut-off rural communities of developing countries, which rely heavily on agriculture (Bilsborrow, 1987) and are faced with increasing nutrient mining due to population growth (Henao and Baanante, 2006).

Membrane distillation is one of the promising techniques to produce potable water from impaired water sources in developing countries, as it only requires low-grade heat (e.g. solar energy) to transfer volatile substances through a hydrophobic membrane by establishing a vapour pressure gradient. As such, theoretically speaking, 100 % rejection of non-volatile substances can be achieved. Specifically for urine, recovery of ammonia and water was already achieved through membrane distillation, albeit at intermediate recovery – 40.6-75.1% for ammonia and 31.9-48.6% for water correspondingly (Zhao et al., 2013). Another study by El-Bourawi et al. (2007) however showed that ammonia removal efficiencies of over 90% from ammonia-water solutions are possible under specific operational conditions.

In this paper, we further investigate the options that membrane distillation and membrane stripping have towards water and ammonia recovery, by varying major operational conditions and testing membranes with various characteristics.

## Background

The vapour pressure gradient across the membrane surface required for mass transfer in membrane distillation can be established in various ways: direct membrane distillation (DCMD), by separating a feed and permeate liquid stream with a membrane, air-gap membrane distillation (AGMD), where the transferred vapour condenses in a separate chamber filled with air, separated from the permeate liquid by a condensing wall, sweeping-gas membrane distillation

(SGMD), where the transferred vapour is transported to a condenser by a carrier gas, and vacuum membrane distillation, where a vacuum pump provides the vapour pressure gradient (Alkhudhiri et al., 2012). This study was conducted using direct-contact membrane distillation, which is the simplest and most researched configuration, used for desalination processes (Hsu et al., 2002), concentration of aqueous solutions in food industries (Calabro et al., 1994) and acid manufacturing (Tomaszewska et al., 1995).

In direct-contact membrane distillation, the bulk vapour pressure gradient of compound i between the feed and permeate is defined as:

$$\Delta p_i = p_{i,f} - p_{i,p}$$

Where $p_{i,f}$ and $p_{i,p}$ are the feed and permeate vapour pressure of compound i respectively. The vapour pressure of volatile compound i within a mixed solution can be calculated using:

$$p_i = p_i^0 \, x_i$$

In which $p_i^0$ is the vapour pressure above a pure solution of compound i at the temperature of interest. This vapour pressure can be calculated by using the Antoine equation:

$$p_i^0 = \exp\left(A - \frac{B}{T + C}\right)$$

In which T is the absolute temperature in K. For water, at temperatures between 274.15 and 373.15 K, the values for A, B and C are 23.1964, 3816.44 and −46.13, respectively.

For ammonia, the calculation is slightly more complex, as it is affected both by the ammonium-ammonia equilibrium as well as the Henry's law coefficient. The pKa of ammonium is 9.24 at standard conditions, meaning that ammonia is only quantitatively present as a dissolved gas above a pH of 7. Additionally, for ammonia to 'strip' from a watery solution, it has to overcome very strong hydrogen bonds. The Henry coefficient of ammonia gas is 60 M/bar at standard conditions. Vapour pressure of ammonia is therefore strongly correlated to solution temperature and pH.

The flux of mass through the membrane is defined as:

$$J_i = \frac{m_{p,i} - m_{f,i}}{A \, t}$$

Where $m_{p,i}$ and $m_{f,i}$ are the mass of substance i in the permeate and feed water specifically, A is the membrane surface in the module and t is the time in h. To compare membrane performance throughout an experiment with variable vapour pressures, membrane permeability (specific per compound) is used:

$$A_{m,i} = \frac{J_i}{\Delta p_i}$$

As various volatile substances may transport through the membrane, and selectivity towards a specific substance is often desirable. To this goal, the separation factor is often introduced:

$$\alpha = \left(1 - \frac{C_p}{C_f}\right) \times 100$$

## Materials and methods

***Experiments***

Urine was collected from healthy female and male candidates and mixed in a 20L disinfected vessel. These vessels were either kept refrigerated at 4°C until experiments were started (non-hydrolyzed urine) or were inoculated with stale urine from previous experiments and left at room temperature to hydrolyze until the pH reached 9. The composition of the three batches used in this study are shown in Table 1.

**Table 1:** Composition of fresh and hydrolyzed human urine batches.

| Batch code | **HYDRO-01** | **HYDRO-02** | **FRESH-01** |
|---|---|---|---|
| Volume (L) | 5 | 12.2 | 4.2 |
| pH (4 °C) | 9.5 | 9.34 | 6.97 |
| TOC (mg/L) | 4660 | 4648 | 34346 |
| IC (mg/L) | 1680 | 1663 | 1538 |
| Na (mg/L) | 1558 | 1752 | 1802 |
| K (mg/L) | 1486 | 1638 | 1363 |
| Ca (mg/L) | 16 | 67 | 65 |
| Mg (mg/L) | 1 | 6 | 54 |
| PO4-P (mg/L) | 381 | 452 | 508 |
| NH4-N (mg/L) | 1335 | 1216 | 296 |

The experiments were conducted in a membrane distillation set-up with an active membrane surface of 0.0056 m² (L: 0.25m, W: 0.05m, D: 0.005m). The initial pH of feed (urine) and permeate (demineralized water) solutions was adjusted using 12 M NaOH or 96% $H_2SO_4$ to minimize dilution. All reagents were analytical grade. The temperature was controlled within ±1°C using Pt100-electrodes. pH of the feed urine and conductivity and total mass of the permeate were logged on a personal computer. Experiments were run for a period of 6 hours (membrane stripping) or until temperature control due to volume reduction became erratic.

The membranes used in this study were flat-sheet PTFE membranes, of which average pore size, thickness, water contact angle and flux are shown in Table 2.

**Table 2:** Characteristics of the membranes used in this study.

| Membrane type | S02 | NS01 | NS02 |
|---|---|---|---|
| Average pore size (µm) | 0.2 | 0.1 | 0.2 |
| Membrane thickness (µm) | 127[1] | 66 | 66 |
| Contact angle (water) (°) | 130 | 134 | 142 |
| Flux (L/m².h)[2] | 13.85 | 16.46 | 38.70 |

5 mL samples of feed urine and permeate were taken at regular intervals and were kept frozen at -18°C until preparation for analysis. Sodium, potassium, calcium and magnesium analysis was performed on a tabletop Vista MPX ICP-OES (Agilent Technologies, USA). Ammonium concentrations were determined using a continuous flow AA3-AutoAnalyzer (BranLuebbe, Germany). Phosphorus was determined spectrophotometrically using the Scheel method. TC and IC concentrations were analysed on a Shimadzu TOC-5000 analyzer.

***Results***

Ammonia recovery was calculated as:

$$Rec_{NH_3}(\%) = \frac{m_{NH3,perm,eq.}}{m_{NH3,feed,0}} \times 100$$

In which $m_{NH3,perm,eq.}$ and $m_{NH3,feed,0}$ are the total masses of ammonia at equilibrium in the permeate, and initially in the feed. Losses are not accounted for, except when comparing to the ammonia removal, which is defined as:

$$Rem_{NH_3}(\%) = \frac{m_{NH3,feed,eq.}}{m_{NH3,feed,0}} \times 100$$

[1] Membrane thickness includes the porous support layer.

[2] Clean water flux was determined at a bulk vapour pressure difference of 9.8 kPa ($T_{feed}$=40°C, $T_{permeate}$=20°C) using demineralized water. Due to temperature polarization effects, membrane permeability is not constant.

## Results and discussion

### *Ammonia recovery*

*Effect of urine hydrolysis on ammonia recovery*

From the urine analysis above, it can be hypothesized that ammonia recovery from human urine should take place after full hydrolysis has taken place. Indeed, even when ammonia volatilization to the surroundings occurs, ammonia levels are still up to 4 times higher in hydrolyzed urine. However, membrane processes are sensitive to (bio)fouling, and as hydrolysis is performed by ureolytic bacteria (Mobley and Hausinger, 1989) their presence may impact membrane performance.

The data in Table 3 shows the average ammonia recovery from non-hydrolyzed, hydrolyzed and hydrolyzed urine with base added, under varying feed temperatures. All experiments were stopped at a concentration factor of 3, at which point permeate samples were taken. Even though we will investigate the effect of operational parameters such as feed pH and feed and permeate temperature on the membrane distillation process further, it is worth mentioning that ammonia recovery is severely impacted by the degree at which ammonia is freely available in urine. Not only is TAN liberated from urea during hydrolysis, but the subsequent pH rise shifts the ammonia/ammonium equilibrium towards ammonia. Membrane distillation as ammonia recovery treatment should therefore focus on hydrolyzed urine. Interesting to note is that ammonia recovery is also severely impacted by the ability of the permeate stream to absorb ammonia: when using demineralized water as a permeate stream, the permeate pH increases severely and stabilizes at 10, at which point no net ammonia transfer is occurring from feed to permeate.

**Table 3:** The effect of hydrolysis and pH adjustments in feed and permeate streams on ammonia recovery.

| Hydrolysis? | Feed pH | Permeate | $T_{perm}$ (°C) | $T_{feed}$ (°C) | Average $Rec_{NH3}$ (%) |
|---|---|---|---|---|---|
| No | unadjusted (pH 7) | DI water | 20 | 45; 50 | 26.7±10.3 |
| No | unadjusted (pH 7) | 0.2M $H_2SO_4$ | 20 | 40;45;50;55 | 68.4±8.6 |
| Yes | unadjusted (pH 8.5) | 0.2M $H_2SO_4$ | 20 | 40;45;50 | 59.3±3.6 |
| Yes | 12 | 0.2M $H_2SO_4$ | 20 | 45;50;55 | 95.6±1.05 |

*Effect of urine pH*

When increasing the hydrolyzed feed urine pH, the ammonia/ammonium equilibrium is shifted towards (volatile) ammonia, effectively increasing the driving force for ammonia flux through the membrane. As can be seen in Figure 1, increasing the feed urine pH from 9 to 10.5 allows almost complete recovery (95%) of total ammonia within 2 hours. Further increasing the pH has little effect on ammonia recovery.

Even though the initial bulk ammonia driving force in this experiment is only in the order of magnitude of 0.5 kPa to 1.5 kPa (calculated based on TAN concentration), the maximal ammonia mass flux is approximately in the order of magnitude of 150-200 $g_{NH3}/m^2.h$ (based on the results in Figure 2), with a maximal separation factor of up to 16. Surprisingly, little difference in the ammonia flux is to be noted in the initial phase of all three experiments, which leads to the conclusion that ammonia flux is largely independent of initial ammonia concentration, corroborating the findings of El-Bourawi et al. in 2007, who investigated nitrogen recovery from aqueous ammonia solutions through membrane distillation.

Even though maximal ammonia fluxes are high, they drop off to almost zero as soon as the ammonia concentration in urine is decreased to 100 mg $NH_3$/L (Figure 3). The driving force has been decreased by at least a ten-fold by the combined effects of lower concentration and pH decrease, explaining the severe decrease in ammonia flux.

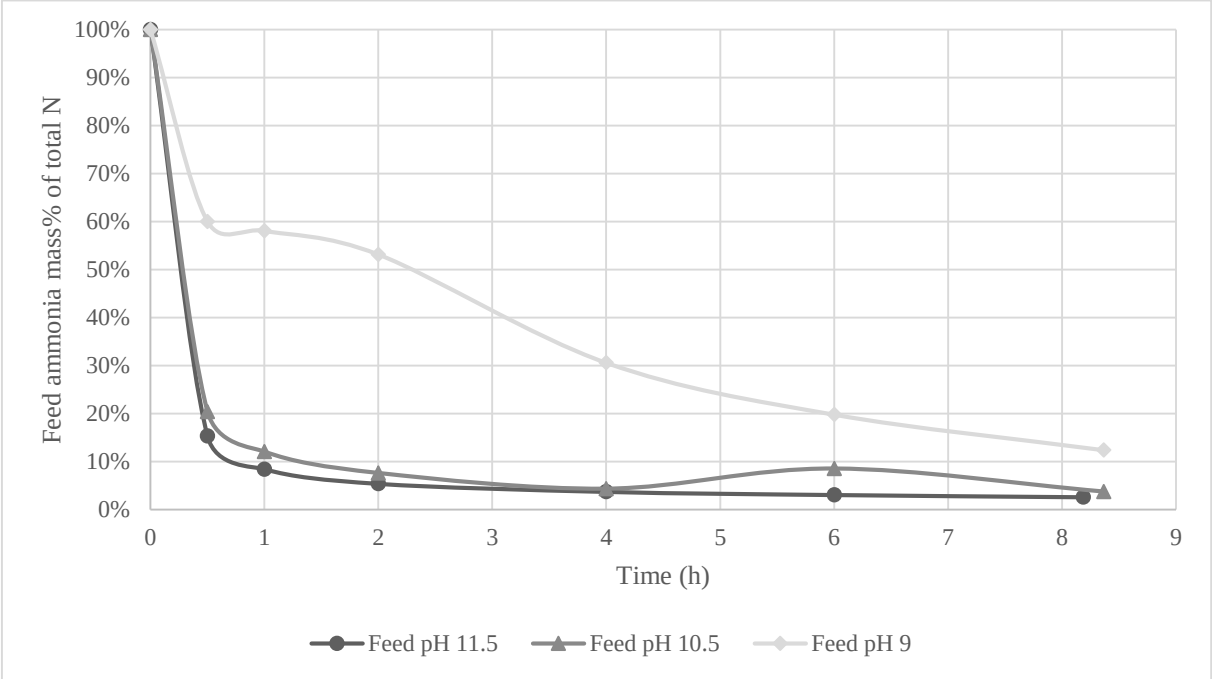


**Figure 1:** Feed mass percentage of total ammonia versus total time of the membrane distillation experiment. Urine of batch Hydro-02 was used, with respective feed and permeate temperatures of 50 and 20°C. The membrane used in this series of experiments came from a single sheet of NS02 membrane.

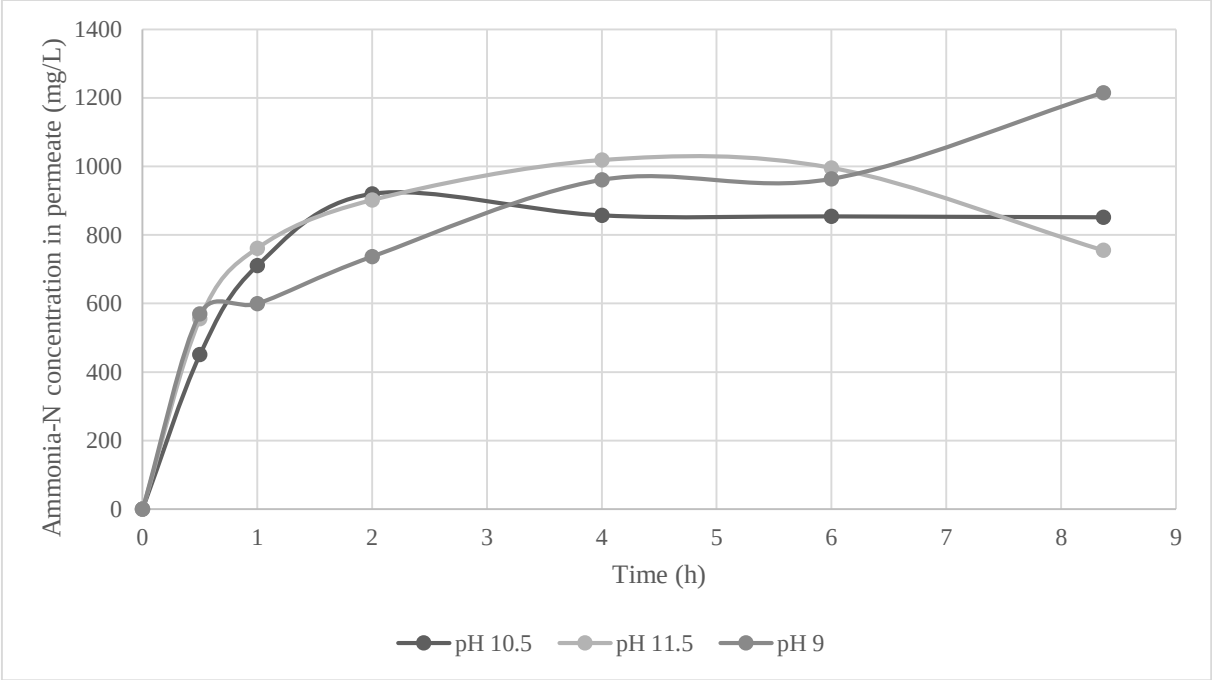


**Figure 2:** Concentration of ammonia in the acidified permeate. Urine of batch Hydro-02 was used, with respective feed and permeate temperatures of 50 and 20°C. The membrane used in this series of experiments came from a single sheet of NS02 membrane.

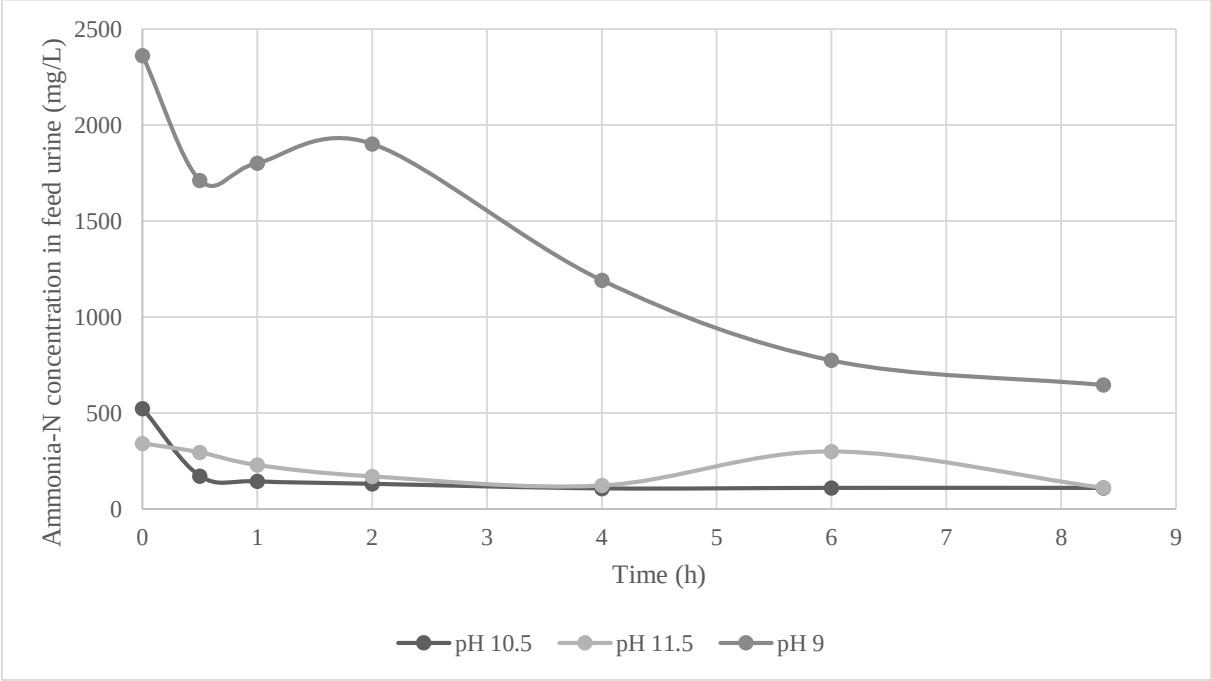


**Figure 3:** Concentration of ammonia in the feed urine. Urine of batch Hydro-02 was used, with respective feed and permeate temperatures of 50 and 20°C. The membrane used in this series of experiments came from a single sheet of NS02 membrane.

*Effect of feed temperature*

In a next set of experiments, the influence of feed temperature was investigated. As membrane distillation is often operated below 60°C, we investigated a temperature range between 40 and 60 °C (Figure 4). Even though within this range the equilibrium driving force of ammonia doubles, the effect on total ammonia recovery is rather small. Especially when the feed temperature is increased from 50 to 60 °C, the gains are marginal.

However, when looking at Figure 5, the equilibrium ammonia concentration in the permeate of the experiment run at a feed temperature of 40°C is higher. Even though efforts were made to prevent ammonia losses, it seems that a higher vapour pressure and driving force also increases the losses encountered. The prevention of losses through gas-tightening should be taken into account when designing larger scale membrane distillation set-ups.

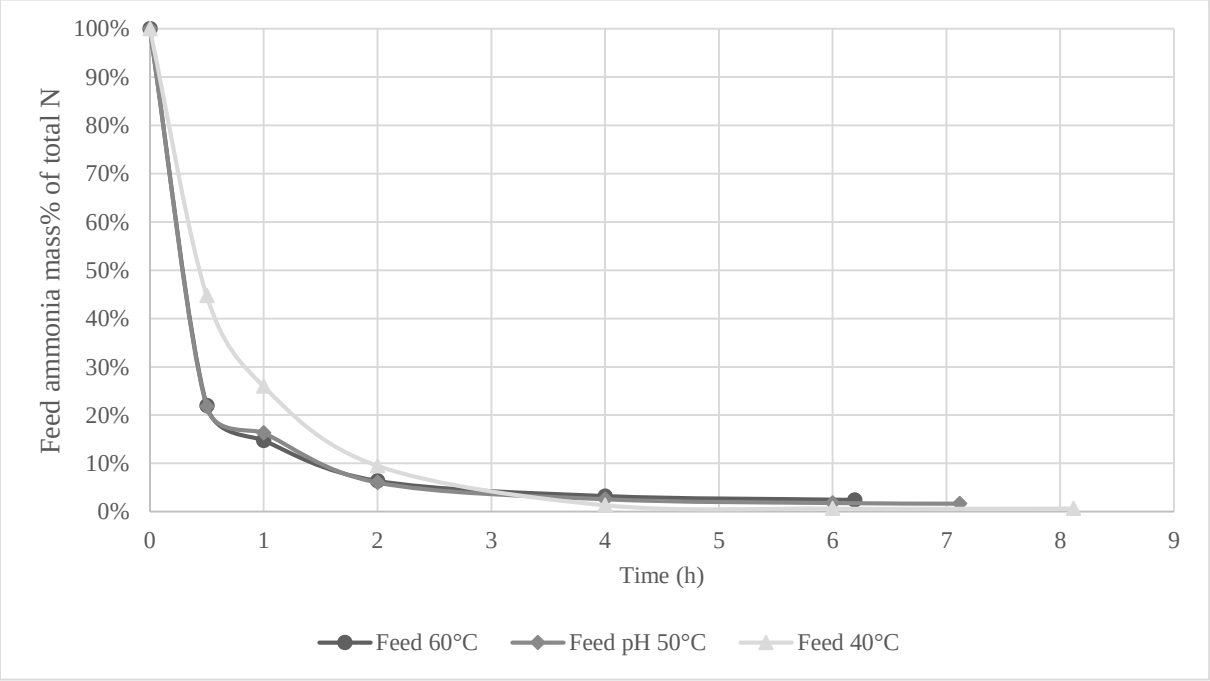


**Figure 4:** Feed mass percentage of total ammonia versus total time of the membrane distillation experiment. Urine of batch Hydro-01 was used, with a feed pH of 10.5. The membrane used in this series of experiments came from a single sheet of S02 membrane.

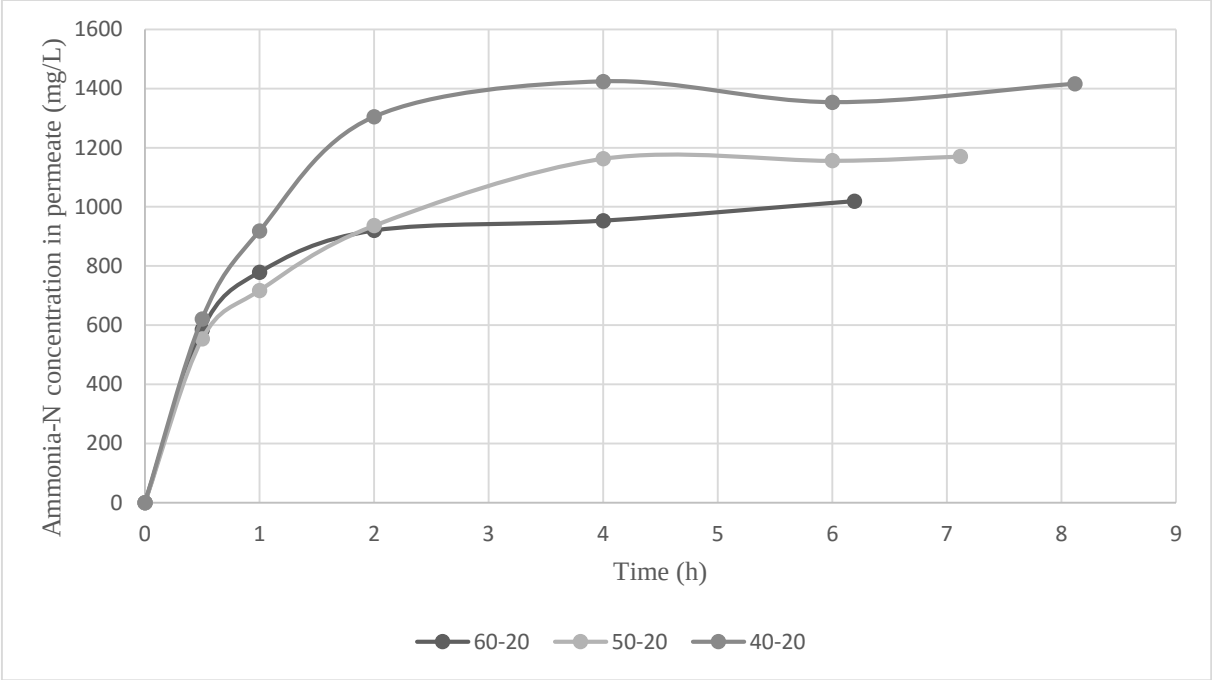


**Figure 5:** Concentration of ammonia in the acidified permeate. Urine of batch Hydro-01 was used, with a feed pH of 10.5. The membrane used in this series of experiments came from a single sheet of S02 membrane.

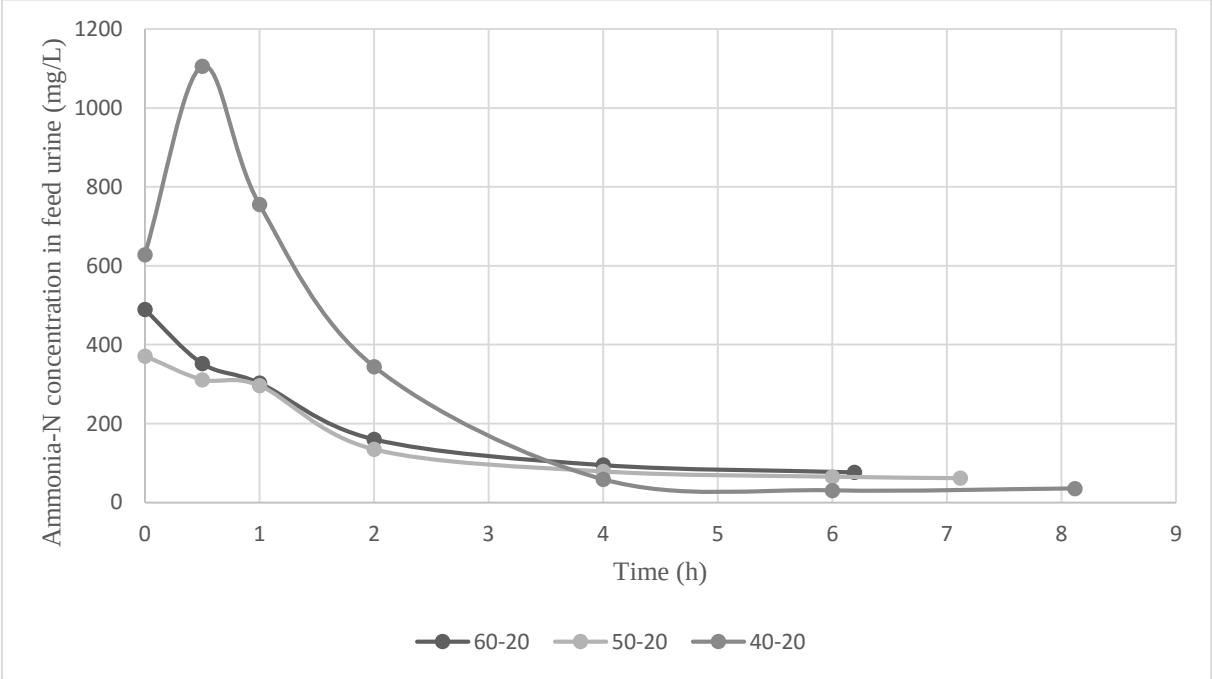


**Figure 6:** Concentration of ammonia in the feed urine. Urine of batch Hydro-01 was used, with a feed pH of 10.5. The membrane used in this series of experiments came from a single sheet of S02 membrane.

***Potable water recovery***

Potable water recovery from urine is a much debated option. Whether undesirable from a purely psychological or ideological point of view, or due to technical aspects, currently (vacuum) distillation is one of the only single-step technologies that meets health and safety guidelines. Membrane distillation, however in many aspects similar to distillation, could provide some interesting advantages over regular distillation. However, safety and water quality standards should be met, and the membrane distillation process should be robust against process failure such as fouling or wetting. Here, we investigate the process stability and fouling resistance, the maximum water recovery and presence of unwanted compounds in permeate water.

*Effect of hydrolysis on membrane fouling*

Due to the large difference in (biological) composition between non-hydrolyzed and hydrolyzed urine, water flux through the membrane may vary heavily when urine of a different hydrolysis degree is used. When comparing Figure 7 and 8, it is clear that water flux is not too heavily impacted by fouling, wetting or scaling in middle term experiments. Indeed, average fluxes of 5.45 and 4.7 L/m².h, respectively are attainable in middle term experiments. However, when looking at membrane fouling (Figure 9), it is clear that some membrane fouling is developing, especially within the zones of low turbulence. Non-hydrolyzed urine that can be kept stable by e.g. acidification is preferable for long-term water production to keep the membrane process stable.

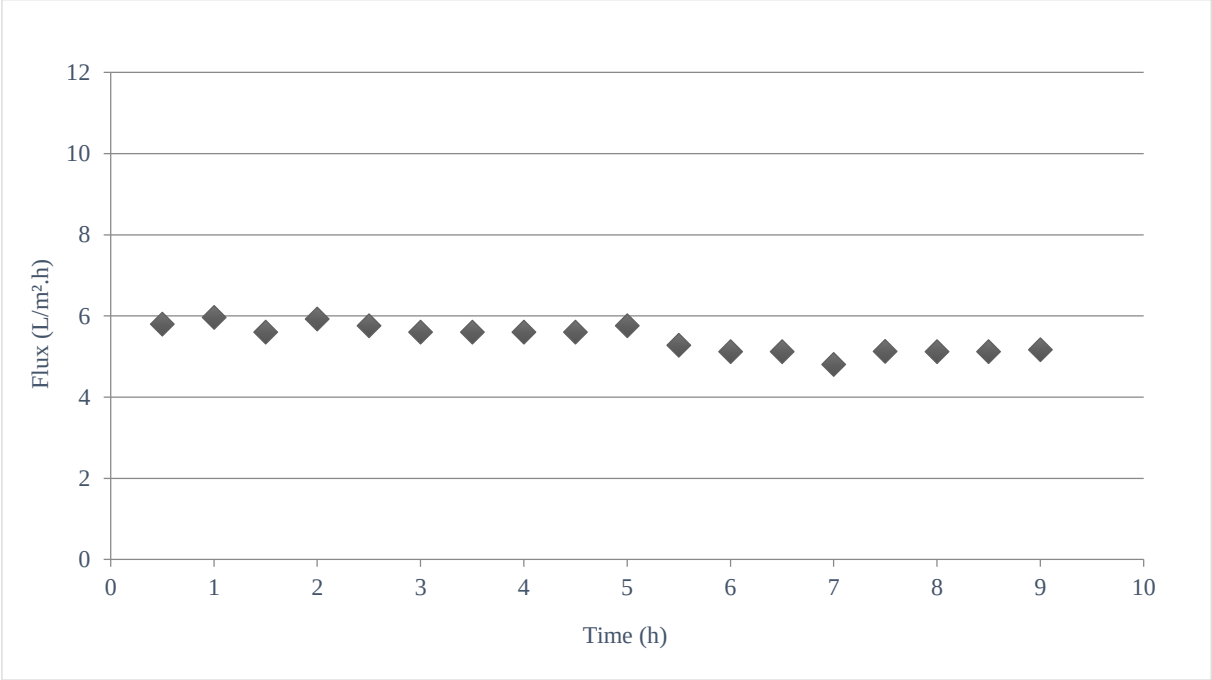


**Figure 7:** Water flux in a membrane distillation experiment using batch FRESH-01 urine. $T_{feed}$=50°C, $T_{permeate}$=20°C, pH unadjusted (pH 7). Experiments were run to a concentration factor of 3, the membrane used is a NS01 membrane.

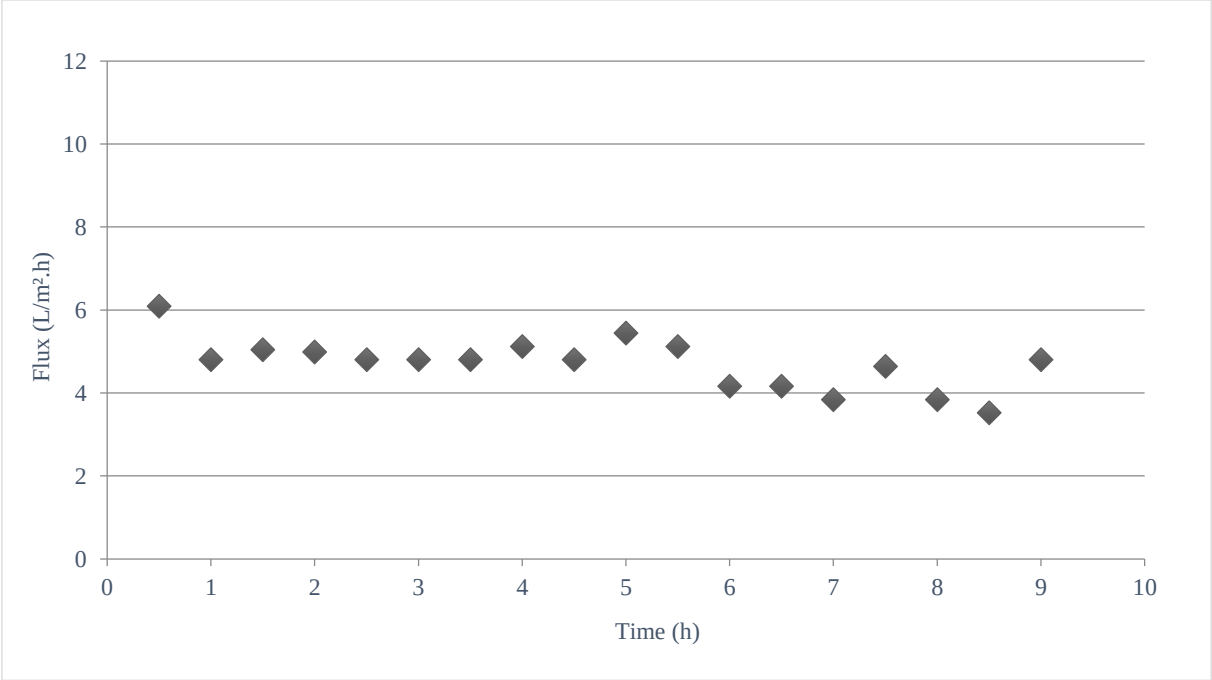


**Figure 8:** Water flux in a membrane distillation experiment using batch HYDRO-02 urine. Tfeed=50°C, Tpermeate=20°C, pH adjusted to 12). Experiments were run to a concentration factor of 3, the membrane used is a NS01 membrane.

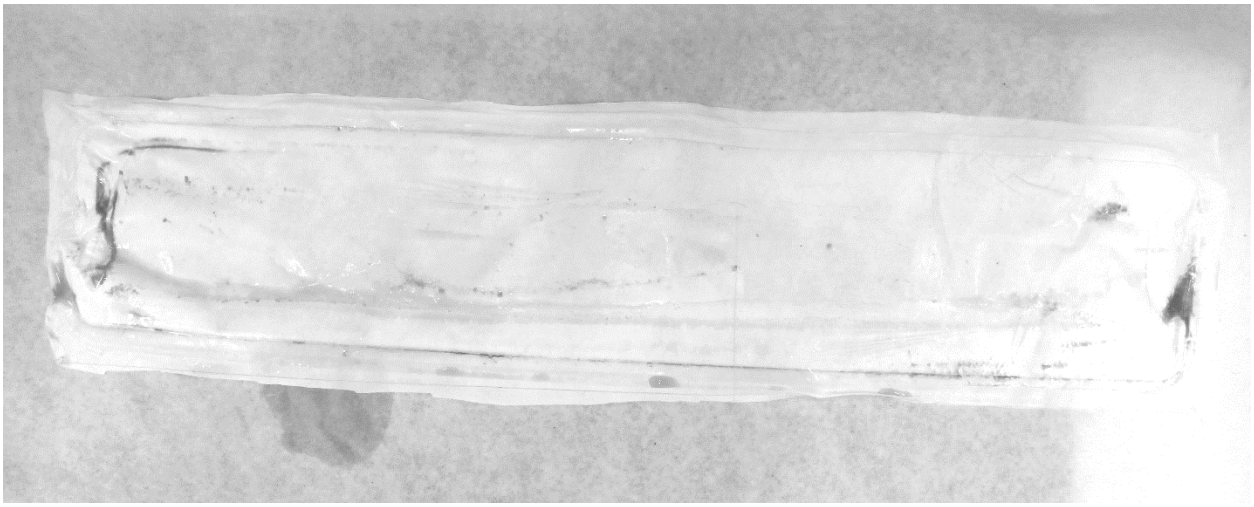

**Figure 9:** Membrane fouling of the membrane used in experiment of Figure 10.

*Maximum water recovery*

As membrane distillation is not too severely impacted by fouling or scaling, high water recoveries should be attainable. However, at a certain point the feed concentration and viscosity will start to increase, reducing the vapour pressure and potentially foul and scale the membrane. In the experiments above, recovery of up to 65% was achievable without significant flux loss. In Figure 12 an experiment was run for 25h, effectively recovering up to 75% of the water in urine, at which point temperature control was becoming erratic.

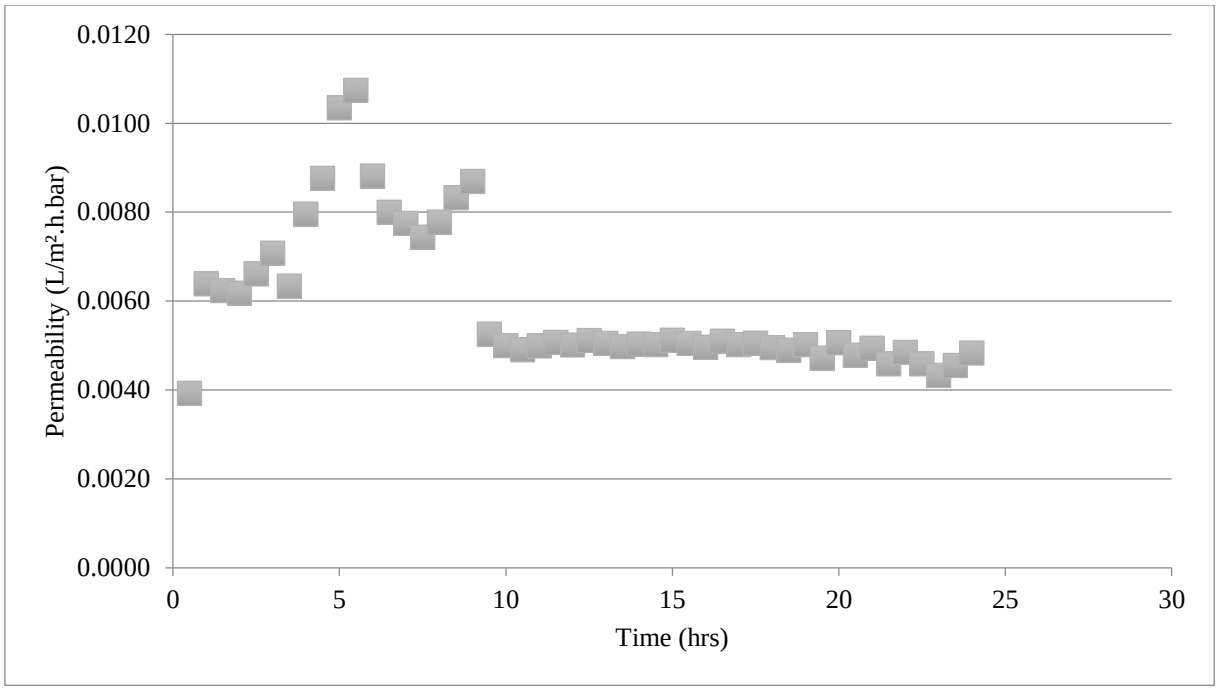


**Figure 10:** Long-term membrane distillation experiment, using 2 L of batch Fresh-01 urine. $T_{feed}$=50°C, $T_{permeate}$=20°C. The pH was unadjusted at 7. Membrane used: S02.

## Conclusions

In this paper, we aimed to investigate the possibilities of ammonia and potable water recovery from human urine through membrane distillation. Membrane distillation could offer some advantages regarding speed and efficiency over existing urine treatment technologies, specifically regarding ammonia recovery. It was shown that up to 95 m% of all ammonia present in hydrolyzed urine could be recovered by increasing the urine pH to 10.5 or higher within a period of 2 hours. As such, membrane distillation offers a very valuable alternative to other ammonia recovery technologies, such as stripping. However, membrane distillation still requires addition of caustics and heat for a full, quick and reliable ammonia recovery from urine, reducing its applicability as a possible nitrogen fertilizer production technique for developing countries.

The possibility of potable water production was investigated in human urine by assessing the permeate water quality, maximum recovery and mid-term process stability. It was shown that at least 75% of the available water could be recovered from non-hydrolyzed human urine without process failure.

Membrane distillation can therefore be classified as a very interesting treatment step for human urine treatment, allowing very high recovery of ammonia and water. Further research should focus on further improving the selectivity towards either water and/or ammonia and investigate long-term flux behaviour of membrane distillation membranes.

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