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Nutrient concentration of blackwater digestate with a pilot-scale low-temperature evaporator

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ABSTRACT

Human excreta contains most of the nutrients consumed in diets, making its recovery essential for sustainable sanitation. Separately collecting blackwater (faeces and urine) enables efficient nutrient and energy recovery. After anaerobic digestion, blackwater produces a nutrient-rich liquid that requires transportation and sanitation for use as fertiliser; concentrating this liquid reduces transport costs and environmental impact. This study investigates nutrient concentration in blackwater digestate using a pilot-scale evaporator designed to operate with waste heat as its primary energy source. Experiments were conducted under two pH conditions (6 and 2.8). The evaporator operated at 60 °C with enhanced heat transfer and gravity-based separation, achieving volume reduction factors of 87 (pH 6) and 85 (pH 2.8). pH strongly influenced nutrient solubility (phosphorus, magnesium, calcium) and equipment leaching. Pharmaceutical residues persisted in concentrates, indicating a need for additional treatment. Final concentrates contained NPK up to 6.7%, 1.5%, and 1.2%, respectively. The specific energy consumption (SEC) increased with concentration, ranging from 0.56 kWh to 1.1 kWh at 60 °C, approximately 1.4–2.8 times higher than the theoretical value. Equipment modifications for low pH and improved material selection could enhance efficiency and regulatory compliance. Future designs may also integrate heat recovery systems to reduce energy demand and improve sustainability.

1. Introduction

By 2050, approximately 70% of the global population is expected to live in urban areas [1]. This means that wastewater treatment needs to remove the excessive nutrient loads (nitrogen and phosphorus) from wastewater before discharging into the receiving waters [2].

Biological nitrogen removal is done through two processes: nitrification and denitrification, or partial nitrification and anaerobic ammonium oxidation (ANAMMOX) [3,4]. These processes imply that most of the nitrogen is returned to the atmosphere. Chemical phosphorus removal and biological phosphorus removal are well-established processes wherein the phosphorus in the chemical process ends up in a precipitate, while the phosphorus in the biological process ends up in the sludge [5]. In addition, ammonia production for fertiliser consumes 1–2% of all of the energy in the world [6] and has a large carbon footprint [7]. The ammonia industry, traditionally focused on fertilisers, is poised to become a competitor in the energy sector as a means of hydrogen storage [7]. Phosphorus in conventional agriculture comes from mining phosphate rocks, which are extracted in only a few countries, thus contributing to geopolitical tensions [8]. Phosphate fertilisers originating from phosphate rocks can contribute to an increase of Pb, Cd and As in the soil and/or crops in the long term [9].

An alternative to using conventional fertilisers and removing nutrients in standard wastewater treatment plants could be to recover nutrients directly from wastewater streams. To do so, separating blackwater (toilet water) from greywater could facilitate the process. Blackwater contains more than 90% of the nitrogen and phosphorus in domestic wastewater [10] as well as other macro and micronutrients [11]. If blackwater is treated separately from greywater, the biogas production potential can also be increased by about 60% with the addition of food waste, compared to conventional biogas-producing wastewater treatment plants [12]. At the same time, biological

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nutrient removal is not required, which has been reported to be representing 49 kWh ($kg_{rem}TN)^{-1}$ and 160 kWh ($kg_{rem}TP)^{-1}$ [13], which in turn decreases electricity use [14]. This kind of separate wastewater treatment has been implemented in several new European urban districts. One such district is Oceanhamnen, Helsingborg, Sweden [15], which consists of approximately 350 apartments, two restaurants, three office buildings and a hotel.

To facilitate the management of the nutrients in blackwater, e.g. for transportation from the recovery facility for further processing and/or to farmland, a nutrient concentration process is needed at the recovery facility [16]. One established process is ammonia stripping and the production of ammonium sulphate in combination with struvite precipitation [17]. These technologies are currently applied in Oceanhamnen to recover nitrogen and phosphorus, but other methods can be used to recover all nutrients in the blackwater. Vacuum distillers are a feasible option tested for concentrating pig slurry digestate in a semi-batch process. The volatilization of ammonia and volatile fatty acids was found to be dependent on the feed pH [18]. However, as the concentration increases significantly, ammonium losses through volatilization may also increase, making it challenging for vacuum distillers [19]. Alternatively, a falling film evaporator powered by a mechanical vapour compressor has been used to concentrate blackwater and wastefood digestate [20]. However, a known challenge with falling film evaporators is that the thermophysical properties of the working fluid such as density, thermal conductivity, and the risk of solid formation change with increased concentration, which can affect performance and stability [21]. Therefore, it is of interest to explore other types of evaporators that may achieve higher nutrient concentrations in the final product [20,22]. To date, reported volume reduction factors for similar digestates concentrated with evaporation or distillation approaches have been from about 3 (vacuum distillation of pig slurry digestate [18]) to about 25 (falling film evaporation of blackwater and waste food digestate [20]), and higher volume reduction has often been limited by ammonia losses, scaling/solid formation, and changes in thermophysical properties [23]. There is currently limited pilot-scale evidence for stable concentration processes for blackwater digestate with high volume reduction while simultaneously quantifying nutrient partitioning/losses, condensate quality, and micropollutant enrichment.

In this context, a pilot-scale evaporator was tested to concentrate blackwater digestate from the Oceanhamnen city district (Helsingborg, Sweden). The unit operates at low temperature (60 °C) and is based on a wetted-surface evaporation concept in which the feed forms a thin film on cellulose sheets and evaporate. The vapour is condensed in a separate condensate tank and collected by gravity. This low-temperature operation enables the heat input to be supplied by low-grade waste heat sources. This study focused on performance of this evaporation process under controlled operating conditions. The objectives were to (1) demonstrate a process for volume reduction of blackwater digestate at pH 2.8 and 6.0, with a pilot-scale low-temperature evaporator (60 °C), (2) quantify nutrient and mineral partitioning using mass balance calculations across the concentrate, condensate, and other losses (e.g., precipitation and volatilization), (3) determine specific evaporation energy demand as a function of the volume reduction, and (4) assess the concentrate quality, including pharmaceutical behaviour and enrichment (160 compounds).

2. Methods

2.1. Experimental set-up

The experimental set-up, provided by Helios Innovations, features two evaporators working in parallel. This set-up, model TESS, is shown in Fig. 1. The evaporators were filled with cellulose blocks from Munter Group AB. Three IBC tanks were used. In the first tank (I), the pH of the digestate was adjusted, while the second tank (II) was used for feed and concentrate. The condensate was collected in the third tank (III). The

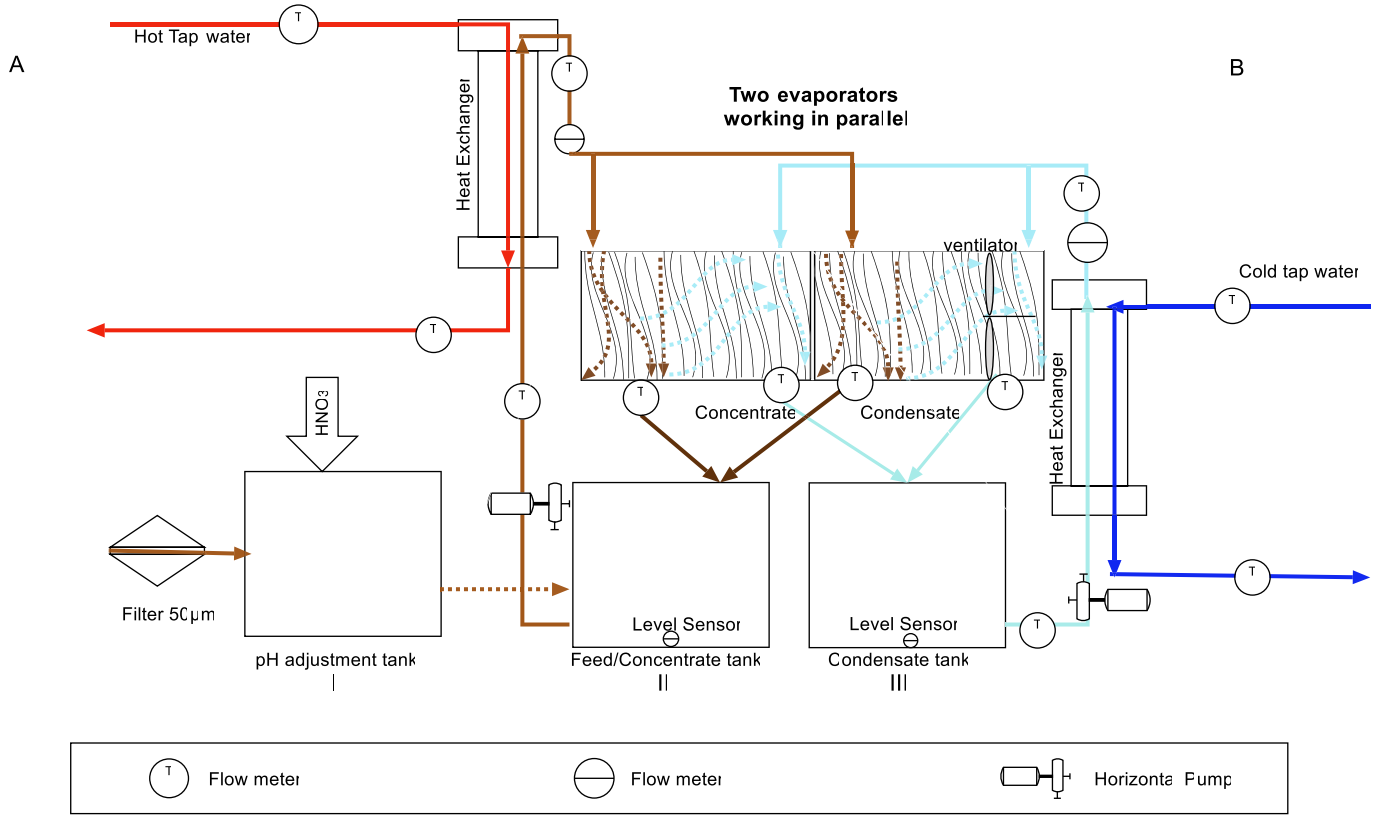

Fig. 1. Experimental evaporator set-up.

feed used was a liquid fraction of the digestate from an up-flow anaerobic sludge blanket (UASB) treating approximately 900 PE of source-separated blackwater from the Oceanhamnen city district in Helsingborg, Sweden.

Two heat exchangers were used, one for the hot side (A) and the other for the cold side (B). The heat source on the hot side was hot tap water (70 °C). The temperatures of the warm feed and the concentrate liquids entering the evaporators A and B were both at 60 °C. The source of the cold medium was tap water (15–18 °C), and the liquid in the condensate tank, after cooling the condensate, was constantly reintroduced into the two parallel evaporators at 20 °C. The condensate produced was cooled in a heat exchanger and then circulated through the evaporator to condense the vapour.

In total, 12 different sensors continuously measured the temperature, marked T in Fig. 1. Four of these were connected to each of the two heat exchangers, two to the outflow of the concentrate and two to the outflow of the condensate.

The feed/concentrate tank was equipped with an Expert 1400 level sensor (MJK Automation), and the condensate tank was equipped with Diver & Baro level sensors (van Essen Instruments B.V). The Diver was placed inside the tank and the Baro was placed in the air next to the tank. The water level was calculated as the difference between the air pressure and the water pressure H_2Ocm. Data from all the sensors were logged each minute.

Calibration curves for both sensors were derived in order to relate the level indicated by the sensors to the volume in the feed/concentrate and condensate tanks. 20-litre buckets were filled and emptied into the tanks, and the level was recorded every 20 l, until the tanks were filled with 1100 l. From this, a linear regression was obtained with an R^2 of 0.998.

The liquid volume in the tanks (V) was then computed over time, t, using the calibration function g and the measured liquid level h (Eq. (1)):

$$V(t) = g(h(t)) \tag{1}$$

Eq. (2) was used as a consistency check for sensor data quality and drift:

$$\Delta V = V_{condensate}(t_{i+1}) - V_{condensate}(t_i) \approx V_{concentrate}(t_i) - V_{concentrate}(t_{i+1}) \tag{2}$$

were the difference in liquid volume ΔV over time t was calculated by the volumes in the condensate and concentration tanks at time t_i and t_{i+1} and then compared.

2.2. Operation and sampling

Two pH conditions were tested, pH 6.0, which is a common pH for liquid fertilisers [24], and pH 2.8, which is below pH 4.5 when the buffering capacity is consumed. Once the buffering capacity is consumed, only a relatively small additional acid dose is required to further decrease pH. The condition of pH 2.8 was selected as an intermediate acidification level, which represents the hydrogen ion concentration midpoint between pH 6.0 and pH 2.5, which has previously been studied with an air gap membrane distillation that achieved VRF of 15 [25].

The pH in the present study was adjusted using nitric acid (≈60% (w/w), analytical grade). To adjust the pH to 6, 4 l of acid were required per $m^3$of blackwater digestate. In contrast, adjusting the pH to 2.8 required 6 l of acid per m^3 of digestate. This means that achieving a pH of 2.8 only required 1.5 times more acid compared to that required to reach a pH of 6. This is because the alkalinity in the water is converted into carbonic acid at pH levels below 4.5. As a result, a small addition of acid at this pH significantly decreases the pH. Additionally, at pH 2.8 the solubility of phosphorus and prevention ammonium volatisation are ensured.

The blackwater digestate was filtered through a 50 μm bag filter before nitric acid was added to Tank I to adjust the pH to either 6 or 2.8. The concentration process was done in batches of 1 m^3, i.e. Tank II was filled and re-filled with blackwater digestate four times (Fig. 2a), with the volume being reduced each time to about 50–30 l of concentrate. Since the condensate was used as cooling water, we were required to start with 100 l of liquid in the condensate tank, and tap water was used as a condensate at the start of the first batch of each experiment. Prior to running the experiments, we intended to use tap water in the condensate tank only at the start of the first batch, and for the consecutive batches, use approximately 100 l of the previous batch's condensate as the starting volume in Tank III. However, a leak was detected in the third batch at pH 6, which required the condensate tank to be fully emptied and cleaned. Next, tap water was used at the start of every batch in Tank III; for the mass balance, it was considered that the tap water's contribution to the nitrogen and phosphorus levels was negligible in comparison to the blackwater digestate.

Composite and grab samples were taken from the feed when the feed was pumped from the pH Adjustment Tank I to the Feed/Concentrate Tank II (Fig. 1). For the grab samples, five 100-mL bottles were collected for different parameters. For the composite sample, 1 l of each batch of feed was collected and 10 mL of each feed was filtered in a 0.45 μm filter for the analysis of dissolved metals. At the very beginning of transferring a new cubic meter from Tank I to Tank II, additional grab samples were taken to characterise the mixture of the previous concentrate and compare it to the new feed from Tank I. Grab samples of feed, concentrate, and condensate were collected once at each sampling occasion and analysed once. To evaluate the representativeness of the analyses of the feed characterizations, we quantified batch-to-batch variability in the feed (mean, SD, CV%) across the individual feed batches (Supplementary Table S1). The variability is summarized in Supplementary Table S1. Additionally, some nutrient parameters of the final concentrates were measure by two different laboratories, compared in Table S2. Both tables in the supplementary material show consistency in the analyses, except for sulphate, see Supplementary information. Furthermore, grab samples of the concentrate and the condensate were taken when approximately 90% of the water in Tank II had evaporated and there were about 100 l of concentrate in Tank II. The final grab samples from each 1 m^3 batch were taken when there were about 50 l in Tank II (before the next batch was added to Tank II). Samples were taken at volumes of 100 l and 50 l because the volume reduction factor at 100 l is half of that at 50 l. A 4-l composite sample of the condensate was made by taking a 1-litre sample from Tank III after each batch of concentrate. After the sample was taken, the condensate tank (III) was emptied. All samples were put in a freezer and stored at −20 °C before their analysis.

Each experiment at different pH consisted of four batches, with eight sampling points shown in Fig. 2. The target volume reduction factor (VRF) was 80. After the third batch, the VRF had already reached 67 at pH 6 and 71 at pH 2.8, which is higher than most values reported in the literature; therefore, this study aimed to push the operational boundary further.

2.3. Chemical analyses

Feed, concentrate, and condensate samples were analysed for NH_4-N, NO_2-N, NO_3-N, Total N, PO_4-P, total P and SO_4-S in each feed batch, composite feed, and in the final concentrate of each batch, and also when there were 100 l in the Feed/Concentrate tank. These compounds were also measured in the final condensate of each batch and in the composite condensate sample when there were 100 l in the Feed/Concentrate tank and after the fresh adjusted feed of a batch was mixed with the concentrate of the previous batch. Metal(oids) were measured in the composite feed and condensate, and in the final condensate of each batch by an accredited laboratory, using the method for metal (oids) (EPA Method 245.7). DOC, TOC, COD-Cr and BOD_7 were analysed according to the EPA Method 415.3, EPA Method 9060A (SW-846), EPA Method 410.4, and EPA Method 405.1, respectively, in samples of the composite feed, composite condensate, and final concentrate.

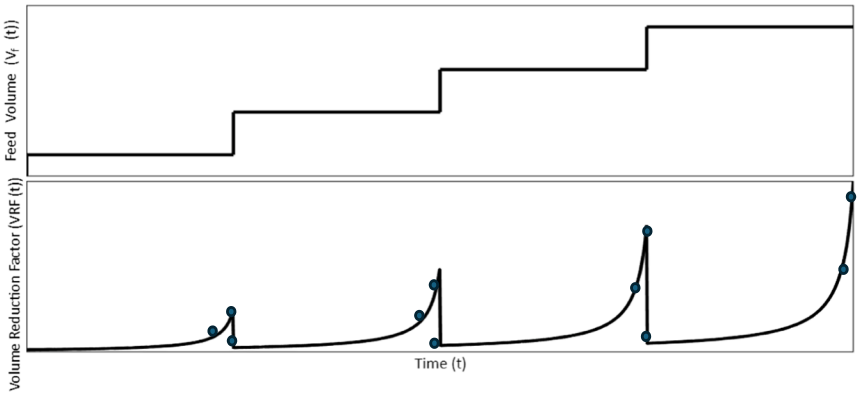

Fig. 2. The behaviour of the total added feed volume (a) and the volume reduction factor VRF (b) over time. Each step in the curves represents a new concentration batch. The blue points indicate sampling occasions in Tank (II) and Tank (III).

Micropollutants were only analysed in the composite feed and the final concentrate using ISO/IEC 17025:2017 by an accredited laboratory.

Nitrite, nitrate, and total nitrogen levels were determined using the QuAAtro Applications Method No. Q-003-04. NH_4-N levels were assessed according to the QuAAtro Applications Method No. Q-001-04. Ammonium, nitrite, and nitrate were also determined using a spectrophotometer according to CSN EN ISO 111732 (EPA Method 350.1), CSN EN USI 13395, SM 4500-NO2 (EPA Method 353.2), and SM-4500-NO3 (EPA Method 353.2), respectively, with filtration applied for turbid samples. Total Kjeldahl nitrogen was determined based on CSN EN 25663 and CSN ISO 7150-1 (EPA Method 351.2), with the total nitrogen calculated as the sum of NO_2-N, NO_3-N, and Kjeldahl nitrogen.

Total phosphorus was digested according to EN-1189:1996 and analysed using QuAAtro Applications Method No. Q-031-04. The phosphate reacted with antimony and molybdate ions, followed by ascorbic acid reduction, producing a blue phosphorus-molybdenum complex measurable at a wavelength of 880 nm in a UV–visible spectrometer.

Alkalinity was determined according to USO 9963-2, CSN 75 7373, and SM 2320, titrated to pH 5.4.

The micropollutant analysis of hormones was performed following the EPA 539:2010 method, while the other micropollutants were measured following the EPA 1694:2007 method. In total, 160 different substances were evaluated.

Metals such as, As, Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, and Zn were analysed on the cellulose sheets were determined by ICP-SFMS according to SS-EN ISO 17294-2:2016 and US EPA Method 200.8:1994, after digestion, according to B-PB29-MW by an accredited laboratory.

2.4. Chemical modelling

Visual MINTEQ 4 [26] was used to determine the mineral composition and solid formation in the blackwater concentrate. Due to the high activities, the activity corrections used were based on specific ion interaction theory (SIT) [26]. The equilibrium temperature was considered 25 °C at 1 atm. Particulate and dissolved metals were used to validate the model. The specific solid formation was determined based on the saturation index given by the software after entering the concentrations of elements measured. This model was used to evaluate possible precipitates but not the metal interactions with organic matter.

2.5. Statistical analysis

The mass of nutrients in the concentrate and condensate at pH 6 and pH 2.8 were compared using a paired *t*-test with Minitab 22. The significance level was $\alpha = 0.05$.

2.6. Calorimetric calculations

The volume reduction factor (VRF) was defined with Eq. (3):

$$VRF(t) = \frac{V_f(t)}{V(t)} \tag{3}$$

where $V_f(t)$ is the feed volume and V(t) is the concentrate volume at time t. The VRF of each batch in both experiments were intended to be as similar as possible for each step, as were the final volumes treated. The behaviour of Vf and VRF from Eqs. (1) and (3) is shown in Fig. 2.

The thermal power P (in Watts) supplied to the process was calculated for both the cold and hot sides of the evaporator using Eq. (4):

$$P = \rho c_w q \Delta T \tag{4}$$

where c_w is the specific heat capacity of water, ρ is the density, q is the volumetric flow rate on the hot side, and ΔT is the temperature difference between the inlet and outlet of the hot tap water stream in the heat exchanger. The inlet temperature of the hot tap water was 70 °C. The heat required for evaporating a mass difference of liquid Δm in kg expressed as dW, is given by Eq. (5)

$$dW = \Delta H_{vap}\, dm \tag{5}$$

where, ΔH_{vap} is the enthalpy of evaporation.

Then

$$dW = Pdt \tag{6}$$

where it is assumed that all the heat entering the system through to the heat exchanger during Δt will be used for evaporation, which equals dW. Furthermore,

$$dm = \delta dV \tag{7}$$

where dV is the evaporated volume during dt, and δ is the density of the water that crossed to the condensate side and is assumed to be the same as that of pure water. Replacing (6) and (7) in (5) gives Eq. (8):

$$SEC = \frac{P}{\delta \dot{V}} = \frac{P}{\dot{m}} \text{ as} \Delta t \to 0 \tag{8}$$

Eq. (8) was used to estimate the specific energy consumption (SEC) as a function of the volume reduction factor as shown in Eq. (9).

$$SEC = f(VRF) \tag{9}$$

where f is assumed to be a linear function.

3. Results and discussions

3.1. Water mass balances

The volume reduction factor (VRF) could be achieved at approximately 20 times the volume reduction per batch; after the fourth batch at pH 6, a VRF of 87 was achieved and at pH 2.8, a VRF of 85 was achieved. The volume reduction factors obtained at the end of each concentration step were similar for the two experiments (at pH 6 and pH 2.8) but not the same (Fig. 3).

Generally, water loss during the experiments was small since the volumes of water measured at the beginning and end of each concentration step (marked as S (Start) and E (End) in Fig. 3) were similar. The maximum mass balance deviation was observed during the third concentration step of the experiment at pH 2.8, where the calculated difference was 52 l. We attempted to keep the volume reduction factor the same for corresponding batches in both experiments as well as the feed volume of each batch. However, the feed volumes at pH 2.8 were slightly higher than those at pH 6; The total volume treated was 3697 l at pH 6 and 3768 l at pH 2.8. Therefore, the last batch at pH 2.8 required less volume to reach a total volume equal to that at pH 6.

For the experiment carried out at pH 6, water leakage was observed during the run of the third batch at a VRF of 71. Therefore, tap water was added to the condensate tank in the third batch (see details in Section 2.2). Tap water was used at the start of each concentration step in the experiment at pH 2.8 (Fig. 3).

3.2. Feed and concentrate quality

The concentrations of most of the parameters measured in the two pH-adjusted feed batches used in the experiment are shown in (Table 1) as blackwater digestate, which is generally a heterogeneous substrate. The nitrate concentrations in the two feed batches differed considerably after the pH was adjusted with nitric acid (to a pH of 2.8 in one experiment and pH 6 in the other). The blackwater digestate without acid addition was analysed in a previous study [11], where the nitrogen and ammonium was estimated to 1030–1120 mg tot-N/L and1060 to 1440 mg NH_4-N/L, respectively. Nitrate and nitrite where not detected in the blackwater digestate, and phosphorus was between 79 and 160 mg Tot-P/L while phosphate phosphorus was between 60 and 74 mg PO_4-P/L. The addition of nitric acid decreased both the COD and alkalinity at both pHs, the latter to below the detection limit at pH 2.8. The reduction in alkalinity could be explained by carbonates in the blackwater digestate transforming to carbon dioxide, and nitric acid, a strong oxidiser transforming some of the organic carbon into carbon dioxide and water 31.

According to chemical simulations, phosphorus tends to form various compounds such as hydroxyapatite, ferrihydrite, struvite and $MnHPO_4$ at pH 6. These simulations also indicated that other compounds may precipitate in the cellulose sheets, resulting in their loss which need to be verified in future studies, solid-phase characterization (e.g., XRD). Interestingly, DOC was concentrated 22 times at pH 6. Therefore, in the final concentration, no mineral formation was observed, but there was sorption of calcium, iron, copper, magnesium, sodium and other metals. This suggests that the non-dissolved fraction of metals in the concentrate at pH 6 is likely attached to organic matter.

Low pH is known to affect the solubility of metals such as copper, calcium and manganese (Table 1). At pH 2.8, calcium and copper are mostly dissolved. Since the concentration of copper and zinc increased dramatically in the concentrate, the hypothesis is that it came from the brass joints in the equipment.

It is interesting that at pH 2.8, the dissolved fraction of manganese is smaller than the total fraction since it usually dissolves at a low pH when the oxidation state is Mn^{2+}. However, in higher oxidation states (Mn^{3+} and Mn^{4+}), manganese can form insoluble oxides or hydroxides [27]. Unfortunately, manganese oxide and manganese hydroxide are not in the mineral database of the software.

The decrease in sulphate in the feed after adding nitric acid may lead to the release of gases such as H_2S, which has low solubility at a low pH [28].

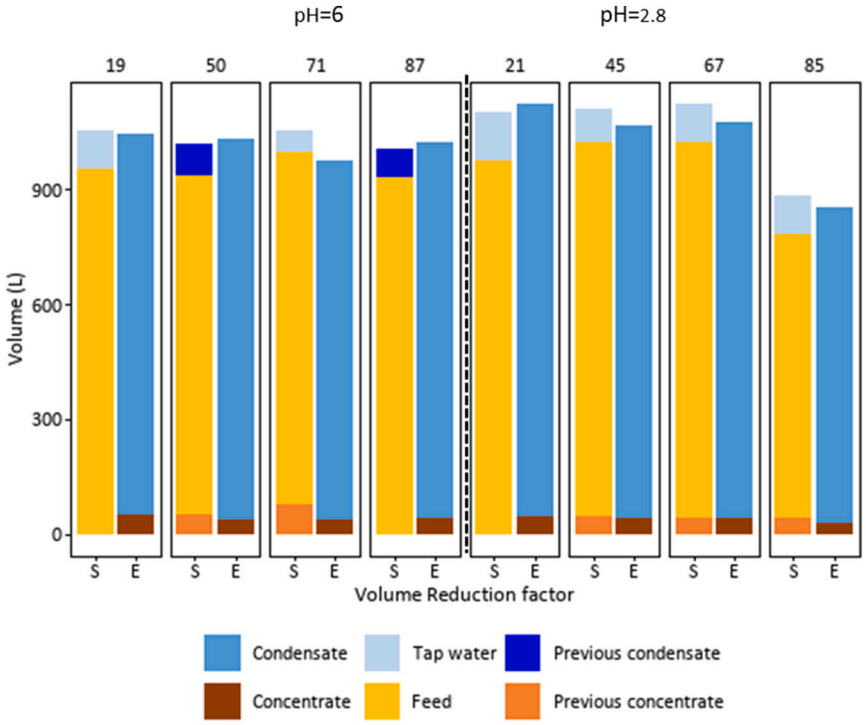

Fig. 3. Water mass balance for the two experiments at pH 6 and pH 2.8. Start refers to the different volumes of water in Tanks II and III at the start of each batch, and End refers to the volumes in the tanks after one batch. The volume reduction factor (VRF) is shown above each batch (Start(*S*)-End(E)).

Table 1
Measured parameters for the composite samples of the blackwater digestate (feed) after the addition of acid, and the final concentrate (after four batches). The composite samples were combined sub-samples from the four batches of feed used at the two studied pH levels.

Parameter	Unit	Feed		Concentrate	
		pH = 6	pH = 2.8	pH = 6	pH = 2.8
Turbidity	NTU	200	138	3420	6000
pH	–	7.5	2.6	7.5	1.3
Conductivity	mS/m	1480	1600	9160	11,000
Alkalinity	mg HCO_3^-/L	<2.4	<2.4	1680	<2.4
DOC	mg/L	155	111	3410	3850
TOC	mg/L	324	140	6560	4840
COD-Cr	mg/L	1630	917	1630	19,000
BOD 7	mg/L	256	176	2310	1950
Total N	mg/L	1460	1860	66,000	76,000
NH_3-N + NH_4-N	mg/L	963	1130	32,300	35,800
NO_3-N	mg/L	780	1000	26,300	42,200
NO_2-N	mg/L	<0.02	<0.07	5.99	<0.05
Total P	mg/L	77.4	87.1	1210	3370
PO_4-P	mg/L	61.3	83.1	1080	2490
Total S	mg/L	65.6	26	1650	875
SO_4-S	mg/L	47.4	19	907	674.18

Metals	Unit	Total	Dissolved	Total	Dissolved	Total	Dissolved	Total	Dissolved
As	µg/L	17	17.2	12.6	13.5	489	404	599	545
Ca	mg/L	69.5	57.1	43	39.7	417	400	1740	1810
Cd	µg/L	0.45	0.15	0.05	<0.05	<2	1.98	16.6	14.7
Co	µg/L	1.21	0.84	1.52	0.32	23.8	18.8	38.6	36.6
Cr	µg/L	3.73	2.53	51.4	0.99	42	22.9	130	138
Cu	µg/L	103	33.80	45.2	3.76	106,000	7920	137,000	129,000
Hg	µg/L	0.102	0.02	<0.02	<0.02	0.297	0.451	0.2	80.1
K	mg/L	402	342	279	282	11,300	10,000	11,500	11,500
Mg	mg/L	23.8	23.20	24.2	23.9	123	138	863	961
Mn	µg/L	90	71.6	201	33.5	571	787	3100	2950
Mo	µg/L	6.92	3.09	2.36	<0.5	82.4	54.4	<50	27.5
Na	mg/L	473	430	354	312	14,400	12,300	12,100	12,600
Ni	µg/L	15.4	8.94	19.4	7.6	390	259	1130	1070
Pb	µg/L	2.06	0.70	1.24	<0.5	127	137	8760	7940
Zn	µg/L	421	144	110	147	3440	3700	132,000	128,000

3.3. Nutrient mass balance

The percentage of ammonium in the concentrate did not differ significantly between the two pH levels regardless of the VRF. There was a higher total concentration of nitrogen and nitrate at pH 2.8 due to the greater addition of nitric acid.

The estimated loss of phosphorus at pH 6 was approximately 82%, and only 18% of the total phosphorus mass was found in the final concentrate (Fig. 4). The phosphorus may have been lost as precipitates on the cellulose sheets in the evaporator. Moreover, at the end of the third batch of the same experiment (VRF 71), the percentage of phosphorus found in the concentrate was 29%. This implies that increasing the volume reduction factor at pH 6 does not directly increase the concentration of phosphorus in the concentrate.

At pH 6 and VRF 87, 42% of the total nitrogen added was kept in the concentrate, while at pH 2.8 and VRF 85, only 39% was kept. For pH 6, the condensate had approximately 12% of the total mass of the nitrogen and around 40% was lost through sorption or to the atmosphere, while at pH 2.8, the condensate had 20% of the total mass of the nitrogen and 41% was lost. At pH 6 and VRF 71, however, 50% was found in the concentrate, 36% in the condensate, and only 14% was lost. The higher losses at VRF 87 compared to VRF 71 can be explained by the leakage detected that led to some water loss in the concentrate.

At VRF 19, the concentration of ammonium at pH 6 and pH 2.8 was 63% and 52%, respectively. At VRF 19, 43% and 83% of the phosphorus was in the concentrate at pH 6 and pH 2.8, respectively. Finally, potassium concentrations at VRF 19 and pH 6 were 28%, while at VRF 21 and pH 2.8, 65% of the potassium was found in the concentrate. Since the chemical properties of the nutrients differ, the concentration process also differs at the same VRF. Therefore, optimising how much should be concentrated is essential. Although the nutrient losses seem quite high, they can be compared with theoretical concentrations and the reverse osmosis of waste food digestate. With a VRF of 85, the estimated concentrations in the concentrate from food waste digestate were 22% of the total nitrogen, 10% of the phosphorus and 85% of the potassium [29]. This means that the evaporator concentrate was able to retain higher percentages of nitrogen and phosphorus, but not potassium.

Mass balances for ammonium, nitrate, total nitrogen, phosphate and total phosphorus were determined and are shown in Fig. 5.

The fate of nitrogen depends on the species. Since ammonium is volatile, the pH affects its concentration. In this study, nitric acid was used to adjust the pH, signifying that the concentration of nitrate was higher at the lower pH. Although nitrate is usually under the detection limit in blackwater digestate, it is stable in water and can be used along with sodium as an indicator of leakage and general mineral losses during the process.

The concentrations of ammonium, nitrate and total nitrogen in the tap water and the previous condensate were low. Mass balance calculations showed that less than 7% of the total nitrogen was lost due to sampling.

A paired *t*-test of both pH experiments for all the volume reduction factors showed that the losses of ammonium were not significantly different. At pH 6.0, it was found that the losses matched the equilibrium curves of ammonium and ammonia [30]. Despite the differences in ammonia's partial pressure in blackwater digestate at pH 6 (1.48E-5 atm at 55 °C and 5.62E-5 atm at 70 °C) and pH 2.8 (9.1E-9 atm at 55 °C and 3.56E-8 atm at 70 °C), the equilibrium at both pH 6.0 and pH 2.8 favours the formation of ammonium over ammonia [11], which may explain the

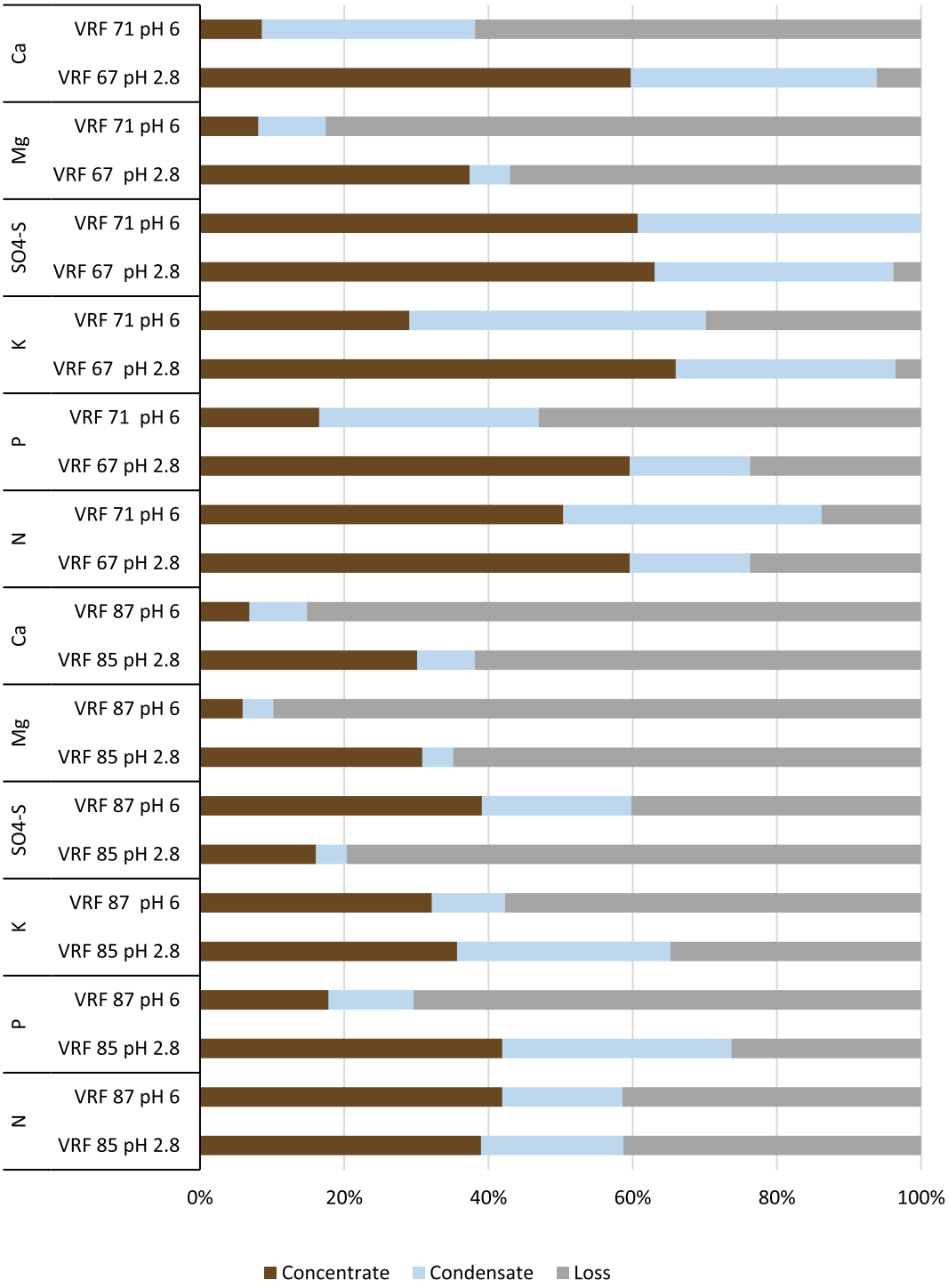

Fig. 4. Percentage of nutrients of the total added ending in the concentrate and condensate or lost in the concentration process of the third (VRF = 67 & 71) and fourth batches (VRF 85 & 87), at pH 2.8 and pH 6. VRF is the volume reduction factor.

similar behaviour of ammonium at both pH levels.

Nitrate was also found in the condensate, indicating that drops of the concentrate crossed into the condensate. The final mass of ammonium at pH 6 was 1100 g, while at pH 2.8 it was 1380 g. This difference of about 300 g was probably due to ammonia volatilization. According to Saracco and Genon [30], temperature does not affect volatilization below pH 5, only the pH does.

The addition of nitric acid introduced nitrate-N and increased the total nitrogen in the concentrate. However, acid addition would increase operating costs due to chemical consumption, as well as introducing hazards into the working environment, and may require downstream neutralization/buffering depending on the intended use of the concentrate. Therefore, acidification is not necessarily the most suitable strategy for ammonium stabilization. As an alternative, stabilization of ammonium could be achieved via biological nitrification, a process already applied for stabilizing urine-based fertilisers [31,32], in which ammonium is aerobically converted to non-volatile nitrate. In addition, nitrification decreases pH, potentially reducing or eliminating the need for mineral acid addition (which may be associated with non-renewable inputs depending on the acid source).

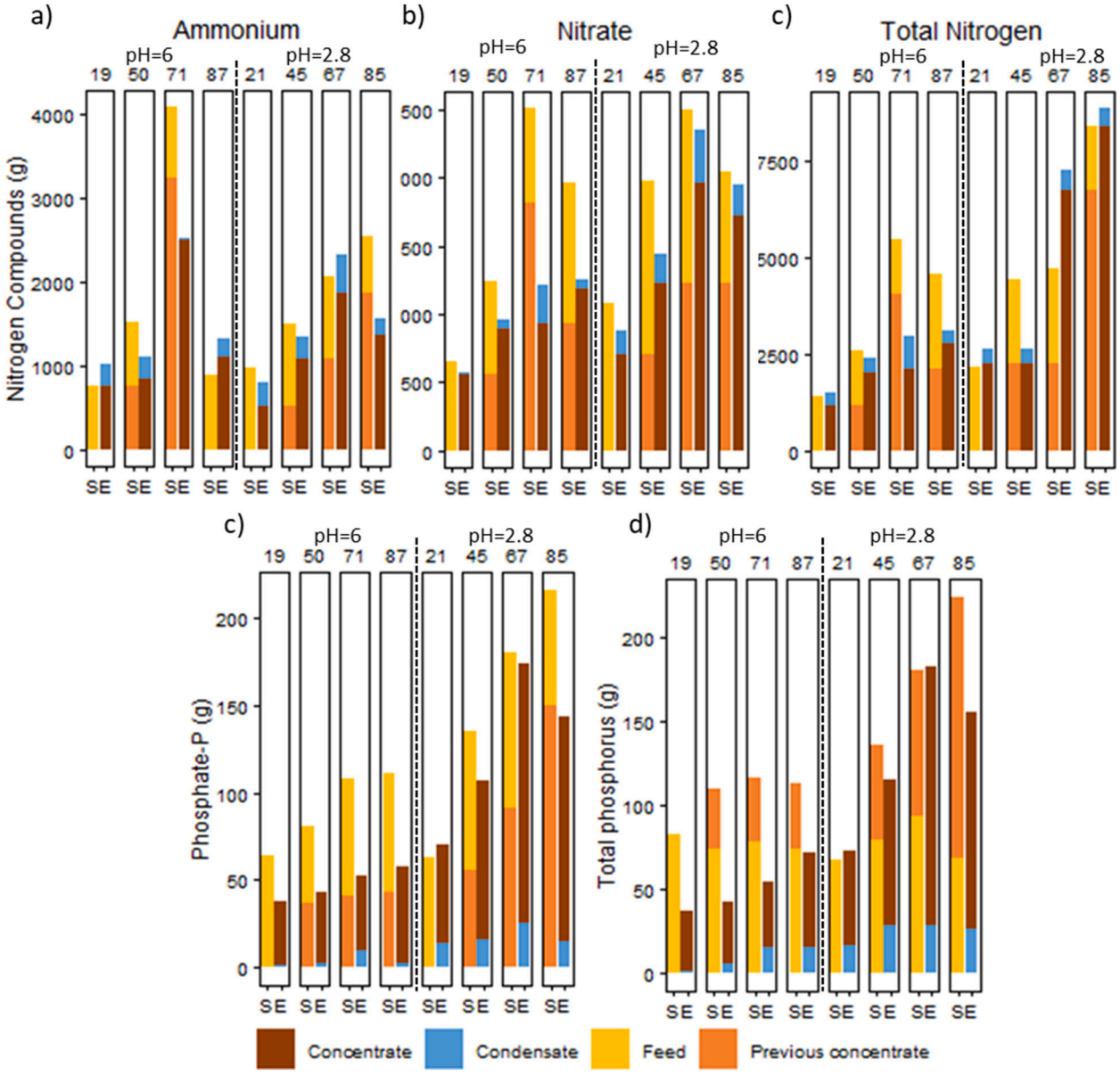

Fig. 5. Measured concentrations for each batch of a) nitrogen- ammonium, b) nitrogen- nitrate, c) total nitrogen, and d) phosphorus -phosphate- and total phosphorus. "S" refers to the different start volumes of water in Tanks II and III at the start of each batch and "E" refers to the end volumes in the tanks after one batch. Above each batch (Start-End), the actual volume reduction factor (VRF) is indicated.

Total phosphorus concentration in each batch's feed at pH 6 ranged from 74 to 86 mg/L, and at pH 2.8 from 69 to 95 mg/L. The mass of phosphate found in the concentrate in the experiment at pH 2.8 was statistically larger than at pH 6. One explanation for this is that phosphorus was precipitating to a higher degree at pH 6, as shown by the chemical modelling. The simulation indicated that at pH 6, phosphorus-containing minerals in the concentrate primarily formed with calcium, copper, aluminium and iron. This occurred before the inclusion of organic matter since the high DOC (dissolved organic carbon) relative to some metals in the concentrate caused most metals to be adsorbed by the organic matter. Interestingly, the total phosphorus mass in the concentrate at pH 2.8 was higher when the volume was reduced by 67 times compared to a VRF of 85.

3.4. Metal leakage and loss

Magnesium and calcium were not found in the concentrate at the concentrations that would be expected after concentrating the blackwater digestate 85 or 87 times (Fig. 4a). They were found in much smaller concentrations than expected at each step, but more so at pH 6 than at pH 2.8 (Fig. 6). The Visual MINTEQ simulations at the measured concentrate conditions indicated that (i) adsorption or association of divalent cations with organic matter and (ii) formation of phosphate-containing mineral phases could be thermodynamically plausible under the high concentrations reached during evaporation. In addition, the simulations suggested that ammonium-containing phosphate solids could form during the concentration process (e.g., Mg, Ca, or other metal phosphate phases incorporating NH_4), potentially contributing to apparent nitrogen losses by retaining ammonium in a solid phase. In further studies this needs to be confirmed with solid-phase characterization. Some hydroxyapatites could form with calcium [11], but the final concentration of DOC in the concentrate was 3410 mg/L, so these metals were likely sorbed to the organic matter. Similar observations were found by Groeneveld et al. [33], where most of the metal and metalloids fraction were found in the sewage sludge. Sorption depends on the pH and ionic strength [34], so at pH 6, sorption is most likely to

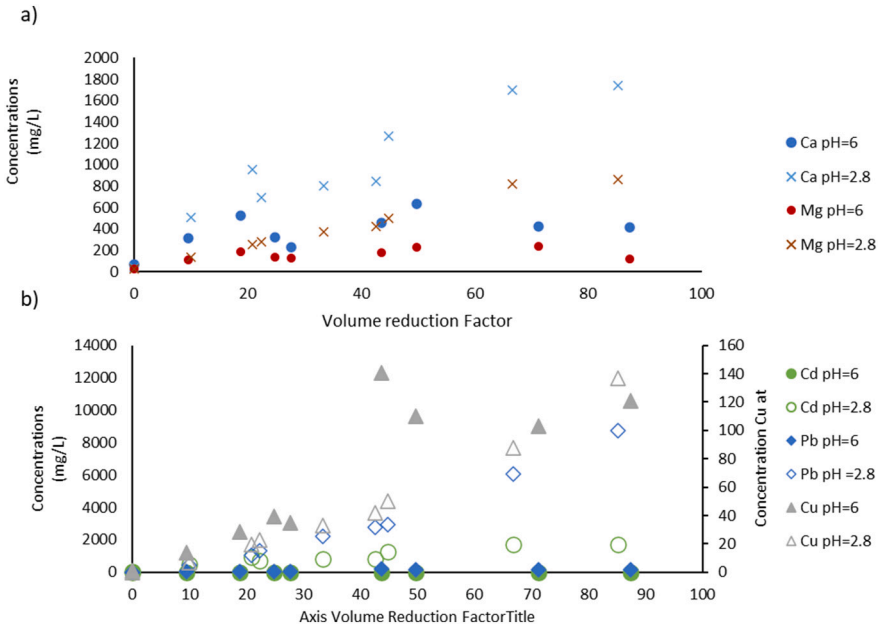

Fig. 6. Total metal concentrations in the concentrate, where a) represents calcium and magnesium, and b) metals that may have leached out from the equipment.

happen rather than at pH 2.8. The sorbed compounds may have stayed on the cellulose sheets, leading to a loss of calcium and magnesium. This is unfortunate since calcium and magnesium are secondary macronutrients [35].

Several metals can be potentially harmful to plants, such as copper, cadmium, lead and zinc. At pH 2.8, the concentrations of copper, lead and cadmium in the concentrate were 302, 7000, and 300 times higher than in the feed, respectively, exceeding the expected concentrate-to-feed ratio. Given a VRF of 85, it is likely that these elevated concentrations came from the equipment or cellulose sheets. For pH 6 (Fig. 6), the high concentrations were also likely due to leaching from the equipment, just not as much as at pH 2.8. For example, the copper could have originated from the brass joints installed in the equipment. Copper and zinc may partially originate from contact with system materials; however, both are also commonly present in digestates. In addition, while elevated concentrations of Cu and Zn can be phytotoxic [36], these elements are also micronutrients [35] and are typically present at low levels in commercial liquid fertilisers; therefore, potential risk depends on the final concentrations and application dose.

The anomalously high Cd enrichment was observed at pH 2.8, but not at pH 6.0, suggesting pH-dependent mobilization (from dissolution/desorption of solids or surfaces). However, the source of Cd could not be resolved from the present dataset. According to the chemical model at pH 6, some plumbogummite, a mineral containing lead and phosphate, was formed. This formation may reduce the final concentration of lead. However, this does not change the likelihood that at pH 2.8, there were lead leakages from the equipment.

The final concentrate at pH 2.8 showed a higher nitrogen concentration than at pH 6.0, mainly because more nitric acid was added to control pH. However, this difference is not apparent when nitrogen is reported as mg/kg dry matter (DM). At pH 2.8, the total solids content was lower, so although the nitrogen concentration in the liquid phase was higher, the total dry mass was smaller. Consequently, after normalization to DM, the nitrogen value appears lower at pH 2.8 than at pH 6.0 (Table 2). In contrast, the concentrations of P, Ca, and Mg on a dry-mass basis were 5.2-, 3.5-, and 6.0-fold higher, respectively, at pH 2.8 compared with pH 6.0. The concentration of sulphur was lower in the concentrate at pH 2.8, since it was released into the atmosphere at low pHs [38]. The mass percentage of elemental NPK was 60:3:12 in the concentrate produced at pH 6, and 67:15:12 in the concentrate produced at pH 2.8. This pH retained more phosphorus than ammonium in the concentrate, considering the percentage weights (Fig. 6).

Most of the micronutrients analysed were found in both concentrates except for Mo. There were high concentrations of micronutrients such as Cu and Zn, but they were at levels still under the permitted limits (European Parliament and the Council 2009).

The concentrations of elements of concern (As, Al, Pb, Cd and Hg) in the concentrate at pH 6 were low (Table 2). To assess the concentrations of these metals, it was important to consider that the concentrate, when applied as a fertiliser, would be diluted and dosed in relation to nutrient loads needed, and the total load of metals would be small. However, the acceptability for fertiliser use would be determined by the competent food/environmental authorities under the applicable local regulatory framework and product classification. At pH 2.8, the concentrations of arsenic, cadmium and mercury were higher but still below the EU limits for metals in fertilising products [37]. The elevated levels of aluminium

Table 2
The concentrations of macronutrients, micronutrients and elements of concern present in the final concentrate at pH 6 and pH 2.8.

	Macronutrients (g/kg DM)		Micronutrients (mg/kg DM)			Elements of concern (mg/kg DM)		
	pH 6	pH 2.8		pH 6	pH 2.8		pH 6	pH 2.8
N	277.1	268.9	Co	3.1E-5	0.14	As	3.4E−3	2.1
P	5.1	27.0	Cu	0.14	485	Al	2.1E−1	46.4
K	47.3	40.7	Fe	0.13	0.03	Pb	8.1E−3	31.0
Ca	1.75	6.2	Mn	0.02	11	Cd	<2.1	0.06
Mg	0.52	3.1	Mo	<0.002	<0.18	Hg	<8E−5	7.6E−4
S	6.93	3.1	Zn	0.13	467	Na	60,450	42,820
						Cl	64,650	79,990

and lead at the lower pH are likely due to leaching from the experimental set-up. However, even at a pH of 6, it is possible that solids containing these metals could form.

A problem with blackwater digestate is its sodium chloride content, which is due to modern society's high salt consumption. The sodium chloride content could be problematic for fertilising in arid areas or when used for hydroponic cultivation. The sodium concentrations in the final concentrates are comparable to those reported for Aurin [39]. In contrast, the chloride content, expressed as a percentage of total mass, is approximately 50% lower in the present study. A different research group has studied nitrified urine with a MBR followed by distillation and activated carbon, which also reported high concentrations of sodium chloride. Since K^+ is an essential macronutrient for plants while Na^+ is non-essential for most crops and can interfere with K^+ nutrition, the K/Na ratio is a practical indicator of fertiliser quality for plant uptake [40]. Table 4 compares potassium (K) and sodium (Na) ratios for the urine fertiliser Aurin, fecal compost, MBR-nitrified urine treated with activated carbon, pH-adjusted blackwater digestate (pH 6 and pH 2.8) evaluated in the present study, as well as the concentrates produced at the same pH values. Across all products, the K/Na ratio falls within a relatively narrow range (0.69–0.95). The blackwater digestate exhibits K/Na ratios of 0.89 (pH 6) and 0.78 (pH 2.8), which are comparable to or higher than those of Aurin (0.84) and the nitrified urine product (0.69). Since elevated Na relative to K can increase salinity stress and antagonize K acquisition in plants, a comparable or higher K/Na ratio indicates that the blackwater digestate provides K with no greater Na-associated constraint than the reference excreta-derived fertilisers. In this respect, the blackwater digestate product is competitive with established urine-based and compost-based alternatives [41,42]. To prevent over-fertilisation in Sweden, the maximum allowable application rates for organic fertilisers are 170 kg nitrogen/ha/year and 22 kg phosphorus/ha/year for [43]. To apply 170 kg nitrogen/ha/yr by the pH 6 concentrate produced at VRF 87, about 2.6 m^3 of concentrate would be needed per hectare and year, which is a smaller volume than would be needed to add 22 kg of phosphorus (18 m^3/ha/yr). The corresponding numbers for the concentrate at VRF 85 and pH 2.8 are 2.2 m^3/ha/yr and 6.5 m^3/ha/yr, respectively. It was expected that nitrogen in the concentrate would be the limiting element for the maximum fertilising rate on agriculture fields, given that nitric acid was used for stabilization.

Table 3 shows how much metal would be added to fields if the concentrate were applied at the maximum nitrogen fertiliser rate allowed. All the metals complied with the regulations except for copper at both pH levels and zinc at pH 2.8, which likely came from the equipment itself. If the materials used in the equipment were replaced, the concentrate would comply with Swedish regulations for fertilisers [43].

Table 3
Calculated metal loads from the concentrate produced at pH 6 and pH 2.8 to agriculture soils at an assumed fertilisation rate of 170 kg N per ha and year in comparison with the maximum metal loads allowed according to regulations (Statens naturvårdsverks författningssamling SNFS 1994:2; MS:72) [43].

Metal	Unit	Metal load at fertilisation rate of 170 kg nitrogen/ha/year		Regulation max
		pH 6	pH 2.8	
Pb	g/ha/year	0.33	19.59	25
Cd	g/ha/year	0.01	0.04	0.75
Cu	g/ha/year	273.03	306	300
Cr	g/ha/year	0.11	0.29	40
Hg	g/ha/year	<0.01	<0.01	1.5
Ni	g/ha/year	1.00	2.53	25
Zn	g/ha/year	8.9	295	600

Table 4
Potassium to sodium ratios in the pH adjusted blackwater digestates and in comparisons to other studies.

Human resource product	K/Na
blackwater digestate pH 6	0.89
blackwater digestate pH 2.8	0.78
Concentrate pH 6.0 VRF 87	0.78
Concentrate 2.8 VRF 85	0.95
Aurin [41]	0.84
MBR nitrified Urine with active carbon [42]	0.69
Fecal compost [41]	0.71

3.5. Micropollutants

The concentrations of the 150 analysed micropollutants can be found in the data repository (SND National Swedish database), and 14 of the compounds analysed in the two composite feed and final concentrate samples are shown in Fig. 7. The detection limits were in many cases high because of the complex matrix of the blackwater digestate and the final concentrate. Due to the high concentrations in the concentrate, it was necessary to dilute the samples 10 to 79,000 times before analysis, which increased the uncertainties of the analyses. The micropollutant concentrations of the 31 compounds quantified (SND National Swedish database) did not differ significantly across the different pH levels in either the feed or the concentrate (Mann-Whitney, $P = 0.086$). This is true even though extra protons in the solution at the lower pH slightly modified some of the micropollutants, thus making it harder to detect them as the spectra of a mass spectrometer [44]. There was a significant difference between the feed and concentrate at pH 6 and at pH 2.8 (Mann-Whitney $p = 0$).

The concentrate's matrix was more complex than the matrix of the feed, and the number of micropollutants quantified at pH 6 decreased by 13, whereas the number of micropollutants detected in the feed and concentrate at pH 2.8 remained at 43. Regardless, the micropollutants in the blackwater digestate and concentrate may require a different method in order to analyse the concentration more precisely. For example, the concentrations of benzathine in both feeds were < 50 µg/L, while in the concentrate at pH 6 < 150 µg/L and at pH 2.8 < 500 µg/L are very high for the analysis. For instance, other methodologies could be tested, such as Ultra-High-Performance Liquid Chromatography (UHPLC) coupled with high-resolution mass spectrometry (HRMS), which has proven its efficacy with wastewater. This method could be tried with blackwater digestate along with a novel solid phase extraction technique [45].

Ciprofloxacin and tetracycline were detected in the feed at both pH levels, while trimethoprim was only found in the feed at pH 2.8. In the concentrate, ciprofloxacin was present at both pH levels, tetracycline was only detected at pH 6, and trimethoprim was found at pH 2.8 (Fig. 7). Additional compounds evaluated were candesartan, irbesartan and metoprolol (blood pressure regulators), carbamazepine, citalopram and venlafaxine (neurologic or psychiatric disorders drugs), ibuprofen and diclofenac (commonly used non-steroidal inflammatory drugs), hydrochlorothiazide (a diuretic), and estradiol (a hormone). The concentration at pH 6 of some micropollutants, such as caffeine and candesartan, presented concentrate-to-feed ratios similar to non-reactive ions such as sodium or nitrate. In contrast, other micropollutants such as carbamazepine and metoprolol had concentrate-to-feed ratios more similar to phosphorus or cobalt. Irbesartan and estradiol had very low concentrate-to-feed ratios, with values of 2 and 6, respectively. At pH 2.8 the only micropollutants that have a concentrate-to-feed ratio. Like non-reactive ions were Hydrochlorothiazide, Venlafaxine and Trimethoprim, while diclofenac was concentrated only 3 times. Interestingly in domestic mix wastewater diclofenac can be found up to 7.1 µg/L [46], while the values on the composite feed at pH 6 were 46 and 3.6 µg/L at pH 2.8 µg/L. Additionally, estradiol was measured below 13.4 ng/L in domestic wastewater [47], and the analysis of the composite feed at pH

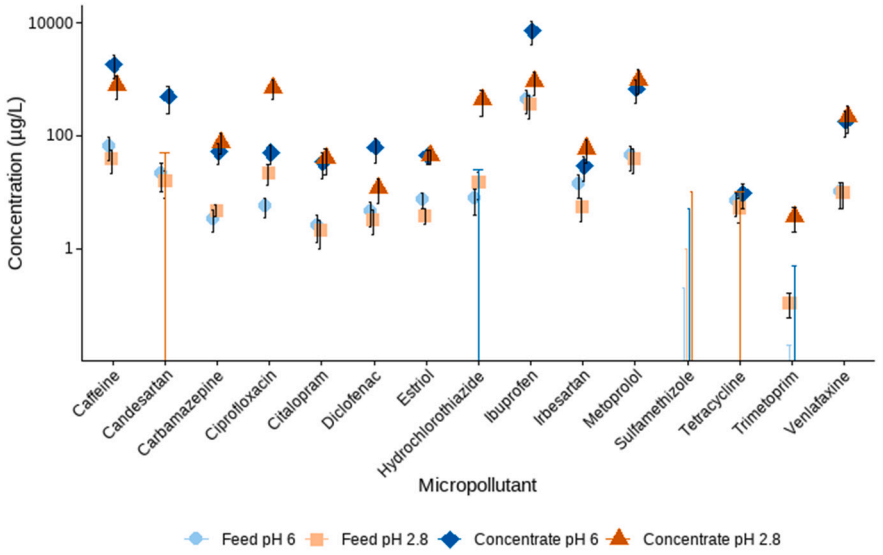

Fig. 7. Concentrations of micropollutants analysed in the feed and concentrate at the two studied pH levels (pH 2.8 and 6). The black error bars indicate the uncertainty of the chemical analysis, while the coloured bars indicate no detection with their limit of quantification.

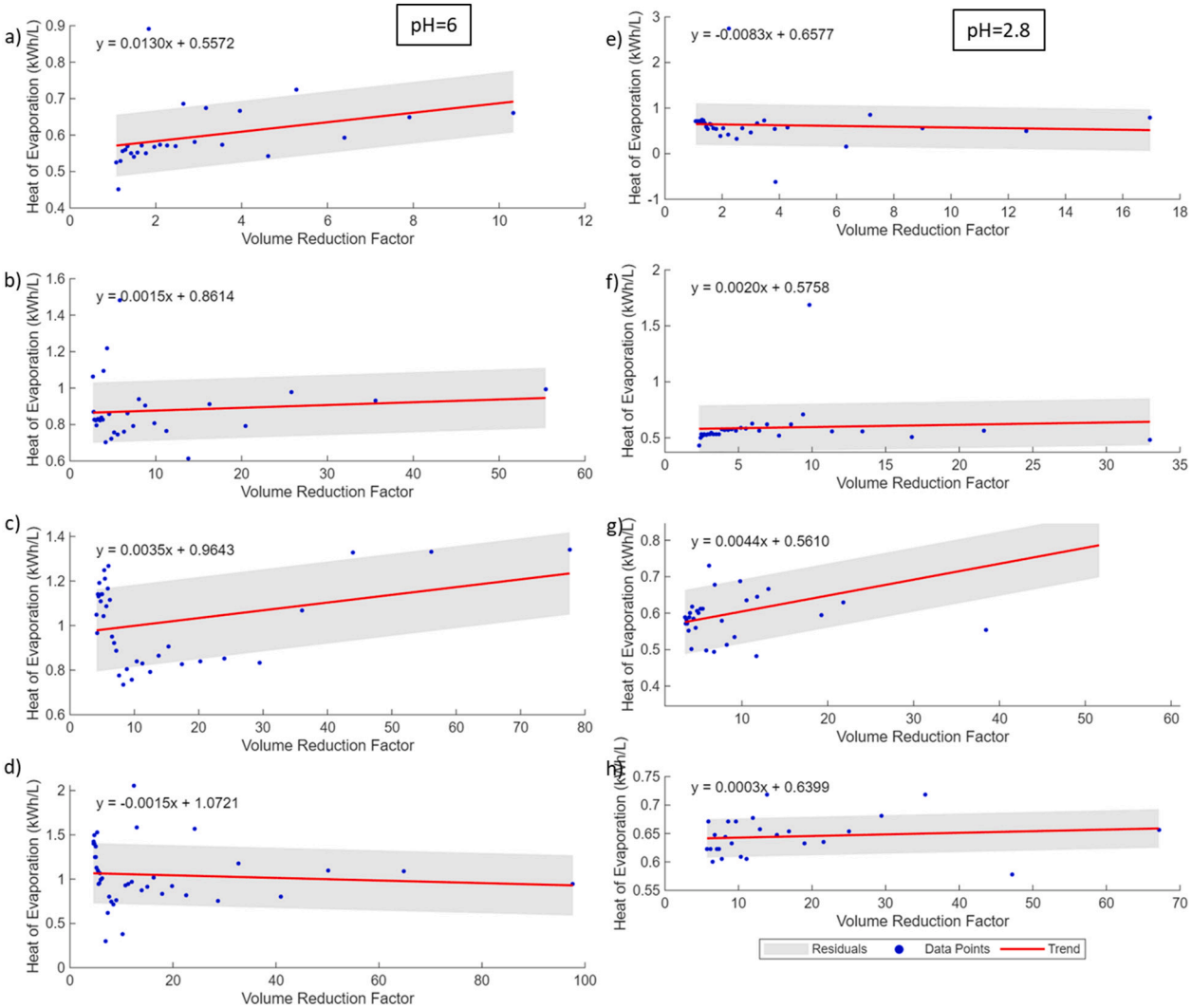

Fig. 8. Energy required to evaporate 1 l of blackwater digestate at different VRF. Panels a–d show experiments at pH 6; panels e–h show experiments at pH 2.8. Final VRF values are indicated in the figure. Data points were recorded every minute, with hourly intervals used for analysis. Trendlines represent linear regressions, and residuals are expressed as standard deviations. Final VRF is defined as the ratio of total feed added to the remaining volume at the end of the evaporation process.

6 was 7.3 μg/L and 3.9 μg/L. These concentrations are likely to remain stable over time because estradiol is a widely used in hormone replacement therapy [46]. Diclofenac treatments, however, can vary from short-term to long-term depending on whether the conditions are chronic [48]. It is expected that the concentration of estradiol will maintain steady in blackwater, whereas diclofenac levels may fluctuate. In contrast, antibiotics are typically used for short durations and may show varying concentrations.

The treatment of pharmaceuticals may be relevant if the blackwater digestate is going to be concentrated since the concentration of pharmaceuticals and potentially PFAs may be accumulated in the concentrate. Some possible post or pretreatments could be nanofiltration, activated carbon, electrodialysis and activated carbon [49,50].

3.6. Energy use

Fig. 8 shows a linear estimation of the SEC based on the measured power, illustrating the energy required to evaporate one litre of blackwater digestate at different VRF. Fig. 8a–d correspond to experiments conducted at pH 6, while Fig. 8e–h represent experiments at pH 2.8. Data points were recorded every minute, but the analysis was performed at hourly intervals. The trendline represents a linear regression, and residuals are expressed as standard deviations. The final VRF is defined as the ratio of the total volume of the feed added to the remaining volume at the end of the evaporation process. The data points, representing each hour of operation, were obtained from Eq. (6). The SEC for six out of the eight batches tended to increase with the volume reduction factor. This phenomenon can be explained by the solute's colligative properties since an increase in the solute concentration led to a higher boiling point and lower vapour pressure regardless of the solute's nature [23]. Leakage of the concentrate into the condensate slightly increases the volume of the condensate, which is mistakenly considered as evaporated water. This can lead to a less pronounced linear regression compared to the actual SEC for the blackwater digestate. Fig. 8 shows some negative heat values due to the volume differences during operation. The sloshing of the concentrate and condensate caused fluctuations, which led to negative volume readings from the sensor. Additionally, there are more data points at the beginning of the graph than at the end due to the volume reduction factor. However, more studies are needed to understand how the process uses energy. Theoretically, 0.67 kWh are required to evaporate 1 l of water at 60 °C [51]. In Fig. 8, the y-intercept ranges from 0.55 kWh to 1.06 kWh, indicating that at the start of each batch, the SEC was close to the theoretical value. The low values of the intercepts can be due to the interaction between the hydrogen bridges decrease slightly in saline solutions at a given temperature [52].

Although the SEC tends to increase with VRF, the slope of the regression line is relatively small, suggesting that the effect is not strongly pronounced. This may be due to an overestimation of water transfer to the condensate side, as well as the nature of VRF, which results in a higher density of data points at the beginning of the curve, that potentially underestimating the influence of VRF. Additionally, the differential method used to calculate volume reduction introduced significant noise into the data. The power required for heat application during the experiment at pH 2.8 was 18.7 kW, while at pH 6.0 it was 15.5 kW. This difference influenced the SEC between the two conditions. The variation in power demand was primarily due to the larger volumes used in the first three batches at pH 2.8 compared to those at pH 6.0. As a result, the final batch at pH 2.8, which was the most concentrated, required significantly less volume, thereby affecting the overall energy needed to reach a volume reduction factor (VRF) of 85.

On average, the SEC was 938 kWh/m^3 of feed at pH 6 and 768 kWh/m^3 of feed for pH 2.8. These values can be compared to those obtained via desalination using vapour compressor distillation. Without heat recovery, it requires 722 kWh/m^3, whereas with heat recovery, it only requires between 5 kWh/m^3 and 42 kWh/m^3, thus obtaining an 85% energy recovery rate [39]. Using multistage evaporators can reduce the minimum energy requirement by up to 20% [53]. In large-scale pulp mills, energy efficiency can be further improved, 20 to 40%, not only by increasing the number of stages, but also by upgrading evaporator vessels or implementing higher solids firing in recovery boilers [54]. However, this last strategy is not applicable for the concentration of blackwater digestate, since it is not suitable, as it is not suitable for recovery boiler firing and contains inorganic components that impose operational limitations at high solids concentrations. In the evaporation process tested in this study, heat recovery from the generated steam could be implemented, and a condenser could replace the use of cold tap water for cooling. This would significantly reduce the energy required for evaporation, thereby improving the overall system efficiency [55]. For comparison with ammonia stripping, the literature reports SEC values of 5–20 kWh per kg NH_4–N recovered across stripping, electrokinetic or hybrid configurations [55]. However, estimating the energy use by SEC per kg NH_4–N recovered is not directly applicable for the studied process, since stripping is nitrogen-selective, while our process is based on evaporation-driven water removal (best benchmarked as kWh/m^3). In the fourth batch of the present study, the measured volumetric SEC was 0.65–1.0 kWh/L distillate (650–1000 kWh/m^3). For comparison, Vondra et al. [56] reported heat consumption for vacuum evaporation of liquid digestate across feed dry-matter levels of about 2–6% and concentrate dry-matter up to 16%, and presented a technology ranking where forced-circulation evaporation had the highest heat demand, while multi-stage falling-film and multi-stage flash were substantially lower due to staging and improved heat recovery. Since the final concentrate in the present study reached about 32% dry matter, it exceeds the concentration range considered by Vondra et al. [59]; therefore, the comparison is only indicative. Within this limitation, the reported SEC in the present study is most consistent with forced-circulation vacuum evaporation and higher than multi-stage falling-film/flash benchmarks, underscoring the strong influence of evaporator configuration and heat integration on SEC. Notably, the system used in this study did not include heat-recovery integration (e.g., multi-effect operation or vapour recompression), contributing to the observed a higher SEC relative to staged/heat-recovered evaporator configurations in the literature.

For a population equivalent of 10,000 people, the daily production of blackwater digestate is estimated to range between 80 and 110 m^3/day [57]. To concentrate this volume to a Volume Reduction Factor (VRF) of 50, the energy required, assuming no heat recovery, would range from approximately 68 MWh/day to 93 MWh/day.

In 2021, it was estimated that 620 exajoules (EJ) of heat energy were used around the world [58]. However, over 60% of this energy was wasted, primarily due to heat loss. Of this wasted energy, 66% was at low temperatures below 200 °C. Therefore, the potential exists to use low-grade waste heat as an energy source for operating the evaporator. However, the availability of this energy is site-dependent. Since the evaporator was studied for the concentration of blackwater digestate, energy integration with the anaerobic digester may be a potential source.

4. Conclusions

This study investigated the nutrient concentration process of blackwater digestate with a pilot-scale low-temperature evaporator.

At pH 2.8, it was possible to retain significantly more ammonium, phosphorus, calcium, and magnesium. The mineral losses were accumulative, so at a higher volume reduction factor, the mineral losses increased. The most critical losses were of phosphorus, magnesium, and calcium at pH 6, probably due to precipitation inside the evaporator. The retention of ammonium was also slightly favoured by a lower pH, while sulphur possibly released into the atmosphere during the pH adjustment.

The concentrate obtained had an elemental mass percentage of N:P:K equalling 60:5:12 at pH 6 and 67:15:12 at pH 2.8. Elements of concern

such as As, Al, Pb, Cd, and Hg were present in low concentrations and are under the limits established by the European Union on dry matter and the Swedish regulations regarding the amount of kg/ha/year at a pH between 6 and 2.8, with particular attention to testing at around pH 4 in order to ensure that phosphorus and other nutrients are retained without causing corrosion to the equipment since corrosion can lead to the release of undesirable metals.

The total energy required including electricity, and the cooling and heating systems was 938 kWh per cubic meter of feed at pH 6 and 768 kWh for the experiment at pH 2.8 [55,59].

CRediT authorship contribution statement

Stephanie Rusch Fehrmann: Writing – review & editing, Writing – original draft, Software, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Hamse Kjerstadius:** Writing – review & editing, Supervision, Resources, Methodology, Funding acquisition, Formal analysis, Conceptualization. **Inga Herrmann:** Writing – review & editing, Visualization, Supervision, Investigation, Formal analysis, Conceptualization. **Annelie Hedström:** Writing – review & editing, Visualization, Validation, Supervision, Methodology, Formal analysis, Data curation, Conceptualization. **Andreas Johansson:** Writing – review & editing, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Elisabeth Kvarnström:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Project administration, Conceptualization.

Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this work, the author(s) used Grammarly and Windows Copilot to check language. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the published article.

Declaration of competing interest

The authors declare that they have no conflicts of interest about the research project.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jwpe.2026.109660.

Data availability

The data has been sent to the national database of Sweden and will be open to the public

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