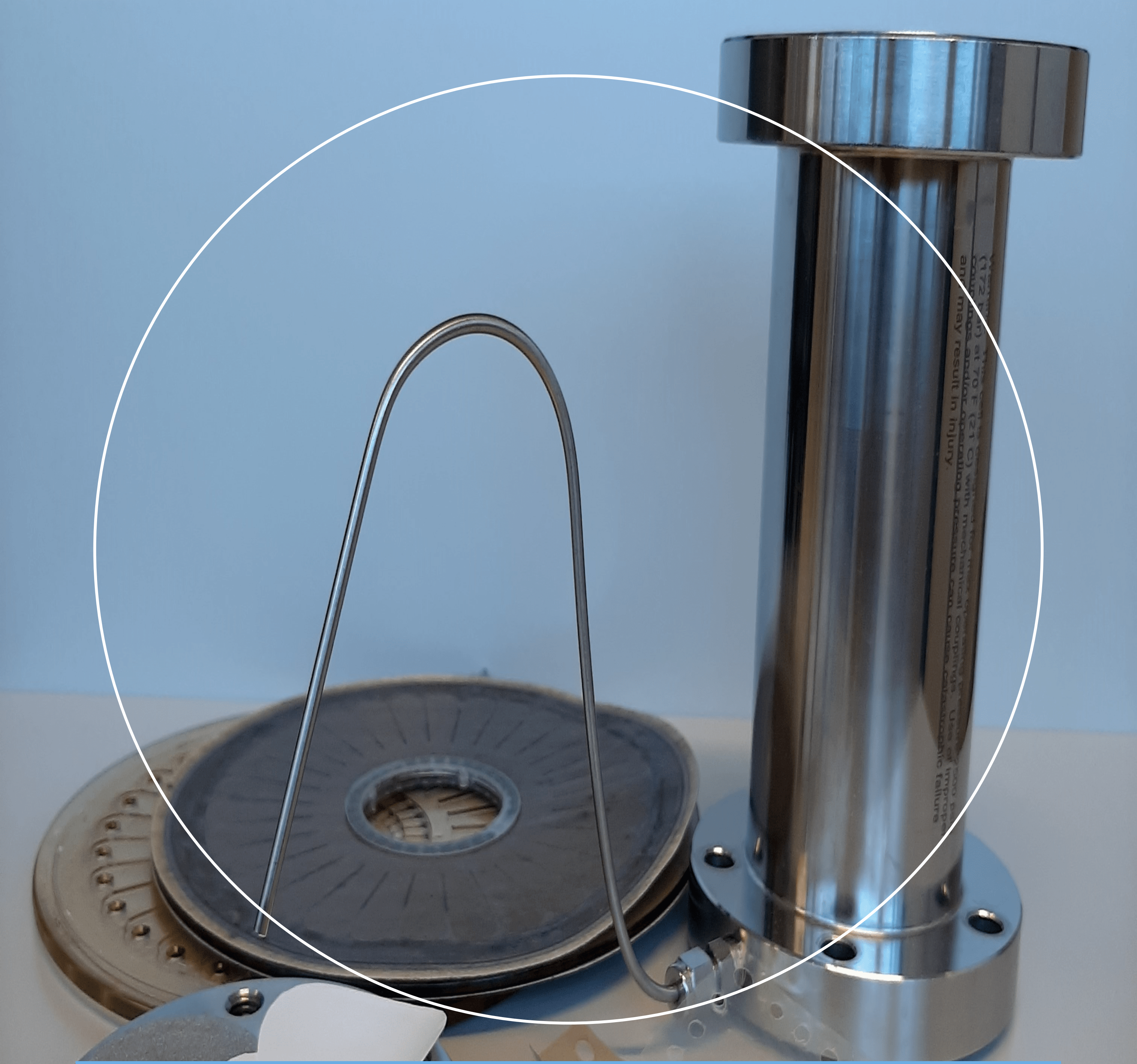

Ammonium recovery from residual aqueous streams

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Authors: T. Mubita[1], W. Appelman[1], L. van Dijk[2], C. Meerman[2], T. van den Eijnde[3], T. Opschoor[4], N. van Linden[4], O.F. Schoumans[5]

[1] Wageningen Food & Biobased Research
[2] Colubris Clean Tech
[3] Nijhuis Saur Industries
[4] Lenntech
[5] Wageningen Environmental Research

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PO box 17, 6700 AA Wageningen, The Netherlands, T + 31 (0)317 48 00 84, E info.wfbr@wur.nl, www.wur.eu/wfbr.

Colophon

Title	Ammonia recovery from residual aqueous streams
Author(s)	T. Mubita (WFBR) W. Appelman (WFBR) O. Schoumans (WEnR) L. van Dijk (Colubris Clean Tech) C. Meerman (Colubris Clean Tech) T. van den Eijnde (Nijhuis Saur Industries) T. Opschoor (Lenntech) N. van Linden (Lenntech)
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Contents

Preface

Nitrogen recovery is becoming an important step in increasing the lifetime of ammonium as a source for food production and in the reduction of the amount of artificial mineral fertilizers. One of the themes in the public-private-collaboration project BIOVALOR (Valorisation of biomass streams; TKI project code LWV20.119), which is funded by Topsector Agri&Food, is nitrogen recovery from organic residual flows and industrial wastewater as a potential source of ammonium. This study was carried out by Wageningen Research (WEnR/WFBR), Lenntech, Nijhuis Saur Industries, and Colubris Clean Tech in the period 2021-2022.

Abstract

This report contains the main findings of studies carried out by different partners in the context of the TKI project: Valorization of Biomass (Biovalor). The studies were focused on evaluating and testing state-of-the-art technologies for ammonium (NH_4^+) removal and recovery from different streams: organic residual flows and industrial wastewater. These streams consisted of digester supernatants, i.e., the liquid fraction of digestates, with and without pretreatment, produced at Attero, Loonwerkersbedrijf, van Amstel, Groot Zevert, and Cosun Beet Company.

One of the evaluated technologies was transmembrane chemisorption (TMCS), which is used for the recovery of NH_4^+ in the form of high-purity ammonium salts, e.g., $(NH_4)_2SO_4$. During TMCS ammonia (NH_3) is stripped from the feed stream and recovered in an acid solution as fertilizer. The complexity of the digestates' matrix—the presence of organic compounds and dissolved salts, among other compounds—in which NH_3 is dissolved greatly influences the recovery efficiency of the TMCS process. Therefore, often pretreatment processes are required to efficiently recover NH_4^+.

The application of pressure and electrically driven technologies as pretreatment for TMCS were evaluated. Electrocoagulation was evaluated to improve the quality of digester supernatants by removing COD before membrane filtration processes. Additionally, microfiltration, high-pressure nanofiltration, conventional electrodialysis, and electrodialysis with bipolar membranes were evaluated for the removal of NH_4^+.

1 Introduction

Digestates produced during fermentation and anaerobic digestion of organic feedstocks and waste streams, such as animal manure and food waste, are commonly nutrient-rich liquids, and hence a good source of carbon, phosphorus, and nitrogen compounds. Of special interest, within the scope of the present study, are the liquid fraction of digestates, often called digester supernatants, of manure and residual waste streams as a nitrogen source. Nitrogen (N) compounds, mostly ammonium (NH_4^+) in these streams, are a viable alternative to producing sustainable/biobased mineral fertilizers as a substitute for synthetic mineral fertilizers (mainly produced by Haber-Bosch technology).

Recently (2022), the European Commission has redrawn the Dutch derogation regarding the nitrogen application as manure of 230 - 250 kg N per ha per year for grassland of dairy farms. In the period 2023 – 2026 the maximum manure application standard will be reduced to 170 kg N per ha per year as mentioned in the Nitrates Directive. To meet with the crop requirements more N is to be bought as fertilizer and more manure has to be exported from the farm. However, nitrogen recovery for manure into a mineral fertilizer, like ammonium sulfate, can help to solve this for the farmers. However, it is important that the European Commission accepts the REcovered Nitrogen from manURE (RENURE) criteria soon.

One approach commonly used to recover NH_4^+ consists of firstly applying reverse osmosis (RO) to concentrate nutrients from digester supernatants, and thereafter stripping ammonia—usually by a temperature increase of feed stream—to produce ammonium sulfate/nitrate salts. The main drawback of this approach is that the energy consumption of the technologies involved is high. Additionally, the stripping process is practically feasible and cost-effective at concentrations higher than 3 g/l NH_4^+-N [1]. Currently, electrically driven processes such as electrodialysis offer the possibility of recovering nutrients and producing fertilizers—when coupled with stripping technologies such as transmembrane chemisorption—at lower energy consumption.

1.1 Electrodialysis (ED)

Electrodialysis (ED) is an electrically driven process in which ions are removed from a dilute stream (diluate), as they are transported through selectively permeable membranes, to a concentrated stream where ions are retained [2]. An ED system comprises a stack with alternating cation- and anion-exchange membranes in between two electrodes, i.e., cathode and anode (**Figure 1**).

The major application of ED has been the production of drinking water from brackish water [3]. However, in the last decade, there has been an increasing use of ED in other applications, such as i) desalination of industrial wastewaters [4], ii) separation of organic acids [5, 6], and iii) selective removal of nutrients from various streams, eg., digester supernatant from wastewater treatment plants (WWTPs) [7].

For ammonium (NH_4^+) removal, ED is considered a viable and more sustainable technology compared to conventional technologies such as biological nitrification/denitrification and anaerobic ammonia oxidation (Anammox). Firstly, removal of NH_4^+ with ED has been reported to be less expensive, in terms of capital and operation costs, than nitrification/denitrification and anammox [8]. Secondly, NH_4^+ can be recovered with ED and used for the production of fertilizers, whereas with the other technologies, NH_4^+ is converted to dinitrogen gas, and hence N is lost to the environment.

With ED systems using selective membranes, e.g., monovalent selective, ions can be not only removed but also fractionated into two streams i) one enriched with multivalent ions, e.g., phosphate (PO_4^{3-}), sulfate (SO_4^{2-}), calcium (Ca^{2+}), and magnesium (Mg^{2+}), and ii) another one enriched with monovalent ions, e.g., NH_4^+, potassium (K^+), and chloride (Cl^-) [9-11].

The technical feasibility of ED as a recovery technology for NH_4^+ has been demonstrated in several studies [10, 12, 13]. However, their practical implementation is still constrained by its power consumption. According to Ward et al. [13], ED needs to have an energy consumption below 10 kWh/ kg NH_4^+-N (36 kJ/g NH_4^+-N) to be considered a feasible solution over the Haber-Bosch process. In ED, energy consumption greatly depends on the feed stream (**Table 1**). In general, it increases as the electrical conductivity of the feed stream decreases, and as ions are recovered during the process.

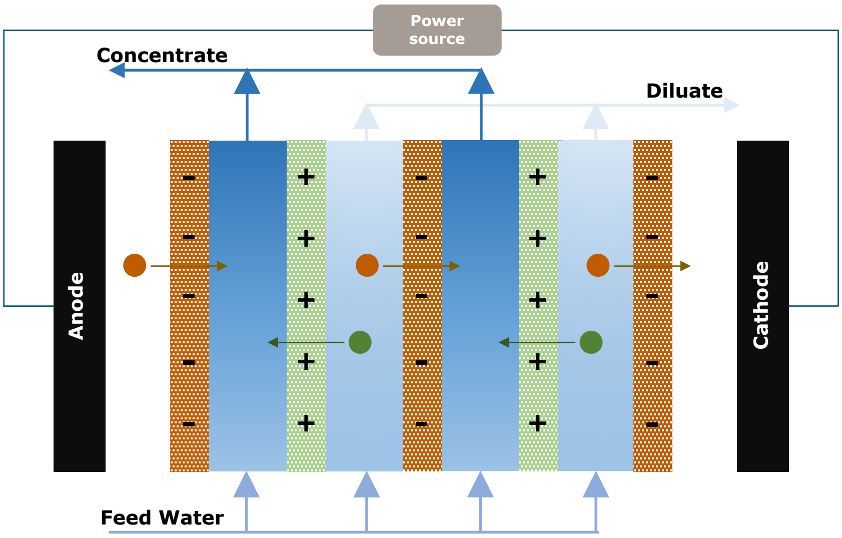


Figure 1. Schematic overview of the electrodialysis principle

1.1.1 Electrodialysis with bipolar membranes (EDBM)

The system configuration of electrodialysis with bipolar membranes (EDBM) includes bipolar membranes (BPMs)—constituted by a cation- and an anion-exchange layer—that allow water dissociation and hence the production of protons (H^+) and hydroxide (OH^-) ions [14]. Three streams are produced in EDBM i) a dilute (desalinated) stream, ii) a base stream, and ii) an acid stream. Cations from the feed stream (dilute stream) are transported to the base stream, whereas anions move to the acid stream.

The potential application of EDBM for NH_4^+ removal has been studied at the lab scale with synthetic waters [15, 16] and the pilot scale with digester supernatant [17].

***Table 1** Energy consumption and other operating parameters of electrodialysis (ED) and electrodialysis with bipolar membranes (EDBM) systems. Information in this table is reported in Ref. [16]*

Study	System	J (A·m²)	Influent/amount N (g/l)	Mode	TAN removal (%)	Energy consumption (kJ/gN)
Desloover et al. [18]	ED	20	Digestate$^{(s)}$~5	C	58	16.7
Tarpeh et al. [19]	ED	100	Urine~4	C	60.6	30.6
Ward et al. [13]	ED	100	Centrate~0.7	C	78	18
Van Linden et al. [15]	EDBM	-	Diluate$^{(s)}$ NH_4HCO_3~ 1.5	B	85	18
Rodrigues et al. [16]	EDBM	100	Wastewater$^{(s)}$ ~2	C	78	18.3

(s) refers to synthetic solution; C and B refer to continuous and batch operation modes, respectively.

1.2 Transmembrane chemisorption (TMCS)

Transmembrane chemisorption (TMCS) is used to remove gas compounds from an aqueous stream by using a hydrophobic membrane. To apply this technology for ammonium (NH_4^+) recovery, dissolved NH_4^+ is transformed into ammonia (dissolved NH_3 gas, i.e. NH_4OH) by raising the pH of the water stream. Thereafter, the resulting alkaline stream is led to the TMCS membrane contactor in which gaseous NH_3 is stripped. A receiving phase, that contains an acid solution, reacts with the NH_3 transported across the membrane forming a salt solution [20]. The TMCS process, therefore, offers the possibility of selective stripping of NH_3 and the production of a high-purity fertilizer. TMCS is also referred to as ammonium membrane stripping (AMS) being developed by Colubris Clean Tech (which took over the developer company Bluetec), one of the partners in the Biovalor project.

However, since TMCS is essentially an integrated stripping process, its efficiency greatly depends on the non-wetting of the membrane, i.e. the prevention of liquid penetration into membrane pores, which only should contain gas. In wetted membranes, the selectivity will dramatically drop and the resistance to gas transport will increase. Various factors can affect the membrane's wettability: operational conditions, e.g., temperature and pressure, the composition of the feed solution, and characteristics of the membrane surface, among others. Adhesion forces within the liquid and the interaction between the membrane surface and liquid (cohesion force) play an important role in determining the membrane's wettability. The surface tension of the liquid is a good measure of the strength of adhesion forces—often, liquids with low surface tension tend to increase the wetting of hydrophobic membrane [21]. A high concentration of organic solutes tends to decrease the surface tension, hence increasing the membrane's wettability. In addition, the interaction membrane-liquid is affected by fouling on the membrane surface increasing membrane's wettability [22].

An important parameter to measure the membrane wettability is the Liquid Entry Pressure (LEP), which is the hydrostatic pressure that needs to be applied to the system before liquid starts flowing through the dry pores of the membrane. The LEP is correlated to the surface tension of the liquid (γ_L), the contact angle of the liquid on the membrane surface (θ), and the size and shape of membrane pores. A mathematical expression for LEP is given below

$$\boldsymbol{LEP = \frac{-2\, B\, \gamma_L\, cos(\theta)}{r_{max}}} \qquad \text{Eq. 1}$$

where B is a dimensionless factor that accounts for irregularities in the membrane pores (with B = 1 for cylindrical pores), and r_{max} is the maximal pore radius.

Throughout the different studies presented in this report, membrane wettability is assessed by measuring either θ, LEP, or γ_L.

2 The study

The recovery of NH_4^+ from organic residual flows and industrial wastewater requires the integration of several technologies. Achieving high removal/recovery efficiencies often depends on improving the quality of the stream to be treated by, for instance, i) reducing COD to decrease fouling on membranes, ii) removing solids to avoid clogging, and iii) increasing NH_4^+ concentration to make the recovery/removal more cost-effective, among others.

In the present study, we evaluate various state-of-the-art technologies, i.e., electrocoagulation (EC), ultrafiltration (UF), nanofiltration (NF), electrodialysis (ED), and electrodialysis with bipolar membranes (EDBM), for the removal and recovery of NH_4^+. Some of these technologies, e.g., EC, are used as pretreatment to improve the quality of digestates before applying membrane filtration processes, whereas other technologies, such as ED and EDBM, are used as pretreatment for transmembrane chemisorption. These technologies were used to treat a variety of feed streams, all of which came from fermentation processes of different organic wastes.

2.1 Methods

The evaluation of the technologies was divided among four different partners of the TKI Project Biovalor as follows:

a. Nijhuis Saur Industries used an electrocoagulation process using iron and aluminum electrodes as pre-treatment for ultrafiltration. The feed stream for EC was
    - Attero's liquid fraction of digestate after press treatment.

b. Bluetec conducted AMS tests to produce $(NH_4)_2SO_4$ from the following streams:
    - Pretreated reverse osmosis (RO) concentrates from the thin fraction of manure digestates produced at Loonwerkersbedrijf van Amstel and Groot Zevert.
    - Permeate streams of microfiltration and high-pressure nanofiltration from the treatment process of the thin fraction of manure.

c. Wageningen Food & Biobased Research used electrodialysis to remove and concentrate NH_4^+ from:
    - Attero's effluent water from the electrocoagulation pilot unit and
    - Cosun's liquid fraction of digestate from Cosun.

The work carried out by Bluetec and WFBR looked into how effective the pretreatment of digestates with pressure-driven technologies and electrically-driven technologies, respectively, is in decreasing the wettability of hydrophobic membranes commonly used for TMCS processes.

d. Lenntech used electrodialysis with bipolar membranes (EDBM) to produce a high pH liquid stream in which NH_4^+ is converted to NH_3 from:
    - Attero's liquid fraction of digestate after press treatment,
    - Attero's effluent water from the electrocoagulation pilot unit, and
    - Cosun's liquid fraction of digestate.

2.2 Electrocoagulation as pre-treatment for ultrafiltration

Nijhuis Saur Industries treated Attero's liquid fraction of digestate (after press treatment). The study with this stream involved i) the application of electrocoagulation (EC), on a lab-scale system, as pretreatment for ultrafiltration (UF) to reduce fouling in the membrane systems, ii) a UF process to evaluate feasible recovery and short-term scaling and fouling of the membranes, and iii) a projection based on the outcome of the UF process to estimate the performance of a reverse osmosis (RO) process.

Results of the EC tests carried out with two different electrodes, i.e., iron (Fe) and aluminum (Al), are shown in **Table 2**. Higher COD removal was achieved with Al electrodes, about 72%, whereas with Fe electrodes the removal was 63%. The effluents of the EC process, i.e., those generated with Fe and Al electrodes, respectively, were further treated using a lab-scale UF system (**Table 3**). Stable operations and recoveries of about 95% were achieved with both EC-effluents. However, membrane fouling occurred, which is likely due to the high residual concentration of metal ions in the effluents of the EC process.

There is a marked difference in the amount of NH_4^+ removed from the two effluents produced with EC. For the effluent produced with Fe electrodes, the UF process achieved about 82% NH_4^+ removal. On the other hand, for the effluents produced with Al, the removal was about 45%.

Table 2 ***Characteristics of the feed stream and effluent of an EC process using iron (Fe) and aluminum (Al) electrodes***

Parameter	**Feed stream**	**Effluent**		**Removal (%)**	
		Fe	**Al**	**Fe**	**Al**
COD (mg/L)	4030	2530	2910	63	72
pH (-)	8.00	8.4	8.5	-	-
Conductivity (mS/cm)	27.40	21.3	21.9	-	-
Me Dosage (mg/L)	-	1736	839	-	-
Current (A)	-	5.0	5.0	-	-
Voltage (V)	-	5.9	7.7	-	-

From the results obtained with UF, a projection was made to estimate the performance of an RO process. This projection was only carried out for the Fe-treated effluents of EC. Results indicate that the system is not limited by the osmotic pressure or scaling of multivalent cations, and theoretically should achieve recoveries of around 60% at 35 barg. However, it is not recommended, and strongly discouraged, to run a RO process with the EC's effluents mainly due to the high concentrations of Fe in the streams. Therefore, further optimization of the EC process is required such that the residual concentration of metal salts is minimized at maximum COD removal.

Results from the projection also show that the concentrate stream from RO is enriched in NH_4^+ and potassium ions (**Table 4**). Nonetheless, further research is needed to determine a suitable treatment to recover the nutrients of this concentrated stream.

Table 3 ***The concentration of different components in i) the streams used as feed streams for ultrafiltration (UF), i.e., stream before EC, effluents produced with Fe and Al electrodes, and ii) permeate produced after UF****.*

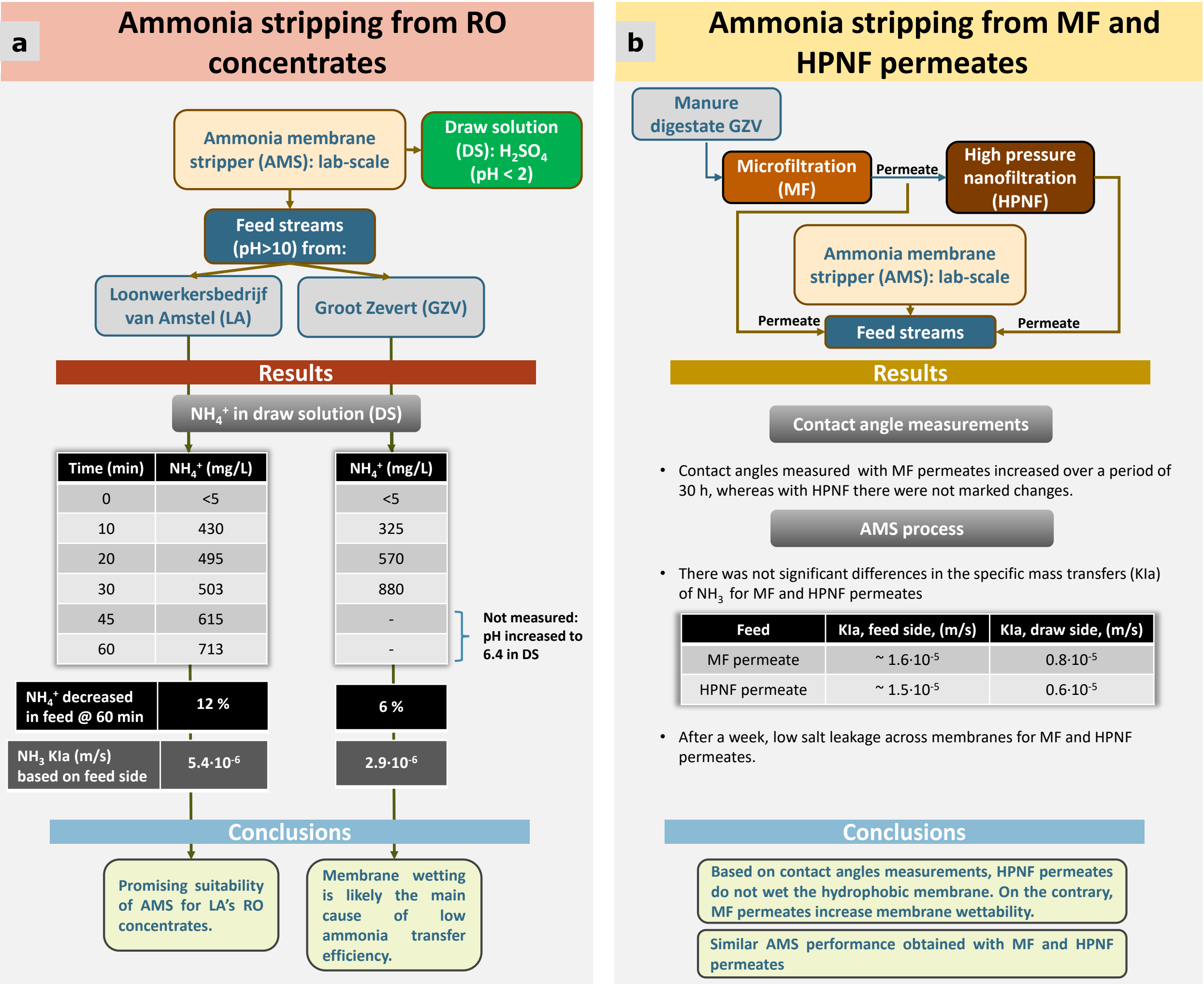
Parameter (mg/l)	Influent (no EC)	Effluent EC with Fe	Effluent EC with Al	UF Permeate (Fe)	UF Permeate (Al)
Sulfide				<0.3	<0.5
Fluoride				0.18	0.07
Nitrogen Kjeldahl		2360	2100	1970	1870
Ammonium-N	2200	2200	2000	1800	890
Nitrate-N		<0.05	<0.05		0.13
Nitrite-N		<0.01	<0.01		0.3
Orto-phosphate-P				0.34	<0.1
Silicate				8.1	2.1
Sulfate				<20	<10
CZV	4030	2530	2910	1720	1640
TSS				<10	<10
TIC	2400		2100	2000	1700
Aluminum				0.06	4.5
Barium				<0.02	<0.02
Boron				0.3	0.3
Calcium		42	8.8	23	4.3
Iron		170	11	5.8	4.5
Potassium				1700	1800
Copper				0.054	0.55
Lead				<0.005	<0.005
Magnesium		49	31	40	25
Manganese				0.18	0.083
Sodium				900	1200
Nikkel				0.46	2.3
Zink				0.035	0.046

Table 4 *Estimated compositions of the streams produced after the RO process. The feed stream for the process was the EC effluent produced with Fe electrodes.*

Parameter (mg/l)	RO concentrate	RO permeate
Sulfide	<1	<0.1
Fluoride	0.5	<0.1
Nitrogen Kjeldahl	4600	250
Ammonium-N	5300	280
Nitrate-N	0	0
Nitrite-N	0	0
Orto-phosfate-P	2.5	<0.05
Silicate	20	0.2
Sulfate	25	<0.1
CZV	4200	100
TSS	0	0
Bicarbonate	24500	200
Aluminium	0.15	<0.05
Barium	0	0
Boron	0.65	<0.2
Calcium	50	0.1
Iron	15	<0.05
Potassium	4200	30
Copper	<2	<0.5
Lead	<0.1	<0.005
Magnesium	100	0.2
Manganese	<0.5	<0.05
Sodium	2500	16
Nikkel	1.3	<0.05
Zink	<0.15	<0.05
Chloride	550	<10

2.3 Pressure-driven technologies as pre-treatment for TMCS

This part of the study was conducted by the company Colubris Clean Tech (Bluetec). Contact angle measurements and TMCS experiments at a lab scale were carried out to evaluate membrane wettability with different feed streams. The TMCS experiments are based on the ammonia membrane stripping (AMS) process as developed by Bluetec. An overview of the experiments, i.e., the tested process streams and main observations, is shown in **Figure 2**.



Time (min)	NH_4^+ (mg/L)
0	<5
10	430
20	495
30	503
45	615
60	713

NH_4^+ (mg/L)
<5
325
570
880
-
-

Not measured: pH increased to 6.4 in DS

	LA	GZV
NH_4^+ decreased in feed @ 60 min	12 %	6 %
NH_3 Kla (m/s) based on feed side	$5.4 \cdot 10^{-6}$	$2.9 \cdot 10^{-6}$

Promising suitability of AMS for LA's RO concentrates.

Membrane wetting is likely the main cause of low ammonia transfer efficiency.

- Contact angles measured with MF permeates increased over a period of 30 h, whereas with HPNF there were not marked changes.

- There was not significant differences in the specific mass transfers (Kla) of NH_3 for MF and HPNF permeates

Feed	Kla, feed side, (m/s)	Kla, draw side, (m/s)
MF permeate	~ $1.6 \cdot 10^{-5}$	$0.8 \cdot 10^{-5}$
HPNF permeate	~ $1.5 \cdot 10^{-5}$	$0.6 \cdot 10^{-5}$

- After a week, low salt leakage across membranes for MF and HPNF permeates.

Based on contact angles measurements, HPNF permeates do not wet the hydrophobic membrane. On the contrary, MF permeates increase membrane wettability.

Similar AMS performance obtained with MF and HPNF permeates

***Figure 2** Schematic overview of the study carried out by Bluetec on membrane's wettability with different process streams.*

2.3.1 Ammonium recovery from pretreated reverse osmosis concentrates

The first set of experiments carried out by Bluetec aimed to remove ammonium (NH_4^+) from pretreated reverse osmosis (RO) concentrates from the thin fraction of manure digestates. The RO concentrates were taken from Loonwerkersbedrijf van Amstel (LA) and Groot Zevert (GZV). For LA's concentrates, the pretreatment consisted of precipitation with iron chloride, flotation, and paper filtration, whereas for GZV's concentrates, it consisted of coagulation with polymer flocculants, centrifugation, and microfiltration.

Contact angle measurements at different times (0 and 4 h) showed that initial values of θ were > 90° and ± 90° with LA's and GZV's concentrates, respectively. After 4 h, θ decreased with both concentrates. However, with LA's concentrates, θ was ± 90°, whereas with GZV's concentrates, it was < 90° (**Figure 3**). Initially, the membrane's surface is hydrophobic for both concentrates (Figure 4), but with GZV's concentrates the surface became hydrophilic over time. For long-term operation, this behavior has a serious impact on the efficiency of the AMS process because the tested membrane is likely to get wetted more easily with GZV's concentrates.

After alkalinization (pH increase to 10 with NaOH solution) of the concentrates, AMS experiments were conducted for 60 min. With LA's concentrates, the process was stable, i.e., no drastic change in the pH of the feed and draw solution (DS). About 750 mg/L of NH_4^+ were removed from the feed, which represents approximately a decrease of 12%. A mass balance showed that not all NH_4^+ ended up in the draw solution: about 5% of the total NH_4^+ removed was likely lost due to leakage in the system.

The AMS process with GZV's concentrates showed to be less stable, sudden increases in pH (from ~2 to ~6.4) were measured in the draw solution after 20 and 35 minutes of running the experiment. Decreasing the pH of the draw solution after 20 min required the addition of a high amount of acid. After 35 min all the measurements were stopped. Overall, the total NH_4^+ removed was about 6%.

To compare the performance of the AMS process for LA's and GZV's concentrates the specific mass transfer coefficient k_L was calculated by

$$\boldsymbol{k_L = J \cdot \frac{\ln\left(\frac{c_0}{c_t}\right)}{c_o - c_t}} \qquad \textit{Eq. 1}$$

$$J = \frac{\dot{m}}{A_m} \qquad \textit{Eq. 2}$$

$$\dot{m} = V \cdot \frac{c_0 - c_t}{\Delta t} \quad for\ V = constant \qquad \textit{Eq. 3}$$

Where k_L is the mass transfer coefficient in m/s, J is the flux in g/m²/s, $\dot{m}$ is mass transfer rate in g/s, A_m is membrane's surface area in m², V is the volume of solution, c_0 and c_t are concentrations at time 0 and at a certain point in time (t).

The k_L values are often used to estimate the total membrane area and flow needed to achieve certain NH_4^+ recovery. **Table 5** shows calculated k_L based on the feed side of the membrane. Overall, for LA's concentrates, results show that recovering NH_4^+ by AMS is possible despite the low k_L**.** However, for GZV's concentrate, AMS is not suitable due to indications of membrane wetting at an early stage of the process.

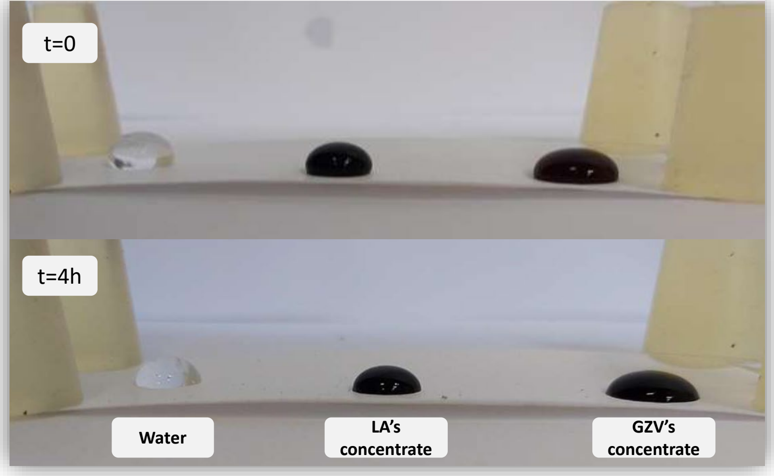


Figure 3 ***Droplets of water and RO concentrates from two different process streams in contact with a hydrophobic membrane at two different times, 0 and 4h****.*

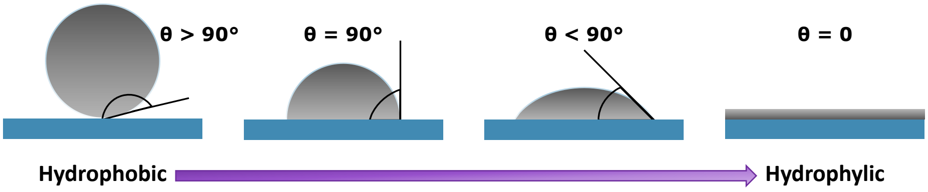


Figure 4. ***Changes in contact angle (θ) with increasing hydrophilicity of the solid surface****.*

Table 5 ***Specific mass transfer coefficient based on the feed side of the membrane obtained with RO concentrates during the AMS process.***

RO concentrate	k_L(m/s)
LA	$5.4\cdot10^{-6}$
GZV	$2.9\cdot10^{-6}$

2.3.2 Ammonium recovery from microfiltration and high-pressure nanofiltration permeates

In the second set of experiments (**Figure 2b**), permeate streams of microfiltration and high-pressure nanofiltration processes were used as feed streams for the AMS system. The permeates came from the manure processing plant located at Groot Zevert (**Figure 5**).

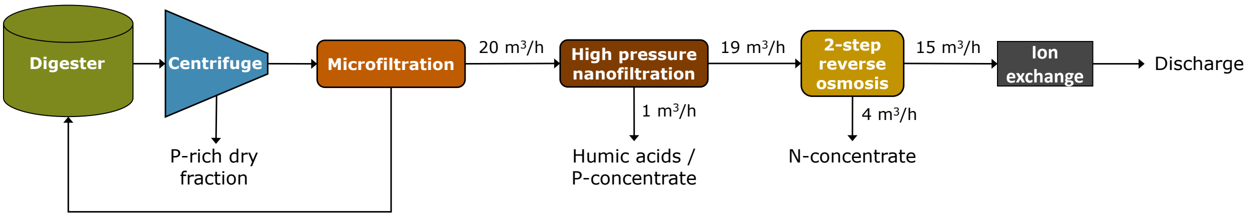


Figure 5 ***Schematic figure of the treatment process of the thin fraction of manure digested carried out at Groot Zevert.***

During the AMS process, membrane performance was similar between the different permeates. There was no marked difference between the MF- and HPNF-permeate in terms of i) the k_L values (**Table 6**), and ii) salt leakage across the membrane after one week of continuous AMS process. However, contact angle measurements showed that the permeates behave differently on the membrane surface, i.e., significant wetting of the membranes with MF permeates but not with HPNF-permeate. A marked flattening of the MF permeate's droplet occurred over time, whereas changes in the HPNF-permeate's droplet are less pronounced **(Figure 6**). From contact angle measurements, it seems that HPNF is a better-suited pretreatment stage for the AMS process.

Table 6 ***Specific mass transfer coefficient for the feed and draw side of the membrane obtained with MF and HPNF permeates during the AMS process.***

Permeate	k_L (m/s), feed	k_L (m/s), draw
MF	$1.6\cdot10^{-5}$	$0.8\cdot10^{-5}$
HPNF	$1.5\cdot10^{-5}$	$0.6\cdot10^{-5}$

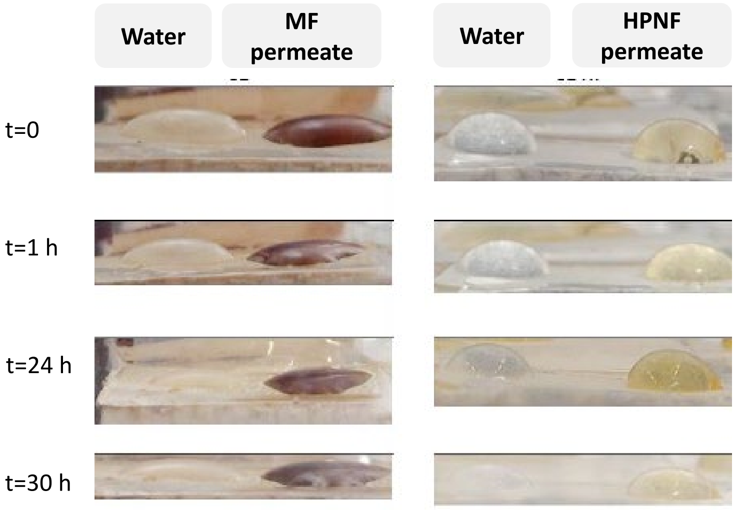


Figure 6 ***Wetting transitions of MF- and HPNF-permeate droplets on the surface of membranes used for AMS.***

Based on the findings of the second set of experiments, Bluetech proposed a process scheme to recover NH_4^+ from the digestate produced at Groot Zevert's facilities (Figure 7). Cost estimates, i.e., capital and operational expenditures (CAPEX and OPEX), for the HPNF and AMS units are shown in **Table 7** and **8**.

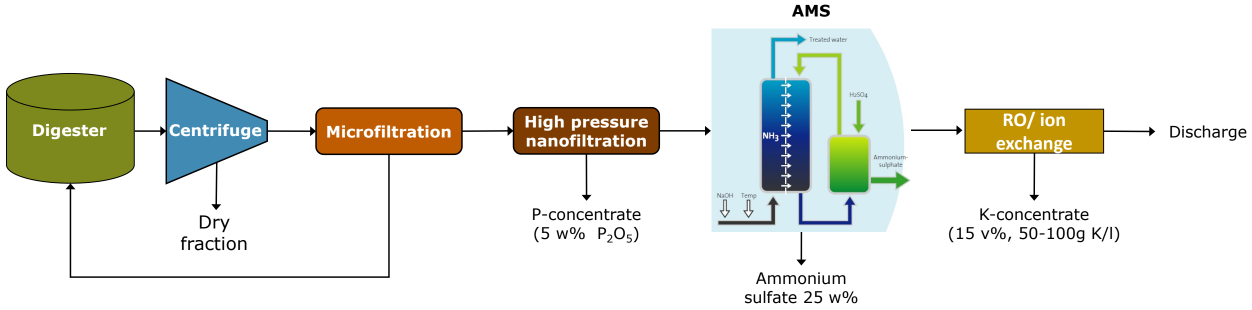


Figure 7 ***Bluetec's proposed process scheme for NH_4^+ recovery from the thin fraction of manure digestate at Groot Zevert facilities.***

Table 7 ***Estimation of capital and operating expenditures (CAPEX and OPEX) for the high-pressure nanofiltration unit (HPNF).***

CAPEX	Cost (€)
Investment	550,000
Inclusive:	
• Delivery complete installation	
• Commissioning	
• Automation	
Exclusive:	
• Connection onsite	
• Integration with existing CIP	
OPEX	**Cost (€) per year**
Energy	21,600
Membrane replacement (2 years)	12,000
CIP and anti-scalant (line item)	35,000
Interest and depreciation	40,900
Total (CAPEX + OPEX)	**109,500 per year (€ 0.78/m³ MF permeate)**

Table 8 ***Estimation of Capital and operating expenditures (CAPEX and OPEX) for the ammonia membrane stripping (AMS) unit.***

CAPEX	Cost (€)
Investment	580,000
Inclusive:	
• Delivery complete installation	
• Dosing stations and storage tanks	
• Commissioning	
• Automation	
Exclusive:	
• Connection onsite	
OPEX	**Cost (€) per year**
Energy	7,900
Membrane replacement (4 years)	35,100
Sodium hydroxide	546,800
Sulfuric acid	458,800
Ammonium sulfate	-196,600
Interest and depreciation	34,500
Total (CAPEX + OPEX)	**786,500 per year (€ 6.75/m³ MF permeate)**

The starting points for the design of the HPNF and AMS unit are based on the following throughputs:

1. HPNF unit

Nominal flow	16 m^3/h
CIP-actions average throughput	14 m^3/h
Hours available for CIP-actions	20 h per week

2. AMS unit

Input parameters

Nominal flow	15.2 m^3/h
NH4-N	4,500 mg/l
Temperature	30 °C
CIP-actions average throughput	1.3 m^3/h
Hours available for CIP-actions	20 h per week

Process conditions

N-removal	75%
Absorbent	96% H_2SO_4
Product	25 w% ammonium sulfate

2.4 Electrically-driven technologies as pre-treatment for TMCS

2.4.1 Electrodialysis to remove and concentrate ammonium

Wageningen Food & Biobased Research (WFBR) was in charge of studying the application of electrodialysis as a pretreatment to improve the quality of the influent stream to TMCS. With ED as pretreatment, it is aimed to not only remove and concentrate NH_4^+ but also produce a concentrated stream with a lower content of non-charged organic solutes compared to the feed stream. The latter aspect is considered because, as mentioned previously, organics solutes tend to decrease the surface tension of liquid streams, hence membrane wettability increases.

Two process streams were treated with ED: i) the effluent water from the electrocoagulation pilot unit from Attero, and ii) the liquid fraction of digestate from Cosun. The ED system to treat these streams consisted of recirculating reservoirs—linear flow velocity in the stack was 5 cm/s—of diluate, concentrate, and electrode rinse solutions. At the start of the experiments, these solutions were a process stream, water, and sodium sulfate 0.5 M, respectively. The ED stack was: i) assembled with five cell pairs of alternating cation- and anion-exchange membranes, and ii) operated in batch mode at constant voltage (15 V).

Liquid entry pressure (LEP) and contact angle measurements were used to evaluate the wetting of two commercially available hydrophobic membranes (**Table 9**) suitable for the TMCS process.

Table 9 Main characteristics of the hydrophobic membranes used for LEP measurements.

Membrane	Material	Pore size (μm)
Solupor PE, Lydall Inc	Ultra-high molecular weight polyethylene	0.32
Sartorius	Polyethersulfone	0.10

2.4.1.1 Attero's effluent from the electrocoagulation process

The study with Attero's stream—as it was received, i.e., no pre-treatment before experiments—was focused on evaluating the performance of two sets of ion-exchange membranes (**Table 10**) for the concentration of NH_4^+ using ED. To that end, different initial diluate to concentrate volume ratios (α=volume diluate/volume concentrate) were used, i.e., α= 2 and 5. The ED experiments were stopped when the conductivity in the diluate compartment decreased by about 95% of the initial value.

Table 10 **Main characteristics of ion-exchange membranes used for the removal of ammonium as reported by manufacturers**

Membrane	Ion-exchange group [23]	Structure [23]	Electrical resistance ($\Omega \cdot cm^2$)*	Application
1st set of tested membranes				
Fujifilm Type II CEM	Sulfonic	Polyamide	8.0	Low water permeating membranes. Mostly used for purifying processes, and wastewater concentration.
Fujifilm Type II AEM	Quaternary ammonium		5.0	
2nd set of tested membranes				
Neosepta CMX	Sulfonic	Styrene-divinyl benzene. Highly crosslinked	3.0	Desalination and concentration of inorganic salts.
Neosepta AMX	Quaternary ammonium		2.4	

* Electrical resistance measured in 0.5 M NaCl

For α=5 at the beginning of the experiments, both sets of membranes, i.e., Fujifilm and Neosepta membranes, had similar performance in terms of NH_4^+ removal and energy consumption (**Table 11**). About 1.8 g of NH_4^+-N was removed from the feed stream, which represents a concentration factor of 2.6 and 2.9 for Fujifilm and Neosepta-membrane (**Table 11**), respectively. The concentration of Na^+, K^+, Ca^{2+}, and Mg^{2+} in diluate and concentrate in the ED system with both types of membranes are shown in **Table S.I.1 and S.I.2**.

For α=7, the total NH_4^+ removed does not differ markedly between the two sets of membranes (**Table 13**). However, with Fujifilm membranes, a lower concentration factor (**Table 14**) was achieved as a result of higher water transport across this membrane compared with that measured across Neosepta-membrane.

Table 11 **Ammonium mass balance and energy consumption after the ED process obtained with two sets of membranes: Fujifilm and Neosepta's membrane, for α=5**

	Fujifilm membranes NH_4^+-N (g)		Neosepta membranes NH_4^+-N (g)	
	Removed from diluate	Measured in concentrate	Removed from diluate	Measured in concentrate
Mass NH_4^+-N	2.1	1.8	2.0	1.8
Energy consumption (kWh/kg NH_4^+-N removed)	17.2		16.8	

Table 12 *Nitrogen-ammonium concentration before and after ED process and NH_4^+-N concentration factor obtained with two sets of membranes: Fujifilm and Neosepta's membrane, for α=5*

	Fujifilm-membranes NH_4^+-N (mg/l)		Neosepta-membranes NH_4^+-N (mg/l)	
	Diluate *	Concentrate	Diluate *	Concentrate
Initial	897	4.5	868	3.4
End**	53.2	2340	57.6	2490
Concentration factor	2.6		2.9	

*Diluate stream before ED: pH=8.80. ** Operation time with Fujifilm membranes was 210 min and with Neosepta-membranes was 180 min.

Table 13. ***Ammonium mass balance and energy consumption after the ED process obtained with two different sets of membranes: Fujifilm and Neosepta's membrane, for α=7***

	Fujifilm membranes NH_4^+-N (g)		Neosepta membranes NH_4^+-N (g)	
	Removed from diluate	Measured in concentrate	Removed from diluate	Measured in concentrate
Mass NH_4^+-N	2.0	1.7	2.0	1.9
Energy consumption (kWh/kg NH_4^+-N removed)	17.4		16.0	

Table 14 ***Nitrogen-ammonium concentration before and after the ED process and NH_4^+-N concentration factor obtained with two sets of membranes: Fujifilm and Neosepta's membrane, for α=7***

	Fujifilm-membranes NH_4^+-N (mg/l)		Neosepta-membranes NH_4^+-N (mg/l)	
	Diluate *	Concentrate	Diluate *	Concentrate
Initial	854	2.9	861	3.7
End**	47.9	2160	46.8	3000
Concentration factor	2.5		3.5	

*Diluate stream before ED: pH=8.80. ** Operation time with Fujifilm membranes and Neosepta-membranes was 180 min.

With regards to wettability, hydrophobic membranes Solupor and Sartorius behave similarly. Results indicate that wetting is reduced with ED-concentrates (L2 & L3 for α=5) compared to that with untreated Atteros' streams (before ED). With ED concentrates, the contact angle with the membrane's surface is higher (**Figure 8**) and the pressure in which liquid starts to significantly flow across the membranes is increased (**Figure 9**).

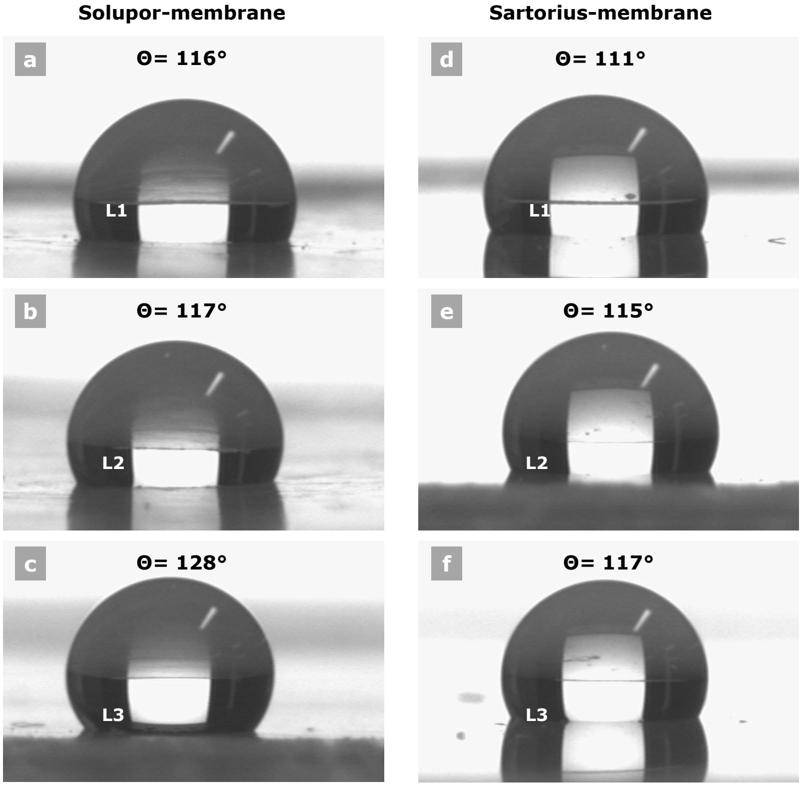


Figure 8 ***Contact angle by sessile drop method of Attero's stream and concentrates produced with electrodialysis (ED) on a-c) Solupor-membranes, and d-f) Sartorius-membranes. L1=Attero's stream before ED, L2 & L3=concentrate stream produced with ED (α=5) with Fujifilm-membranes and Neosepta-membranes, respectively.***

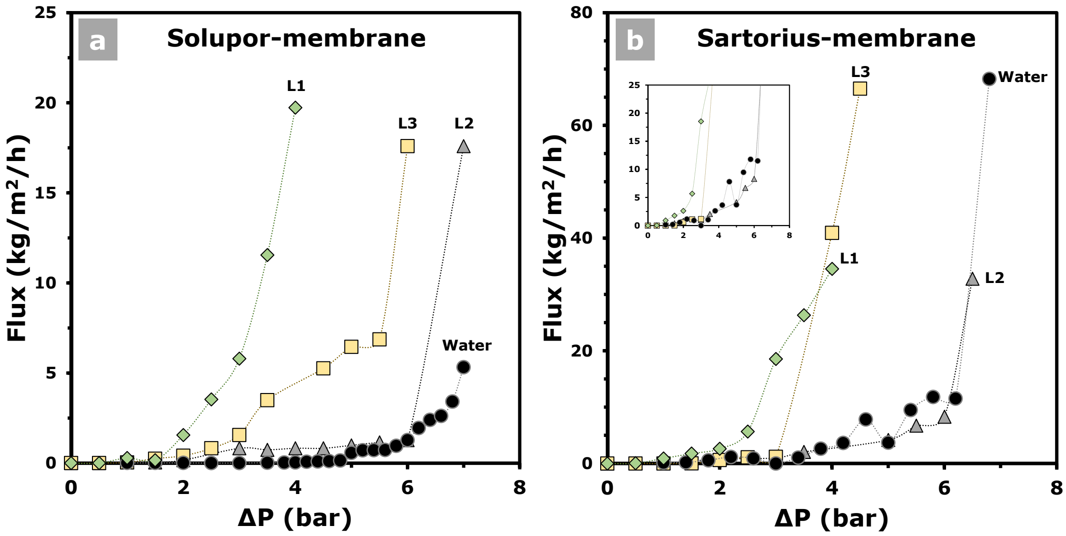


Figure 9 ***Flow of liquid through hydrophobic membranes, a) Solupor and b) Sartorius-membrane, as a function of the applied pressure during liquid entry pressure (LEP) measurements. Data points correspond to the following liquids: i) Water, ii) L1= Attero's process stream before ED, iii) L2 & L3= Concentrate stream produced with ED (ɑ=5) with Fujifilm and Neosepta membranes, respectively. Dotted lines are to guide the eye. LEP measurements were performed using a stirred cell (Sterlitech HP4750X) that can hold a membrane with an area of 14.6 cm^2.***

2.4.1.2 Cosun's effluent stream

The liquid fraction of digestate produced at Cosun was also treated with ED to concentrate NH_4^+. Before ED experiments, large particles were removed by centrifugation (17000xg for 15 min.) The supernatant produced was used as the feed solution for the ED assembled with Neosepta AMX and CMX membranes (

Table 10). The initial volume ratio diluate to concentrate (ɑ) was 5. Electrodialysis achieved 72% ammonium removal and a concentration factor of this ion of 2.6 (**Table 15**). The concentration of other ions, i.e., Na^+, K^+, Ca^{2+}, and Mg^{2+}, is shown in **Table S.I. 3**. The energy consumption of the process was 20 kWh/kg NH_4^+-N.

The wetting study through liquid entry pressure (LEP) measurements showed that concentrates produced with ED increased the wettability of Solupor-membranes, but did not cause any marked change in Sartorius-membranes wettability compared to Cosun's stream before ED (**Figure 10**). To minimize membrane wetting, the pressure difference in systems with Solupor and Sartorius-membranes should be kept below 3 and 2 bar, respectively.

Table 15 ***Nitrogen-ammonium concentration before and after the ED process and NH_4^+-N concentration factor obtained with Cosun's stream as feed solution***

	Neosepta-membrane			
	Diluate		**Concentrate**	
	Initial	**Final***	**Initial**	**Final***
EC (mS/cm)	13.9	2.6	0.2	35.2
pH	8.1	7.3	9.4	8.0
NH_4^+-N (mg/l)	1070	295	5.5	2760
NH_4^+-N removed from diluate (g)	2.0			
NH_4^+-N measured in concentrate (g)	1.6			
Concentration factor	2.6			
Energy consumption (kWh/kg NH_4^+-N removed)	20.0			

* Operation time= 210 min

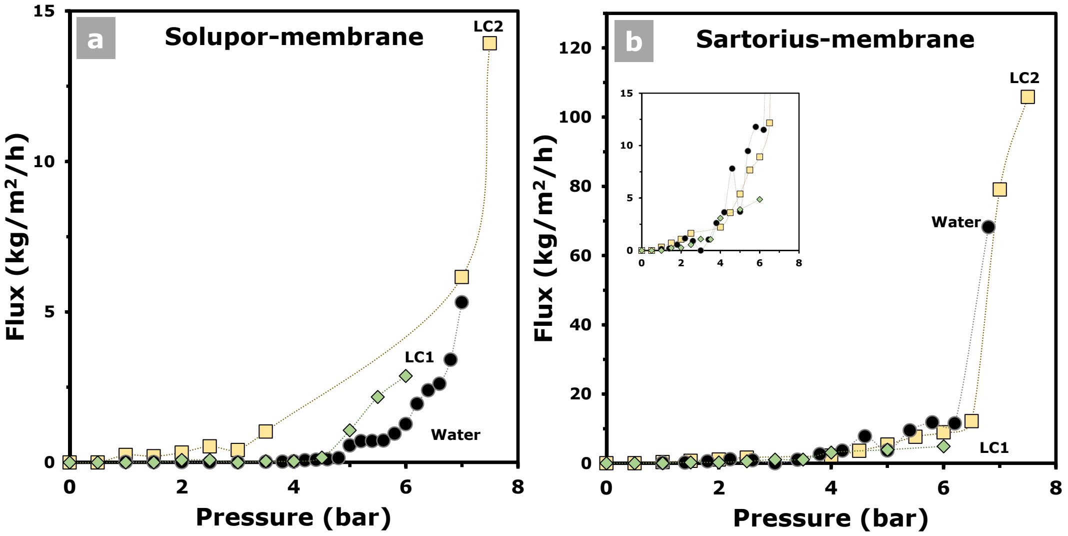


Figure 10 Flow of liquid through hydrophobic membranes, a) Solupor and b) Sartorius-membrane, as a function of the applied pressure during liquid entry pressure (LEP) measurements. Data points correspond to the following liquids: i) Water, ii) LC1= Pretreated (centrifugation) Cosun's stream before ED, iii) LC2= Concentrate stream produced with ED (α=5) with Neosepta membranes. Dotted lines are to guide the eye. LEP measurements were performed using a stirred cell (Sterlitech HP4750X) that can hold a membrane with an area of 14.6 cm².

2.4.2 MEZT-EDBM process

In this part of the study, Lenntech applied the MEZT-EDBM process, on a lab scale, to remove total ammoniacal nitrogen (TAN) from three rejected waters: i) Attero's digestate after press treatment (Stream At1), ii) Attero's digestate after electrocoagulation (Stream At2), and iii) Cosun's digestate after decantation (Stream Co1). All three rejected water were filtered with 25 μm filter bags before the EDBM experiments. With the MEZT-EDBM process (Figure 11), NH_4^+ is recovered in a solution with high pH (> 9.0) to obtain dissolved ammonia gas. The electrodialysis system was operated in batch mode and experiments were run until the electrical conductivity (EC) of the feed water decreased by at least 70%.

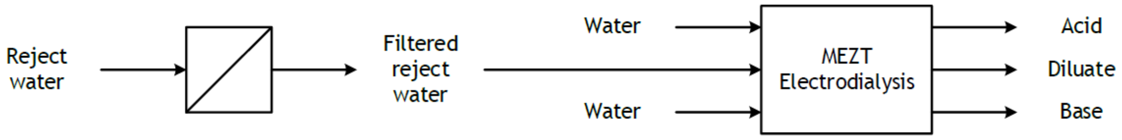


***Figure 11** Schematic overview of the MEZT-EDBM process.*

Tables 16-18 show the NH_4^+ concentration in each of the treated feed streams at the beginning and end of the MEZT-EDBM process, and in the produced streams, i.e., acid and base. For Atteros's rejected waters, the MEZT-EDBM process achieved a TAN removal from streams At1 and At2 of 87% and 90%, respectively. Water recovery of the process was 95% with stream A1 and 98% with stream A2. The energy consumption for the total process (ion removal and recovery) was calculated to be 25 kWh/kg-N for stream A1 and 34 kWh/kg-N for stream A2. For Cosun's rejected water (Stream Co1), the TAN removal was 73%, while the water recovery is 94%. The energy consumption for the total process (removal and recovery) was 26 kWh/kg-N.

Table 16 *Characteristics of Attero's digestate after press treatment (Stream At1) and produced streams during MEZT-EDBM process*

	Feed stream		Product	
	Initial	**Final**	**Acid**	**Base**
EC (mS/cm)	20.7	3.5	26.9	32.5
pH	8.1	7.3	1.3	13.6
NH_4^+ (mg/L)	2510	350	233	1680
Energy consumption (kWh/kg NH_4^+-N removed)	25			

Table 17 *Characteristics of Attero's digestate after electrocoagulation (Stream At2) and produced streams during MEZT-EDBM process*

	Feed stream		Product	
	Initial	**Final**	**Acid**	**Base**
EC (mS/cm)	13.5	2.0	36.8	30
pH	8.8	7.8	0.9	13.1
NH_4^+ (mg/L)	1115	118	85	746
Energy consumption (kWh/kg NH_4^+-N removed)	34			

Table 18 *Characteristics of Cosun's digestate after decanter (Stream Co1) and produced streams during MEZT-EDBM process*

	Feed stream		Product	
	Initial	**Final**	**Acid**	**Base**
EC (mS/cm)	15.6	7	7.0	17.4
pH	8.1	7.8	1.9	13.4
NH_4^+ (mg/L)	2040	570	90	860
Energy consumption (kWh/kg NH_4-N)	26			

2.5 Summary of the results obtained in the different studies

Feed stream	Technology used	NH_4^+ removed (%)	Energy consumption (kWh/kg NH_4^+-N)	Characteristics of the produced stream	Notes
Attero's liquid fraction of digestate after EC treatment with Fe-electrodes	UF	82		• Multicomponent mixture • NH_4^+:K^+=1.1 • NH_4^+:Na^+=2.0 • TSS<10 mg/L	
Attero's liquid fraction of digestate after EC treatment with Al-electrodes	UF	49		• Multicomponent mixture • NH_4^+:K^+=0.5 • NH_4^+:Na^+=0.7 • TSS<10 mg/L	
Pretreated RO concentrates from the thin fraction of manure digestates produced at Loonwerkersbedrijf van Amstel	AMS (TMCS)	12		$(NH_4)_2SO_4$ solution, purity not measured	Low wettability of the membranes, which indicates AMS might be suitable for this stream. However, the mass transfer coefficient is low, and hence more research is needed to evaluate if upscaling to treat this stream is cost-effective.
Pretreated RO concentrates from the thin fraction of manure digestates produced at Groot Zevert	AMS (TMCS)	6		$(NH_4)_2SO_4$ solution, purity not measured	Membrane wetting is high. AMS is not suitable to recover NH_4^+ in this stream.
Permeate streams of UF and HPNF processes from the thin fraction of manure	AMS (TMCS)			$(NH_4)_2SO_4$ solution, purity not measured	• Similar membrane performance with the permeates from MF and HPNF processes. • The technology can potentially recover 70-85% of NH_4^+.
Attero's effluent water from the EC pilot unit	ED	94	17	• Multicomponent mixture • NH_4^+:K^+=1.2-1.7 • NH_4^+:Na^+=0.4-0.5	• Suitable for removing and concentrating NH_4^+ at least 2.5-3.5 times.

Feed stream	Technology used	NH_4^+ removed (%)	Energy consumption (kWh/kg NH_4^+-N)	Characteristics of the produced stream	Notes
				Sodium sulfate was used as the electrode rinse solution. Part of the Na^+ ended up in the concentrate stream	- An increase in pH is required before the TMCS process.
	MEZT-EDBM	90	34	- Multicomponent mixture - $NH_4^+:K^+=0.2$ Potassium nitrate was used as the electrode rinse solution. Part of the potassium ended up in the base stream. - $NH_4^+:Na^+=0.5$ - Multivalent ions are removed before the MEZT-EDBM process	The produced stream can be directly used for the AMS process.
Cosun's liquid fraction of digestate	ED	73	20	- Multicomponent mixture - $NH_4^+:K^+= 0.5$ - $NH_4^+:Na^+= 8.0$	- Suitable for removing and concentrating NH_4^+ at least 2.6. - An increase in pH is required before the AMS process.
	MEZT-EDBM	72	26	- Multicomponent mixture - $NH_4^+:K^+=0.3$ Potassium nitrate was used as the electrode rinse solution. Part of the K^+ ended up in the base stream. - $NH_4^+:Na^+=4.3$ - Multivalent ions are removed before the MEZT-EDBM process	The produced stream can be directly used for the AMS process.
Attero's liquid fraction of digestate after press treatment	MEZT-EDBM	87	25	- Multicomponent mixture - $NH_4^+:K^+=0.4$ - $NH_4^+:Na^+=1.9$ - Multivalent ions are removed before the MEZT-EDBM process	The produced stream can be directly used for the AMS process.

3 Concluding remarks

In this report, preliminary results on the removal and recovery of ammonium from different organic residual flows and industrial wastewater are reported. The efficiency and effectiveness of the removal/recovery process depend, among other things, on the composition of the feed stream and pre-treatment process. Studies conducted with different feed streams and technologies showed that:

- the ammonia membrane stripping (AMS) process can be applied to recover ammonium (NH_4^+) from reverse osmosis (RO) concentrates produced at *Loonwerkersbedrijf van Amstel*. However, additional experiments are required to determine the full potential of AMS with this stream because the specific mass transfer coefficient of NH_4^+ across the membrane is markedly low ($5.4 \cdot 10^{-6}$ m/s), which implies that high membrane surface areas and/or long operational times are required to remove NH_4^+.
- the AMS process was not efficient when RO concentrates produced at *Groot Zevert (GZV)* were used as feed stream because of increased membrane wetting. However, wetting was markedly reduced when permeates of a high-pressure nanofiltration process, also produced at *Groot Zevert (GZV)*, were used instead, which led to an increase in recovery efficiency.
- long-term operation of the AMS process might be a predictor of the wetting behavior of the membranes compared to the liquid entry pressure and contact angle measurements used in this study.
- with digester supernatants (after press treatment) produced at *Attero*
    - electrocoagulation (EC) with iron electrodes contributed to removing approximately 63% of COD, which helps to mitigate fouling on membrane systems. However, effluents produced with EC exhibited high iron concentrations. Therefore, the EC process is not recommended as pretreatment for ultrafiltration and reverse osmosis processes because scaling, due to the formation of iron oxides and hydroxides, is likely to occur onto membranes.
    - electrodialysis with bipolar membranes (EDBM), specifically the MEZT electrodialysis (ED) process, achieved 87% removal of NH_4^+.
- with the digestate stream after EC produced at *Attero*
    - electrodialysis removed 94% of NH_4^+ and concentrated it 3.5 times.
    - the MEZT ED process removed 90% of NH_4^+.
- with the liquid fraction of digestate produced at *Cosun*
    - electrodialysis removed 73% of NH_4^+ and concentrated it 2.6 times.
    - the MEZT ED process removed 72% of NH_4^+.

Overall, it appears technologically feasible to remove and recover NH_4^+ from residual streams. However, the complex nature of the streams requires the implementation of an integrated process in which various technologies are coupled to maximize recovery. For instance, an integrated process might include a combination of technologies such as

- sequential pressure-driven processes followed by AMS, as proposed by Bluetec (see **Figure 7**).
- electrodialysis followed by AMS. ED is used i) to decrease the amount of non-charge organic compounds that can increase membrane wetting in AMS systems, and ii) to concentrate NH_4^+.
- electrodialysis with bipolar membranes followed by AMS. The stream produced with EDBM can be directly used in the AMS because it does not require the addition of chemicals to increase the pH.

Regardless of the combination, currently, the overall energy consumption for NH_4^+ recovery is estimated to be higher compared to the widely applied process to produce ammonia from fossil fuels, i.e., the Haber-Bosch process. For single technologies such as ED and MEZT-EDBM process the energy consumption for NH_4^+ removal ranges from 17 to 34 kWh/kg NH_4^+-N, whereas the energy consumption for ammonia production, including conversion from primary sources, is about 8 kWh/kg NH_4^+-N (28 GJ/t) [24, 25].

The results shown in this report are preliminary, and hence several efforts to reduce the total energy consumption of the NH_4^+ recovery can still be implemented. However, it is important to have a more comprehensive view of the future potential of an integrated process for NH_4^+ recovery by evaluating total energy consumption and assessing economic feasibility and environmental impact.

There are incentives to investigate further and implement processes for NH_4^+ recovery. In the production of ammonia, more marginal fossil fuels, e.g., coal and extra heavy oil, are being used [26], which typically have a higher carbon footprint and energy demand. Technologies for NH_4^+ recoveries, such as those here evaluated, can be operated using clean and sustainable energy sources, which would have a reduction in energy demand and adverse effects on the environment. Additionally, these technologies are mature and their implementation in resource recovery is on the way to deployment. For instance, EDBM, specifically the MEZT process for nitrogen (and potassium) recovery, has shown positive results. Improvements have been realized in the pre-treatment process that enables the treatment of digestates at the farm level. The first commercial applications, likely for large farms, with the MEZT process are expected in early 2023. These initial large-scale applications will enable optimizing designs and cost levels to make the process also attractive for medium – and small farms as well.

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5 Supplementary information (SI)

The following Tables S.I. 1-3 show the composition of the streams at the beginning and end of the ED process and the mass balance. Precipitates of calcium (Ca^{2+}) are likely to occur in the concentrate stream. Therefore, the amount of Ca^{2+} measured in this stream is significantly lower than that removed from the diluate stream.

Table S.I. 1 ***Attero's process stream:*** *Composition of diluate and concentrate streams before and after ED process obtained with Fujifilm membranes for α=5*

Ion	Before ED (mg/L)		After ED (mg/L)	
	Diluate	Concentrate	Diluate	Concentrate
Ca^{2+}	12.2	1.3	1.6	6.4
K^+	638	3.1	34.8	1720
Mg^{2+}	33.2	0.18	2.6	104
Na^+	1616	12.2	191	6758

*Process stream before ED: pH=8.86; electrical conductivity= 14.86 mS/cm

Table S.I. 2 ***Attero's process stream:*** *Composition of diluate and concentrate streams before and after ED process obtained with Neosepta membranes for α=5*

Ion	Before ED (mg/L)		After ED (mg/L)	
	Diluate	Concentrate	Diluate	Concentrate
Ca^{2+}	14.5	0.8	2.2	5.5
K^+	647	2.2	34.1	1970
Mg^{2+}	34.0	0.1	3.2	124
Na^+	1594	7.9	166	6945

*Process stream before ED: pH=8.86; electrical conductivity= 14.86 mS/cm

Table S.I. 3 ***Cosun's process stream:*** *Composition of diluate and concentrate streams before and after ED process obtained with Neosepta membranes for α=5*

Ion	Before ED (mg/L)		After ED (mg/L)	
	Diluate	Concentrate	Diluate	Concentrate
Ca^{2+}	129	11.7	28.7	22.4
K^+	2153	11.3	358	6093
Mg^{2+}	356	1.18	71.6	1274
Na^+	311	3.76	157	350

*Process stream before ED (stream pretreated by centrifugation): pH=8.07; electrical conductivity= 13.87 mS/cm

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Wageningen Food & Biobased Research
Bornse Weilanden 9
6708 WG Wageningen
The Netherlands
E info.wfbr@wur.nl
wur.nl/wfbr

Report 2395

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