

# Closed-loop air recirculation architecture for alkaline urine dehydration and water recovery using regenerable superabsorbent polymers

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## HIGHLIGHTS

- Closed-loop air recirculation system for low-temperature urine dehydration
- Regenerable SAP-based desiccants enable cyclic air-vapour moisture absorption.
- Eliminates CO<sub>2</sub>-driven acidification and nitrogen loss without excess Ca(OH)<sub>2</sub>
- >99 % retention of major endogenous organics confirmed by targeted metabolomics
- Produces a dry fertiliser and clean water suitable for various reuse applications

## ARTICLE INFO

### Keywords:

Water reuse  
Safe nutrient recycling  
Decentralised sanitation  
Source separation  
Wastewater treatment  
Fertiliser production

## ABSTRACT

We evaluated a novel closed-loop evaporative system designed to concentrate alkalinised human urine while simultaneously recovering water using regenerable superabsorbent polymers (SAPs). This architecture recirculates air and physically isolates urine from atmospheric CO<sub>2</sub>, thereby maintaining high alkalinity and preventing enzymatic urea hydrolysis. The system was operated at ~30 °C using sodium polyacrylate, potassium polyacrylate, or a 1:1 (w/w) blend of both SAPs across eight absorption–desorption cycles. All treatments exhibited high initial water uptake (>1.4 kg m<sup>-2</sup> day<sup>-1</sup>) and gravimetric absorption (>0.8 g g<sup>-1</sup>), with performance declining due to polymer fatigue after repeated use. FT-IR spectra revealed the depolymerisation of the acrylate backbone leading to the formation of acrylic acid residues, confirming chemical deterioration during the thermal regeneration of the SAPs. Notably, the closed-loop design eliminated the need for supersaturating urine with Ca(OH)<sub>2</sub>, which is required in open evaporative systems to buffer against CO<sub>2</sub>-induced acidification. Colorimetric and targeted metabolomic analyses confirmed complete nitrogen retention and > 99 % recovery of the 30 most abundant endogenous organic solutes in urine, including urea, creatinine, and hippuric acid. These results demonstrate that low-temperature evaporation can preserve the full biochemical complexity of urine, producing a dry, sanitised fertiliser as well as water with extremely low organic content.

## 1. Introduction

Human urine constitutes 1 % of total domestic wastewater by volume but contains approximately 80 % of nitrogen, and 50 % of both phosphorus and potassium [31]. Urine separation at source and subsequent recycling presents an opportunity to close nutrient loops, reduce dependence on synthetic fertilisers, and enhance food system sustainability [1]. Given that fertiliser production accounts for 2–3 % of global energy use and 1–2 % of global CO<sub>2</sub> emissions [33], environmental arguments in favor of urine recycling are compelling [2]. However, urine's

high water content, ~95 % [3], remains a major barrier to its large-scale recycling, as it makes storage, transport and fertiliser application inefficient. As the field of source-separated sanitation matures, its next frontier lies in developing methods for concentrating urine, particularly at a larger scale in decentralised and semi-centralised settings [4], where urine from multiple toilets is conveyed to a shared treatment unit (e.g., in the basement of a multi-storey residential or institutional building) [5]. Superabsorbent polymers (SAPs) offer a novel alternative route to address this challenge. These crosslinked hydrogels, composed of three-dimensional networks of hydrophilic functional groups, absorb several

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hundreds of times their weight in water without dissolving or losing structural integrity [8]. The absorbed water can be released through mild heating, allowing SAPs to undergo multiple stages of dehydration and rehydration [6]. Regeneration performance varies across SAP chemistries, with some SAPs maintaining performance for more than ten absorption-desorption cycles [7], whereas SAPs such as sodium polyacrylate show a reduction in absorption after five cycles [8]. While SAPs are widely used in agriculture and hygiene products [34], their potential as functional materials in sanitation systems remains largely underexplored.

Here, we propose to use SAPs as passive, regenerable desiccants within a sealed urine dehydration system that recirculates air in a closed loop. In this architecture, humid air generated during urine evaporation passes over SAPs, which absorb the moisture. The resulting dry air is then recirculated back over the urine, sustaining a humidity gradient while physically excluding the ingress of ambient  $\text{CO}_2$ . This “closed-air-loop” configuration enables phase-separated dehydration, wherein nutrients are retained in the urine concentrate, while evaporated moisture is captured within the SAP matrix. Upon SAP regeneration, the absorbed moisture can be recovered for reuse.

A key design principle of our proposed system is to preserve the nutrient content of urine throughout the drying process. Urea, which accounts for the majority of nitrogen in freshly excreted urine, is thermodynamically unstable and hydrolysed to ammonia by the enzyme urease [9]. Urease is produced by bacteria commonly present in human microbiota, urine collection pipes, storage tanks and the ambient environment, and its presence significantly increases the risk for nitrogen loss through ammonia volatilisation [10]. To prevent urea degradation, fresh urine can be stabilised by adding sparingly soluble alkaline earth hydroxides, such as calcium and magnesium hydroxide, which raise the pH above 10. At such pH, urease is reversibly deactivated [11], and pathogenic organisms are also inactivated [12]. Dehydrating alkalised urine at temperatures below 40 °C results in complete retention of plant-essential macronutrients in the concentrated solid fertiliser produced by the process [13]. However, exposure of alkaline urine to air during drying introduces  $\text{CO}_2$  in urine, forming carbonic acid and lowering pH [13]. This acidification reactivates urease and increases the risk of ammonia volatilisation [10], a vulnerability clearly observed in field-scale alkaline urine dehydrators. In a pilot trial in Finland, nitrogen recovery fell to ~30 % when open-air forced convection caused air temperatures to exceed 60 °C, leading to base-catalysed thermal urea degradation [14]. Similarly, long-term field tests with ambient-air drying achieved only ~10 % nitrogen recovery because sustained  $\text{CO}_2$  dissolution reduced urine pH from 12.5 to 8, thereby enabling biological urea hydrolysis [15]. Our proposed system addresses these vulnerabilities by physically isolating urine from ambient air, thus maintaining alkalinity throughout the dehydration process, and thus, preserving nutrient alongwith other urine nutrients.

In this study, we evaluate the performance of the proposed closed-air-loop urine evaporator, focusing explicitly on its chemical, physical and functional aspects. We do not assess the fate of macronutrients as our prior studies have already demonstrated complete retention of nitrogen and other plant-essential nutrients in urine during alkaline drying below 40 °C [13,16]. Here, we specifically test the closed-loop architecture’s ability to maintain a stable urine pH (>10) throughout the drying process, including during repeated polymer reuse cycles. We characterise the moisture absorption and water recovery efficiencies of two superabsorbent polymers, potassium and sodium polyacrylate, across multiple absorption-desorption cycles. Finally, we examine the recovered water by quantifying the fate of >200 endogenous organics present in urine, both before and after treatment with activated carbon filtration. Our findings provide new insights into the feasibility of integrated nutrient and water recovery from human urine using a regenerable, low-energy evaporation system tailored for decentralised sanitation contexts.

## 2. Methods

### 2.1. Materials

Fresh human urine was collected from anonymous adult donors, both male and female, aged between 18 and 65, using 500 mL polypropylene flasks. All samples were pooled, homogenized, and stabilised by adding calcium hydroxide at 10 g L<sup>-1</sup>, a dosage previously shown to effectively prevent enzymatic urea hydrolysis [17]. The mixture was stirred for 30 min at 300 rpm using an overhead stirrer (OHS 60, Velp Scientific, Italy) and left to settle overnight to remove suspended solids and undissolved  $\text{Ca}(\text{OH})_2$  by sedimentation.

Two commercially available superabsorbent polymers with different counter-ion affinities, sodium polyacrylate and potassium polyacrylate (Alquera Ciencia SL, Spain), were used as regenerable desiccants in the study. Potassium polyacrylate typically exhibits higher swelling capacity and absorption in ion-rich media such as urine due to the weaker interaction of K<sup>+</sup> with carboxylate groups, whereas sodium polyacrylate forms stronger ionic bridges that can suppress swelling [18]. No pre-treatment or additives were applied to modify their properties. The dehydration system consisted of three airtight glass containers connected by silicone tubing (Ø 12 mm) forming a closed-loop airflow circuit. Container 1 (cylindrical, Ø 12.5 cm, 1.75 L volume) served as the urine evaporation chamber, while containers 2 and 3 (rectangular, 21 × 15 × 7 cm, 1 L volume) housed the SAPs for dehumidifying the moist air. A total of 100 g SAP was used per cycle, distributed equally between the two absorption chambers. To ensure airtightness, petroleum jelly was applied to all the rubber seals, and each lid was fitted with air inlet and outlet ports using O-ring sealed connectors. A 200 W aquarium pump (nominal air flow rate 6.6 m<sup>3</sup> h<sup>-1</sup>) was used in both configurations. In the closed-loop configuration, the pump recirculated air through all the containers in series, with air leaving the SAP chambers (containers 2 and 3) routed back to the pump inlet and then to the urine evaporation chamber (container 1), forming a fully sealed loop that prevented contact with ambient air. An air pressure drop test was done to confirm the airtightness of the sealed setup. In the linear (open) configuration, the outlet of container 3 was vented to the room, and the pump inlet drew in fresh ambient air. The same pump therefore provided a comparable volumetric airflow, but in a once-through mode, allowing continuous ingress of atmospheric  $\text{CO}_2$ .

### 2.2. Experimental procedure

Three SAP treatments were tested across trials: sodium polyacrylate, potassium polyacrylate, and a 1:1 (w/w) mixture of the two. In all cases, the polymers were evenly distributed between the two moisture absorption chambers.

Two sets of experiments were conducted. The first aimed to compare the performance of the circular, closed-air-loop configuration versus a linear, open-air configuration to maintain alkaline pH during progressive urine dehydration. In both systems, urine was dried at ~30 °C to mass concentration factors (CFs) of 1.25, 2, 2.5 and 5, corresponding to reductions to 75 %, 50 %, 40 % and 20 % of the original mass, respectively. At each CF, the pH of the urine in container 1 was measured.

The second experiment aimed to evaluate the moisture absorption and water recovery performance of the SAPs. In each trial, 50 g of alkaline urine was loaded into container 1 and dehydrated until a constant weight was achieved, typically corresponding to the removal of ~46 g (± 2 %) of the original urine mass. A total of 100 g of SAP was used per cycle (50 g per absorption chamber). After each drying cycle, the water-laden SAPs were removed and desorbed using a rotary evaporator (Büchi R-150, Germany) and the mass of recovered water was recorded. The recovered water was then divided into two aliquots. One aliquot was analysed directly, while the other was treated with activated carbon (0.1 g L<sup>-1</sup>, Merck KGaA, Germany) for 30 min (by mixing over a magnetic stirrer), followed by filtration through a 0.45 µm syringe filter

(Filtropur S, Sarstedt, Germany). Both aliquots were stored at 4 °C for further analysis of endogenous organic content.

To assess the reusability of the SAPs, each polymer batch was subjected to eight full absorption–desorption cycles. After each cycle, the SAPs were thermally regenerated in a hot air oven (Electrolux, Sweden) at 60 °C for 6 h. Changes in absorption capacity and water recovery efficiency were tracked across cycles. All experiments were performed in duplicate or triplicate.

### 2.3. Characterization

Urine pH was determined using an iUnitrode (6.0278.300, Metrohm Switzerland) connected to a 914 pH/Conductometer (2.914.0020, Metrohm, Switzerland). The concentration of COD was measured using Spectroquant® COD cell tests (500–10,000 mg L<sup>-1</sup> range). Urea analysis was performed using a Thermo Scientific™ Gallery™ discrete analyser with the manufacturer's urease-GLDH method, where urea is first hydrolysed by urease and the resulting ammonium is quantified via the glutamate dehydrogenase (GLDH) reaction; the decrease in absorbance at 340 nm is stoichiometric with urea concentration. The urea concentration was calculated as total ammonia after urease hydrolysis minus free ammonia, which was quantified separately using the Gallery™ ammonia method. To assess chemical changes in the SAPs over multiple reuse cycles, Fourier-transform infrared (FT-IR) spectroscopy was performed. Spectra were recorded in absorbance mode on a Nicolet FT-IR Impact 410 spectrometer (Madison, USA) over the range of 400–4000 cm<sup>-1</sup> at a resolution of 8 cm<sup>-1</sup>. The SAPs were first milled and pressed into KBr pellets using a laboratory-grade hydraulic press.

A targeted metabolomics approach was used to quantify endogenous organic compounds in urine, recovered water, and recovered water post-treatment with activated carbon. A custom assay combining direct-injection mass spectrometry with reverse-phase LC-MS/MS was used to screen and quantify 255 metabolites, including amino acids, phospholipids, biogenic amines, organic acids, acylcarnitines, sphingomyelins, and nucleotides/ nucleosides. The assay was performed on an ABSciex 5500 QTrap® mass spectrometer coupled to an Agilent 1290 series UHPLC system (Agilent Technologies, Palo Alto, USA).

Sample preparation followed the protocol provided with the custom 96-well plate assay. It involved derivatization and extraction of analytes with selective mass-spectrometric detection using multiple reaction monitoring pairs. For all analytes except organic acids, thawed samples were vortexed, centrifuged (13,000 ×g), and applied to the centre of a filter plate. Filters were dried under nitrogen, derivatised with phenyl-isothiocyanate, and following incubation, dried again, and extracted with 300 µL of solvent. Extracts were transferred to a 96-deep well plate via centrifugation and diluted with LC-MS running solvent. For analysis of organic acids, 10 µL aliquots were derivatised with 3-nitrophenylhydrazine and internal standards, incubated for 2 h, and stabilised with BHT and water before LC-MS injection. Data analysis was performed using Analyst 1.6.3 (Sciex, USA).

### 2.4. Calculations

The performance of the dehydration system was evaluated using the following metrics, calculated as follows:

#### Drying rate

$$\text{Concentration Factor (CF)} = \frac{\text{Mass of concentrated urine before evaporation (g)}}{\text{Mass of concentrated urine after evaporation (g)}} \quad (6)$$

The urine evaporation/drying rate normalised to the surface area was calculated using:

$$\text{Drying rate} = \frac{W_u}{T \bullet A_u} \quad (1)$$

where  $W_u$  is the mass of urine dehydrated (kg),  $T$  is the drying time (days), and  $A_u$  is the surface area of urine exposed to air (m<sup>2</sup>).

#### Moisture absorption rate

The rate of moisture uptake by the SAPs, normalised to SAP surface area, was computed as:

$$\text{Moisture absorption rate} = \frac{W_m}{T \bullet A_p} \quad (2)$$

where  $W_m$  is the mass of moisture absorbed (kg) and  $A_p$  is the polymer surface area exposed to moist air (m<sup>2</sup>).

#### Moisture absorption efficiency

To quantify how effectively SAPs absorbed moisture from urine vapour, the following equation was used:

$$\text{Moisture absorption (\%)} = \frac{W_m}{W_u} \times 100 \quad (3)$$

where  $W_m$  is the mass of moisture absorbed (g).

#### Water extraction efficiency

The proportion of absorbed water that could be recovered from SAPs via desorption was calculated using:

$$\text{Water recovery (\%)} = \frac{W_w}{W_m} \times 100 \quad (4)$$

where  $W_w$  is the mass of water extracted (g).

#### Partitioning of endogenous organic compounds

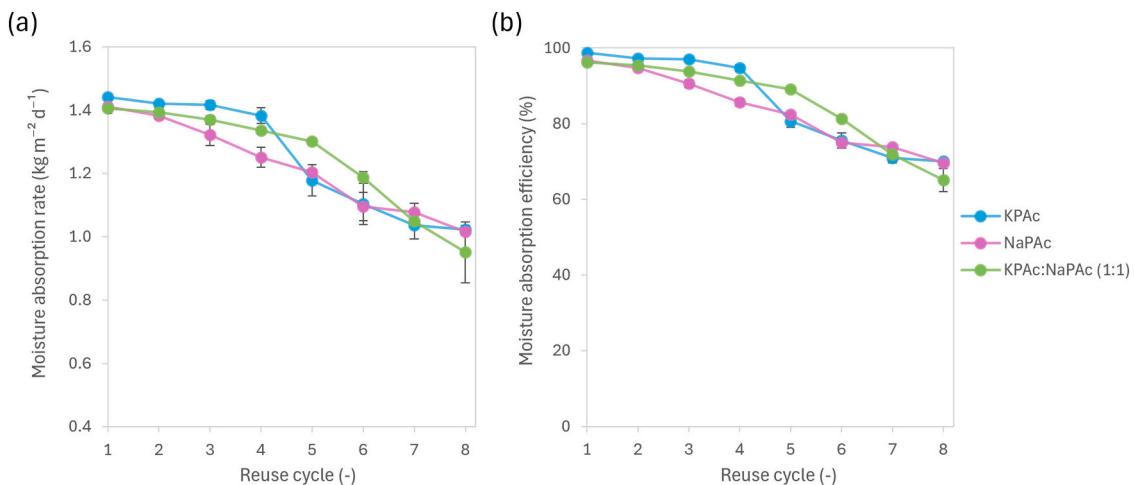
To determine the extent to which endogenous organic compounds present in urine partitioned into the vapour phase and were captured in the recovered water, the following metric was calculated:

$$\text{Partitioning (\%)} = \frac{\sum C_w}{\sum C_u} \times 100 \quad (5)$$

where  $\sum C_u$  is the total concentration of quantified endogenous organic compounds in fresh urine, and  $\sum C_w$  is the total concentration of the those same compounds detected in the recovered water (with or without activated carbon filtration).

#### Mass concentration factor of concentrated urine

The concentration factor (CF) was calculated as the ratio of the initial mass of unconcentrated urine before evaporation to the final mass of urine after evaporation, following the approach described by Gao et al. [32].



**Fig. 1.** (a) Moisture absorption rate ( $\text{kg m}^{-2} \text{d}^{-1}$ ) and (b) moisture absorption efficiency (%) of potassium polyacrylate (KPAc) and sodium polyacrylate (NaPAc) and their 1:1 (w/w) mixture during repeated urine dehydration cycles in a closed-loop setup. Values are mean  $\pm$  SD ( $n = 3$ ) for each drying cycle.

## 2.5. Statistical analyses

Experiments were performed in duplicate or triplicate, and results are presented as mean values with associated standard deviations. Differences in physicochemical parameters (e.g. pH, drying rate, absorption capacity, and water recovery) were assessed using one-way analysis of variance (ANOVA) followed by Tukey's Honest Significance Difference (HSD) post-hoc test to identify pairwise differences. All statistical analyses were performed in R version 4.0.0 [19] using RStudio (version 1.2.5042), with the packages *car* [20] and *agricolae* [21].

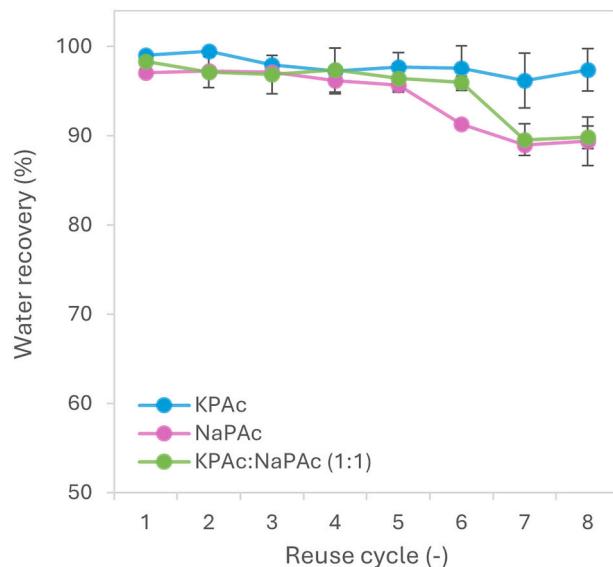
## 3. Results

### 3.1. Evaporation and moisture absorption dynamics

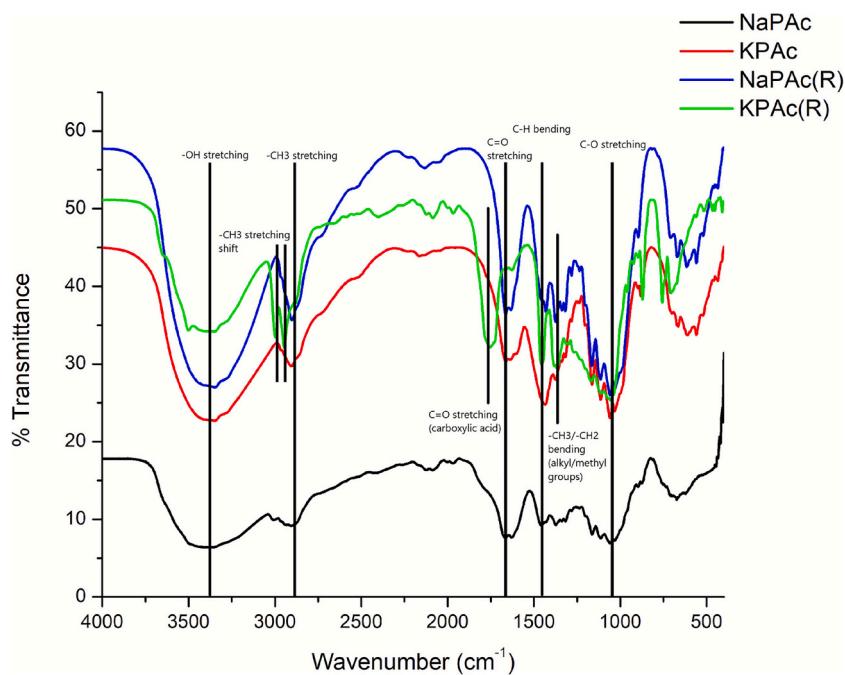
In the closed-loop drying system, overall performance is governed by two interdependent processes: the evaporation of urine in the source chamber and the subsequent absorption of water vapour by

superabsorbent polymers (SAPs) in the downstream air path. In contrast, the linear system does not include a recirculating air loop as humid air is vented out and continuously replaced by ambient air. To evaluate system performance, both SAP vapour absorption and urine evaporation were quantified as area-normalised rates. Urine evaporation rates ranged from  $6 \text{ kg d}^{-1} \text{ m}^{-2}$  in the closed-loop system to  $9 \text{ kg d}^{-1} \text{ m}^{-2}$  in the linear setup (Table S1 in the SI). Moisture absorption by SAPs declined over successive reuse cycles, with the rate decreasing from  $1.44 \text{ kg d}^{-1} \text{ m}^{-2}$  to  $1.02 \text{ kg d}^{-1} \text{ m}^{-2}$  across eight cycles (Fig. 1 (a)).

Both SAPs initially demonstrated high water uptake per gram, exceeding  $0.8 \text{ g}$  of moisture per gram of polymer across all treatments. Potassium polyacrylate achieved  $>0.84 \text{ g g}^{-1}$  in the first four cycles, with sodium polyacrylate performing comparably for the first three. All treatments showed reduced performance over time. By cycle eight, moisture absorption dropped to  $0.64 \text{ g g}^{-1}$  for both SAPs, and to  $0.60 \text{ g g}^{-1}$  for the 1:1 mixture (Table S2 in the SI). The mixture maintained  $>0.80 \text{ g g}^{-1}$  absorption for five cycles, indicating favourable short-term performance but a steeper decline thereafter. When expressed as a percentage of the moisture absorbed, potassium polyacrylate retained the highest efficiency during early reuse, whereas sodium polyacrylate



**Fig. 2.** Water extraction efficiency (%) from potassium polyacrylate (KPAc), sodium polyacrylate (NaPAc), and their 1:1 mixture over eight dehydration-regeneration cycles of urine treatment. Values are the percentage of absorbed moisture recovered as water using a rotary evaporator (mean  $\pm$  SD,  $n = 3$ ).



**Fig. 3.** FT-IR spectra of fresh and regenerated potassium polyacrylate (KPAc) and sodium polyacrylate (NaPAc) before and after eight absorption–desorption cycles (NaPAc(R) and KPAc(R)).

showed marginally better performance in the later cycles (Fig. 1(b)). The crossover between the two occurred around cycle 5.

### 3.2. Water recovery efficiency

The water recovery from moisture-laden SAPs was consistently high across all treatments during the early cycles, with values exceeding 96 % from cycles 1 to 4 (Fig. 2). Potassium polyacrylate exhibited the most stable performance throughout the eight-cycle experiment, maintaining efficiencies above 97 % even in the final cycle. In contrast, sodium polyacrylate had a noticeable decline in water recovery after cycle 5, falling to below 90 % for the last two cycles. The 1:1 SAP mixture showed a similar trend, pointing to sodium polyacrylate as the limiting component in the blend.

### 3.3. Structural changes in SAPs over multiple reuse cycles

FT-IR spectroscopy was used to evaluate chemical changes in the structure of sodium and potassium polyacrylate after eight cycles of moisture absorption and thermal regeneration. Spectra were compared between fresh polymers and samples regenerated at 60 °C (Fig. 3). Fresh SAPs displayed all the characteristic polyacrylate peaks: a broad peak around 3350 cm⁻¹ and sharp –CH₃ stretching around 2800 cm⁻¹, strong C=O stretching at ~1700 cm⁻¹, and C—O and –CH₃/-CH₂ bending vibrations between 1450 and 1050 cm⁻¹ [22]. Thermal stress due to repeated regeneration induced several spectral shifts indicative of partial degradation of the polymers. In the regenerated samples [NaPAc(R) and KPAc(R)], the C=O stretching around 1700 cm⁻¹ intensified and shifted to 1750 cm⁻¹, indicating the formation of acrylic acid, a known degradation product of polyacrylates. Altered intensity and position of the –CH₃ bond stretching (near 3000 cm⁻¹) further suggest changes in the polymer backbone, whereas changes in the –CH₃/-CH₂ bending region (~1400 cm⁻¹) imply modifications to alkyl side chains (Fig. 3). Overall, FT-IR results confirm that thermal regeneration led to partial

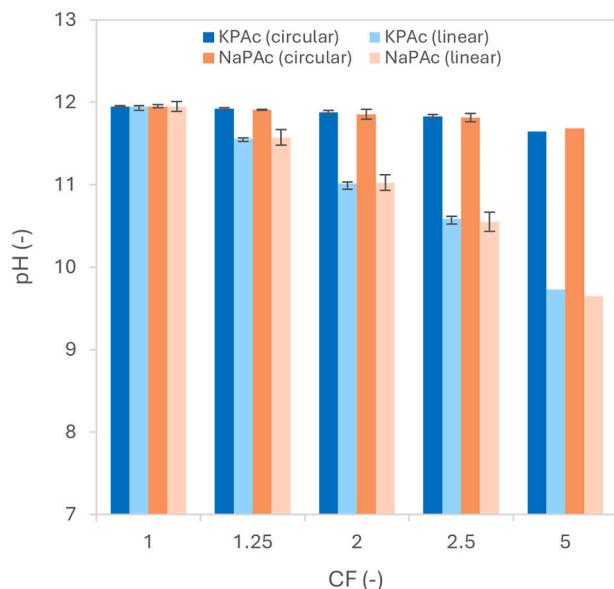
chemical transformation of the SAPs, with sodium polyacrylate appearing more susceptible to degradation than potassium polyacrylate.

### 3.4. Partitioning of endogenous organic compounds

Targeted metabolomics provided insights into the fate of urine-derived organics during dehydration and subsequent water recovery from SAPs (Table S3 in SI). Out of 255 screened compounds, ~210 were quantifiable in the unconcentrated alkaline urine, with a total organic solute concentration of 2.15 g L⁻¹. Of these, ~90 were detected in the recovered water, however, most were present at very low concentrations, with a total quantified organic load of less than 23 mg L⁻¹. In both sodium and potassium polyacrylate treatments, nearly half of the detected compounds in water were either at or marginally above the detection threshold, and only a small subset appeared in clearly quantifiable amounts. Differences between the SAP chemistries were minimal. Although condensates from sodium polyacrylate yielded slightly more metabolite detections than those from potassium polyacrylate, total concentrations were nearly identical (22.5 vs. 21.9 mg L⁻¹).

Urea concentration in urine remained stable throughout the process, with no detectable nitrogen loss observed across concentration factors (Fig. S1 in SI). The 30 most abundant organics aside from urea, including glucose, hippuric acid, lactic acid and creatinine, which together contributed over 90 % of the total organic load, were retained with >99 % efficiency in concentrated urine (Table S3 in the SI). These findings were supported by COD measurements, which decreased from 6120 mg L⁻¹ in urine to 75 ± 1.4 mg L⁻¹ in the recovered water, and further to 25 ± 0.7 mg L⁻¹ following activated carbon treatment.

The few compounds that did partition into the recovered water, such as creatinine (~7.2 mg L⁻¹), lactic acid (~0.36 mg L⁻¹), and creatine (~1.1 mg L⁻¹), were small, polar, and highly water-soluble. Their presence likely reflects co-evaporation or entrainment during vacuum desorption of the SAPs, rather than volatilisation. Subsequent treatment of the recovered water with activated carbon reduced the total organic



**Fig. 4.** Change in pH of alkalized urine dehydrated in circular (closed-loop, CO<sub>2</sub>-excluded) versus linear (open-air, CO<sub>2</sub>-accessible) setups using potassium (KPAC) and sodium polyacrylate (NaPAC). Urine initially alkalised to a pH of ~12.0 was dried to various concentration factors (CF = 1.25, 2, 2.5, and 5). Error bars show standard deviation ( $n = 3$ ). The closed-loop setup maintained significantly high pH compared to the linear setup at all tested CFs ( $p < 0.001$ ).

content to ~11 mg L<sup>-1</sup> in both treatments. These findings confirm that dehydration at temperatures below 40 °C preserves nearly all endogenous organic solutes in the fertiliser product, while generating water with extremely low organic content.

### 3.5. pH stability during alkaline urine dehydration in closed-loop versus linear airflow setups

The initial pH of alkalized urine (CF 1) was ~12.0, and maintaining this high pH is essential to prevent enzymatic urea degradation to ammonia. During dehydration to CF 5, the closed-loop system preserved high alkalinity in urine, with final pH values of 11.64 (potassium polyacrylate) and 11.68 (sodium polyacrylate). In contrast, there was significant pH drop in the linear setup to ~9.7 at CF 5. Differences between closed-loop and linear setups were statistically significant at every CF ( $p < 0.001$ ), confirming the critical role of CO<sub>2</sub> exclusion provided by the closed-loop airflow configuration. No significant difference was observed between the two SAP types within each airflow configuration, suggesting that SAP chemistry had little influence on pH stability (Fig. 4).

## 4. Discussion

### 4.1. Performance trade-offs in closed-loop vs. linear dehydration systems

The closed-loop dehydration system successfully maintained urine alkalinity (pH >11.5 at CF = 5) by preventing atmospheric CO<sub>2</sub> ingress, a known cause of urine acidification and nitrogen loss during dehydration [11]. This chemical stability also ensured complete retention of urea, with no detectable nitrogen loss observed across concentration factors. By contrast, the linear system exhibited significant acidification (pH ~9.7 at CF = 5), since humid air is continuously replaced by ambient air in this configuration. However, preserving alkalinity in the closed-loop system involved inherent performance trade-offs, notably in the evaporation kinetics. In the linear system, continuous exchange of humid air with drier ambient air maintains a high vapour pressure gradient at the urine-air interface, whereas in the closed-loop system,

dehumidification depends on the vapour absorption efficiency of the SAPs, which exhibited lower uptake rates for water vapour (< 0.8 g g<sup>-1</sup>) compared to their liquid-phase absorption capacities (>300 g g<sup>-1</sup>) [23]. The absorption efficiency of the SAPs also decreased from ~1.4 to ~1.0 kg d<sup>-1</sup> m<sup>-2</sup> over successive reuse cycles. FT-IR analysis corroborated this, showing spectral changes in the polymer consistent with its degradation and partial depolymerisation to acrylic acid residues.

### 4.2. Sodium polyacrylate vs. potassium polyacrylate as regenerable dessicants

The closed-loop drying system evaluated here relies critically on the performance of SAPs as regenerable desiccants. Between the two tested polymers, potassium polyacrylate demonstrated higher absorption capacity and faster vapour uptake compared to sodium polyacrylate, particularly in early reuse cycles. This advantage can partially be explained by particle size differences: the potassium polyacrylate used in this study had a dry granule size of ~2–3 mm, whereas sodium polyacrylate particles were significantly finer (150–850 µm). The coarser morphology of potassium polyacrylate likely facilitates looser swelling and a more open hydrogel structure (Fig. S2 in the SI), aiding internal vapour diffusion and deeper absorption before surface saturation [24]. By contrast, the finer particles of sodium polyacrylate gel more densely and rapidly upon hydration, limiting air-polymer interaction and reducing the surface area available for dehumidification. Potassium polyacrylate also exhibits better moisture adsorption as compared to sodium polyacrylate, and is more stable when exposed to ion-rich media, such as urine, because K<sup>+</sup> binds more weakly to –COO- groups [18]. The performance differences observed in our study therefore reflect both the as-supplied particle size distribution and the counter-ion chemistry of the two materials.

Both SAPs exhibited declining moisture absorption with repeated reuse, decreasing from 1.44 to 1.02 kg m<sup>-2</sup> d<sup>-1</sup> in surface area-normalised moisture uptake, and from >0.8 to ~0.64 g g<sup>-1</sup> in gravimetric absorption. This reduction reflects cumulative polymer fatigue resulting from repeated wetting-drying and thermal stress. Although the operational drying temperature was moderate (< 30 °C), each cycle concluded with thermal regeneration of the SAPs at 60 °C for 6 h to desorb moisture. Repeated thermal cycling is known to accelerate degradation in polyacrylates [25]. Thermal stress can induce chain scission, dehydration and rearrangement in polymer backbone structure, leading to reduced swelling capacity and internal diffusivity [26]. The FT-IR spectra of the polymers showed new absorption bands near 1750 cm<sup>-1</sup> and 1400 cm<sup>-1</sup>, indicative of carbonyl-containing degradation products and formation of acrylic acid [27], while shifts near 3000 cm<sup>-1</sup> reflected altered –CH stretching consistent with alkyl chain breakdown [28]. These spectral signatures point to the partial collapse of the crosslinked network, contributing to the observed loss in water absorption efficiency.

### 4.3. Fate of endogenous organic compounds

This study provides the first comprehensive account of how endogenous organic compounds behave during alkaline urine dehydration in a closed-loop system. Although trace amounts of organics were detected in water recovered from urine, the total load was less than 1 % of that originally present in urine. This confirms that alkaline drying not only conserves urea and inorganic solutes in the concentrated urine, as previously demonstrated [14,29], but also preserves the full spectrum of organic constituents in urine. The retention of macronutrients, micronutrients, and endogenous metabolites supports the development of complete fertilisers derived from source-separated urine. The few compounds that were detected in the recovered water, such as creatinine and lactic acid, likely partitioned due to physical entrainment during vacuum distillation. The recovered water, with or without polishing with activated carbon filtration, may be suitable for secondary uses such as

toilet flushing, handwashing, or irrigation of crops.

#### 4.4. Design implications of a closed-loop architecture

Although the volume of recoverable water per person is modest, about  $1.4 \text{ L d}^{-1}$ , even small quantities can be meaningful in arid and water-scarce settings. As an illustrative comparison, hydroponic systems require about  $0.5 \text{ L m}^{-2} \text{ day}^{-1}$  for leafy greens like lettuce [30], so urine-derived water from one person could theoretically support the irrigation demand of  $3 \text{ m}^2$  of hydroponic crops.

Beyond the recovery of clean water, the broader significance of this system lies in its closed-loop air recirculation architecture. By physically isolating urine from ambient air and atmospheric  $\text{CO}_2$ , the system preserves high alkalinity throughout the drying process, preventing acidification and enzymatic urea hydrolysis. This addresses a key barrier in alkaline urine dehydration, pH instability, which has contributed to nitrogen losses and system failure in past field trials of the technology [14,15]. Unlike open-airflow configurations that expose the urine to fluctuating environmental conditions, a recirculating air loop ensures both chemical and operational stability. It also eliminates the need for excessive  $\text{Ca}(\text{OH})_2$  dosing, which is commonly used in open systems to buffer against acidification [14,17]. In our system, urine was only saturated, not oversaturated with  $\text{Ca}(\text{OH})_2$ , and undissolved  $\text{Ca}(\text{OH})_2$  was removed before drying. This reduced the chemical demand, which may enhance the nutrient density of the final fertiliser product while lowering operational costs. An important next step would be to evaluate how these mechanistic advantages translate into practical design choices. A detailed techno-economic analysis would help assess the implementation potential and scalability of the system and identify the most suitable deployment contexts.

The system may also offer thermodynamic advantages that could be explored in future studies. As humid air passes over the SAPs, water vapour condenses into the polymer, releasing latent heat. While we did not quantify this effect, future prototypes could be designed to harness this energy exchange, potentially improving the system's overall efficiency.

## 5. Conclusion

This study introduced a closed-loop air recirculation architecture for dehydrating alkali urine using regenerable superabsorbent polymers (SAPs). This design addresses key limitations of open-air urine drying systems, particularly pH drift and nitrogen loss due to  $\text{CO}_2$ -driven acidification. By integrating urine evaporation with SAP-based absorption-desorption cycling, the system physically excluded atmospheric  $\text{CO}_2$ , maintained high urine alkalinity ( $\text{pH} > 11.5$ ), and prevented enzymatic urea hydrolysis. Notably, this stability was achieved without oversaturating urine with  $\text{Ca}(\text{OH})_2$ , thereby reducing chemical demand and potentially increasing the nutrient density and agronomic value of the final fertiliser.

Metabolomic and colorimetric analyses confirmed that >99 % of the 30 most abundant endogenous organic compounds, including urea, creatinine, and hippuric acid, were retained in the concentrated urine, while the recovered water contained  $<23 \text{ mg L}^{-1}$  of organic solutes and  $< 75 \text{ mg L}^{-1}$  COD. These results show that the full biochemical complexity of human urine can be stabilised and preserved in a dry, sanitised fertiliser product.

Both sodium and potassium polyacrylate SAPs were effective over multiple regeneration cycles, though performance declined due to polymer fatigue and thermal degradation. FT-IR spectra showed characteristic signatures of backbone depolymerisation, highlighting the need for materials research to improve SAP longevity under repeated thermal cycling. Future iterations of this system could also explore harnessing latent heat released during vapour absorption, opening

opportunities for passive urine drying in decentralised settings.

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.desal.2025.119786>.

## CRediT authorship contribution statement

**Anuron Deka:** Writing – review & editing, Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Björn Vinnerås:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Rupam Kataki:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Conceptualization. **Prithvi Simha:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Methodology, Funding acquisition, Formal analysis, Conceptualization.

## Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Björn Vinnerås reports financial support was provided by Swedish Research Council. Björn Vinnerås reports financial support was provided by Horizon Europe Research and Innovation Programme. Prithvi Simha reports a relationship with Sanitation360 AB that includes: board membership. Björn Vinnerås reports a relationship with Sanitation360 AB that includes: board membership. Prithvi Simha and Björn Vinnerås are co-owners of Sanitation360 AB, a spin-off from the Swedish University of Agricultural Sciences that commercialises technologies for recycling human urine. The authors declare that the work reported in this study was conducted independently and was not influenced by their involvement in the company. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

**Data availability** All data supporting the findings of this study are available within the article and its Supplementary Information (SI). Table S3 in the SI provides the complete targeted metabolomics dataset, including quantified concentrations of over 200 endogenous organic compounds in urine before and after dehydration and activated carbon treatment. Additional experimental data, including drying rates, moisture absorption, and urea recovery, are presented in the SI.

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## Acknowledgments

Colleagues at the Swedish University of Agricultural Sciences (SLU) are acknowledged for donating urine and assisting with laboratory work. This research was funded by a grant from the Swedish Research Council (Vetenskapsrådet) for the project NECESSITY (Nästa generationens källsorterande avloppssystem) (grant number 2022-04188). PS and BV were supported by the European Union's Horizon Europe Research and Innovation Programme for the project "P2Green: Closing the gap between fork and farm for circular nutrient flows" (Grant number 101081883). The Sophisticated Analytical Instrumentation Centre (SAIC) at Tezpur University is thanked for their assistance with

FT-IR analyses. Targeted metabolomics analyses were performed by The Metabolomics Innovation Centre (TMIC) in Canada.

## References

- [1] U. Badeti, J. Jiang, S. Kumarasingham, A. Almuntashiri, N.K. Pathak, A. Chanan, S. Freguia, N. Ghaffour, H.K. Shon, S. Phuntsho, Source separation of urine and treatment: impact on energy consumption, greenhouse gas emissions, and decentralised wastewater treatment process, *Desalination* 583 (2024) 117633.
- [2] A. Aliahmad, P. de Morais Lima, H. Kjerstadius, P. Simha, B. Vinnerås, J. McConville, Consequential life cycle assessment of urban source-separating sanitation systems complementing centralized wastewater treatment in Lund, Sweden, *Water Res.* 268 (2025) 122741.
- [3] E. Friedler, D. Butler, Y. Alfiya, Wastewater composition, in: *Source Separation and Decentralization for Wastewater Management*, 2013, pp. 241–257.
- [4] M. Garrido-Baserba, D.L. Sedlak, M. Molinos-Senante, I. Barnosell, O. Schraa, D. Rosso, M. Verdaguera, M. Poch, Using water and wastewater decentralization to enhance the resilience and sustainability of cities, *Nat. Water* 2 (10) (2024) 953–974.
- [5] A. Aliahmad, P. Simha, B. Vinnerås, J. McConville, Comparative environmental assessment of three urine recycling scenarios: influence of treatment configurations and life cycle modeling approaches, *Environ. Sci. Technol.* 59 (39) (2025) 21160–21173.
- [6] N. Adsul, S.-T. Kang, Exploring the evolution of superabsorbent polymer in cementitious materials: insights into testing methods and their impact on properties, *Polym. Bull.* 81 (17) (2024) 15395–15468.
- [7] A.S. Gadtya, D. Tripathy, S. Moharana, Recycling and reuse of superabsorbent polymers, in: *Bio-based Superabsorbents: Recent Trends, Types, Applications and Recycling*, Springer, 2023, pp. 161–183.
- [8] B.S. VS, L.K. Gujala, A. Althuri, A review on superabsorbent polymers: application and recent advances in environmental and economic profiling, *Discover Appl. Sci.* 7 (11) (2025) 1258.
- [9] B. Krajewska, Ureases I. Functional, catalytic and kinetic properties: a review, *J. Mol. Catal. B: Enzym.* 59 (1–3) (2009) 9–21.
- [10] P. Simha, C. Friedrich, D.G. Randall, B. Vinnerås, Alkaline dehydration of human urine collected in source-separated sanitation systems using magnesium oxide, *Front. Environ. Sci.* 8 (2021) 619901.
- [11] P. Simha, C. Lalander, A. Nordin, B. Vinnerås, Alkaline dehydration of source-separated fresh human urine: preliminary insights into using different dehydration temperature and media, *Sci. Total Environ.* 733 (2020) 139313.
- [12] J. Senecal, A. Nordin, P. Simha, B. Vinnerås, Hygiene aspect of treating human urine by alkaline dehydration, *Water Res.* 144 (2018) 474–481.
- [13] A. Vasiljev, P. Simha, N. Demisse, C. Karlsson, D.G. Randall, B. Vinnerås, Drying fresh human urine in magnesium-doped alkaline substrates: capture of free ammonia, inhibition of enzymatic urea hydrolysis & minimisation of chemical urea hydrolysis, *Chem. Eng. J.* 428 (2022) 131026.
- [14] P. Simha, C. Karlsson, E.-L. Viskari, R. Malila, B. Vinnerås, Field testing a pilot-scale system for alkaline dehydration of source-separated human urine: a case study in Finland, *Front. Environ. Sci.* 8 (2020) 570637.
- [15] M.E. Riechmann, B. Ndwandwe, E.E. Greenwood, E. Reynaert, E. Morgenroth, K. M. Uder, On-site urine treatment combining Ca (OH)<sub>2</sub> dissolution and dehydration with ambient air, *Water Res.* X 13 (2021) 100124.
- [16] J. Senecal, B. Vinnerås, Urea stabilisation and concentration for urine-diverting dry toilets: urine dehydration in ash, *Sci. Total Environ.* 586 (2017) 650–657.
- [17] D.G. Randall, M. Krähenbühl, I. Köpping, T.A. Larsen, K.M. Uder, A novel approach for stabilizing fresh urine by calcium hydroxide addition, *Water Res.* 95 (2016) 361–369.
- [18] D.D. Pinzon-Moreno, I.R. Maurate-Fernandez, Y. Flores-Valdeon, A.A. Neciosup-Puican, M.V. Carranza-Oropeza, Degradation of hydrogels based on potassium and sodium polyacrylate by ionic interaction and its influence on water, *Polymers* 14 (13) (2022) 2656.
- [19] Team, R, RStudio: Integrated Development for R [Software], RStudio, Inc., Boston, MA, USA, 2016.
- [20] J. Fox, S. Weisberg, Visualizing fit and lack of fit in complex regression models with predictor effect plots and partial residuals, *J. Stat. Softw.* 87 (2018) 1–27.
- [21] F. De Mendiburu, *AGRICOLAE: Statistical Procedures for Agricultural Research*, Version 1.2-4, 2020.
- [22] I. Karagöz, G. Yücel, Use of super absorbent polymers with Euonymus plants (*Euonymus japonicus* 'Aureomarginatus') in ornamental plant cultivation, *J. Agric. Sci.* 26 (2) (2020) 201–211.
- [23] A. Mignon, J. Vermeulen, D. Snoeck, P. Dubrule, S. Van Vlierberghe, N. De Belie, Mechanical and self-healing properties of cementitious materials with pH-responsive semi-synthetic superabsorbent polymers, *Mater. Struct.* 50 (2017) 1–12.
- [24] X. Ma, Q. Yuan, J. Liu, C. Shi, Effect of water absorption of SAP on the rheological properties of cement-based materials with ultra-low w/b ratio, *Construct. Build Mater.* 195 (2019) 66–74.
- [25] R. Mahon, Y. Balogun, G. Oluymi, J. Njuguna, Swelling performance of sodium polyacrylate and poly (acrylamide-co-acrylic acid) potassium salt, *SN Appl. Sci.* 2 (2020) 1–15.
- [26] F.K. Metze, S. Sant, Z. Meng, H.-A. Klok, K. Kaur, Swelling-activated, soft mechanochemistry in polymer materials, *Langmuir* 39 (10) (2023) 3546–3557.
- [27] L. Feng, H. Yang, X. Dong, H. Lei, D. Chen, pH-sensitive polymeric particles as smart carriers for rebar inhibitors delivery in alkaline condition, *J. Appl. Polym. Sci.* 135 (8) (2018) 45886.
- [28] G. Lavrić, A. Miletić, B. Pilić, D. Medvešček, S. Nastran, U. Vrabić-Brodnjak, Development of electrospun films from wastewater treatment plant sludge, *Coatings* 11 (6) (2021) 733.
- [29] P. Simha, J. Senecal, A. Nordin, C. Lalander, B. Vinnerås, Alkaline dehydration of anion-exchanged human urine: volume reduction, nutrient recovery and process optimisation, *Water Res.* 142 (2018) 325–336.
- [30] V. Carter, N. Verbrugge, F. Lobos-Roco, C. Del Río, F. Albornoz, A.Z. Khan, Unlocking the fog: assessing fog collection potential and need as a complementary water resource in arid urban lands—the Alto Hospicio, Chile case, *Front. Environ. Sci.* 13 (2025) 1537058.
- [31] H. Jönsson, T.A. Stenström, J. Svensson, A. Sundin, Source separated urine—nutrient and heavy metal content, water saving and faecal contamination, *Water Sci. Technol.* 35 (9) (1997) 145–152.
- [32] Y. Gao, B. Vinnerås, P. Simha, Partitioning behavior and crystallization of urea, salts and water during stepwise dehydration of acidified human urine by evaporation, *Sci. Total Environ.* 966 (17870) (2025) 9.
- [33] S. Menegat, A. Ledo, R. Tirado, Greenhouse gas emissions from global production and use of nitrogen synthetic fertilisers in agriculture, *Scientific Reports* 12 (1) (2022) 14490.
- [34] M.S. Ostrand, T.M. DeSutter, A.L. Daigh, R.F. Limb, D.D. Steele, Superabsorbent polymer characteristics, properties, and applications, *Agrosyst. Geosci. Environ.* 3 (1) (2020) e20074.