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# Nutrients in a Circular Economy: Role of urine separation and treatment

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*Abbreviations:* WWTP, wastewater treatment plant; NiCE, nutrients in a circular economy; ARC, the Australian Research Council; FAO, The Food and Agriculture Organization; UDFT, urine diversion flush toilets; UDDT, urine diversion dry toilets; UD, urine diversion; AOB, ammonia oxidizing bacteria; NOB, nitrite oxidizing bacteria; FA, free ammonia; FNA, free nitrous acid; PBBR, packed-bed bioreactors; MBBR, moving-bed biofilm reactors; MBR, membrane bioreactor; SBR, sequencing batch reactors; MAP, magnesium ammonium phosphate; GPHM, gas permeable hydrophobic membrane; HFMC, hollow fibre membrane contactor; PAC, powder activated carbon; HRT, hydraulic retention time; GAC, granular activated carbon; MFC, microbial fuel cell; MEC, microbial electrolysis cell; ED, electrodialysis; CDI, capacitive deionisation; AEM, anion exchange membrane; CEM, cation exchange membrane; EDMBR, electrodialysis membrane bioreactor; RED, reverse electrodialysis; MCDI, membrane capacitive deionization, FCDI, flow-electrode capacitive deionisation; LMC, liquid membrane chamber; MD, membrane distillation; DCMD, direct contact membrane distillation; VMD, vacuum membrane distillation; RO, reverse osmosis; NF, nanofiltration; FO, forward osmosis; CI, chief investigators.

## **Abstract**

The increasing global population, over the last few decades, brought about the rise in synthetic fertiliser market prices along with explosive agricultural demand. Source separation of urine can be one of the most effective solutions for nutrient recovery as a fertiliser, transforming the conventional linear economy into a circular economy. The urine diversion from wastewater can improve conventional wastewater treatment plants to be energy-efficient and cost-effective, as a considerable quantity of nutrients in wastewater is derived from urine. The stabilisation and sanitisation of source separated urine have been studied earlier by applying physical, biological, chemical, and electrochemical treatment processes. Further processes such as evaporation, freeze-thawing, and membrane processes have been investigated to recover as well as concentrate nutrients as a urine-derived fertiliser. As such, this review discusses the urine diversion and collection systems as well as previous technologies for the urine treatment and nutrient recovery. In addition, the industrial research hub funded by Australian Research Council (ARC) which focuses on the Nutrients in a Circular Economy (NiCE) is introduced in this paper. The ARC NiCE hub aims to achieve four themes, including urine collection, processing, fertiliser optimisation, and fertiliser end-use, moving towards a closed circular nutrient economy.

**Keywords:** NiCE, nutrient, urine, circular economy, resource recovery

## 1. Introduction

Scientific understanding of the types of nutrients required by the plants and the discovery of synthetic fertiliser nutrients actually revolutionised agricultural productivity and food production that helped explosive global population growth. Nutrients are finally becoming part of the domestic wastes (solid and liquid) that need treatment and management which otherwise can have a significant impact on the receiving environment. Excessive nutrients discharged along the coastal areas cause eutrophication and this environmental challenge is global. Traditionally, nutrients in the solid and liquid wastes are considered as a waste that needs to be removed however the recent concept has been changed to look into nutrient as a resource for recovery and reuse (circularity) rather than as a waste (linearity) [1, 2]. This new concept is driven mainly by two major issues: very high energy and carbon footprint of the synthetic fertiliser nutrients (mainly nitrogen-based fertiliser) and phosphorus being a finite resource with mine reserves limited to only few countries. Efforts have been made to recover nutrients from the solid organic wastes (such as food, sludge, plants, etc. converted into composts and bio solids) and wastewater (such as wastewater reuse for irrigation, or treatment to remove and recover nutrients) [3-5]. Nutrient in the wastewater is present in highly diluted form and their low concentration makes the nutrient recovery less economical and commercially viable technology. Human urine contributes 85-90% of nitrogen, 50-80% of phosphorus and 80-90% of potassium although it contributes only 1% of the total wastewater volume. As urine is the major source of nutrient in wastewater, there has been a paradigm shift in the approach to recover nutrients from the urine directly instead of wastewater effluent. Diverting urine from the wastewater through the separation of urine at source from the domestic wastewater and subject to appropriate treatment is the most economical approach to nutrient recovery. The source separated urine is much concentrated form for more effective nutrient recovery and reduces nutrient loading in the WWTP which can help reduce wastewater treatment costs.

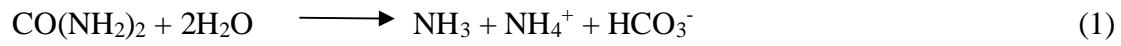
There has been a global wave of efforts to separate urine from sewage and recover nutrients as fertiliser, with initiatives observed in countries such as the United States, Australia, Switzerland, South Africa, and Ethiopia. However, urine diversion toilets, which are the first step in this paradigm shift, are still limited to off-grid locations with poor toilet infrastructure and low-income settings. Pilot projects in Europe and Thekwini, South Africa have shown the limitations of urine diversion toilets, which include being unwieldy, smelly, and unreliable. Moreover, it is impractical to retrofit urban settings with separate sewer pipes to transport urine to a central location. It is worth noting that the widespread adoption and success of nutrient recovery from urine would also be contingent upon the socio-economic context of a region or country, including factors such as cultural acceptance and regulatory frameworks to support sustainable practices [6].

This paper reviews the important role of which a urine-focused research can play in the achieving the nutrients in a circular economy (NiCE) and how the industry research hub jointly funded by the Australian Research Council (ARC) and the industries can contribute to realising this aim. The paper describes various urine treatment technologies recently under development and what more works are needed that could help make a significant contribution towards realising a closed circular economy for the nutrients.

## **1.1 Urine composition and properties**

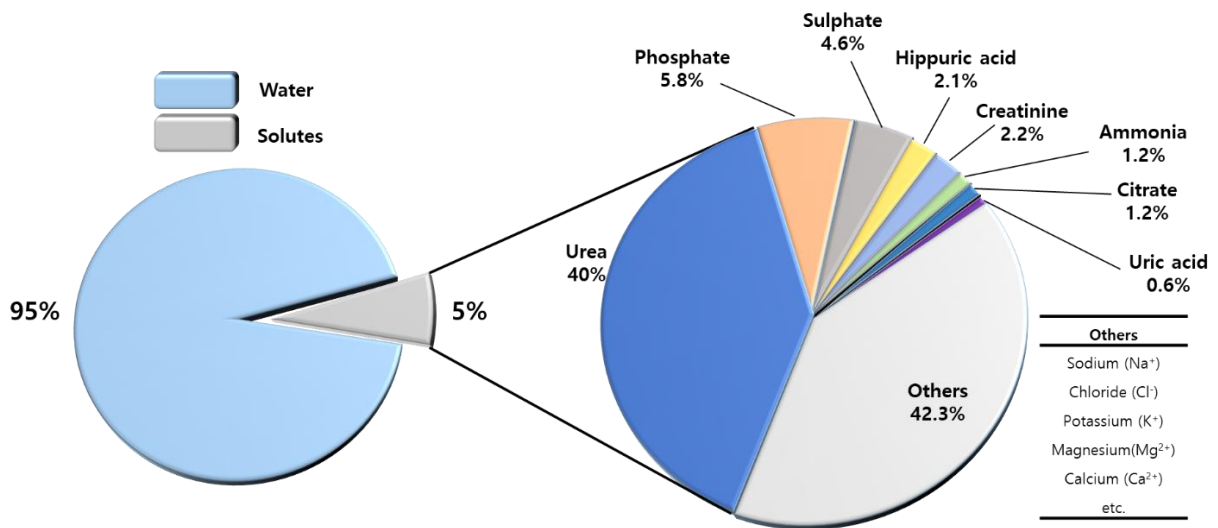
The biological and chemical composition of fresh urine varies with fluid intake, diet, temperature, blood pressure, general health, etc. of the individual person. The chemical composition of source separated urine changes during storage (Table 1), as urea ( $\text{CO}(\text{NH}_2)_2$ ) is hydrolysed to form ammonia and bicarbonate in the presence of urease (Equation 1). This

hydrolysis increases pH (around 9), conductivity and osmotic pressure of the stored urine. The increased pH also results in at least 30% of phosphorous precipitation to form of calcium phosphate, struvite, and calcite [7-9]. Figure 1 describes the composition of urine based on the mass ratio with respect to the total mass of 1.5 L urine [10].



**Table 1.** The composition of fresh and stored human urine [11].

Component (unit)	Fresh urine	Hydrolysed urine
Total nitrogen (mg <sub>N</sub> .L <sup>-1</sup> )	9200	9200
Total ammonia (mg <sub>N</sub> .L <sup>-1</sup> )	480	8100
Ammonia NH <sub>3</sub> (mg <sub>N</sub> .L <sup>-1</sup> )	0.3	2700
Urea (mg <sub>N</sub> .L <sup>-1</sup> )	20	0
Total phosphate (mg <sub>P</sub> .L <sup>-1</sup> )	740	540
COD (mg <sub>O2</sub> .L <sup>-1</sup> )	10000	10000
Calcium (mg L <sup>-1</sup> )	190	0
Magnesium (mg L <sup>-1</sup> )	100	0
Potassium (mg L <sup>-1</sup> )	2200	2200
Total carbonate (mg <sub>C</sub> .L <sup>-1</sup> )	0	3200
Sulphate (mg <sub>SO4</sub> .L <sup>-1</sup> )	1500	1500
Chloride (mg L <sup>-1</sup> )	3800	3800
Sodium (mg L <sup>-1</sup> )	2600	2600
Alkalinity (mM)	22	490
pH	6.2	9.1

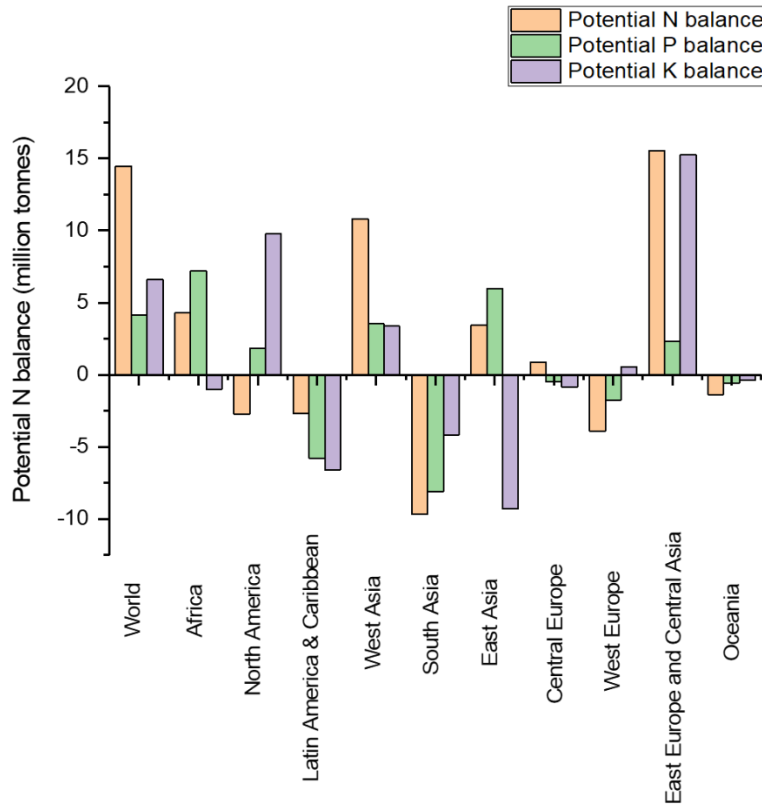


**Figure 1.** The composition of fresh human urine using the mass ratio of the urine component with respect to the total mass of 1.5 L urine.

## 1.2 Global fertiliser demand and production

The Food and Agriculture Organization (FAO) reported that the total consumption of synthetic fertiliser has increased almost 300% by 2019 compared to 1961. Moreover, the global fertiliser demand of the three primary plant nutrients –ammonia ( $\text{NH}_3$ ) as N, phosphate ( $\text{P}_2\text{O}_5$ ) as P, potash ( $\text{K}_2\text{O}$ ) as K– increased to 201 million tonnes by 2022 [12]. This is optimistically predicted to exceed 210 million tonnes by 2026 [13]. Although the world total nutrient capacity of fertilizers was expected to increase further to 273.38 million tonnes by 2020, the world and regional potential balance over five years (Figure 2) suggested that regions such as Latin America & Caribbean, South Asia, West Europe, and Oceania would solely rely on fertilizer import. In addition, Maurer et al. (2003) reported that the energy consumption in the conventional biological ammonium removal processes and struvite precipitation to remove phosphorous is 12.5 kWh/kgN and 8.1 kWh/kgP, respectively [14]. As urine contains high

concentration of nitrogen (on average 8,180 mgN/L), phosphorous (on average 670 mgP/L) and potassium (on average 2160 mgK/L) [14-18], the cost of treating urine is significant [19].



**Figure 2.** World and regional potential nutrient balance between 2016 and 2020. Modified from [20].

## 2. Linearity of nutrient economy

History shows that the development of wastewater treatment technologies has mostly progressed in tandem with the required level of treatment followed by progressive legislation on water pollution. Earlier, BOD and coliforms were considered the main target pollutants for removal from sewage but this later progressed to nutrient removal following the advancement in the science between the nutrient discharge and eutrophication. Nutrient is mostly considered as a pollutant that needs to be removed from the wastewater and limit their discharge to the



environment. Later as the discharge standard became more and more stringent for the nutrient, treatment processes for nutrient removal became more expensive. Effluent irrigation was seen as one of the cheapest options to reduce wastewater treatment through crop uptake although water reuse became a reliable source of water for irrigation especially in the arid regions. Several effluent irrigations are still practised in Australia such as Melbourne water in Victoria, Adelaide in South Australia, Sydney water in New South Wales, etc [21].

In many countries still, wastewater is treated to a high standard only to be discharged to the environment after meeting the target nutrient concentrations. Most of the major wastewater treatment plants in the world have adopted conventional activated sludge process involving biological nitrification-denitrification processes to remove nitrogen from the wastewater. The biologically secondary treated wastewater effluent is either discharged to the environment directly after modest disinfection or subjected to further advanced treatment depending on the end use of the effluent. Wastewater treatment plants involving nutrient reduction below effluent discharge standard are often capital and energy intensive. For example, about 0.13-0.79 kWh of energy per m<sup>3</sup> wastewater treated is used at the WWTP to remove nutrients from the wastewater [22]. Nitrogen is removed from the wastewater effluent through biological denitrification by process subjecting the wastewater to anoxic conditions. The nitrate N in the wastewater is then converted to nitrogen gas following the Equation (2) for denitrification.



Although this approach has significantly helped in removing and lowering the nutrient concentration in the wastewater effluent, the nutrient is simply lost to the environment released as N<sub>2</sub> gas after treatment at significant cost. In this way, the fate of nutrients is one way (linear economy) from the synthesis and back to the environment although it closes the nitrogen cycle. Although most wastewater treatment plants meet the nutrient effluent discharge standard of

each country, cumulative discharge to the aquatic environment is a big concern which ultimately can be a global problem. Eutrophication has been increasingly occurring owing to the intensive fertigation and the following over-enrichment of the agricultural soils and water bodies. The linearity of nutrient economy has also had a significant impact on the natural resources such as phosphorous depletion. In fact, over 95% of the mined phosphate rock in the U.S. has been used for fertiliser production and it is predicted that the phosphorous will be fully depleted by the end of the 21<sup>st</sup> century [23].

### **3. Circular economy for nutrients**

Recently, nutrient in wastewater is seriously considered as a resource that needs to be recovered rather than considered as a waste to be removed and eliminated from wastewater treatment plants. Nutrients generated from the food and human wastes were once synthesised using energy-intensive processes and spending additional energy to just get rid of it from the wastewater makes no sense. Recovering and reusing the nutrient can have a significant environmental benefit while at the same time closing the nutrient cycle. Since the majority of nutrients after consumption becomes part of the wastewater in highly diluted form, nutrient recovery from urine is therefore seen as one of the ultimate approaches to closing the nutrient cycle and achieving circular economy. Urine diversion helps separate collection and treatment of urine from faeces, exemplifying the high potential of energy and resource recovery from source-separated urine at the level of a single household device [24, 25]. In addition, the urine diversion systems contribute less to toilet-related groundwater contamination than conventional sanitation systems [26].

Nutrient recovery from urine involves following steps: source separation (urine diversion from wastewater), designated urine collection system, urine storage, urine treatment and conversion to urine derived fertiliser. Each component of the nutrient recovery from urine is discussed in the following sections.

### **3.1 Source separation of urine and diversion from wastewater**

The most common types of urine diversion in urban areas are urine diversion flush toilets (UDFT) and urine diversion dry toilets (UDDT) which have been commercially available worldwide.

#### **3.1.1 Urine-Diverting Flush Toilet (UDFT)**

In this approach, a separate plumbing system is used to collect urine from the urine diverting flushed toilet. The urine is collected from the flushed urinals and flushed urine diverting pots without mixing the urine with the faeces and other domestic wastewater (see Figure 2). Urine is significantly diluted with flushing water although the dilution can be significantly lower than the normal wastewater without urine diversion. The idea of integrating a sewage system with UDFT was firstly invented in the late 20<sup>th</sup> century in Sweden [27]. The Federal Ministry of Education and Research funded a two-phase collaborative project. The GTZ (German agency for Technical cooperation) program (2005-2006) was to design and construct urine separating system. This was followed by Sanitary Recycling Eschborn (SANIRESCH) program to study the feasibility of implementing ecological sanitation (ecosan) concept for separate treatment and recycling of urine, brown- and greywater from an urban office building [28]. The project

concluded that the urine product is hygienic and safe for use as fertiliser, the social acceptance shows both farmers' and consumers' willingness to use urine as a liquid fertilizer and the project is economically feasible in favourable conditions.

### **3.1.2 Urine-Diverting Dry Toilet (UDDT)**

The first modern version of the waterless system with urine diversion became available in 1970s as a sustainable sanitation system [27]. It reduces the unpleasant odour and vectors (flies), easy to build using locally available materials, suitable for all users and groups and locations (sitters, squatters, washers, and wipers), safe and hygienic faeces handling, and longer product life cycle. However, issues such as pipe blockage due to inappropriate maintenance, presence of foul-smelling, and visible excreta pile constrain its application and promotion [29].

The Ecological Sanitation Ethiopia Project funded by the GTZ started in 2006, Ethiopia, has been demonstrated the technical requirements and economic benefits of UDDT implementation in high population density areas. It was concluded that the use of UDDT in multi-storey buildings and reuse of human excrete produces in agriculture would contribute to self-sufficiency and food-security as well as provide additional income in terms of 1.4 times higher crop yield and productivity. About 25% capital and operational cost reduction was achieved compare to conventional sanitation systems. This shows the advantage for using alternative sanitation systems in higher density areas [30-33].

The Compost Toilet Trial Project conducted by the Wellington Regional Emergency Management Office (WREMO) in 2012, New Zealand, has been undertaken to evaluate the service reliability of urine separating emergency compost toilets under crisis situations where the sewage system is damaged and conventional toilets may not be functioning, such as

earthquake [34]. The trial was conducted for one month. 11 households and workplaces were engaged during that time. It was found that, the compost toilet is safe and hygienic for use inside during exceptional circumstances. A result from the evaluation of user acceptance showed an increase in users' satisfaction as the trial progressed. As here space is limited for urine and the compost disposal as well as manual removal of compost is required at all-time which is labour intensive [35, 36]. The true operation cost and implementation difficulties of composting toilets have only been researched at household level or at the single household device level, but lack of investigation at the large scale [37].

The UTS (University of Technology Sydney) sustainable sanitation project conducted by the Institute for Sustainable Futures (ISF), Australia in 2013, investigated the barriers associated with replacing existing sanitation systems to more sustainable urine diversion (UD) systems [38]. The project consisted of UDDT, water free urinals, piping systems for urine sampling, and tanks for urine storage and transportation. The performances of project were analysed in accordance with regulations and institutions, user practices, operation and implementation, agriculture trials, market, and socio-cultural acceptance. The project observed that the development of urine diversion system could be hampered by: (1) lack policies to support or promote the urine diversion and reuse; (2) no guidelines to regulate source-separated urine practices and (3) low market share in sustainable sanitation business could slow down development of existing product.

### **3.2 Urine collection system and storage system**

Urine diversion requires a separate and designated plumbing system which will be an additional plumbing cost for urine collection and conveyance to the urine storage system. A normal

sanitary plastic pipes such as PVC can be adopted for urine collection system as installed at the UTS FEIT building in Australia. While an additional urine collection plumbing work may not be a significant additional cost for a new building however, the cost can be significant for retrofitting an existing building to install urine diversion system.

Urine storage is another additional infrastructure that may be required and the required storage volume would depend on the treatment capacity and the end use of the urine. Urine is generally required to be stored between 3 to 6 months before application in order to achieve natural disinfection under highly alkaline conditions of the hydrolysed urine [39]. Such a long-term storage will require adequate space which may not be a luxury in urban settings and therefore may require immediate treatment and sent for end use. Urine can be stored in normal plastic tanks. Metals may not be an ideal material for urine storage tanks because of the corrosive nature of the highly alkaline urine.

#### **4. Technologies for urine treatment for nutrient recovery**

Like any wastewater treatment, urine treatment cannot be done using a single process and hence is likely to involve multiple processes depending on the treatment objectives. The urine treatment can be classified based on the purpose of treatment: i) urine stabilisation and sanitisation treatment and (ii) nutrient recovery process. The urine stabilisation involve treatment to prevent ammonia volatilisation to reduce pungent odour, nitrogen loss and achieve sanitisation. Nutrient recovery process involves converting the stabilised and sanitised urine into a safe urine-derived fertiliser with or without concentrating the urine. They are discussed in more details below.

## **4.1 Urine stabilisation and sanitisation treatment**

Stored urine has a very unpleasant smell due to volatilisation of ammonia which is one of the main reasons people are reluctant to use urine as fertiliser. Therefore, urine has to be properly treated to stabilise and prevent the release of pungent odour and loss of nitrogen before it can be used as a fertiliser. Urine treatment processes can be further grouped into physical, chemical, biological and physicochemical processes and the nutrient composition (NPK) and concentration may not significantly change during this treatment step.

### **4.1.1 Physical treatment processes**

#### **Urine storage**

Long duration urine storage has been used as the simplest way of urine treatment since ancient times [11, 40, 41]. Urea is hydrolysed into ammonia which results in a significant increase in pH (up to 9.2) of the urine. This high pH environment provides a natural disinfecting effect for the pathogens present in the urine. The storage period, temperature, and pH are the crucial parameters affecting the inactivation rate of pathogens [11, 16, 42]. It has been recommended that, urine should be stored for up to 6 months or even more before it can be safely used as fertiliser [39]. However, ammonia ( $\text{NH}_3$ ) volatilisation can be an issue resulting in nitrogen loss and odour problem if the storage tank is not tightly sealed [43].

In fresh urine,  $\text{H}_2\text{PO}_4^-$  and  $\text{HPO}_4^{2-}$  accounts for more than 95% of phosphate but under high pH (>9.0),  $\text{HPO}_4^{2-}$  becomes a dominant resulting in the precipitation of phosphate with other minerals such as calcium, potassium, and magnesium [44]. Almost 30% of phosphate can be spontaneously precipitated as struvite and hydroxyapatite, together with all the calcium and

magnesium with around 1% of the total ammonia [11, 40]. Other minerals can also be precipitated as KCl, CaCO<sub>3</sub>, and Mg<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> at high pH. Overall, urine storage by itself is not suitable for direct use as fertiliser due to gaseous ammonia spreading and micropollutant residues, thus further treatments for stabilisation and concentration of nutrients are needed.

#### 4.1.2 Biological treatment

##### Biological nitrification

One of the most effective urine stabilisation methods is biological nitrification, which reduces ammonia volatilisation by converting volatile ammonia-N into more stable form nitrate-N which helps solve odour problem, reduces N loss and also removes organic matters present in urine. As in any biological process, nitrification is two step sequential biological oxidation processes. The first step of this process involves ammonia oxidizing bacteria (AOB) that oxidises ammonia to form nitrite (Equation 3). The second step involves the oxidation of nitrite to nitrate by nitrite oxidizing bacteria (NOB) according to equation 4. The complete nitrification process is shown by equation 5. The formation of nitrous acid releases hydrogen ions, consumes alkalinity and cause the pH decrease [45].



The desired product of stable urine nitrification is a solution contains equal parts of NH<sub>4</sub><sup>+</sup> and NO<sub>3</sub><sup>-</sup>, which requires that nitrite is oxidised at the same rate as ammonia [46]. The growth of



both AOB and NOB can be inhibited by temperature, pH, organic load, DO concentration, free ammonia (FA) and free nitrous acid (FNA) concentration, alkalinity, hydraulic and sludge retention time, and the previous history of the biosystem [47]. It was reported that NOB grow faster than AOB in lower temperature, while AOB grow more rapidly at temperature above 16°C. The pH represents the acid-base equilibria of  $\text{NH}_3$ ,  $\text{NO}_2$  and hydroxylamine during the nitrification process, thus pH fluctuation indicates the instabilities of nitrification process [48]. It is known that NOB has greater pH resistance than AOB, and AOB will result in an irreversible activity loss during pH shocks, while NOB activity stays unaffected. The nitrite accumulation may occur if AOB activity is more favourable than NOB activity. The build-up of nitrite can be detrimental, as it will have fatal impact on both AOB and NOB bacteria and hence, the nitrification process is generally recommended at relatively low temperature and pH [49]. Apart from nitrite accumulation, high concentration of FA and FNA are also important factors for nitrification inhibition.

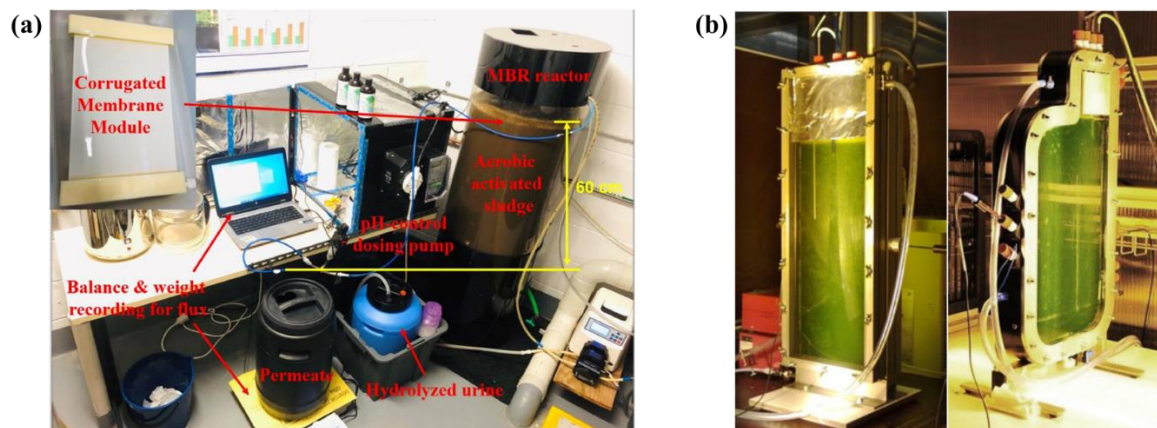
Biological nitrification of hydrolysed urine has been reported by using packed-bed bioreactors (PBBR), moving-bed biofilm reactors (MBBR), membrane bioreactor (MBR), membrane-aerated biofilm reactors (MABR) and sequencing batch reactors (SBR) [49-53]. Urine nitrification is also being studied for long-duration space missions as a promising system which can recover nutrients for soilless cultivation in the spaceship as well as hygienic shower water by treating urine from crews [54, 55]. While attached growth systems such as PBBR and MBBR using biofilm carriers have been widely studied for nitrification from small-scale to pilot-scale, suspended growth systems have also been studied in terms of SBR, showing a superior volumetric nitrification rate up to 1100 mg N/L·d, compared to 640 mg N/L·d in MBBR [46, 51]. However, the difficulty in suspended solids separation for excess biomass removal in SBR could be overcome by the combination with membrane filtration. As MBR

has been an attractive option for both municipal and industry wastewater treatment, it was also applied for the nitrification of real hydrolysed urine [56, 57]. MBR start-up is critical and is usually done starting with dilute urine (using activated sludge from a WWTP for seeding) and then gradually increasing the concentration until the full urine strength at a stable optimal nitrification rate of  $447 \pm 50$  mgN/L·d at around pH 6.2 and FA concentration below 0.5 mg/L [58]. Urine MBR is generally associated with a long start-up period due to the slow-growing nitrifying bacteria in high salinity condition including issues with nitrite accumulation. A 100 L/day pilot urine MBR has been recently installed and tested at the University of Technology Sydney, faculty of engineering and information technology building connected to a source separated urine from the waterless male urinals [59].

### **Nutrient uptake by microalgae**

The high lipid or protein content in various algal species can be utilised as a biofuel or animal food, respectively, as well as a biofertilizer [56, 60]. As cultivation of microalgae requires large amounts of nitrogen and phosphorus, urine can be an effective source of nutrient for recovery by converting into organic bio-fertiliser. Microalgae cultivation via urine media has been mainly conducted in suspended growth systems, either in open-raceways or closed-photobioreactors. The microalgae growth rate and nutrient uptake rate may be largely different depending on the day and night cycles [61, 62]. On the other hand, the indoor photobioreactor has been operated on a comparatively smaller scale with high energy input for lighting and pumping, achieving a higher biomass density and nutrient uptake rate up to 84.1% N and 96.6% P from fresh urine [63, 64]. Innovative design of photobioreactors in recent studies, such as short light-path photobioreactors, bubble column photobioreactors, or submerged membrane photobioreactors intensified the photosynthetic efficiency as well as biomass yield [63, 65-67].

While the previous studies reported that highly diluted urine can effectively cultivate microalgae and cyanobacteria, Tuantet et al. (2014) firstly investigated the effects of undiluted fresh urine and hydrolysed urine on the growth of *Chlorella sorokiniana* [68]. Nitrified urine from the MBR was also demonstrated as a superior growth medium for *Arthrospira platensis* using nitrate as a nitrogen source. [69]. Figure 3 shows the pilot-scale MBR for source-separated urine stabilization as well as photobioreactors for cultivation of *Chlorella sorokiniana* on urine.



**Figure 3.** (a) Pilot-scale MBR for source-separated urine stabilization; (b) photobioreactors for cultivation of *Chlorella sorokiniana* on urine. (a) reprinted from [59] and (b) reprinted from [68] with copyright permission from Elsevier.

#### 4.1.3 Chemical and electrochemical treatment

##### Acidification

Acidification is used to stabilise the highly alkaline urine. The addition of strong or weak acid can prevent the urea hydrolysis of fresh urine, as well as ammonia volatilisation of hydrolysed

urine. It was reported that the former can be more economically advantageous, since the latter requires a larger amount of acid (650 mEq H<sup>+</sup>/L) for neutralisation than that of fresh urine (60 mEq H<sup>+</sup>/L), due to the ammonia and carbonates causing high buffer capacity [11, 40]. It was found that the acidification to pH level 4 is efficient for nitrogen retention, and the subsequent urine odour can be removed [43, 70]. Acidification is also effective for inactivation of pathogens and pharmaceuticals like antibiotics under pH 4 [16]. The issue of malodour and pipe blocking is inevitable for the implementation of non-water urinals in a building-scale urine diversion system, due to the urea hydrolysis and the following mineral precipitation of struvite, calcium carbonate, and calcium phosphate. As such, acidification via acid addition to non-water urinals has been proposed in the previous studies [71]. At the same time, this immediate acidification could increase phosphate recovery by 43% via MgCl<sub>2</sub> addition, as the low pH allowed most of phosphate to remain in an ion state [72].

## **Alkalisiation**

As an alternative to acidification, fresh urine or hydrolysed urine can be stabilised by alkaline addition. Unlike acidification, alkalisiation above pH 10 results in phosphorus precipitation which can produce a P-fertiliser separately. Alkalisiation of urine can be done by either adding alkaline chemicals into liquid phase urine or using dry bed with alkaline agent. Ca(OH)<sub>2</sub> addition to fresh urine has been investigated in previous studies for maintaining the pH above 11 and thereby inhibiting the urea hydrolysis by urease [9, 73]. Since the addition of Ca(OH)<sub>2</sub> promotes the precipitation of calcium phosphate, sustainable fertiliser-producing urinals containing Ca(OH)<sub>2</sub> (10 g/kg urine·day) for instant urine alkalisiation have been developed, which could produce 11.23 g/kg urine of solid fertiliser as well as 998 g/kg urine of liquid fertiliser in a day [74]. Recent studies investigated the urinals combined with the alkaline

dehydration process which can stabilise fresh urine prior to pipe transport and storage, thereby facilitating nutrient valorisation as a solid fertiliser. Wood ash, alkalised biochar, wheat bran, desert soil, and calcium hydroxide, as well as  $\text{MgCl}_2$  and  $\text{Mg}(\text{OH})_2$  have been used as drying media [75, 76]. It was found that wood ash and calcium hydroxide were good alkalising agents producing 9.7% N, 1.5% P, and 8.4% K final solid fertiliser [75].

## **Precipitation**

The pH increase of hydrolysed urine triggers the spontaneous precipitation of phosphorous with magnesium, calcium, iron, and aluminium forming precipitates such as struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ), hydroxyapatite ( $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ ), etc. [40, 77]. While iron and aluminium phosphates are possibly toxic to plants, struvite, magnesium ammonium phosphate (MAP), can be a good fertiliser. Thus, many researchers studied the external addition of magnesium and calcium sources such as  $\text{MgCl}_2$ ,  $\text{Mg}(\text{OH})_2$ ,  $\text{Ca}(\text{OH})_2$ , and  $\text{CaO}$ , to promote nearly all the phosphorus precipitation in urine [78]. Although calcium salt like  $\text{Ca}(\text{OH})_2$  has been widely used as a low-cost material over magnesium salt, calcium salt addition is only recommended for fresh urine, since there is a high possibility of calcium carbonate precipitation rather than calcium phosphate owing to the existence of carbonate after urea hydrolysis [9].

The main mechanisms of phosphorous precipitation with magnesium addition are nucleation and crystal growth [79, 80]. As a low-cost magnesium source, seawater and brine containing a certain amount of  $\text{Mg}^{2+}$  can be applied. While 90% of phosphorous could be recovered by mixing 1:1 of urine and RO brine, 99% of the recovery rate could be achieved by combining seawater and urine into 3.3:1.0 ratio [81-83]. Since the presence of other cations such as calcium and sodium could reduce the struvite purity, high Mg/Ca ratio in the Mg source can be

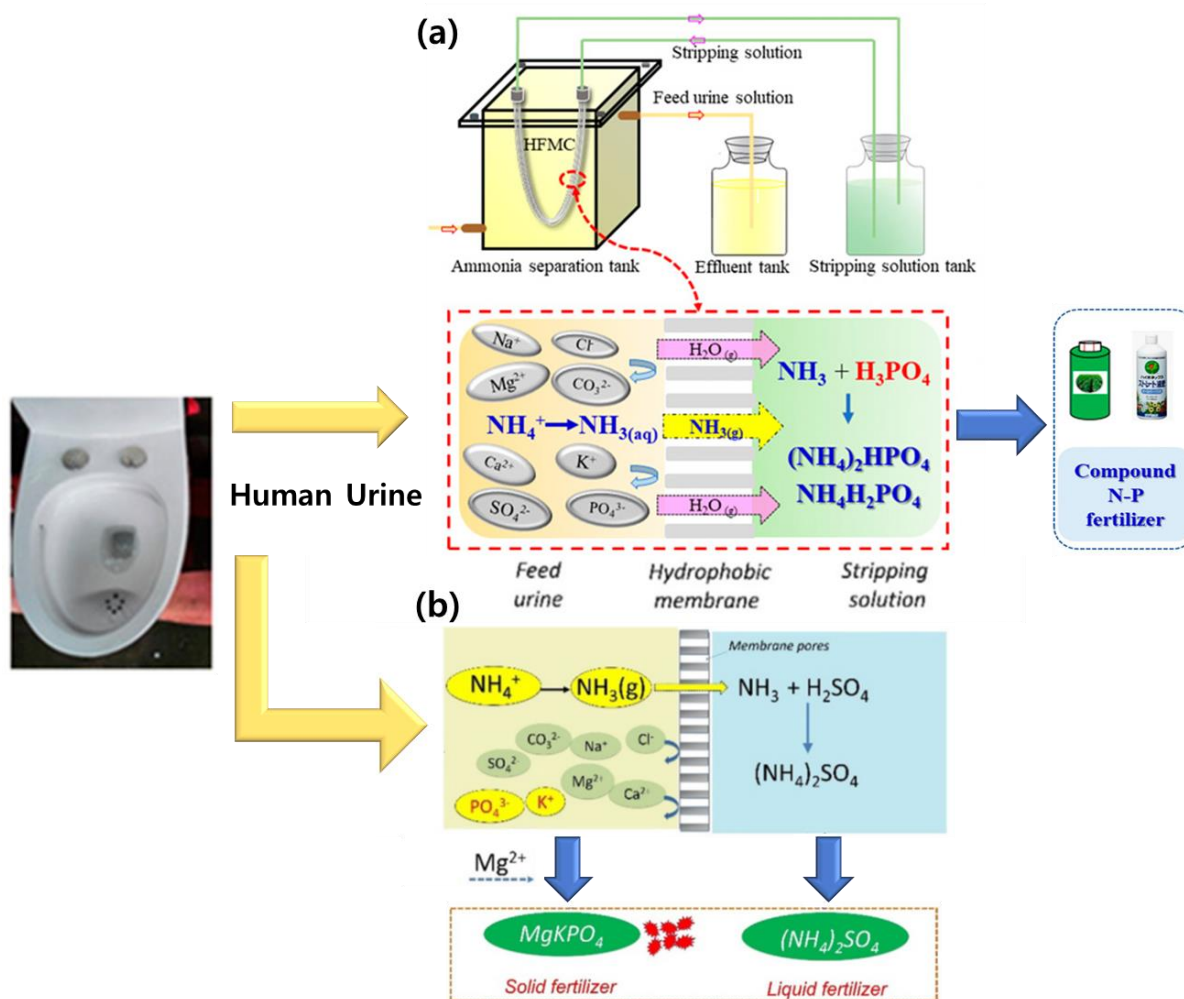
one of the most important factors [84, 85]. Since the nitrogen fraction recovered from struvite is very small (N:P ratio in struvite is 0.5), the remaining nitrogen after struvite precipitation can be further recovered by other processes like air stripping. In case of nitrogen being firstly recovered prior to struvite precipitation, potassium can be recovered along with phosphorous as a K-struvite ( $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$ ), thereby leading to efficient N, P, and K nutrient recovery. Recent studies revealed that fluidized bed crystallisation process under pH 9.5–10.5 and  $\text{Mg}^{2+}/\text{K}^+$  ratio of 1.00–1.25 resulted in maximum 98.4% P and 70.5% K recovery rate by K-struvite production [86-91].

### **Ammonia stripping**

Stored urine, which has a high concentration of volatile free ammonia at around pH above 9.2, is favourable for air stripping at high temperatures. The combination of stripping and an acid absorption process facilitates free ammonia to be stripped in the air column and then to be absorbed to the acid solution. As sulfuric acid has been commonly used for acidification, stripped ammonia is recovered as the liquid form of ammonium sulphate. Although the hydrolysed urine has high pH, additional chemical alkalisation can be beneficial to increase free ammonia fraction in the urine. High pH (10-12), high operation temperature (35–40°C) and air flow rates (0.21–8 m<sup>3</sup>/h) have been applied for high ammonia recovery efficiency [92-96].

Novel technologies, such as vacuum thermal stripping and membrane stripping, have emerged in recent studies. In vacuum thermal stripping process, the urine can generate bubbles due to the lower boiling point than normal, which can strip free ammonia out of urine, while conventional air stripping uses a packed column allowing the air injection. It was found that

pilot-scale vacuum thermal stripping for 12 hours could recover up to 77% of ammonia [97, 98]. Membrane stripping using a gas permeable hydrophobic membrane (GPHM) has been gaining attention, owing to its low energy-consumption and high recovery efficiency as well as the generation of micropollutant-free final product [99]. When GPHM is submerged in hydrolysed urine, the ammonia gas can pass through the membrane because of the ammonia concentration gradient and form ammonia sulphate by contacting acid solution circulating the other side of the membrane [100, 101]. As the free ammonia fraction increases according to pH increase, higher pH in urine can lead to enhanced driving force for ammonia mass transfer [102]. A hollow fibre membrane contactor (HFMC) accompanied with GPHM has been developed in recent studies, which can overcome several drawbacks of conventional air stripping such as flooding and entrainment owing to the complete separation of the feed and acidic solution by the hollow fibre membrane. The development of open-loop HFMC enabled continuous flow of feed urine and the highest ammonia recovery of 80.13% under pH 12 and  $2.61 \times 10^{-5}$  m/s of feed flow velocity [103]. Submerged HFMC facilitated the production of a liquid N and P fertiliser containing up to 55.24 g/L N and 281 g/L P by using  $\text{H}_3\text{PO}_4$  as an absorbing solution [102]. Figure 4 illustrates the schematic diagram of ammonia stripping process using a hollow fibre membrane contactor (HFMC) using  $\text{H}_3\text{PO}_4$  and  $\text{H}_2\text{SO}_4$  as an absorbing solution, respectively.



**Figure 4.** Schematic diagram of ammonia stripping process, (a) a hollow fibre membrane contactor (HFMC) using  $\text{H}_3\text{PO}_4$  as an absorbing solution; and (b) an open loop HFMC using  $\text{H}_2\text{SO}_4$  as an absorbing solution. (a) reprinted from [102] and (b) reprinted from [103] with copyright permission from Elsevier.

## Adsorption

Adsorbents such as activated carbon, ion-exchange resin, zeolite and biochar, have been commonly used for nutrient adsorption from urine. Biochar can be produced by thermal decomposition of waste biomass like agricultural residue, food waste, or sludge. This makes biochar environmentally friendly as well as cost effective, as it can be directly used as a fertiliser as well as a soil conditioner after being saturated by nutrients. The porous and



heterogeneous surface of biochar facilitates physical adsorption activities such as surface precipitation and pore-filling, and in addition, electrostatic attraction, hydrogen bond, and ion-exchange are the main routes of adsorption [104]. Various types of environmentally friendly modified biochar, such as goethite dispersed biochar, eggshell bead-biochar, and magnesium-rich corn cob biomass derived biochar, have been developed in recent studies and showed superior adsorption capability on phosphate recovery and possible use as a slow-release P-fertiliser [105-109]. On the other hand, natural zeolites, which can be obtained from mining, have high ion-exchange capacity due to the presence of cations on the surface such as  $\text{Na}^+$ ,  $\text{Ca}^{2+}$ , and  $\text{Mg}^{2+}$  [110]. Natural zeolite or loess, and modified biochar with faecal sludge or pineapple peel showed 86–87.5% of ammonium removal by adsorption, and the subsequent ammonium saturated adsorbents can be applied as a soil conditioner [111-116].

### **Microbial fuel cell (MFC)**

The combination of biological processes and electrochemical processes facilitates the simultaneous nutrient recovery and energy production. In microbial fuel cells (MFCs), microbial catalysis enables the transformation of chemical energy into electrical energy. As such, human urine was firstly suggested as a fuel source for MFCs by Ieropoulos et al. (2012) owing to its high conductivity and buffering capacity [117]. Many previous studies focused on energy production from urine by using different designs of MFCs such as stacked MFC or membraneless MFCs [118-121]. When the urine is used as an organic source in MFC, ammonium recovery and electricity generation are possible. The ammonium ions in hydrolysed urine can pass through the cation exchange membrane towards the cathode and convert into free ammonia because of the high pH in the cathode chamber. Thus, previous studies have investigated the integration of MFCs and ammonia stripping for ammonia recovery [122].

However, a struvite precipitation on the cathode surface is problematic as it can lead to the reduction of energy production [123]. Thus, MAP precipitation followed by MFC or three-stage process of MFC before and after precipitation could enhance energy production [124, 125]. While most MFC research has been conducted in lab-scale less than 1 L, Sharma et al. (2021) recently scaled up to a working volume of 10 L stacked MFC in a three-staged system which can be integrated to household urinals for onsite nutrient and energy recovery [126].

### **Microbial electrolysis cell (MEC)**

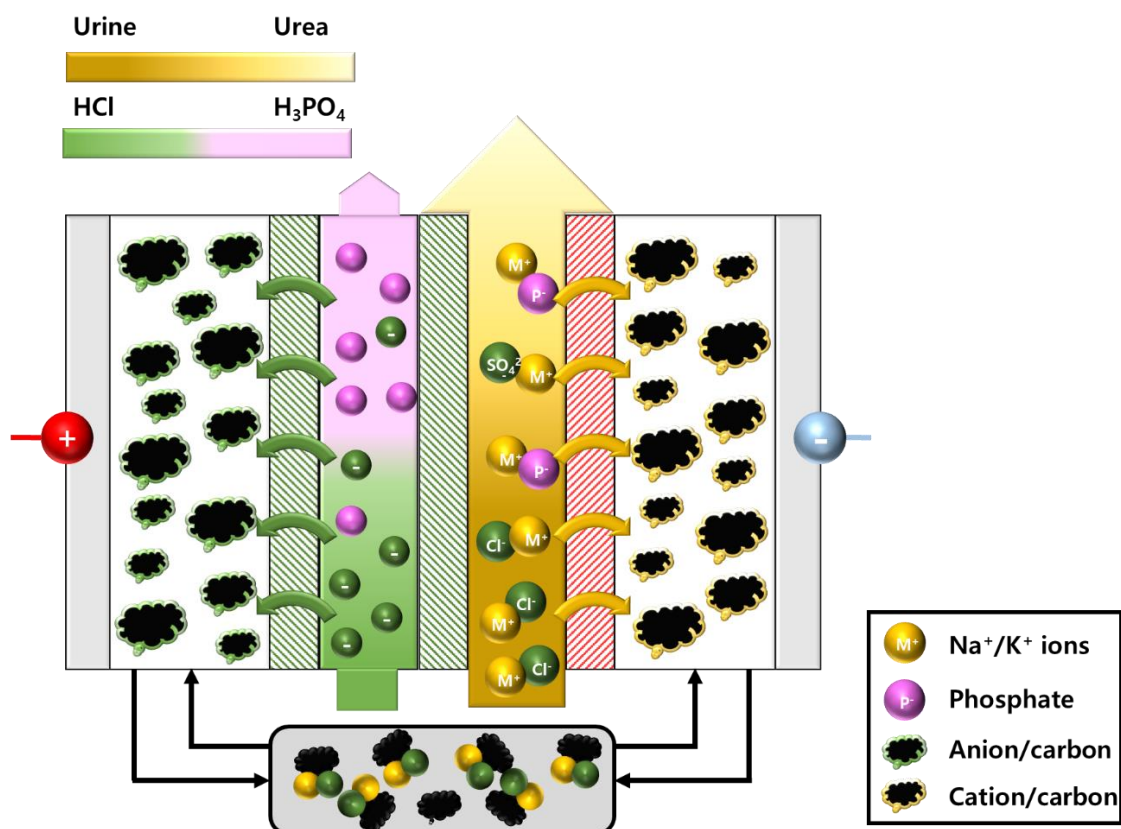
The major difference between MFCs and microbial electrolysis cells (MECs) is the aeration in the cathode, while the other settings are more or less the same. Unlike the cathode compartment in MFC which should be kept in aerobic condition to generate electricity, that in MEC is anaerobic or anoxic condition to produce hydrogen or methane with an additional energy input for electrical current [127]. Overall, the current density in MECs ( $1.7 \pm 0.21 \text{ A/m}^2$ ) was higher than that in MFCs ( $0.5 \text{ A/m}^2$ ) [122, 128]. In addition, the recovery rate of ammonium nitrogen from urine also showed better results than that of MFCs [129, 130]. One of the major challenges for nitrogen recovery in MECs can be a high energy requirement for gas stream supply and recycling for ammonia stripping in the cathode compartment. The introduction of gas-permeable hydrophobic tubular membranes in the cathode could lead to efficient ammonia recovery up to 49%, thereby reducing the energy demand by removing the gas flow for stripping [128]. A scaled-up MEC coupled with gas permeable hollow fibre membranes investigated for the first time by Zamora et al. (2017) [131]. Recent studies combined biological treatment before or after the MEC process in order to enhance nitrogen recovery efficiency as well as avoid nitrogen loss [50, 132].

## **Electrodialysis (ED) and capacitive deionisation (CDI)**

Electrodialysis (ED), which consists of at least a pair of anion exchange membrane (AEM) and cation exchange membrane (CEM), relies on electrophoretic forces to remove ions from an aqueous solution [133]. This process functions as an electrosorbent without requiring any additional pressure or chemical. Although ED requires an external electrical energy, the energy demand is around 14 Wh/L·urine, which is much lower than that of distillation (up to 700 Wh/L·urine). However, ED could result in 8–13% of nitrogen loss and over 30% of N, P, and K could remain in the diluate, while distillation showed a superior concentration capability with nearly no P and K loss [134]. In addition, the concentration factor could be limited by osmosis and electro-osmosis affecting the water transport across the membrane. Notwithstanding, ED was reported as effective in the removal of micropollutants such as diclofenac, carbamazepine, and ibuprofen via adsorption on the membrane, though the removal efficiency varied depending on the compounds and operation times [135, 136]. Reverse electrodialysis (RED) and selective electrodialysis membrane bioreactor (EDMBR) were recently developed in order to efficiently recover energy from the ED process as well as nutrients [137]. RED process with urine demonstrated the feasibility of a household or building-scale energy recovery system along with partial urine sanitisation before its discharge into WWTP [138].

Compared to ED, capacitive deionisation (CDI) has a high adsorption capacity for salt separation as well as a high water recovery rate and can be more stable during long-term operations. Advantages of the application of this technology include efficient desalination capability for saline water without chemical addition as well as low operation cost and energy requirements [139-141]. Although the water recovery rate of conventional CDI has been

reported to be 50%, it can be further increased up to 85-95% when applied with membrane or flow-electrode [142]. Since the ion selectivity and degree of ion electrosorption capacity principally depend on the hydrated size, ionic charge, ion concentration, and degree of ion complexation [143], more than half of the research activities in ED and CDI processes are focused on the development of novel electrode to increase ion sorption/desorption performance and reduce energy consumption [144, 145]. Flow-electrode capacitive deionisation (FCDI) has emerged as a novel CDI technology facilitating continuous deionisation with high desalting rates [146]. However, its low selectivity limits the selective separation of nutrients from undesired ions such as  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$ . The introduction of a pair of AEMs with a liquid membrane chamber (LMC) configuration could selectively recover phosphate ions by extracting them into an acidic solution, leading to uncharged  $\text{H}_3\text{PO}_4$  trapped in an anode chamber in separation from  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$  ions [147, 148]. Figure 5 shows a schematic diagram of FCDI system with LMC configuration.



**Figure 5.** Schematic diagram of flow-electrode capacitive deionisation (FCDI) with a liquid membrane chamber (LMC) configuration.

## 4.2 Nutrient recovery as urine-derived fertiliser

Nutrient from the stabilised and sanitised urine can be further treated for the removal of micropollutants before recovering nutrients to use as a safe and suitable urine-derived fertiliser either as a liquid fertiliser or solid fertiliser. While the stabilised urine can be directly used as a liquid fertiliser however, their transportation cost may become significant if it has to be used far from the place of generation. In such a case, dehydration of stabilised urine must be performed to reduce its volume to produce highly concentrated liquid fertiliser or even solid fertiliser.

#### 4.2.1 Evaporation and distillation

As urine consists of approximately 95% of water and 5% dissolved solids such as urea, uric acid, and other dissolved ions, it is necessary to reduce the urine volume to increase the nutrient density and reduce transportation cost. Evaporation has been suggested for the application to an on-site volume reduction system in urine-diverting toilets at a household or building level [149, 150]. However, ammonia volatilisation and chemical urea hydrolysis during these processes, which are highly related to the temperature and operation duration, are the major concern, as it can lead to nitrogen losses. In fact, the high temperature during evaporation above 40 °C promoted chemical urea hydrolysis and caused 5–30% nitrogen loss during the operation up to 60 °C [9]. Ammonia volatilisation can be minimised by stabilising urea by acidification, alkalisation which mainly inhibits enzymatic urea hydrolysis by urease and nitrification. A recent study showed that the combination of struvite precipitation and alkaline dehydration using magnesium based drying media could capture 99% of total nitrogen including free ammonia. Alkalisation using  $\text{MgCl}_2$  and  $\text{Mg}(\text{OH})_2$  promoted struvite formation facilitating the recovery of all the free ammonia excreted from urine, and then dehydrated under 40 °C producing solid fertiliser containing 11% N, 2% P, and 3% K [76].

Compared to evaporation, distillation could lower the nitrogen loss below 3%, at higher operation temperature when the urine is nitrified [51]. Pilot-scale distillation with nitrified urine could produce 8.7% nitrogen concentrated liquid fertiliser at 165 °C while removing 97% of water and 50% of sodium chloride [151]. One of the biggest advantages of the distillation process is the comparatively low energy consumption of the distiller while using vapour compression or heat exchange, which can recover approximately 85–90% of energy consumed. It was discovered that small-scale vapour compression distillers could reduce energy demand from 710 Wh/L to 110 Wh/L for nitrified urine [49]. It was also reported that a high nitrification

rate (640 mg N/L/d) demanded 80% lower electrical energy (11 Wh/g N) than that of low nitrification rates of urine (120 mg N/L/d) [51]. Thus, although evaporation can be applied to an on-site bathroom scale, nitrification followed by distillation with energy recovery may be more feasible for pilot-scale fertiliser production in terms of low energy consumption and nitrogen loss.

#### **4.2.2 Freeze-thawing**

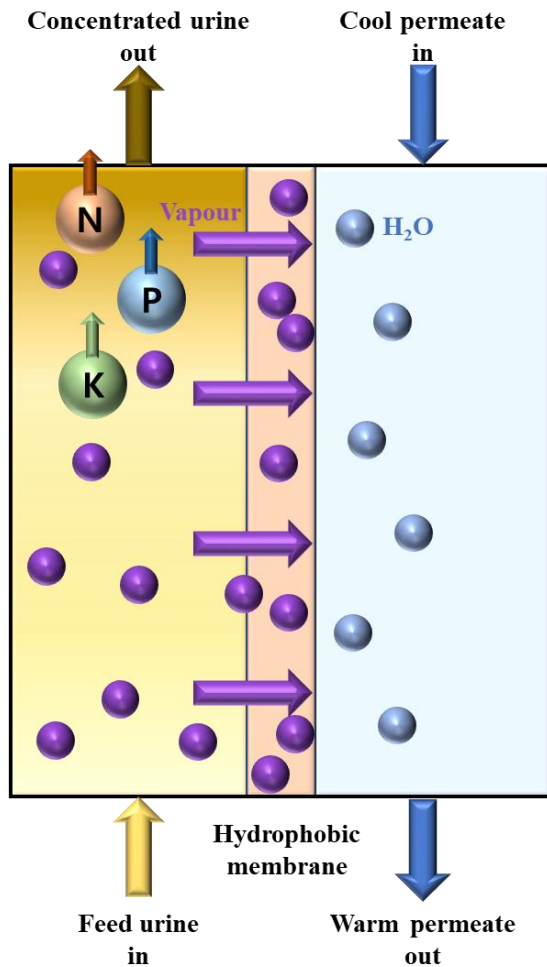
Freeze-thawing basically uses the formation of ice crystals which are free from ions in the urine and can subsequently obtain concentrated nutrient solutions. The vapour pressure of urine is generally lower than that of pure water due to the presence of salts, and similarly the freezing point of urine is also lower than that of pure water. Due to this difference of freezing points, a concentrated nutrient solution can be effectively separated from ice crystals. Thus, this simple method can be effective for urine storage and its transportation as well as energy-effective in cold regions. For the freeze-thawing method, bath type and column type were used in a previous study [152]. These two simple methods froze the urine at -14 °C, which could concentrate nearly 80% of nutrients in 25% of the original urine volume [153]. When the freeze-thawing method was integrated with precipitation by MgO addition, 60% nutrient initially concentrated by freezing in 40% of the original volume, while the nitrogen recovery was enhanced and more than 95% of phosphorus recovered as struvite [154]. Freeze-thawing approach is suitable in a cold country where natural cold outside temperature can be utilised for separation of water from the urine.

### **4.2.3 Membrane treatment process**

#### **Membrane distillation**

Membrane distillation (MD) is an emerging alternative membrane-based technology driven by vapour pressure to separate volatile substances based on their volatilities. Only vapour molecules move across a hydrophobic membrane pores from hot temperature side to low temperature side. Compared to pressure driven membrane processes, MD has potential advantage to be operated at low temperature (30-80 °C) and pressure, thereby reducing energy use and cost. However, the high ammonia concentration of hydrolysed urine at high pH level as well as high temperature can affect more ammonia transfer through the porous membrane [155, 156]. Therefore, urine stabilisation, such as acidification or nitrification of hydrolysed urine is necessary before the MD process. MD process can be categorised into direct contact membrane distillation (DCMD) and vacuum membrane distillation (VMD). VMD has lower nitrogen rejection can be employed as a water regeneration system from urine in space station where the vacuum and solar power resources are sufficient. Membrane fouling, membrane wetting and temperature polarisation are some of the challenges of MD process besides the high heat energy consumption. Pre-treatment, such as ultrafiltration or nitrification in the membrane bioreactor, could mitigate the membrane fouling by removing particulate matters and organics, thereby enhances the flux and quality of permeated water [57, 157]. However, with the use of waste heat and solar heating, MD can be highly effective for concentrating urine by MD process. Figure 6 shows the schematic diagram of MD process for concentration of urine feed.



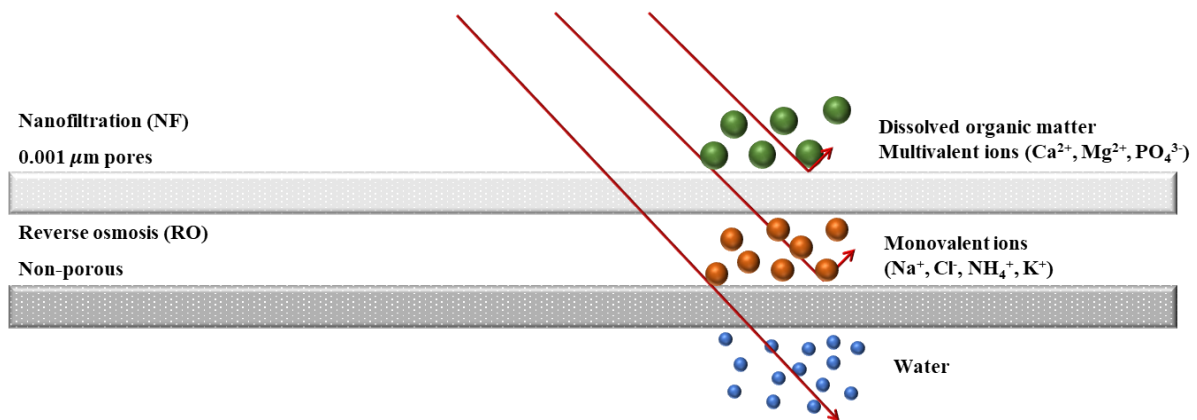


**Figure 6.** Schematic diagram of MD system for concentration of urine feed.

### Reverse osmosis and nanofiltration

Reverse osmosis (RO) and nanofiltration (NF), which are pressure-driven membrane separation technologies, can be effective for concentration of urine although only limited studies are reported recently [158-160]. Figure 7 illustrates the NF and RO processes implicated for the urine. Phosphorus is effectively rejected by RO and NF membranes, while nitrogen rejection is highly pH dependent. Hydrolysed urine has a high pH where majority of nitrogen exists as ammonia ( $\text{NH}_3$ ) rather than ammonium ( $\text{NH}_4^+$ ) and this results in poor nitrogen rejection by the RO or NF membranes. In case of fresh urine, urea rejection is low

(42–57%) by RO and NF [159, 161-163]. However, more recent study by Courtney and Randall (2022), achieved 90.7% and 82.5% of urea rejection by applying seawater RO membrane for real acidified urine and air-bubbled base-stabilised urine, respectively [158, 160]. The application of RO membrane with nitrified urine showed up to 80% of nitrogen rejection [164]. Besides nutrients, RO or NF membranes can also reject other dissolved ions present in the urine alongside the nutrients [165]. NF was found to be effective in retaining 92% of pharmaceuticals, including carbamazepine, diclofenac, propranolol, and ibuprofen, from non-hydrolysed urine at around pH 5 [166]. Membrane fouling and scaling remain as other challenges of the membrane-based technologies.



**Figure 7.** Nanofiltration (NF) and reverse osmosis (RO) processes implicated for the urine.

### Forward osmosis

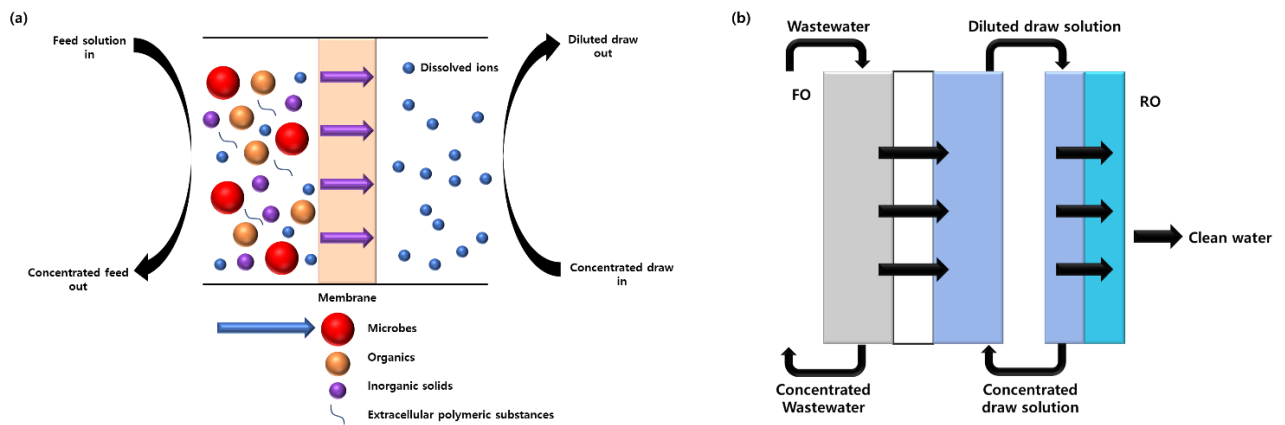
Forward osmosis (FO), an osmotically driven membrane processes can be a promising low-energy technology option for nutrient concentration. However, a cheap, and suitable draw solution which can be easily regenerated must be used to keep the overall energy down. Waste brine (mostly containing NaCl) from seawater or desalination is normally a low-cost draw

solution [167]. A novel vertically arranged up-flow FO reactor and the application of aquaporin membranes were recently introduced for membrane fouling reduction and higher permeate water quality, respectively [168-170]. The high osmotic pressure of hydrolysed urine (up to 2000 kPa) can be used as a driving force for microalgae dewatering, facilitating the use of the diluted urine for microalgae cultivation [162].

In addition, the occurrence of reverse salt flux (RSF) is also one of the main obstacles inhibiting the rejection efficiency [161]. In fact, it was reported that charged ions such as  $\text{NH}_4^+$  and  $\text{K}^+$  showed higher retention rates in RO processes than in FO, owing to the ion exchange mechanism caused by the reverse flux of sodium ions from the draw solution [167]. Many researchers spent efforts to increase the nitrogen rejection in FO processes, while overcoming these drawbacks at the same time. It was found that both the poor rejection of urea from fresh urine and RSF issues can be considered as benefits for simultaneous generation of struvite and N-enriched fertiliser by adapting magnesium-based fertiliser as a draw solution. The result showed that the reverse  $\text{Mg}^{2+}$  flux and high urea permeation facilitated struvite formation in the feed side and urea-rich magnesium fertiliser in the permeate side, respectively [161].

The integration of FO and other membrane technologies have been suggested for compact portable systems, which can produce high quality water as well as concentrated nutrient solutions, in situations where potable water production is not readily available such as a space mission or cave exploration. The FO-MD hybrid system firstly concentrated the source-separated urine for nutrient recovery by the FO process and the diluted draw solution was reconcentrated by the MD process for continuous operation, generating distilled water as well [171]. Conversely, the FO system might be a necessary pre-treatment for MD, because the effective removal of potential foulants in the FO processing step was beneficial to membrane wetting and fouling reduction in MD [172, 173]. RO process as a post-treatment, which could

recover the draw solution and produce reclaimed water, can be integrated to the toilet system and used for parks or sightseeing places where the sewage pipelines are uninstalled [163]. Figure 8 shows the schematic diagram of FO process as well as hybrid FO-RO system. In conclusion, the hybrid system of FO and MD or RO can effectively recover water and produce concentrated fertiliser from urine even in extreme circumstances like space stations, but further research is required regarding scaled-up application and its energy efficiency improvement.



**Figure 8.** Schematic diagram of (a) forward osmosis (FO) and (b) hybrid forward osmosis-reverse osmosis (FO-RO) system

**Table 2.** Remarks and challenges of previous technologies on nutrient recovery from source separated urine.

Treatment type		Major outcomes	Remarks	Challenges	Reference
Physical	Storage	1% N and 30% P of spontaneous precipitation.	<ul style="list-style-type: none"> <li>• Due to the rapid hydrolysis of urea by the urease enzyme, 90% of N exists in ammonia form.</li> <li>• Prolonged storage can reduce pharmaceuticals and pathogens.</li> </ul>	<ul style="list-style-type: none"> <li>• Ammonia volatilisation occurs at the high pH level at 9.2, which causes a strong odour and nitrogen loss.</li> <li>• Storage itself is not enough for urine treatment for fertiliser.</li> </ul>	[11, 40]
	Evaporation /distillation	By alkaline dehydration at 40 °C, 11% N, 2% P, and 3% K as a solid fertiliser. 8.7% N in liquid fertiliser by distillation.	<ul style="list-style-type: none"> <li>• Evaporation can be performed 40°C-60°C while distillation can be performed at higher temperature and low pressure (200-500 mbar).</li> <li>• Urine dryer can be installed in existing or future bathrooms for on-site alkaline dehydration and solid fertiliser production.</li> <li>• Vapor compression or heat exchange distillation facilitates energy recovery, reducing energy consumption by up to 90%.</li> </ul>	<ul style="list-style-type: none"> <li>• Both evaporation and distillation need nitrogen stabilisation (acidification, alkalization, or nitrification) to prevent the nitrogen loss via volatilisation.</li> </ul>	[76, 151]
	Freeze-thawing	60-80% of nutrient concentration in 25-40% of the original volume.	<ul style="list-style-type: none"> <li>• Freezing can avoid odour problems and ammonia loss, which is beneficial for urine management and transportation.</li> <li>• Can be advantageous in cold regions.</li> </ul>	<ul style="list-style-type: none"> <li>• Energy consumption is high to cool down the temperature.</li> </ul>	[153, 154]

<b>Membrane processes</b>	<b>MD</b>	80% water recovery and 95% ammonia and 97% P and K recovery from hydrolysed urine.	<ul style="list-style-type: none"> <li>• Acidification pre-treatment of urine prior to DCMD reduced ammonia transfer and membrane fouling, and the ammonia-concentrated retentate can be used as a N-fertiliser.</li> <li>• VMD could remove 99% COD and up to 75% ammonia from urine, which can be applied for water regeneration from urine in space station where vacuum and solar power is sufficient.</li> </ul>	<ul style="list-style-type: none"> <li>• High feed temperature can reduce ammonia and COD rejection.</li> <li>• Membrane fouling and wetting might be inevitable due to high organic contents and micropollutants in urine, but this can be mitigated by pretreatment like ultrafiltration or nitrification.</li> </ul>	[174, 175]
	<b>RO/NF</b>	90.7% urea recovery from acidified urine by RO. >90% N (hydrolysed urine at pH 6.5), >99% P and K recovery by RO and NF.	<ul style="list-style-type: none"> <li>• RO process could completely reject P, K, and S, while the rejection of N highly depends on the pH.</li> <li>• NF could selectively separate <math>\text{NH}_3</math> from urine while producing a micropollutant-free nutrient solution.</li> <li>• Monovalent mineral salts such as NaCl and KCl, which is essential substance in space, can be recovered from urine by NF.</li> </ul>	<ul style="list-style-type: none"> <li>• Direct application of urine to NF and RO can cause severe membrane fouling and scaling, thus pretreatment is required.</li> </ul>	[158, 160]
	<b>FO</b>	80% $\text{NH}_4^+$ , >90% P and K recovery from hydrolysed urine.	<ul style="list-style-type: none"> <li>• Seawater or desalination brine can be applied as a low-cost draw solution in FO, producing biofertilizer, while urine itself can be a draw solution for microalgae dewatering.</li> <li>• By using magnesium contained draw solution in FO, spontaneous recovery of N</li> </ul>	<ul style="list-style-type: none"> <li>• <math>\text{NH}_4^+</math> and <math>\text{K}^+</math> showed lower retention rate than RO process.</li> <li>• Reverse salt flux can inhibit the rejection efficiency.</li> </ul>	[167]

Biological			<p>and P, and subsequent production of N-fertiliser and struvite is possible.</p> <ul style="list-style-type: none"> <li>• Simultaneous production of liquid fertiliser and reclaimed water is feasible by a light-weight portable FO system for parks, sightseeing places, or temporary circumstances such as cave exploration.</li> </ul>		
	<b>Nitrification</b>	<p>Nitrification rate of 1.8 g N/m<sup>2</sup>·d in MABR, 930 mg N/L·d in MBBR, 447 ± 50 mg N/L·d in MBR.</p>	<ul style="list-style-type: none"> <li>• Nitrification stabilises urine by biological oxidation of ammonia to nitrate by nitrifying bacteria.</li> <li>• Nitrification in MBBR or MBR can be a good pretreatment for distillation and MD for concentration.</li> <li>• Urine nitrification in MBR, MABR or SBR can be feasible for the nutrient use in space.</li> <li>• It can recover all the nutrients in one final product as a liquid fertilizer.</li> </ul>	<ul style="list-style-type: none"> <li>• The start-up time and hydraulic retention time of bioreactor can be long.</li> <li>• High concentration of nitrite, FA, and FNA are crucial factors for AOB and NOB inhibition.</li> </ul>	[49, 51, 58]
	<b>Nutrient uptake by microalgae</b>	<p>52% N and 38% P of uptake rate in raceway pond. 84.1% N and 96.6% P of uptake rate in photobioreactor from fresh urine.</p>	<ul style="list-style-type: none"> <li>• Urine can be an efficient growth medium for microalgae.</li> <li>• Microalgae can be cultivated by nutrients from urine, resulting more than 90% of both N and P recovered from urine.</li> <li>• Addition of trace elements or nitrification pre-treatment could enhance the growth rate.</li> </ul>	<ul style="list-style-type: none"> <li>• Direct use of undiluted urine is not suitable for microalgae growth.</li> </ul>	[62, 64]
<b>Chemical/ Electro-chemical</b>	<b>Acidification</b>	<p>Acidification below pH 4 retained 99.5% N</p>	<ul style="list-style-type: none"> <li>• Acidification used for the stabilisation of urine to prevent nitrogen loss.</li> <li>• It can reduce odour by preventing the formation of NH<sub>3</sub>, as well as organic content of urine.</li> </ul>	<ul style="list-style-type: none"> <li>• Economically disadvantageous due to the chemical addition.</li> </ul>	[43, 70]

		<ul style="list-style-type: none"> <li>• Can be beneficial for the inactivation of pathogens and pharmaceuticals like antibiotics under pH 4.</li> <li>• In case of waterless urinals in a building, acidification can effectively prevent pipe blockage by redissolution of phosphorus precipitates.</li> </ul>		
<b>Alkalisisation</b>	Alkalisisation with wood ash/lime retained 74–90% of urea. 11.23 g/kg urine of solid fertiliser recovered with 10 g/L of $\text{Ca}(\text{OH})_2$ .	<ul style="list-style-type: none"> <li>• Alkalisisation (pH &gt; 10) can be another way to stabilize urine by urease inhibition.</li> <li>• Calcium, magnesium, potassium hydroxides, wood ash, biochar, and anion change resin can be used.</li> <li>• Phosphorous precipitation at high pH can produce a P-fertiliser separately.</li> </ul>	<ul style="list-style-type: none"> <li>• Economically disadvantageous due to the chemical addition.</li> <li>• The pH increase may cause the <math>\text{NH}_3</math> volatilisation.</li> </ul>	[74, 150, 176]
<b>Precipitation</b>	Max. 99% of P and 70.5% of K recovery.	<ul style="list-style-type: none"> <li>• For struvite precipitation, various magnesium sources such as <math>\text{MgO}</math>, <math>\text{MgSO}_4</math>, and <math>\text{MgCl}_2</math> can be added.</li> <li>• Magnesium ammonium phosphate (MAP) and magnesium potassium phosphate (MPP) are good slow-release fertilisers for plant.</li> </ul>	<ul style="list-style-type: none"> <li>• Further process is required for higher nitrogen recovery, such as ammonia stripping.</li> <li>• Calcium salt addition is not suitable for hydrolysed urine due to the calcium carbonate precipitation.</li> </ul>	[89-91, 177]
<b>Adsorption</b>	Max. 87.5% of $\text{NH}_4^+$ and 97% of $\text{PO}_4^{3-}$ recovery.	<ul style="list-style-type: none"> <li>• In terms of ammonium adsorption, resins showed the highest efficiency compared to zeolite and biochar.</li> <li>• Biochar can be made from waste biomass, which makes it environmentally friendly and cost effective.</li> </ul>	<ul style="list-style-type: none"> <li>• Depending on the type of adsorbent, only one nutrient can be adsorbed.</li> </ul>	[114, 178, 179]



		<ul style="list-style-type: none"> <li>• Nutrient saturated biochar can be directly applied as a slow-release fertiliser and soil conditioner.</li> <li>• Activated carbon hardly adsorbed nutrients but pharmaceuticals.</li> </ul>	
<b>Stripping</b>	Max. 99% of N recovery via ammonium sulphate.	<ul style="list-style-type: none"> <li>• High pH (10-12) and high temperature (35-40 °C) are favourable for ammonia stripping due to the volatile ammonia.</li> <li>• Sulfuric acid is typical absorption medium, by forming ammonium sulphate.</li> <li>• Vacuum thermal stripping allows lower boiling point at high absolute pressure.</li> <li>• Membrane stripping can reduce energy demand as they use less heat and enhance the N recovery rate.</li> </ul>	<ul style="list-style-type: none"> <li>• Economically disadvantageous due to the alkalisation and usage of acid solution.</li> <li>• Separate recovery of nitrogen and phosphorous.</li> </ul> <p>[95]</p>
<b>MFC</b>	31% NH <sub>3</sub> recovery by MFC followed by stripping Max. 94.6% PO <sub>4</sub> <sup>3-</sup> -P and 46% NH <sub>4</sub> <sup>+</sup> -N recovered by hybrid precipitation and MFC	<ul style="list-style-type: none"> <li>• Urine can be a good fuel source for MFC due to its high conductivity and buffering capacity.</li> <li>• Precipitation as pre- and post-treatments of MFC could increase P recovery rate and energy production.</li> <li>• Stacked MFC in a three-staged system can be integrated to household urinals for onsite nutrient and energy recovery.</li> </ul>	<ul style="list-style-type: none"> <li>• Due to the high pH in cathode chamber, ammonia stripping process is required to recover transferred ammonium.</li> <li>• Struvite precipitation on the cathode surface is problematic, leading to the reduction of energy production.</li> </ul> <p>[122, 124, 126]</p>
<b>MEC</b>	49.5% N, 42.8% P, and 54.7% K recovery by hybrid electrolysis/MEC	<ul style="list-style-type: none"> <li>• MECs showed overall higher current density and NH<sub>4</sub><sup>+</sup> recovery rate than MFCs.</li> <li>• MEC with ion exchange membranes concentrated nutrient in diluted urine, with</li> </ul>	<ul style="list-style-type: none"> <li>• The gas stream supply and recycling for ammonia stripping in the cathode</li> </ul> <p>[180]</p>

		<p>4.5 and 3.0 of concentration factor for N and P, respectively.</p> <ul style="list-style-type: none"> <li>• Anaerobic digestion as a pre-treatment or nitrification as a post-treatment increased TAN recovery and reduced nitrogen loss.</li> </ul>	compartment requires high energy.	
<b>ED/CDI</b>	Max. 70% of N, 67% of P, 83% of K recovery from five-fold diluted urine	<ul style="list-style-type: none"> <li>• ED was reported as effective in the removal of micropollutants.</li> <li>• Pre-treatment of precipitation and nitrification prior to ED process reduced scaling and biofouling.</li> <li>• Compact RED system can be applied in a household or building scale energy recovery with partial urine sanitization.</li> <li>• Flow-electrode capacitive deionisation (FCDI) can selectively recover phosphate from chloride-rich urine without any addition of chemicals.</li> </ul>	<ul style="list-style-type: none"> <li>• ED and CDI showed relatively high nitrogen loss and low nutrient recovery rate compared to other processes.</li> </ul>	[134]

## **5. NiCE: ARC industrial research Hub**

### **5.1 Background**

The NiCE hub is a four-year national program (2022-2026) of cross-disciplinary research to transform Australia's wastewater industry from waste removal to resource recovery and from centralised to decentralised, at urban precinct scale. Funded by the Australian Research Council and the consortium of industries, the project is aimed at transformation by providing the wastewater industry with capability to compete with conventional manufacture or extraction processes for nutrients (nitrogen, phosphorous, micronutrients). Competitiveness will derive from new technology, new products and markets, and newly developed regulatory regimes. The transformation will underpin a circular nutrient economy.

### **5.2 Research themes**

The NiCE research Hub will take a holistic approach to tackling the challenge of urban, decentralised processing of urine to fertilisers, tailored for Australian conditions by engaging all end-users across the system to deliver a roadmap to precinct-scale implementation of urine diversion, enabling and launching a viable circular nutrient economy that is scalable to a large-size city. Its vision is for Australia to have a resilient wastewater industry, resilient agriculture and horticultural sectors, a healthy environment, and a strong manufacturing industry for advanced membrane, biotech and sensor technologies—all underpinned by a circular economy of nutrients. NiCE industry Hub is built on four broad research themes:

#### **Theme 1: Urine collection**

Theme 1 is to investigate the collection of urine at the source, and the effects that doing so has on the management, operation and planning of wastewater conveyance (sewers) and treatment systems. This theme addresses the need to understand drivers that will increase use of waterless urinals and urine-diverting toilets by members of the public and the characteristics of a technical, commercial and regulatory environment conducive to installation of urine collecting devices by plumbers, builders and developers in green-field builds and retrofits. Importantly, it will also quantitate the effects of decentralised urine collection on wastewater infrastructure.

### **Theme 2: Urine processing to liquid fertiliser**

Urval and Ugold have been demonstrated by chief investigators (CIs) Shon & Freguia at small scale with real urine in the field, making their technology readiness level (TRL) 5-6. Both need further substantial testing and validation under field conditions and at a larger scale to progress their development. That will be done in the activity under Theme 2. To avoid the risk that the circular economy of nutrients will be constrained by the limited number of technology options, new options need also to be explored.

### **Theme 3: Fertiliser optimisation**

Liquid fertilisers may require specific N:P:K ratios, or certain concentrations of trace elements or organics, to meet specific plant/crop and soil requirements and be able to maximise nutrient use efficiency. We will develop protocols to establish fertiliser formulations so that these requirements can be met by optimising nutrient ratios. We will establish what nutrient ratios/concentrations are required through activity in Theme 4. From a human health risk perspective, human enteric pathogens (viruses, bacteria) and chemical hazards (macro- and micro-pollutants) likely to be present in the collected, stored and processed urine only pose a risk to human health if people are exposed. If adequately treated and appropriate risk management strategies are implemented at critical control points, environmental pollution and

public health issues resulting from the use of Ugold and Urval can be minimised. We need to know what those critical control points are (Theme 3).

#### **Theme 4: Fertiliser end-use**

We need to know the behaviour of Urval, Ugold and their derivatives in the soil-plant-atmosphere continuum and integrate this with existing knowledge regarding interactions among soil physicochemistry and hydrology. This synthesis will be used to assess conditions that provide maximum benefits while also minimising negative impacts. We also need to know how urine-based fertilisers compare to conventional fertilisers for their ability to promote healthy plants and produce other desired outcomes, including carbon farming and reduced environmental losses. We will directly study the uptake and mobility of nutrients in different soil and management systems and in the presence of different plants. Effects of urine-derived fertilisers on soil salinity and pH, solute movement in soil, nutrient availability and residual effects, and biological fertility will be studied systematically over years of application, to enable long-term assessment and recommendations on their use. Different methods of fertiliser application will be assessed depending on the specific crop type and soil management practices.

### **5.3 Research consortium: collaborations and opportunities**

The NiCE Hub is a consortium of seven Australian Universities (University of Technology Sydney, University of Melbourne, Victoria University, Western Sydney University, Griffith University, University of Southern Queensland, University of Queensland) and 18 industries involving CIs with multi-discipline skills including engineering, science, technology, agriculture, health, policy, social science and economics with stakeholders across every component of this new value chain (urine-fertiliser-plant growth).

## **5.4 Expected research outcomes and benefits**

The NiCE Hub is specifically designed to drive competitiveness for the wastewater industry sector in recovering nutrients from urine in distributed systems (recovery at an urban building, rather than a centralised sewage treatment plant) and recycling them as a concentrated liquid fertiliser for peri-urban agriculture and city parks. In turn this will help build resilience for the wastewater industry in Australia in the face of climate change, aging infrastructure, increasing urban populations and higher expectations for effluent water quality. Secondly, it will satisfy emerging policy imperatives for implementation of a circular bio-economy. Circular economies are increasingly being sought across the globe by industry, government and societal groups [181].

NiCE is also designed to grow Australia's fertiliser industry and manufacturing industries by demonstrating the technical capability, market demand and economic viability for new products—new 'home-grown' fertilisers tailored to Australian soil and non-soil crops, and new components of distributed wastewater systems. Australia imports majority of the fertiliser it uses and hence the fertilisers market size in Australia is quite large with domestic market value of approximately \$1.5B [182]. The liquid fertiliser market is growing at a significant rate due to the attractive properties of liquid fertilisers over conventional solid fertilisers in targeted irrigation applications (e.g. foliar and aerial), and its amenability with herbicides and pesticides.

NiCE will take a circular approach to achieving the project aims. Each of four central themes (Urine collection, Urine processing, Fertiliser optimisation, and Fertiliser end-use) will directly interact with the themes immediately upstream and downstream of it. Each of these central themes reflects a portion of the circular economy and is represented by a set of Partner

Organisations who are stakeholders in the circular nutrient economy. Two overarching themes—whole-of-system economics; engagement, learning and communication—will interact simultaneously with the central four themes.

NiCE hub is currently engaged in lab-scale and pilot-scale demonstrations of source-separated urine treatment technologies, including assessment of environmental impacts through simulation studies. These demonstrations aim to evaluate the effects of urine diversion in decentralized wastewater treatment plants, providing valuable insights into the potential benefits and challenges of implementing this technology at scale [183, 184]. Furthermore, the ongoing research project conducted by NiCE Hub focuses on utilizing the produced fertiliser from urine diversion for crops, plants, and horticulture, which holds significant economic value. This hub involves evaluating the potential of resource recovery from urine through life cycle assessment (LCA), with the aim of identifying the environmental impact and potential benefits of this approach. The findings of this continuous research project are expected to contribute to the development of sustainable and environmentally-friendly solutions for urine management, with the potential for widespread adoption in the future.

## **6. Summary and future perspective**

The wastewater industry has come a long way in its effort to make the water and nutrient circular. Earlier efforts have been focussed on the recovery of nutrients from the wastewater where it is present in much diluted form however, nutrient recovery from urine is seen as one of the most effective approach for nutrient recovery since 80% N and 55% P in the wastewater actually originate from urine. This review paper provides an overview of technologies for urine stabilisation, sanitation, and nutrient recovery, categorized into physical, biological, and

electro-chemical treatments, with a focus on concentrated urine as a potential fertiliser. Despite the considerable research efforts dedicated to urine stabilization and nutrient recovery, a single technology that can efficiently recover nutrients, energy, and water from human urine remains elusive. Integration of appropriate technologies, such as nitrification and membrane distillation (MD), or forward osmosis (FO) and MD, may offer more promising solutions. However, it should be noted that the state of knowledge and technology readiness varies across different treatment methods, ranging from lab-scale to pilot-scale demonstrations. Additionally, studies conducted under real urine conditions and addressing pharmaceutical residues removal are still lacking. Hence, further research is needed to overcome the limitations of existing technologies and facilitate their implementation at higher scales, thereby increasing the technology readiness level. Moreover, considering that volume reduction processes such as distillation tend to be energy-intensive, efforts should be directed towards reducing energy consumption, which should be a specific focus of future research endeavours.

While the source separation of urine and its treatment become essential, in rural environments, urine can be stabilised using simple and low-cost methods such as onsite storage for aging and pathogen removals, then apply directly to fields as a fertiliser, which do not require extensive infrastructures. Additionally, households in rural areas with on-site processing and storage systems, such as urine-diversion toilets with evaporation, can extract valuable nutrients from urine and be collected for transportation to facilities such as farms or factories. On the other hand, in urban areas, they involve additional and designated plumbing infrastructure for urine collection and storage which can be significant challenge for the existing buildings. However, for new or renovated buildings, a separate network of pipes in the basement can be installed to process and concentrate urine for easy collection, avoiding extensive modifications to existing urban infrastructure. Urine treatment and processing to convert urine into more concentrated urine-derived fertiliser is an ongoing research focussed at commercialisation opportunities. The



ARC NiCE Hub brings together researchers from multi-disciplinary background from Australia and overseas to work towards achieving nutrient in a circular economy.

### **Declaration of competing interest**

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

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