Sanitation and dewatering of human urine via membrane bioreactor and membrane distillation and its reuse for fertigation

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List of Abbreviations

MBR	Membrane bioreactor
HRT	Hydraulic retention time
MD	Membrane distillation
DCMD	Direct contact membrane distillation
DOC	Dissolved organic carbon
UF	Ultrafiltration
TMP	Transmembrane pressure
MLSS	Mixed liquor suspended solids
LC-OCD	liquid chromatography coupled with organic carbon

Abstract

Source separation and recovery of human urine have often been proposed as an effective way to achieve a more sustainable waste-to-resource cycle. Its high density of available macronutrients (N-P-K) in urine makes it an ideal raw material for the production of fertiliser. However, to improve the safety and public acceptance of urine-based fertilisers, odour and pathogens must be removed. In this work, low-temperature DCMD was investigated a mean to produce a non-odorous high-concentration liquid fertiliser. The effectiveness of urine-fertiliser in hydroponically growing leafy vegetables was benchmarked with a commercial solution. Also, prior to the DCMD, urine was biologically oxidised through an MBR which removed over 95% of the DOC and converted almost 50% of the NH_3 into NO_3^-. The results showed that, despite the high salinity and high LMW organics in human urine, MD was still able to achieve a final product with TDS concentration up to 280 $g.L^{-1}$. A sharp flux decline was measured after 80% water recovery, but alkaline cleaning effectively removed the thick fouling layer and fully recovered the initial flux. When used to grow lettuce and Pak Choi hydroponically, the produced urine fertiliser achieved promising performances as the biomass from the aerial part of the plants was often similar to the one obtained with commercial fertilisers. Overall, this article investigates the whole urine-to-biomass cycle, from collection to treatment to plant growth tests.

Keywords: Urine nitrification, nutrient recovery, membrane bioreactor, membrane distillation, urine fertiliser, resource recovery.

1 Introduction

Most of the excess nutrients that human ingest are excreted through urine, which makes it a suitable raw material for fertiliser production. As such, urine to fertiliser conversion is becoming increasingly investigated as a means to reduce wastes, and reliance on finite mineral fertilisers. In this context, a growing number of agricultural trials were performed, showing that crops' yields significantly increased when fertilised with urine, and they were often similar in quantity to the one obtained with synthetic fertilisers (Bonvin et al., 2015; McHunu et al., 2018; Pandorf et al., 2019; Viskari et al., 2018). The use of urine as fertiliser source has also shown to be an effective way to achieve both biomass production as well as wastewater phytoremediation (Kamyab et al., 2017; Rezania et al., 2016; Yang et al., 2015).

Besides, an upstream urine separation is also expected to benefit the downstream sewage treatment processes. As urine contributes up to 80% of the NH_3-N in wastewater, its separation would reduce the aeration energy required for biological oxidation of ammonia to form nitrate thereby resulting in smaller, less energy-intensive and more effective wastewater treatment plant (Jacquin et al., 2018; Larsen et al., 2009; Larsen et al., 2016; Maurer et al., 2003; Udert and Wächter, 2012). Additionally, urine separation could help in reducing the pharmaceutical compounds coming with the sewage before and after its treatment and discharge (Lienert et al., 2007; Pronk et al., 2006).

Given that untreated urine is generally a poorly accepted fertiliser solution, the best chance to enhance its fertiliser value is to produce safe, effective and odourless fertilisers product (Mkhize et al., 2017; Okem et al., 2013; Udert and Wächter, 2012). As such, several signs of progress were made by the scientific community in developing and optimising a wide range of technologies to produce safe fertilisers from urine (Larsen et al., 2013; Maurer et al., 2006; Randall and Naidoo, 2018). Still, one major bottleneck for producing commercial fertilisers from urine is its low nutrient concentration (i.e., 10-1000 times lower depending on the dilution factor) compared to the commercial liquid fertiliser (Maurer et al., 2006). Therefore, much attention was put on selectively extracting single elements (i.e., N, P and K) from urine, or on recovering all the ions by stabilising and concentrating urine directly. When focusing on the recovery of single ions, adsorption, electrochemical ammonia stripping and struvite precipitation have been the most widely investigated approaches (Etter et al., 2011; Tarpeh et al., 2018; Tarpeh et al., 2017). Even though these approaches were successful in the

recovery of P as struvite or N as $(NH_4)_2SO_4$ or NH_4HCO_3, each process recovers mainly one single ion, neglecting all the others (Jermakka et al., 2018). While this can be useful for some applications, complete urine re-use would mean no discharge of waste by-products, a higher degree of nutrients recovery and above all, environmentally more sustainable.

In this context, (Udert and Wächter, 2012) proposed a combination of biological urine oxidation to reduce organics and pH, followed by thermal distillation to produce a stable, highly concentrated fertiliser solution with only distilled water as a by-product. In the first stage of the process, a sequence batch reactor was used to achieve partial NH_3/NH_4^+ conversion to NO_3^- and removal of malodorous organics. During nitrification, the alkalinity in the urine was also consumed, causing a reduction in pH from about 9.2 (typical pH of hydrolysed urine) to 5.5 - 6.5. The pH of the reactor was maintained by adjusting the HRT as feeding raw urine increased the pH while nitrification reduced it. In the second stage, the stabilised and acidified urine was thermally evaporated to remove water and increase its dissolved solids concentration. (Fumasoli et al., 2016) later investigated the effect of pH set-point in the nitrification performance and biomass selection, pathogens inactivation performance and mechanisms (Bischel et al., 2015) and operational costs of the system. Recently, other groups have looked at the use of an MBR followed by electrodialysis (De Paepe et al., 2018) or by microalgae cultivation (Coppens et al., 2016) to achieve full nutrients recovery. However, only the nitrification – distillation process is currently operated in a semi-large scale, by the company VUNA GmbH, and the urine fertiliser commercialised under the trademark of “Aurin”(Etter, 2019). Aurin production is a clear demonstration of the real potential of urine separation and re-use. Nonetheless, the high energy required to operate such system i.e., 107 ± 31 $Wh.L^{-1}$ for distillation and 35 ± 24 $W{\cdot}g\ N^{-1}$ for nitrification reduces the net revenue from the sale of the fertiliser product, thereby hindering the expansion of such system (Fumasoli et al., 2016).

As up to 82% of the energy demand to run the process is due to the mechanical vapour compression distillation unit, this work aimed at investigating the potential use of a lower temperature thermal process to concentrate the nitrified urine. Direct contact membrane distillation was chosen as an alternative to mechanical vapour compression as it can operate at temperatures of 50-70˚ C, and this thermal requirement could be met by either solar or waste heat integration (Al-Obaidani et al., 2008). However, to match the current system, DCMD should be able to dewater urine to over 250 $mS.cm^{-1}$ of electric conductivity which, in

turn, means DOC concentrations up to 1000 mg.L^{-1}. These conditions imply a high risk of severe organic fouling and inorganic scaling on the porous hydrophobic membrane (Naidu et al., 2017). As such, for the first time, an in-depth investigation of the nature and reversibility of MD fouling, after 20 times concentration of nitrified urine, was conducted. Additionally, the effectiveness of the produced N-P-K fertiliser was tested in growing lettuce and Pak Choi hydroponically. Ultimately, this research linked all the key steps of the urine-to-biomass process, from collection to treatment and finally to plant growth tests.

2 Methodology

A UF - MBR reactor was operated using real, undiluted urine collected from the male's urinals at the University of Technology Sydney (UTS), and its permeate was dewatered using MD. UF-MBR was chosen over the sequence batch reactor to have an improved quality of the stabilised urine as UF can reject bacteria and viruses (that would not be killed in the MD process due to the low operating temperatures). MD permeate flux, maximum urine concentration factor, nutrients rejection rates, membrane fouling and cleaning requirements were all measured in the study.

Finally, the concentrated fertiliser was used to grow lettuce (*Lactuca Sativa L.*) and Pak Choi (*Brassica Chinensis)* in a vertical garden. These plants were selected for their adaptability to hydroponic systems, short life-cycle and extensive data on their cultivation under different conditions and scenarios (Chekli et al., 2017; Yang et al., 2015). The biomass weight produced using urine fertiliser was benchmarked with the biomass from a commercial nutrient's solution and a standard formulation as a control.

2.1 Urine collection and storage

The University of Technology Sydney, Faculty of Engineering and IT has built-in, a separate sewage piping network that allows to sample and store the urine collected from over 30 male urinals. Urinals are waterless and are cleaned periodically using a commercial biodegradable and chlorine-free detergent and, occasionally, a sulphuric acid-based anti-scaling solution. The urine was collected from both the sewage network and, during low flow periods (i.e., weekends and vacation period), from anonymous lab members. As the urine collected from the sewage network was generally already hydrolysed, probably due to the presence of urease-producing bacteria in the pipes, it was decided to store it in 10 L airtight water drums before using in the experiments. The first batch of raw urine was collected the 15/02/2019 and fed to the MBR. New batches were periodically collected depending on reactor performances. Every new batch was analysed for the pH, electric conductivity (EC), DOC, NH_3, anions (PO_4^{3-}, SO_4^{2-}, Cl^-) and cations (Na^+, K^+, Mg^{2+}, Ca^{2+}).

2.2 Nitrification membrane bioreactor start-up and operation

A 5 L MBR was employed for the biological oxidation of urine as presented in Fig. S1. Braided

polyvinylidene fluoride (PVDF) hollow fibre membrane modules from Lotte Chemical (Daejeon, Republic of Korea) were potted, in house, with an active filtration area of 0.042 m^2. The nominal pore diameter of the membrane was 0.03 µm with an inner and outer diameter of 0.8 cm and 2.1 cm, respectively. The compressed air (0.2 m^3/h) was supplied through an air diffuser to provide fine bubble aeration. The aeration served to guarantee levels of O_2 always higher than 6 $mgO_2.L^{-1}$, full reactor mixing and membrane air scrubbing. As the pH in the reactor decreases, due to the consumption of alkalinity during the oxidation of NH_3 to NO_3^-, raw urine was pumped to bring back the pH to the desired set-point. A dosing pump (BL7916-1, Hanna Instruments, Australia) connected to a pH meter (HI6100405, Hanna, Australia) was used to regulate the reactor's pH (Fig. 1). Given that urea hydrolyses giving 1 mol of HCO_3^- and 2 mol of NH_3, without additional alkalinity, the maximum theoretical NH_3 to NO_3^- conversion rate in urine is 50% (Udert and Wächter, 2012). A water level switch was used to activate a peristaltic pump, (Longer BT100 2 J) connected to the UF membrane, to pump the treated urine out of the reactor (Fig. 1). Also, a high resolution (±0.1 kPa) pressure sensor (Keller, Reinacherstrasse, Basel, Switzerland) was installed to monitor the TMP, ensuring a sub-critical flux operation to avoid fouling of the UF module. Given the high fouling potential of urine (Jacquin et al., 2018), the UF module was operated at a flux of about 0.5 $L.m^{-2}.h^{-1}$ which led to low TMP development (3.2 ± 2.3 kPa) which obviously is not an optimised operational flux. The MLSS of the reactor was measured weekly and maintained to approximately 5 ± 1 $g.L^{-1}$ that allowed to keep the MLSS/MLVSS ratio above 0.85. To do so, about 10 mL of sludge was discarded daily (i.e., solid retention time of ~500 d). HRT changed depending on the NH_3 concentration and pH set-point. During stable operations, at pH of 6.2, the measured HRT was 14 ± 5 d^{-1}. Finally, the UF-MBR permeate was collected in a 2 L glass bottle (Fig. S1) and stored at 4° C in a 20 L canister ready to be used for the MD tests.

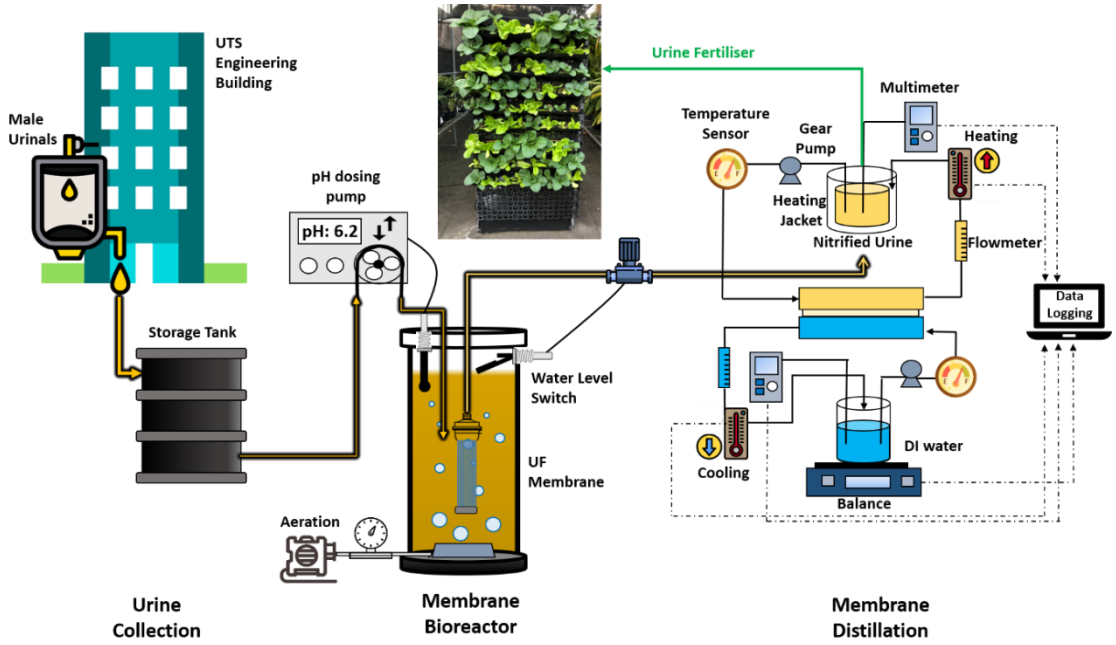

Figure 1 Schematic diagram showing the collection, nitrification via MBR, dewatering via DCMD and testing of urine fertiliser on a vertical garden.

2.3 Membrane distillation set-up and operation

DCMD configuration was chosen due to its higher flux and ease of set up (Lee et al., 2017). Fig. 1 schematically shows the bench-scale DCMD setup used for the experiments. In each experiment, 3 L of urine collected from MBR (MBR effluent) and 1 L of de-ionised (DI) water were recirculated, in counter-current flow mode, inside the acrylic membrane cell. The acrylic membrane cell used had two symmetric channels, each with 7.7 cm length, 2.6 cm width and 0.3 cm depth, allowing for an active membrane area of 0.002 m^2. Durapore®-GVHP PVDF flat sheet membranes were used for the experiments. Their nominal pore size is 0.22 µm, thickness of 125 µm, the contact angle of 131 ± 1˚ and active layer porosity of 75% (Volpin et al., 2018). The dense layer of the membrane always faced the feed solution. A heating jacket, connected to a heater, was used to maintain the urine temperature at 55˚ C while a heat exchanger, connected to a chilling unit, was used to keep the DI water temperature at 20˚ C. A moderate feed temperature was chosen to minimise organic fouling and shift the NH_3 (g) / NH_4^+ (l) equilibrium towards the latter (Bates and Pinching, 1950; Naidu et al., 2017; Naidu et al., 2014). Before starting the experiments, feed and permeate were recirculated, bypassing the membrane cell, for about 30 min to reach the targeted temperature. After that, the cross-flow velocity used to flow the solutions inside the membrane cell was of 8.5 $cm.s^{-1}$. All the

experiments were then run upon reaching 95% water recovery. Finally, as real nitrified urine contains HCl (even if in low concentrations), only acid resistance plastic tubing and fittings were used for the experiments to prevent any corrosion (Etter and Udert, March 2016).

2.3.1 Membrane performance evaluation and cleaning

The performance of the MD process was measured in terms of water flux and solute transport. The flux was calculated based on the step increase in the permeate weight over time. This was recorded with the aid of a balance connected to a computer for continuous data logging. Permeate conductivity and pH were also continuously recorded as they are considered useful parameters to assess the NH_3 transport (measured by the pH increase) and membrane wetting (sharp permeate conductivity increase). Additionally, permeate samples were taken at a regular interval to measure for the anion/cation, NH_3 and DOC rejection. The rejection was calculated based on Eq. S1 in the SI.

Membrane fouling reversibility was evaluated by inspecting the performance and morphology of the membrane before and after the cleaning. First, a membrane coupon was used to concentrate urine over 95% (i.e., EC > 250 $mS.cm^{-1}$). The coupon was then cut in three pieces, each cleaned with a different solution: (I) DI water, (II) 0.1 M citric acid or (III) 0.1 M NaOH. Cleaning was done by placing the coupon in a beaker, filled with the cleaning solution and agitated on an orbital shaker. After 4 h, the membrane was extracted, washed with DI water and dried. After drying, the coupon was soaked in ethanol, frozen with liquid nitrogen and cut with a sharp blade. The resulting cross-sections were analysed using a scanning electron microscope (SEM) (SEM, Zeiss Supra 55VP, Carl Zeiss AG, Germany). Based on the visual inspection of the cleaned membranes, NaOH showed the highest degree of “brown layer” removal. However, to assess the effectiveness of NaOH in restoring the initial flux, the repeated cycles of MD operation and cleaning were performed while keeping the same membrane.

2.4 Analytical measurements

Anions (NO_3^-, NO_2^-, PO_4^{3-}, SO_4^{2-}, Cl^-), cations (Na^+, K^+, Mg^{2+}, Ca^{2+}) and NH_3 were measured at every stage of the process. Cations concentrations were measured using Microwave Plasma Atomic Emission Spectroscopy (MP-AES 4100, Agilent, USA) while anions using Ion

Chromatography (IC, Thermo Fisher Scientific, USA). NH_3/NH_4^+ was measured via colourimetric analysis (Merck KGaA, Darmstadt, Germany) using a spectrophotometer (Spectroquant NOVA 60; Merck KGaA, Darmstadt, Germany). Each sample was first filtered with a 0.45 µm cellulose esters filter. Given the high nitrogen, sodium and potassium concentration in the urine, prior to the NH_4^+, Na^+ and K^+ analysis the samples had to be diluted 50 times.

2.4.1 Organic characterisation

Each sample was filtered with a 0.45 µm cellulose esters filter, and the pH adjusted to be within 5-8. DOC was measured in the urine before and after the MBR and in the MD permeate. Analytikjena Multi N/C 2000 was used for the DOC analysis while liquid chromatography coupled with organic carbon (LC-OCD) (DOC-Labor, Germany) was used to investigate the organic fractionation across the processes.

2.5 GSky Versa Wall and plant response assessment

The GSky Versa Wall (TPR Group, Sydney, Australia) system was chosen, among others, due to its flexibility, versatility and wide application in vertical gardens. The experiment was carried out from 23-04-2019 to 13-06-2019 at the Royal Botanical Garden Sydney Nursery on a west-facing outdoor GSky Vertical Wall module, covered with a twin-wall polycarbonate sheet to minimise the effect of rainfall on nutrient solutions. Lettuce 'All Year Round' (*Lactuca sativa*, Mr Fothergill's seeds, Australia) and Pak Choi 'Hei Xia F1' (*Brassica rapa* Var. chinensis, Johnsons-Seeds, Australia) were germinated in different growing media, grown in a controlled temperature (18-27° C) glasshouse for about 3 weeks and fed with the respective nutrient solutions before being transferred to the wall. Climatic data were recorded, and they could be summarised by a minimum temperature of 11 ± 3° C, a maximum of 26 ± 4° C and a 9 am humidity of 67 ± 9%.

Urine fertiliser performance as nutrient solution 1 (Us) was benchmarked with a commercial hydroponic solution (Optimum Grow twin pack hydroponic nutrient, Growth Technology, Australia) as nutrient solution 2 (Cs) and a control hydroponic solution tailored for lettuce and Pak Choi (i.e., Half-strength Hoagland's solution) as nutrient solution 3 (Hs). The composition of Optimum Grow and Half-strength Hoagland's solutions can be found elsewhere (Chekli et

al., 2017). Three different growing media were used for the experiments and their detailed characteristics and pictures, as well as the Versa Wall operation, is described in section 1 of the supporting information and Fig. S5.

Overall, each combination of nutrient solution-substrate-plant was grown in triplicate, meaning that 81 plants were gown in total (Fig. 6). Each nutrients solution was diluted with deionised water to achieve a nitrogen concentration of about 110 mg-N.L^{-1} (~500 times concentrate urine dilution), and their final elemental composition is displayed in Table S2. Finally, the diluted solutions were replenished every week based on the water level and EC, while the pH was adjusted using KOH or H_3PO_4 to keep it at 6.1 ± 0.2 (TPS -Aqua CPA, V6819).

2.5.1 Data collection and analysis of growth performance

At the end of the experiment, roots and shoots of each plant were separated to assess fresh biomass. Fresh weight (FW) of samples was recorded using an EJ Series scale (AND, EJ-610, A&D Co LTD, Korea). To measure the dry biomass, leaves and roots samples were placed in paper bags in a Memmert GmbH oven (Model 400, Germany) at 60˚ C for 72 h (Chekli et al., 2017; Li et al., 2018) and then weighted again (DW). Finally, roots to shoots ratio (RSR) was also calculated as the ratio between roots dry weight and shoots dry weight.

3 Results and Discussion

3.1 Nitrification bioreactor

The MBR reactor was firstly started in October 2018. The seed sludge was collected from a wastewater treatment plant (Central Park Sydney, Ultimo, NSW, Australia) and, for the first 20 weeks of operation, the sludge was gradually acclimatised to the new substrate. As the salinity, C/N ratio and pH of urine differed significantly from municipal wastewater, urine was initially diluted 100 times and the pH corrected to 7 by addition of HCl (Fumasoli et al., 2016; Jacquin et al., 2018; Udert and Wächter, 2012). Following the increase in the abundance and activity of ammonia and nitrite oxidising bacteria (AOBs and NOBs), estimated through the amount of NO_3^- produced per gram of MLSS, urine dilution was gradually decreased. Once 4 times urine dilution was reached, automatic urine dosing to the bioreactor was introduced. In this mode of operation, the high alkalinity of raw urine was enough to keep in check the pH of the reactor. To reduce the risk of NO_2^- accumulation, the pH set-point was set between 5.8 and 6.6 (Fumasoli et al., 2017). High throughput operation was achieved by increasing the pH set-point to 6.4 – 6.6, thereby reducing the HRT of the system. That is because as the bioreactor pH approaches neutral the nitrification rate increases, which causes higher alkalinity consumption and, therefore, faster urine dosage to buffer the pH drop (Udert and Wächter, 2012). However, higher urine dosage is associated with higher NH_3 loading rate which can then result in higher NO_2^- accumulation rate due to an imbalance between AOBs and NOBs, in favour of the first one (Fumasoli et al., 2017; Fumasoli et al., 2016). When the AOBs population outnumbers the NOBs, the excess NO_2^- produced by the AOBs would inevitably accumulate in the bioreactor which over time further inhibits the NOBs activity (Fumasoli et al., 2016; Udert and Wächter, 2012). Conversely, operating at lower pH range (pH 5.8 – 6.2) minimised the risk of NO_2^- accumulation but at the expense of the HRT. (Fumasoli et al., 2017) also showed that, at low pH, (i.e., lower than 5.4), acid-tolerant γ-proteobacterial AOB accumulates while the microbial community responsible for high-rate nitrification reduces which possibly causes the emission of hazardous NO compounds.

3.1.1 Nitrite accumulation events

Two nitrite accumulation events occurred during the reactor operations. The first NO_2^-

accumulation event (NO_2^--N > 100 mg.L^{-1} in the MBR permeate) happened when urine dilution was reduced from 4 to 2 (i.e. NH_3-N increased from 800 to 1600 mg.L^{-1}). Here, a failure in the pH regulation system caused the pH to increase to 7.5-8 for over 24 h. Feeding was then interrupted to allow for the conversion of the NO_2^- in the bioreactor system to NO_3^-, and the pH was corrected using HCl. For the following 6 weeks, the reactor was operated at high HRT of 35 ± 5 days and a pH of 6.1 ± 0.1. During this period, the NO_2^- concentrations were still higher than 20 mg-N.L^{-1} but slowly decreased to less than 1 mg-N.L^{-1}. The second NO_2^- accumulation event, visible in Fig. 2(B), happened when operating the bioreactor with undiluted urine. In this case, the reactor was re-stabilised in about three weeks only by pH set-point adjustment. Although, both the DOC removal and NH_4^+ to NO_3^- conversion rate dropped during this event, the DOC reduction always remained above 85% (Fig. 2(A)).

In the last 8 weeks of stable operation, only minimal pH set-point adjustments had to be made and an average HRT of 20 ± 3 days was reached with an ammonia conversion rate of 65.2 ± 21.5 mg-N.L^{-1}.d^{-1}. Overall, an average DOC removal rate of 95% and 50% NH_4^+ to NO_3^- conversion rate (which is the theoretical maximum conversion indicated by urine's alkalinity) was achieved. Overall, once acclimatised, the MBR was able to achieve a stable and high DOC removal with about 50% NH_4^+ to NO_3^- conversion rates (Fig. 2(B)). Table 1 also shows that the essential plant macronutrients (i.e., N, P and K) were all preserved in the final permeate.

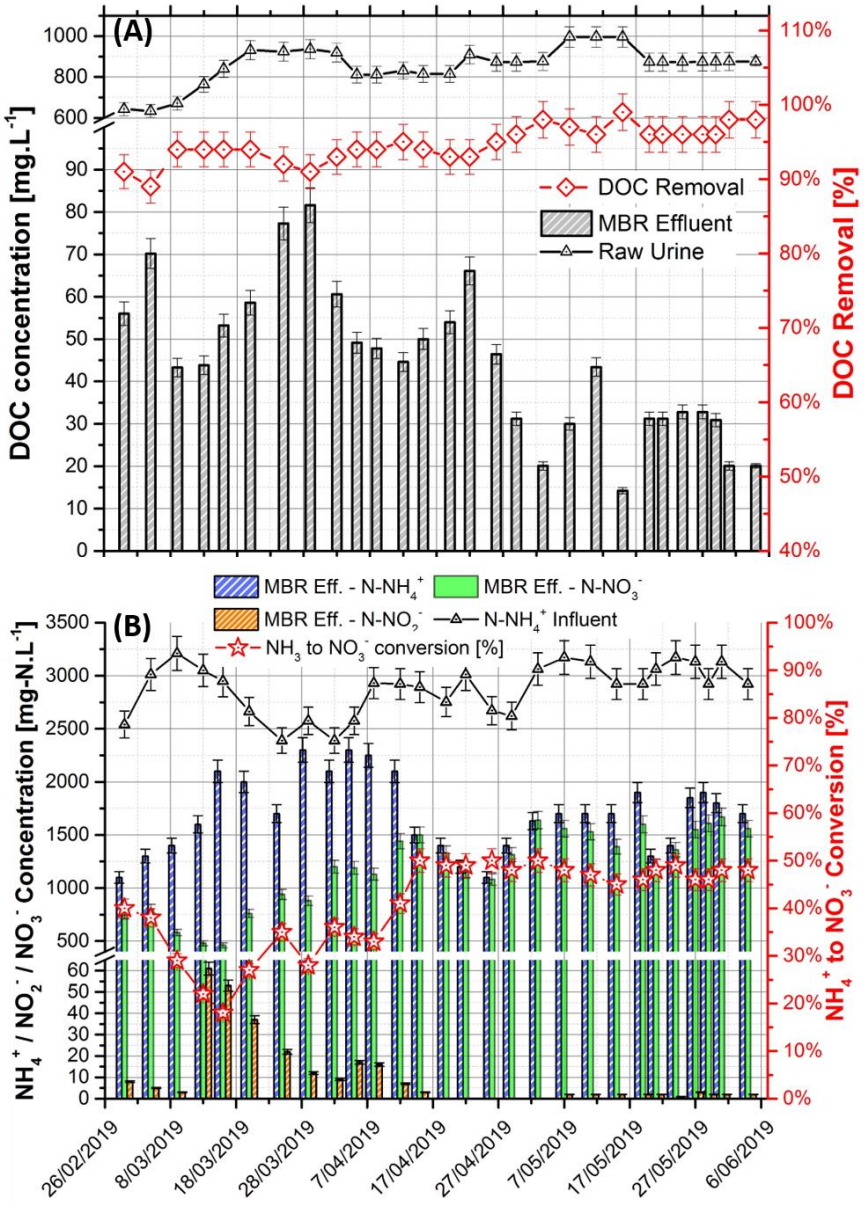

Figure 2 Performance of the MBR when using raw undiluted urine as feed. The top graph (A) shows the removal of DOC while the bottom (B) the conversion of NH_3 to NO_2^- and NO_3^-. The operating pH was 6.2 ± 0.2.

Table 1 Characteristics and ionic composition of urine before and after MBR, after MD and of MD permeate.

		Raw untreated urine	Urine after MBR	Concentrated urine	MD Permeate*
EC	[$mS.cm^{-1}$]	32 ± 5	39 ± 10.5	> 250	0.16 ± 0.1
pH	[-]	9.1 ± 1	6.1 ± 3	4.5 ± 2	9.1 ± 1
DOC	[$mg.L^{-1}$]	865 ± 265	40 ± 15	920 ± 115	1.1 ± 0.1
NH_3-N	[$mg.L^{-1}$]	3038 ± 271	1736 ± 475	31250 ± 1650	22 ± 1.1
NO_3^--N	[$mg.L^{-1}$]	n.d.	1560 ± 104	29641 ± 1865	2.9 ± 0.1
NO_2^- N	[$mg.L^{-1}$]	n.d.	<1	n.d.	n.d.
Cl^-	[$mg.L^{-1}$]	1410 ± 71	1410 ± 71	24800 ± 740	n.d.
PO_4^{3-}-P	[$mg.L^{-1}$]	230 ± 51	247 ± 34	4770 ± 230	n.d.
SO_4^{2-}	[$mg.L^{-1}$]	780 ± 120	800 ± 58	12000 ± 320	n.d.
Na^+	[$mg.L^{-1}$]	1220 ± 258	979 ± 278	19431 ± 5471	1.7 ± 0.1
K^+	[$mg.L^{-1}$]	1023 ± 240	896 ± 180	17920 ± 896	2.4 ± 0.1
Mg^{2+}	[$mg.L^{-1}$]	2 ± 5	3 ± 2	30 ± 21	n.d.
Ca^{2+}	[$mg.L^{-1}$]	51 ± 12	31 ± 21	465 ± 23	n.d.

* MD permeate was measured after 95% urine concentration rate. n.d. = not detected.

3.1.2 Organics transformation within the reactor

The fractionation of organics present in the raw urine and the MBR effluent was characterised via LC-OCD analysis. Depending on the elution time, the instrument identifies four hydrophilic DOC fractions: (I) biopolymers (BPOCD), with size < 20,000 $g.mol^{-1}$, (II) Humic substances and their degradation by-products (HS + BBOCD), with size between 1000 and 300 $g.mol^{-1}$, (III) low molecular weight acids (LMW-AOCD) and neutrals (LMW-NOCD), having size smaller than 350 $g.mol^{-1}$. Based on mass balance, the remaining DOC is categorised as hydrophobic (Huber et al., 2011). Table 2 shows that urine is mainly composed of LMW organics (i.e., < 350 $g.mol^{-1}$), with little $HS+BB_{OCD}$ and almost a negligible amount of BP_{OCD}. A distinct signal from the OCD was detected after 75 min of raw urine elution time (Fig. 3). This peak, attributed to LMW-A compounds, was probably due to the high concentration of organic acids such as uric acid, hippuric acid, citric acid, glucuronic, oxalic acid and lactic acid which have a concentration

ranging from 30 to > 500 mg.L^{-1} (Putnam, 1971). However, the LMW-A peak is restricted to monoprotic acids, diprotic or triprotic acids such as oxalic or citric acid will elute in the building block fraction due to their charge density (Huber et al., 2011). This might be the cause for the high building blocks fraction in raw urine (14%).

After the MBR the OCD signal of urine shows a higher fraction of humic-like substances and building blocks. This could be due to microbial by-products (SMP or EPS) released in the reactor as well as the aggregation of smaller organics (Jacquin et al., 2018). Organics with MW > 1000 Da were all retained by the UF membrane. (Jacquin et al., 2018) also found that the retention of DOM compounds by the membrane was essential to avoid substrate limitation, which could affect biomass stability. Overall, whatever the organic fraction, the concentrations measured in raw urine were reduced 80-99% by the UF-MBR. Among the different fractions, the reduction of LMW-A was the highest, 99.6%, while 94.4% of the LMW-N were removed.

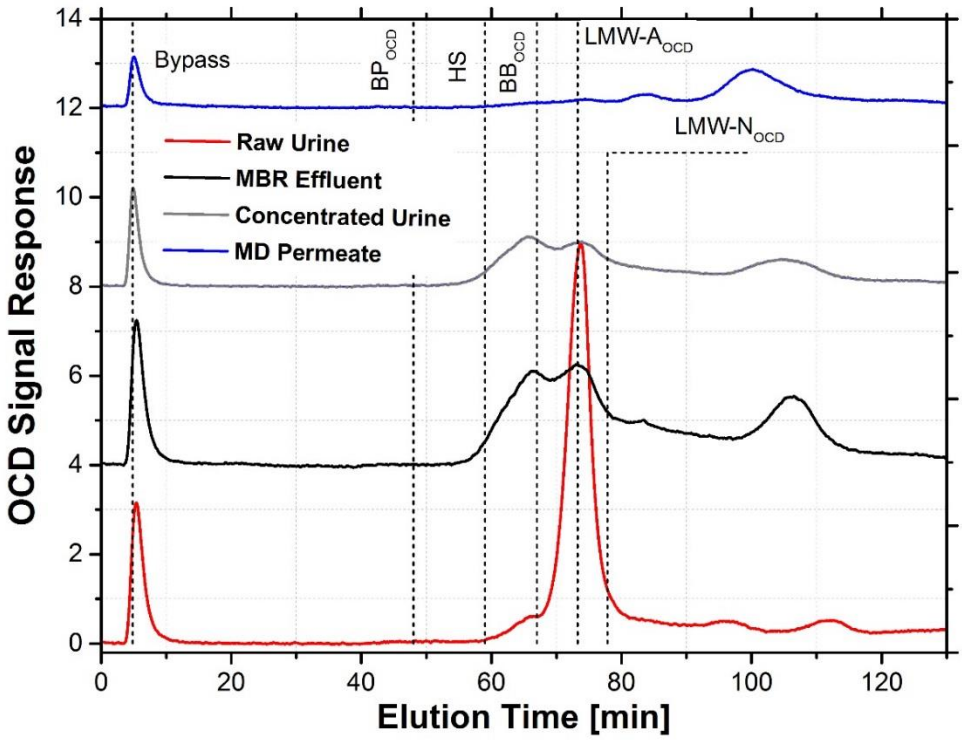

Figure 3 OCD signals obtained by analysing urine before and after the MBR as well as the MD concentration and permeate.

Table 2 LC-OCD data of raw urine, MBR permeate (during stable operation), and MD permeate. Samples from MD permeate and concentrate were collected at 95% urine concentration factor.

			Approximate molecular weights (g/mol):					
			>> 20,000	*~1000*	*~300-500*	*< 350*	*< 350*	
		DOC	**Biopolymers**	**Humic substances**	**Building blocks**	**LMW neutrals**	**LMW acids**	**DOC Reduction**
Raw Urine	mgC/L	987.0	7.8	26.7	138.1	360.4	453.9	
	% DOC		0.8%	2.7%	14.0%	36.5%	46.0%	
Urine after MBR	mgC/L	38.7	0.1	5.2	11.2	20.2	2.0	96.1%
	% DOC		0.3%	12.4%	26.5%	47.9%	4.6%	
Urine after MD	mgC/L	1920.5	20.1	640.1	242.5	985.4	32.4	
	% DOC		1.0%	30.4%	11.5%	46.9%	1.5%	
MD Permeate	mgC/L	1.13	0.01	n.q.	0.13	0.99	0.01	99.9%
	% DOC		0.4%	--	11.1%	87.8%	0.7%	

3.2 Membrane distillation performance

In this section, the urine dewatering performance of a DCMD system was investigated. MD flux decline, permeate quality, and membrane fouling and cleaning were investigated using both synthetic and real nitrified urine. For the real urine experiments, the MBR effluent was fed to the DCMD system without any additional pre-treatment.

3.2.1 MD Flux decline and maximum concentration factor

At a temperature difference (ΔT) between urine and permeate water of 35˚ C, an initial water flux of about 15 $L.m^{-2}.h^{-1}$ was achieved for both synthetic and real urine as MD feed. Within 120-130 h of MD operation, 4.75 L of the urine (of initial 5 L used) permeated to the cold side, thereby achieving 95% water recovery from the urine. Fig. 4(A) clearly shows the marked difference in the urine colour before (light gold) and after the concentration (black colour). In both cases, the EC of concentrated urine exceeded 250 $mS.cm^{-1}$, which is the upper limit of the EC meter used.

Additionally, the permeate conductivity in Fig. 4(A), and the ions rejection values in Fig. S4(A), indicates that membrane wetting did not occur during the 130 h of MD operation. The initial

increase in the permeate EC was due to the pervaporation of NH_3 to the permeate side. As the permeate has little to no buffer capacity, the NH_3 permeation caused a steep pH increase, which stabilised at 9.2 after about 5% urine concentration. The EC stabilisation after about 30% urine concentration was probably because, as pH increases, a higher fraction of NH_4^+ is changed in the form of uncharged NH_3.

It was clear that the MD flux decline was also very severe, especially after about 80% water recovery. In fact, by the end of both experiments, MD fluxes were less than 1 $L.m^{-2}.h^{-1}$(i.e., >94% flux decline). While the water flux for synthetic urine was almost constant until about 80% recovery rate after which it showed a sharp decline, the real urine induced a flux decline throughout the whole experiment, gradual at the beginning and rapidly towards the end. In both cases, the high salinity reached after 80% concentration (>150 $mS.cm^{-1}$) caused a reduction in the vapour pressure, which affected the transmembrane flux (Tijing et al., 2015). Though, in the case of real urine, flux decline was also due to the high amount of organics deposited on the surface of the membrane, and of the spacers (Fig. S2). When approaching 90% concentration, however, inorganic scaling would also occur. Organic fouling accompanied by salt crystals was observed also by (Zhao et al., 2013). In fact, by computing the scaling tendencies of synthetic urine at a function of the concentration factor, through OLI Studio Analyser (Version 9.5, Oli Systems Inc., USA), it was found that after 80% urine concentration, compounds like KNO_3, K_2SO_4 and $NaHCO_3$ would start to precipitate. For example, at 55° C and 90% water recovery, about 6.6% of the KNO_3 is expected to be in crystal form, and its solubility would also drastically decrease at a lower temperature. The precipitation of these compounds is expected to be the reason for the sharp flux decline, for both synthetic and real urine, in the low end of the dewatering process.

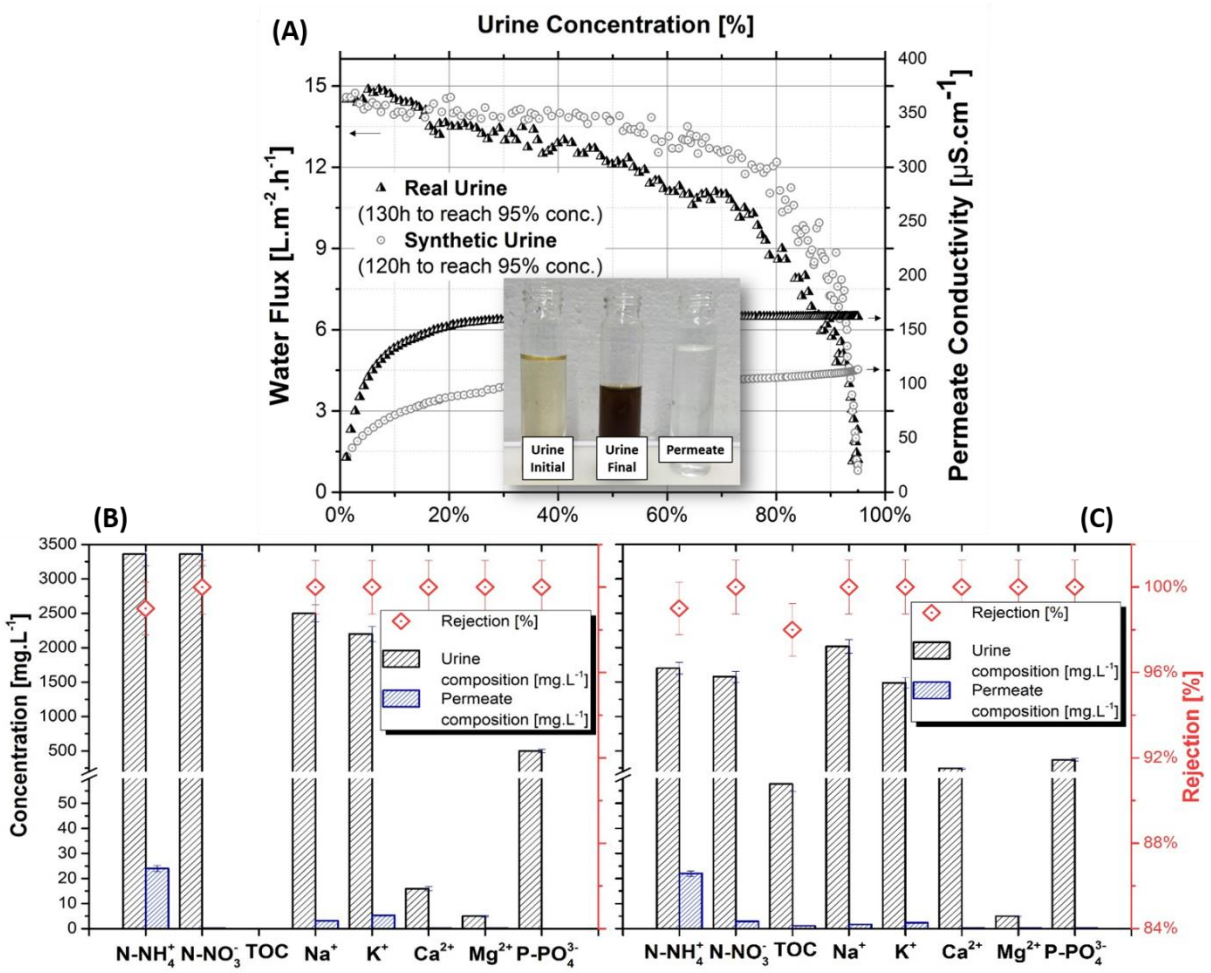

Figure 4 Above: Permeate flux and electric conductivity of synthetic and real nitrified urine as a function of urine concentration factor (Tf = 55 ˚ C, Tp = 20 ˚C, Vf,p = 0.06 m.s^{-1}). Below: Anions, cations and organics concentration in the urine and the final MD permeate using synthetic urine (Fig. B) and real urine (Fig. C). Relative rejection is plotted in red.

3.2.2 Quality of MD permeate and concentrate

Although the presence of ammonical nitrogen in the permeate water from MD is not an issue for water reuse such as toilet flushing or irrigation, in the latter might even be desirable, curtailing the ammoniacal nitrogen transport out of the urine would allow for higher nutrients recovery. The acid dissociation constant (pKa) of NH_3 is temperature-dependent, and it decreases at higher temperatures (Bates and Pinching, 1950). As such, at higher urine temperatures, the equilibrium $H_2O + NH_3\,(gas) \overset{pK_a}{\Longleftrightarrow} OH^- + NH_4^+(liquid)$ was dominated by the reverse reaction, causing more volatile NH_3 gas to vaporise and become part of the permeate (Fig. S4(B). By operating at lower temperatures (55˚C) compared to conventional

MVC distillation (70-100° C) and low pH (6.2), less than 2% of the ammonia was, theoretically, in the form of volatile NH_3 (Fig. S4(B)). This explains why only 1.4% of the initial ammoniacal nitrogen was lost in the process (Fig. 4). Fig. 4 (B) and (C) also showed that all the other ions (NO_3^-, Na^+, K^+, Ca^{2+}, Mg^{2+}, PO_4^{3-}) were entirely rejected by both MD, which confirms that membrane wetting did not occur (Naidu et al., 2017). Finally, Table 1 shows that the ions concentration in concentrated urine is proportional to the ions in diluted urine, meaning that the nutrients were all well retained in the process. Though, the pH of urine decreases from 6.2 to 4.5 after MD, which might be due to the alkali NH_3 lost in the process. As commercial fertilisers are generally acidic, this is not expected to be an issue when it comes to the use of urine fertiliser.

3.2.3 Organics rejection

Table 2 and Fig. 3 show that the vast majority (87.8%) of the organics in the MD permeate were LMW-N. This was expected as between the different organic fractions, LMW-N had the highest concentration, followed by BB and humic-like substances. However, as volatility is generally inversely proportional to the MW, LMW-N is most likely to move to the gas phase and permeate through the membrane. This is expected to be the leading cause for the visible fouling layer that persists on the membrane even after DI-water flush (Fig. 5(C)). (Naidu et al., 2014) demonstrated that LMW humics could penetrate the membrane pores causing severe membrane fouling in a DCMD operation. As the large majority of organics in the nitrified urine are LMW-N, likely, organic foulants in urine also caused membrane pore blockage. Overall, the organic fraction that permeated through the membrane was less than 2%, which explains why the OCD signal of concentrated urine in Fig. 3 shows peaks very similar to diluted nitrified urine.

3.2.4 Membrane cleaning and flux recovery

Fouling reversibility tests were conducted by comparing two successive MD operation cycles of real urine concentration each time up to 95%, followed by DI water (control) or 0.1 M NaOH cleaning before using the same membrane for the next cycle. NaOH was chosen as the pore blocking foulants described in the previous section are expected to be LMW organics (Srisurichan et al., 2005; Tijing et al., 2015). Our previous studies on urine concentration using

hydrophilic membranes also showed that NaOH could entirely remove the organic fouling layer (Volpin et al., 2019). As Fig. 5(C) clearly shows that the membrane was still fouled after DI water cleaning, the cyclic flux recovery tests were conducted only after NaOH cleaning.

The SEM cross-section of the membrane before and after alkaline cleaning showed that the fouling deposits indicated by rough and irregular surface profile on the membrane seem to have been eliminated (Fig. 5(C and D)). The SEM image of the cleaned membrane was, in fact, similar to the pristine membrane (Fig. S(3)). This can also be visually confirmed through naked eyes observation of the membrane before and after cleaning. Also, similar MD permeate flux pattern was observed for the two subsequent cycles of operation (Fig. 5(A)), thereby demonstrating the effectiveness of NaOH cleaning strategy.

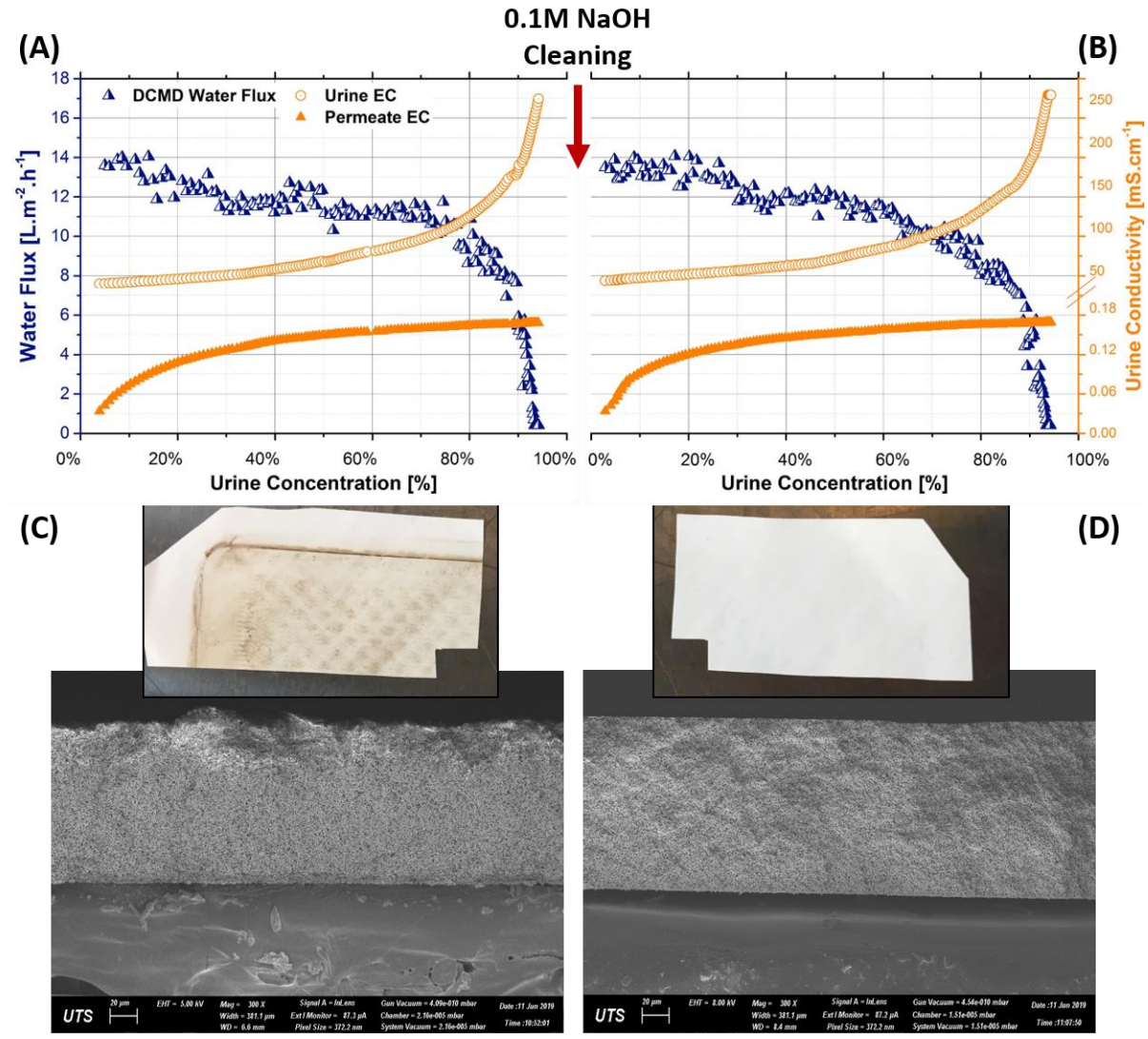

Figure 5 Above: MD permeate flux, feed and permeate conductivity for two repeated cycles (A and B) of real urine concentration, with 0.1 M NaOH cleaning procedure. Below: SEM images of the MD used for real urine concentration, after DI-water flushing (C) and after alkaline with 0.1 M of NaOH (D).

3.3 Lettuce and Pak Choi plants growth

The concentrated nitrified urine product (95% of the water removed or concentrated 20 times) obtained from a combined MBR-MD process was compared with the half-strength Hoaghland solution and Optimum Grow (commercial hydroponic nutrient solution) for growing lettuce and Pak Choi in a vertical garden module at the Sydney Royal Botanical Garden (Fig. 6). The concentrated urine and the Optimum Grow solutions were diluted to reach 110 mg-N.L^{-1} and matching the nitrogen in the half-strength Hoagland solution. At this dilution, the diluted urine solution was found to have about 4 to 7 times less phosphorous, 4 to 5 times less potassium and over 100 times less calcium and magnesium compared to the other two nutrient solutions (See Table S2). This is due to the exceptionally high concentration of nitrogen in the urine compared to other elements. The nitrogen concentration is used as a benchmark for dilution because higher nitrogen concentrations are detrimental to plants as it can cause foliage dehydration and burn (Broadley et al., 2003). Because of the high dilution required for the urine to lower the nitrogen concentration, the EC of the diluted urine is also 3 to 4 times lower compared to the others (i.e., 336 ± 20 vs 1055 ± 79 mS.cm^{-1}) (Fig. S6). However, this could be an advantage for the farmers using urine fertiliser with lower salt-index, as farmers must meet strict EC discharge guidelines to prevent environmental pollution.

As similar growth values were obtained using the three different potting mixes, for the sake of brevity, only the results from PM1 are here presented (Fig. 7) while the others can be found in the SI (Fig. S7). Fig. 7 shows that the Pak Choi roots and aerial parts biomass results obtained with urine fertiliser were statistically the same as ones obtained with commercial fertiliser and the half-strength Hoagland solution. The Pak Choi fresh aerial parts yield obtained using just urine was 41.7 g (± 13.3 g) while the weight of the root was 5.7 g (± 1.6 g). Additionally, Fig. 6 shows that there are no apparent signs of yellowing or scorching of older leaves, a sign of nitrogen, potassium or calcium deficiency, or stunting, a sign of phosphorous or nitrogen deficiency (Chekli et al., 2017). However, the plants grown in the bottom tray are visibly smaller. This could be due to uneven flow distribution in the module. As the water flowed in the trays from top to bottom, plants growing in the top tray likely captured and used more nutrients compared to plants in trays 2 and 3. The installation of drippers in every tray might be more suitable to provide similar nutrients for the downstream plants. At this stage,

however, that is still quite speculative.

The biomass yield of lettuce using urine nutrient solution was, on average, slightly lower than the biomass yield produced with Half-strength Hoagland's solution, 16.1 g (± 2.9 g) and 34.4 g (± 9.5 g) respectively but similar to the biomass yield with the commercial fertiliser 24.4 g (± 4.8 g). This could be due to the lower concentration of phosphorous and potassium in the urine. Neocleousa and Savvasb (2019) showed that at least up to 0.028 $g_P.L^{-1}$, *Lactuca sativa* improves the P use efficiency to cope with the deficiency (Neocleous and Savvas, 2019). Here, however, urine has a P concentration of 0.005 $g_P.L^{-1,}$ which might be the cause for the reduced yield. Nonetheless, the lettuce roots/shoots average values were found to be similar or higher compared to the one reported in the literature (i.e., 0.11 ± 0.2) (Li et al., 2018).

Overall, urine fertiliser showed to be more effective in growing Pak Choi compared to lettuce. When using urine for growing lettuce, the addition of phosphate and potassium might be required to enhance the biomass yield. When looking at a larger scale implementation of fertiliser production from urine, it is essential to optimise both the size of urine equalisation tanks and the EC set-point of the thermal dewatering. This is to achieve a product which is consistent in N-P-K concentration. That, together with the quantification of pathogens and personal care products contained in the produced fertiliser, is paramount to ensure a safe and consistent product.

Figure 6 Picture of the GSky Versa Wall set up with the different growing media. The first three rows (A) were fertilised with Half-strength Hoagland's solution (Hs), the second three (B) with urine (Us) and the bottom three (C) with the commercial nutrients solution (Cs).

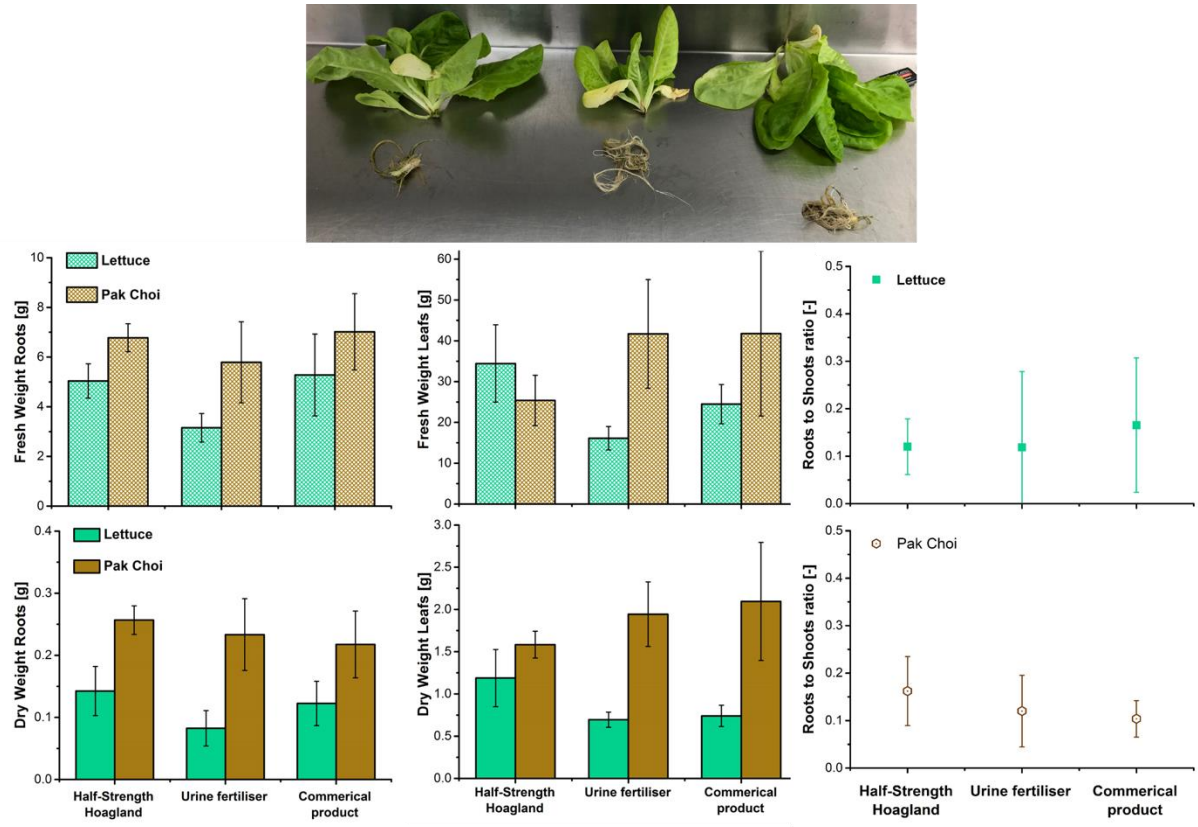

Figure 7 Fresh and dry weight of aerial parts and roots for lettuce (green) and Pak Choi (brown) grown in PM1. Roots to shoots ratio are also displayed in the column on the right. A picture of the separation between leaves (aerial parts) and roots of lettuce is shown at the top.

4 Conclusions

In this work, the human urine effluent from a UF-MBR was dewatered using DCMD process and tested as a nutrient solution to grow lettuce and Pak Choi in a vertical garden. Overall, it was proven that DCMD is able to reach 20 times urine concentration, achieving a dissolved solids concentration of up to 280 $g.L^{-1}$. Also, the NO_2^- accumulation events frequent during urine nitrification, were easily counteracted by tuning the pH set-point of the reactor. This allowed achieving a stable 95% DOC removal and 50% NH_3 to NO_3^- oxidation. LC-OCD analysis also found that, while most of the LMW organics in raw urine are LMW-A, the MBR effluent was mainly composed of LMW-N. The high fouling potential of LMW organics had a significant impact on the MD flux decline during the dewatering step. In fact, a thick fouling layer deposited on the membrane surface, especially after 80% water recovery, causing the water flux to decline from 15 $L.m^{-2}.h^{-1}$ to about 1 $L.m^{-2}.h^{-1}$. Even though alkaline cleaning successfully restored the initial DCMD performances, the chemical cleaning cost should be accounted for when benchmarking MD with distillation.

Finally, encouraging results were observed when growing lettuce and Pak Choi using just MD concentrate as a nutrient solution. In fact, all three solutions tested yielded similar Pak Choi biomass. This was despite the lower EC of urine fertiliser compared to the commercial nutrient solutions and control solution. However, more extensive trials are recommended to understand how to enhance the yield of crops fertigated with urine, and whether changes in the upstream process could improve the quality of the fertiliser.

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