Results in Engineering 31 (2026) 111355

Contents lists available at ScienceDirect

Results in Engineering

journal homepage: www.sciencedirect.com/journal/results-in-engineering

Research paper

Nutrient recovery from agricultural digestate by integrated ultrafiltration and reverse osmosis: effect of pretreatment strategies from laboratory to demonstrative scale

Fabio Rizzioli *, Tommaso Della Gatta, David Bolzonella, Federico Battista

Department of Biotechnology, University of Verona, Strada Le Grazie 15, 37134, Verona, Italy

ARTICLE INFO

Keywords:
Agricultural digestate
Pressure driven membranes
Nutrients recovery
Pretreatment strategies
RENURE compliance

ABSTRACT

Agricultural digestate is a nutrient-rich by-product of anaerobic digestion whose sustainable management is increasingly constrained by environmental regulations and emerging criteria for mineral fertilizers, such as "REcovered Nitrogen from manURE" (RENURE). This study systematically investigates the influence of different pretreatment strategies, namely coagulation, polymeric flocculation, acidification and moderate heating, on an integrated pressure-driven membrane treatment train based on ultrafiltration (UF) and reverse osmosis (RO) applied to agricultural digestate. The pretreatments were assessed in terms of mass distribution, membrane permeability, nutrient partitioning and compliance with RENURE-related thresholds. The results demonstrate that pretreatment selection plays a decisive role in governing both filtration performance and nutrients recovery. Coagulation improved solid removal and moderately enhanced ultrafiltration stability, while polymeric flocculation increased fouling propensity without significantly improving dewatering efficiency. Thermal conditioning reduced viscosity but caused substantial ammonium losses due to volatilization (up to ~20% of the initial nitrogen mass). In contrast, acidification at pH 4 markedly enhanced ultrafiltration permeability and improved ammonium concentration in the final RO retentate, representing the best operational compromise among the tested acidification conditions. The most effective strategy identified at laboratory scale was subsequently validated at technology readiness level 7 in a demonstrative plant operating under real biogas plant conditions. The demonstrative results confirmed the laboratory-scale trends, achieving ammonium concentrations above 17.5 g kg^{-1} and ammonium mass flows of around 45.9 kg d^{-1}, and highlighted acidification as a key enabling step for achieving stable membrane operation and improved nitrogen recovery.

1. Introduction

Livestock manure and agricultural digestate (AGRD) management is both an environmental challenge and an opportunity across the European Union (EU). Large volumes of nutrients-rich, in particular nitrogen, phosphorus, and potassium effluents are generated every year from intensive livestock and biogas production. If not controlled, this nutrients surplus may cause eutrophication of surface and groundwater, and greenhouse gases emissions in the atmosphere [1,2]. However, if properly treated and valorized, it could potentially supply a substantial share of the EU fertilizer demand, thus contributing to nutrient circularity and reducing dependency on synthetic fertilizers [3].

In October 2025, the European Commission welcomed the positive opinion of the Nitrates Committee on the proposal for recovering nutrients from "REcovered Nitrogen from manURE" (RENURE) materials [4]. The Commission proposal Ares (2024) 2885619 explicitly identifies three processing routes that can qualify processed manure as RENURE: (i) ammonia stripping and scrubbing to produce ammonium salts, (ii) reverse osmosis (RO) to produce mineral concentrates, and (iii) struvite precipitation. Under the proposed amendment, Member States may authorize the use of RENURE materials beyond the standard 170 kg N ha^{-1} limit, set up by the EU Nitrates Directive (91/676/EEC) [5] in the Nitrate Vulnerable Zones, up to an additional 80–100 kg N ha^{-1}, provided that strict quality criteria are met, such as a mineral N to total N ratio $\geq$ 90% or organic carbon to total N ratio $\leq$ 3) [6].

Within this evolving policy landscape, pressure driven membranes technologies (PDMT), such as ultrafiltration (UF) and reverse osmosis (RO) emerge as a promising technique to recover and concentrate

* Corresponding author.
E-mail address: fabio.rizzioli@univr.it (F. Rizzioli).

https://doi.org/10.1016/j.rineng.2026.111355
Received 12 January 2026; Received in revised form 25 May 2026; Accepted 1 June 2026
Available online 2 June 2026

mineral nutrients from AGRD. These techniques effectively remove suspended solids (SS) and allow the extraction of N, K and P in a concentrated form, suitable for direct agricultural use or further processing into commercial fertilizers [7]. Specifically on RO, it separates water from dissolved ions through a semi-permeable membrane, concentrating ammonium, phosphates and other soluble macronutrients (such as potassium), thereby producing a potential liquid fertilizer that could meet RENURE specifications. Despite increasing attention, experimental research on treating digestates from animal manure by PDMT and its possible application on pilot or higher technology readiness level (TRL) remain scarcely explored for some bottlenecks correlated to the adoption of PDMT [8]. Although PDMT offer the advantage of requiring very little use of chemicals, compared to ammonia scrubbing or membrane contactors, the high capital and operating costs associated with their energy-intensive processes (2-6 kWh m^{-3}) remain a major barrier to their widespread adoption [9]. Another main issue associated with these processes is represented by the fouling of the membranes which may cause severe clogging or significantly reduce filtration rates [9,10]. Several laboratory-scale studies have investigated PDMT for nutrients recovery from agricultural digestates, demonstrating high rejection efficiencies for nitrogen and phosphorus when UF and RO are applied in sequence. Waeger et al. [11], achieved 85 % of COD removal using only UF, while keeping the nitrogen removal below 20 % to be concentrated by RO. Gienau et al. [12] applied the full process of UF and RO on AGRD, alongside an enzymatic pretreatments, achieving a maximum ammonium, and K concentration in the RO retentate of 5.6, and 9.2 g kg^{-1} respectively; while recovering P in the solid phase up to 10.4 g kg^{-1}. Zielinska et al. [13] achieved 81 and 87 % of nitrogen and phosphorus respectively. But these works have also highlighted membrane fouling as a major operational limitation, strongly influenced by digestate composition and upstream conditioning strategies [11–14]. These issues emerged also during some recent pilot scale experiences. Proskynitopoulou et al. [15] tested combination of sequential processes, including microfiltration, UF, RO at pilot level to a digestate coming from a mixture of animal wastes, agricultural waste and food industry by-products. The authors demonstrated the efficacy of solids and colloids removal with the obtaining of a clarified digestate rich in nutrients and water as final outputs, only when these systems are associated with electrodialysis and UV/ozonation. In another recent work by Qi et al. [16], working on the valorization of swine liquid digestate by applying an integrated UF-membrane reactor and RO system, the authors observed that the system did not achieve good performance in nitrogen and phosphorus recovery, as consequence of the high turbidity of swine digestate, even if they obtained a concentrate rich in K, Ca and Mg.

Pretreatments can improve the efficiency of PDMT and, consequently, the nutrients recovery from digestate. Chemical conditioning, pH adjustment and thermal treatments, have therefore been proposed to reduce the turbidity of digestate, the solids and colloids concentration in digestate. Specifically, the role of flocculation and acidification in digestate-based systems was investigated on improving solid liquid separation (SLS), demonstrating the good ability to reduce the amount of SS in the UF [17]. In a work by Meixner et al. [18] it was demonstrated that the use of precipitating agents significantly improved SS separation efficiency from 46% to 75%. It led to increasing the ultrafiltration flux from 45–50 to 79 L m^{-2} h^{-1}. In addition, these agents enabled an almost complete removal of phosphorus from the supernatant. However, it is important to emphasize that this pretreatment was tested on digestate derived from stillage, rather than on livestock effluents or AGRD. Van Puffelen et al. [19] investigated an integrated treatment cascade for swine digestate, combining SLS, membrane filtration, and chemical-assisted separation steps to evaluate nutrients partitioning recovery. They observed that pretreatment steps are generally required before RO treatment of digestate, particularly SLS and MF/UF filtration, removing about 73% TS and 77% VS overall. However, the specific effect of chemical additives is not always clear. For instance, the use of $MgCl_2$ or iron sulphate as flocculants combined with polymer flocculants did not show a clear improvement in specific nutrients separation under certain operating conditions. Among other pretreatment options, acidification is of particular interest because it can alter nitrogen speciation and nutrients partitioning in digestate [20], yet its integration with PDMT processes has not been systematically assessed, especially beyond laboratory scale. Acidification has already proven to be effective in minimizing NH_3 emissions from digestate [21]. Another work by O'Connor et al. [22] the acidification was applied to stabilize ammonium in digestate and limit nitrogen losses through ammonia volatilization during the concentration step. Different mineral acids were used to enhance ammonium retention and produce nutrient-enriched solid products. However, the investigated matrix was a stillage-derived digestate, and the concentration route was based on evaporation rather than on the integrated UF–RO membrane treatment considered in the present study.

Finally, while previous studies, which mainly focused on single pretreatments or isolated separation steps, this work provides a systematic comparison of multiple pretreatment strategies within an integrated UF–RO process for agricultural digestate and validates the selected configuration under real biogas plant conditions (TRL 7). Moreover, a focus has been addressed on the mechanistic insights into nitrogen speciation under acidification and its direct impact on permeability and RENURE compliance represent a novel contribution to the design of scalable, regulation-oriented digestate valorization schemes. By bridging laboratory-scale experimentation with demonstrative operation, this work advances both the scientific understanding of membrane-based nutrient recovery and its practical implementation within the evolving European regulatory framework.

2. Materials and methods

2.1. Origin of the AGRD

The AGRD used in this study was collected from a full-scale anaerobic digestion plant located in Northern Italy (Isola della Scala, Verona, Italy), where bovine manure and crop residues are co-digested under mesophilic conditions. The main characteristics of the raw AGRD were reported in Table 1. The feedstock employed for the laboratory tests corresponded to the supernatant of the agricultural digestate, obtained after a first SLS of 1.0 kg of raw AGRD assured through the manual separation with a 2 mm filter followed by the centrifugation step at laboratory scale to remove the fibers, the biggest particle and the suspended solids. This clarified liquid phase was used as the feedstock for all subsequent pretreatment and membrane filtration tests (UF and RO).

2.2. Upstream processes applied to the agricultural digestate

To improve filterability and nutrients recovery efficiency during the membrane processes, several pretreatment strategies were applied to the liquid digestate. The treatments included acidification, thermal conditioning, coagulation, flocculation, and thermal treatments. Each pretreatment was tested either before centrifugation or after centrifugation

Table 1
Physical and chemical main characterization of raw AGRD.

pH	8.04 ± 0.05
Total Solids (TS) (% w/w)	7.35 ± 0.20
Volatile Solids (VS) (% w/w)	5.04 ± 0.11
VS/TS (%)	68.57 ± 0.14
Electrical Conductibility (EC) (mS cm^{-1})	13.97 ± 0.33
Total Kjeldahl Nitrogen (gN L^{-1})	4.12 ± 0.24
NH_4^+-N (gN L^{-1})	3.51 ± 0.20
PO_4^{3-}-P (gP L^{-1})	0.28 ± 0.04
Total Phosphorus (gP L^{-1})	1.07 ± 0.08
Total Potassium (gK L^{-1})	4.15 ± 0.45

(on the supernatant entering UF). The operating conditions adopted for each pretreatment (Table 2) were selected based on values commonly reported in the literature for digestate and manure conditioning, as well as on preliminary trials aimed at ensuring process stability and comparability among treatments [23,24]. For example, the study by Beggio et al. [23] was used as a starting point to define the dosage ranges tested in the present work for coagulation and flocculation tests. Since the operating conditions in that study were reported in g L^{-1}, they were converted into %TS by taking into account the total solids content of the digestate matrix investigated by those authors, in order to obtain comparable reference ranges for the AGRD used in the present research. Specifically, aqueous stock solutions of the selected reagents were prepared at 1% w/w. The stock solutions were subsequently dosed into the AGRD to achieve the dosage levels reported in Table 2.

For acidification, the work of Camilleri-Rumbau et al. [24] was considered as a conceptual basis, as it showed that pH reduction prior to membrane treatment can enhance the recovery of soluble nutrients such as ammonium and potassium. On this basis, different target pH values (Table 2) were experimentally tested in the present study in order to identify the most suitable operating condition for the AGRD.

While thermal conditioning (HEAT test, Table 2) was not selected on the basis of a specific literature protocol, but was introduced in this work as an exploratory pretreatment to assess whether a moderate increase in temperature could improve the physical properties, such as the viscosity, of the digestate and, consequently, the efficiency of the subsequent membrane separation steps.

Each pretreatment condition was tested in three independent experimental runs. For each run, analytical measurements were performed in triplicate and results are reported as average values. A summary of these preliminary trials and of the rationale used to define the final operating conditions is reported in the Supplementary Material (Table S1).

2.3. Laboratory-scale process description

The overall treatment scheme consisted of four sequential stages (Fig. 1):

a) SLS of the raw AGRD: this first step was assured by the manual separation of the AGRD though a sieving mesh of 2 mm, which led to the obtaining of a Solid Fraction (SF) and a Liquid Fraction (LF), respectively indicated with SF and LF.
b) Centrifugation (CF): the LF was centrifuged to remove SS and obtain a clarified supernatant (SUR) for membrane processing, while the other stream was represented by the sedimentate (SED).
c) Ultrafiltration (UF): the supernatant was filtered using a Vibro-Lab35P unit (SANI Membranes, Denmark) equipped with a 150 kDa molecular weight cut-off (MWCO), 35 cm^2 NADIR® UP150P (MANN+HUMMEL) polyethersulfone membrane. The system operated at a transmembrane pressure (TMP) of 1.8-2.0 bar, with feed and retentate flow rates of 380 rpm and 260 rpm, respectively. The UF produced a retentate (UF-RET) and a permeate (UF-PER). The UF stage represented the critical bottleneck of the process due to severe fouling phenomena and the resulting decline in membrane permeability [12].
d) Reverse osmosis (RO): the UF-PER was subsequently treated in a RO step represented by the equipment Sterlitech HP4750 stirred cell at 40 bars under nitrogen pressurization, using TRISEP® ACM2 (MANN+HUMMEL) polyamide membrane with 17 cm^2 of surface area. The objective was to concentrate soluble nutrients (NH_4^+, PO_4^{3-}, K^+) on the RO retentate (RO-RET) while recovering high-quality water in the permeate (RO-PER).

The proposed membrane-based configuration does not aim at producing final fertilizer products, but rather at generating nutrient-enriched streams that can be subsequently processed using established recovery technologies.

2.4. Evaluation parameters

The performance of each pretreatment was assessed based on the following parameters:

$$R_{i,\ RO-RET} = \frac{m_{i,\ RO-RET}}{m_{i0}} \cdot 100 \quad (1)$$

Where $R_{i,\ RO-RET}$ is the recovery percentage of nutrient *i* from RO-RET, calculated with the mass recovery of the nutrient *i* from RO-PER and the total starting mass of the nutrient *i* (i_0) in the AGRD.

$$R_{W,RO-PER} = \frac{m_{w,RO-PER}}{m_{w0}} \cdot 100 \quad (2)$$

Where $R_{w,RO-PER}$ is the water recovery percentage from RO-PER, calculated with the water mass recovery from RO-PER and the total water mass (w_0) in the AGRD.

$$L_P = \frac{J}{\Delta P} \quad (3)$$

Where L_P is the membrane permeability (L h^{-1} m^{-2} bar^{-1}), J the trans-membrane flux (L h^{-1} m^{-2}), and ΔP is the trans-membrane pressure (bar).

The best-performing pretreatment was further evaluated for compliance with the RENURE quality criteria, as defined in the European Commission proposal Ares [6]: i) *Mineral N / Total N* $\geq$ *90 %*, or ii) *Organic C / Total N* $\leq$ *3 %*. These indicators were used to assess whether the RO-RET could be classified as a RENURE-compliant fertilizing product. Organic C was measured as Total Organic Carbon. At this scope, the following parameter was considered:

$$\text{Organic C} \Big/ \text{Total N (w / w)} = \frac{TOC}{TKN} \quad (4)$$

Where TOC is the Total Organic Carbon (g L^{-1}), and TKN is the Total Kjeldahl Nitrogen (g L^{-1}).

Table 2
Pretreatment conditions applied to AGRD.

Code	Pretreatment type	Description	Application phase	Reagents and conditions	Ref.
CTR	Control	Untreated liquid AGRD	—	—	—
COA	Coagulation	Addition of hybrid Fe–Al coagulant (PIX-323 + PAX)	Before centrifugation	Different concentration from 0.25% to 1.00 w/w of TS	[23]
FLO	Flocculation	Addition of *Superfloc C-496*	Before centrifugation	Different concentration from 0.25% to 1.00 w/w of TS	[23]
POL	Flocculation	Addition of *FLOPAM 4650*	Before centrifugation	Different concentration from 0.25% to 1.00 w/w of TS	[23]
ACD 3–7	Acidification	pH adjusted to 3, 4, 5, or 7	After centrifugation, before UF	H_2SO_4 (1 M) until target pH reached	[24]
HEAT	Thermal conditioning	Heating at 50°C	After centrifugation, before UF	—	—

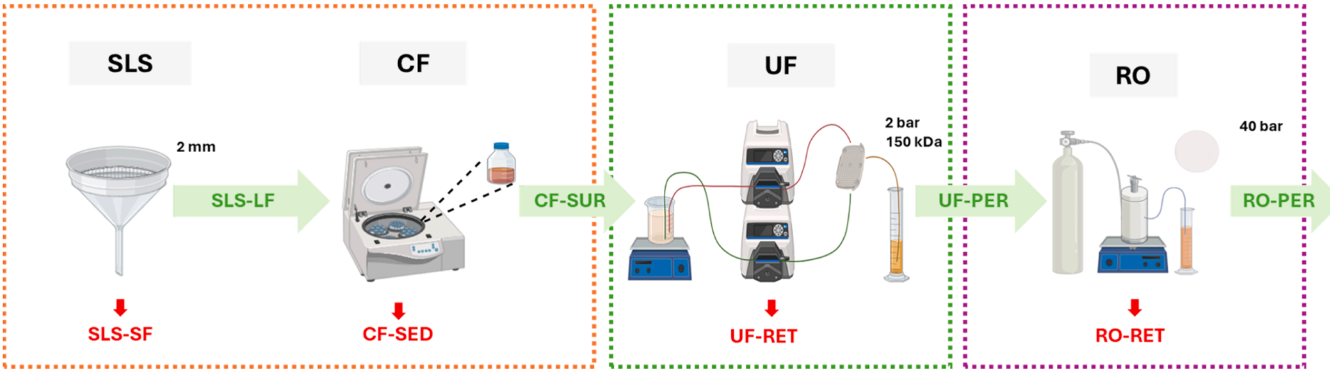

Fig. 1. The lab-scale scheme for the valorization of the AGRD composed by: i) the Solid-Liquid Separation, whit the obtaining of a Solid Fraction (SLS-SF) and a Liquid Fraction (SLS-LF), ii) the Centrifugation (CF) of the SLS-LF, with the obtaining of a Supernatant (CF-SUR) and a Sedimentate (CF-SED), iii) the Ultrafiltration (UF) of the CF-SUR with the obtaining of a permeate (UF-PER) and a retentate (UF-RET) and iv) the Reverse Osmosis (RO), with the obtaining of a permeate RO-PER and a retentate (RO-RET).

2.5. *Validation of the best pretreatment at TRL 7*

The best pretreatment was then tested in a demonstrative plant (DEMO) which was designed, constructed and installed within the LIFE DIMITRA project to upgrade AGRD into concentrated nutrient streams through sequential mechanical and membrane-based separations. DEMO was implemented at the agricultural company La Torre (Isola della Scala, Verona, Italy), which operates a 0.99 MWe anaerobic digestion facility treating cattle manure and lignocellulosic residues. The DEMO plant was installed inside a compact modular container (2.4 × 3.0 × 5.8 m) to ensure transportability and operability under real farm conditions. DEMO 2 consists of three consecutive unit operations (Fig. 2): i) a Solid–Liquid Separation (SLS), ii) an UF, and iii) a RO step. This configuration was selected following an extensive laboratory campaign aimed at identifying the most effective combination of filtration technologies for nutrient concentration and water recovery from AGRD [3].

The SLS stage is performed using a conical-disc filtration system (PRIMESCREEN®), which retains fibers, large particles, and colloidal aggregates (100 – 2000 μm) while producing a clarified liquid fraction suitable for membrane processing. This step ensures the removal of most phosphorus-rich solids and reduces the fouling potential of downstream membranes. The UF unit (ceramic membranes, total filtration area 1.4 m^2, operated at 4 bar) concentrates microorganisms, organic nitrogen, and residual phosphorus in the retentate, while allowing monovalent salts such as ammonium and potassium to permeate. The final RO stage (polyamide membrane, up to 70 bar) treats the UF permeate to simultaneously recover purified water and a nutrient-rich concentrate enriched in ammonium and potassium salts. All equipment is integrated with automated control, level sensors, and cleaning procedures, enabling continuous operation under real working conditions. The system treats approximately 20 tonnes of AGRD per day.

The performance of the best pretreatment was compared with the one from AGRD without any previous upstream process (CTR-DEMO test). The parameters considered for the evaluation of the CTR-DEMO and the best pretreatment were the same ones used for the laboratory tests and reported in Section 2.4.

2.6. *Analytical methods*

Total (TS) and volatile solids (VS), the Chemical Oxygen Demand (COD), the Total Kjeldahl Nitrogen (TKN) ammonium content, the Total Phosphorous (TP) compounds and the Total Organic Carbon (TOC) of the AGRD and the derived streams from the different filtration steps were analyzed according to APHA Standards Methods [25]. pH and electrical conductibility (EC) were measured using CO 3000H portable analyzer by VWR. While potassium (TK) and TOC contents were measured by a spectrophotometric method supplied by Hach Lange.

2.7. *Statistical analysis*

Pretreatments and filtrations were made in triplicate. Data were analyzed using Microsoft Office Excel and RStudio (Posit Software) for ANOVA. The sequent significance levels were used: statistically significant ($p < 0.05$); highly significant ($p < 0.01$); very highly significant ($p < 0.001$).

3. Results and discussion

Figs. 3, 4, 5, 6 and 7 show the TS, COD and ammonium, potassium and phosphorus distributions along all the sequential filtration steps for the CTR test and the different pretreatments on AGRD.

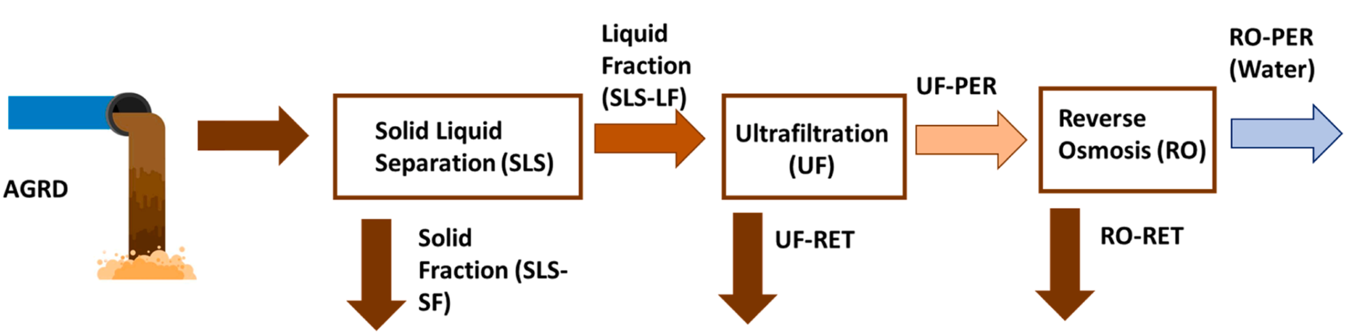

Fig. 2. Scheme of the mechanical and the pressure driven membranes technologies and of the corresponding inlet and outlet streams for the DEMO plant.

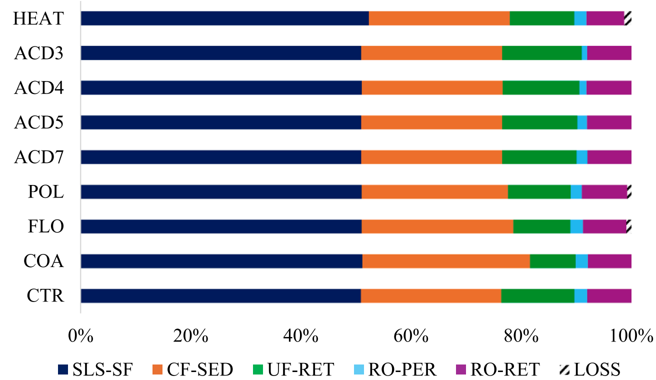

Fig. 3. Mass distribution of TS along the biorefinery line for the different pretreatment conditions applied to AGRD. Bars represent the fraction (%) of the initial TS mass recovered in each process stream. Bars represent the fraction (%) of the initial TS mass recovered in each stream. Process steps and streams include solid–liquid separation (SLS), solid fraction (SF), centrifugation (CF), sediment (SED), ultrafiltration (UF), retentate (RET), permeate (PER) and reverse osmosis (RO).

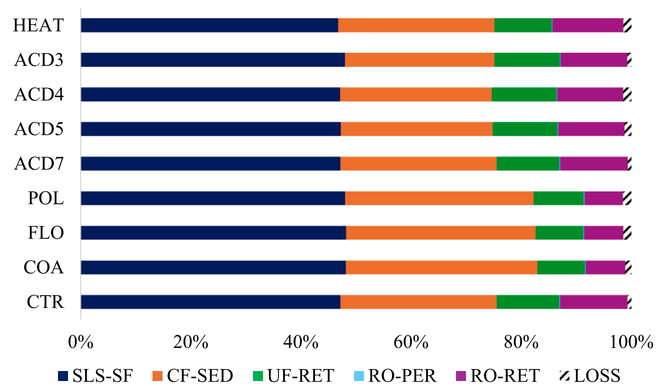

Fig. 4. Mass distribution of COD along the biorefinery line for the different pretreatment conditions applied to AGRD. Bars represent the fraction (%) of the initial COD mass recovered in each process stream. Process steps and streams include solid–liquid separation (SLS), solid fraction (SF), centrifugation (CF), sediment (SED), ultrafiltration (UF), retentate (RET), permeate (PER) and reverse osmosis (RO).

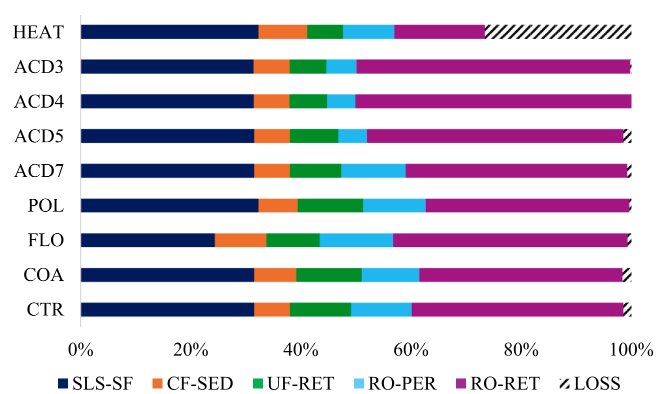

Fig. 5. Mass distribution of ammonium along the biorefinery line for the different pretreatment conditions applied to AGRD. Bars represent the fraction (%) of the initial ammonium mass recovered in each process stream. Process steps and streams include solid–liquid separation (SLS), solid fraction (SF), centrifugation (CF), sediment (SED), ultrafiltration (UF), retentate (RET), permeate (PER) and reverse osmosis (RO).

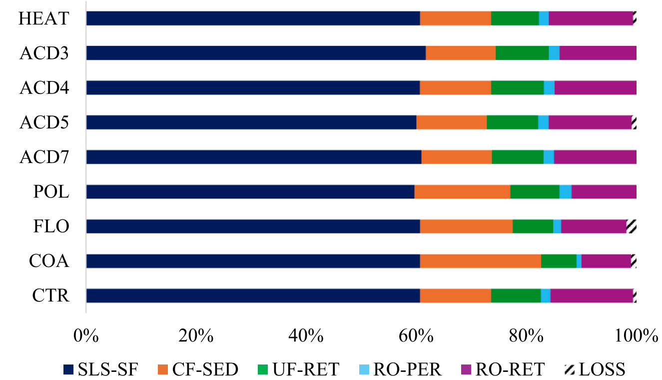

Fig. 6. Mass distribution of phosphorus along the biorefinery line for the different pretreatment conditions applied to AGRD. Bars represent the fraction (%) of the initial phosphorous mass recovered in each process stream. Process steps and streams include solid–liquid separation (SLS), solid fraction (SF), centrifugation (CF), sediment (SED), ultrafiltration (UF), retentate (RET), permeate (PER) and reverse osmosis (RO).

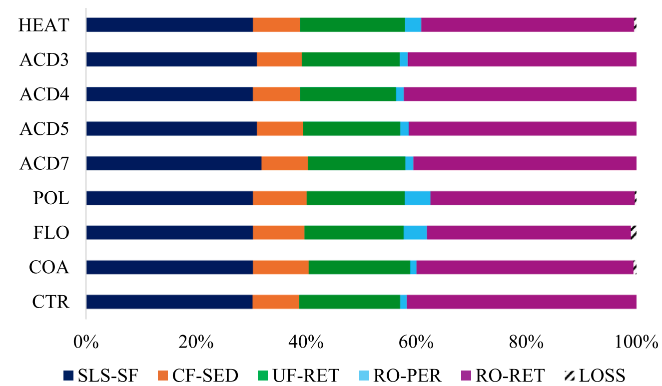

Fig. 7. Mass distribution of potassium along the biorefinery line for the different pretreatment conditions applied to AGRD. Process steps and streams include solid–liquid separation (SLS), solid fraction (SF), centrifugation (CF), sedimentate (SED), ultrafiltration (UF), retentate (RET), permeate (PER) and reverse osmosis (RO). Bars represent the fraction (%) of the initial potassium mass recovered in each process stream.

3.1. Performance of CTR AGRD tests

AGRD was treated through four sequential filtration stages, without any pretreatment: SLS, CF and the two PDMT steps, the UF followed by RO. SLS and CF was used to separate the fibers, biggest solids and colloidal particles from the AGRD, allowing the obtaining of a SF and a LF. Specifically, SF and LF accounted for 35.06 ± 1.02 and 64.94 ± 1.97% w/w, respectively. The LF was sent to a CF step which led to a SUR and SED fraction of about 54.16 ± 2.01 and 10.78 ± 1.54% w/w calculated based on the initial amount of the AGRD. TS were essentially recovered in these two first separation steps: the SF from SLS and the SED from CF exhibited a TS concentration of 11.35 ± 0.36% w/w and 18.50 ± 0.47% w/w, respectively. While the TS removal from SLS and CF was 51.02 ± 1.93% and 25.57 ± 3.41% w/w for a total of about 76.6% w/w of the total TS (Fig. 3). UF provided a physical clarification step, effectively removing SS and colloidal particles, while the RO stage was responsible for the concentration of dissolved salts and nutrients. Specifically, during UF, the SUR from CF was subjected to a selective separation based primarily on size exclusion [26]. Particles and macromolecules larger than the membrane pores were retained, whereas water and dissolved ions readily passed through. This step efficiently removed fine colloids, organic residues, and microbial debris that remained after the preliminary centrifugation, thereby producing a clarified effluent with reduced turbidity. The removal of these colloidal fractions was particularly important because such particles are deformable and tend to form compressible cakes on membrane surfaces, which would otherwise accelerate fouling and flux decline in the subsequent RO unit [27]. By eliminating this fouling potential, the UF step created favorable hydrodynamic conditions for RO operation, allowing stable fluxes and extended membrane life. Chemically, the UF process did not significantly alter the ionic composition of the digestate. Ammonium, potassium ions, and soluble phosphates were unaffected by UF and thus remained in the permeate [27,28]. As a result, the UF stage served as a clarification rather than a nutrient-removal step, transmitting the dissolved species to the downstream RO system for concentration. The clarified UF-PER exhibited an overall reduction of TS of about 90% w/w compared with the raw AGRD, confirming that most particulate matter had been effectively eliminated before entering the RO unit.

In the RO, the separation mechanism shifted from mechanical sieving to molecular diffusion through a dense, semi-permeable membrane [29]. Driven by high hydraulic pressure, water molecules diffused across the membrane while ions and dissolved organics were largely rejected, resulting in a clean water permeate and a nutrient-enriched retentate. The RO membrane, characterized by extremely small free-volume pathways (< 0.001 μm), imposed a strong barrier to the passage of hydrated ions. Electrostatic repulsion between the negatively charged membrane surface and the ionic solutes (the so-called Donnan exclusion effect) further enhanced selectivity, particularly for multivalent species [30]. In this stage, most of the ammonium, potassium and soluble phosphorus were concentrated, while the permeate displayed a very low electrical conductivity, confirming its high purity. Consequentially, in terms of nutrients behavior, the CTR tests achieved recoveries in the RO-RET of approximately 38.45 ± 1.74% w/w for ammonium with a NH_4^+/TKN value of 78.05% w/w, 41.81% w/w for potassium, all expressed based on amount of these nutrients in the initial AGRD. These recovery yields corresponded to 12.80 ± 1.01 g kg^{-1}, 1.67 ± 0.04 g kg^{-1} and 17.43 ± 1.25 g kg^{-1} for ammonium, phosphorous and potassium, respectively in RO-RET stream (Supplementary Materials, Table S1). The near equivalence between ammonium and TKN confirmed that most nitrogen was present in the ammonium form, as expected after AD. EC values supported this interpretation: they remained nearly constant across the UF step, indicating that dissolved salts were not retained, and rose markedly in the RO-RET, reaching about 40 ± 1 mS cm^{-1}. The concentration effect therefore resulted exclusively from the osmotic rejection of solutes by the RO membrane, not from any prior removal.

Phosphorus followed a different pattern: 61.59 ± 2.84% w/w of the TP was already captured in the SF from SLS while 14.95 ± 1.76% w/w was removed in the next CF step. This is attributed to the strong tendency of phosphate ions to adsorb onto fibrous or organic matter and to co-precipitate with calcium, magnesium or iron naturally present in the digestate [31,32]. In the UF + RO sequence, only the remaining soluble phosphate fraction was available for concentration in the RO permeate. Thus, in this configuration, phosphorus separation was primarily governed by earlier solid–liquid interactions rather than by membrane selectivity.

The RO-RET obtained in the present configuration did not yet comply with the new RENURE regulatory criteria, which require NH_4^+/TKN ratio higher than 90% and COD/TKN ratio lower than 3. In our case, the RO-RET showed an NH_4^+/TKN ratio of approximately 80% and a TOC/TKN ratio of about 3.2, indicating that without additional pretreatments or conditioning steps, the recovered concentrate does not yet meet the RENURE requirements. This justifies the need for further upstream treatments of the AGRD to enhance the mineralization of organic nitrogen and reduce the residual organic load before RO concentration. Regarding the water recovery (Equation 2), RO-PER represented 27% w/w of the raw AGRD.

3.2. Performance of coagulation and flocculation on AGRD valorization

In the context of this study, the addition of the different coagulants and flocculants, COA, FLO and POL, each of one tested in a TS concentration range among 0.25 and 1.00% w/w (Table 2), was carried out before the CF, that is, immediately after the initial SLS step. In this configuration, the reagents acted directly on the LF of the AGRD, promoting aggregation of residual colloids and fibrous particles lower than 2 mm, while facilitating the removal of organic-bound nitrogen and the residual phosphorus species. Consequently, the CF was expected to yield a clearer SUR with reduced SS and organic load, improving the performance of subsequent pressure driven membranes processes (UF and RO) in terms of permeability and nutrients concentration efficiency.

COA, FLO and POL tests were optimized with an addition of the correspondent coagulants and flocculants of the 0.5% w/w of TS on AGRD. At 0.25% w/w of TS, the performances in terms of solids removal were lower, while at higher concentrations (0.75 and 1.00% w/w of TS) non-significant improvements were appreciated.

Specifically, coagulants addition (COA test, Table 2) had a first effect on solid removal from SUR during the CF unit operation. The TS concentration on SED phase increased significantly from 18.50 ± 0.47% w/w of the untreated AGRD to 19.80 ± 0.09% w/w in COA test ($p < 0.05$), while the COD concentration increased from 171.5 ± 10.6 to 189.6 ± 20.4 g kg^{-1}, respectively. The amount of the SED phase also increased of 9% w/w compared to the no pretreated AGRD (CTR test). Consequently, the fractions of the initial TS and COD which are recollected in the SED phase increased from 25.5 ± 0.41 to 30.6 ± 0.46% w/w and from 28.3 ± 0.82 to 34.8 ± 0.45% w/w compared to CTR tests ($p < 0.05$), respectively (Figs. 3, 4). These results can be justified considering the principles of the mechanisms governing the coagulants. The LF of AGRD typically contains a complex suspension of organic and inorganic colloids, residual fibers, humic substances, microbial cells, and soluble macronutrients such as ammonium, potassium, and phosphates. These colloidal particles are negatively charged and stabilized by electrostatic repulsion and hydration forces, which prevent their aggregation and settling under natural conditions [33]. Coagulants, such as ferric or aluminum salts, act primarily through charge neutralization and sweep flocculation. When added to the liquid digestate, hydrolyzed metal ions (e.g., Fe_3^+, Al_3^+) form polymeric hydroxides capable of destabilizing negatively charged colloids. This reduces the zeta potential, promoting particle aggregation and sedimentation during the CF step [34,35]

Regarding the main nutrients, the ammonium, potassium and phosphorous recoveries in the RO-RET decreased compared to CTR tests, passing from 38.45 ± 1.74 to 36.89 ± 0.78% w/w for ammonium ($p < 0.05$) (Fig. 5), from 41.89 ± 1.80 to 39.42 ± 0.68% w/w for potassium compounds ($p < 0.05$) (Fig. 7), and from 15.05 ± 0.60 to 9.01 ± 0.06% w/w for phosphorus ($p < 0.01$) (Fig. 6). These recovery yields corresponded to 12.70 ± 1.00 g kg^{-1}, 1.00 ± 0.06 g kg^{-1} and 16.43 ± 2.01 g kg^{-1} for ammonium, phosphorous and potassium, respectively in RO-RET stream (Supplementary Materials, Table S1). Considering the scope to maximize the ammonium and potassium recovery in the RO-RET, these results are not good. On the contrary, the lower amount of phosphorous in RO-RET can be considered a positive aspect, as it is usually recovered for other applications, such as struvite production, in the more solid fractions of the digestates, such as the SF from SLS and SED from CF step.

From a mechanistic standpoint, metal-salt coagulation targets particulate and colloidal fractions and promotes orthophosphate removal by charge neutralization, sweep flocculation and formation of poorly soluble Fe/Al phosphates; thus, it shifts P from the liquid to the solid phase prior to membranes [36,37]. In contrast, NH_4^+ and K^+ remain largely dissolved and are not directly removed by coagulation, as their separation typically requires ion exchange/adsorption, air stripping, or crystallization (e.g. struvite) rather than charge destabilization of colloids [38,39]. Consistent with this theory, the slight decrease in RO-RET recoveries of NH_4^+ and K^+ observed after coagulant dosing is best explained by indirect retention: a minor fraction of NH_4^+ can associate with organic–mineral flocs (electrostatic association/ion pairing and physical entrapment), and K^+ may be weakly co-entrained, so that enhanced floc formation and subsequent centrifugation remove more of these floc-bound species upstream, leaving less to be concentrated by RO [3,40].

FLO and POL tests adopted two different flocculants to try to removal solids from the LF of AGRD after SLS (Table 2). Flocculants, typically long-chain organic polymers such as polyacrylamides, act through bridging and entrapment mechanisms. Their high molecular weight and flexible structure allow them to adsorb onto multiple colloidal surfaces simultaneously, creating inter-particle bridges and forming large, dense flocs that settle rapidly during CF [41]. However, their performances in TS (Fig. 3) and COD removal (Fig. 4) were lower than coagulant in COA test, whose improvements were of only few percentual values compared to CTR test. It is consistent with the underlying mechanisms of action of these reagents. Cationic flocculants act mainly through polymer bridging and inter-particle aggregation, adsorbing onto negatively charged colloids and linking them into larger but relatively porous flocs. Their role is therefore to enhance particle aggregation and settling velocity, rather than to destabilize colloids or induce precipitation [42]. In contrast, metal-based coagulants such as ferric or aluminum salts, such the ones used in COA test, promote charge neutralization and sweep flocculation, leading to the formation of denser, more compact aggregates with higher water-drainage capacity. These mechanisms not only enhance solid–liquid separation but also induce chemical precipitation of phosphorus compounds (e.g., $FePO_4$, $AlPO_4$) [43], as previously reported. Similar findings have been reported for agricultural digestates and manure-derived slurries, where polyacrylamide-based flocculants primarily improve particle aggregation but have limited effect on the final dry matter content, whereas ferric or aluminum coagulants lead to higher solids recovery and more effective dewatering [43].

Regarding nutrients recovered in the RO-RET, the addition of cationic flocculants in FLO and POL tests before CF step did not significantly affect their distribution in the subsequent membrane process (Figs. 3-7). Ammonium, present as a soluble monovalent ion, remained almost entirely in the liquid phase and its recovery in the RO concentrate was unchanged, confirming that polymeric flocculants do not interact chemically with NH_4^+ [44]. A slight decrease of about three percentage points in the recovery of potassium and phosphorus was observed compared with the untreated digestate. Specifically, for FLO tests, the concentrations for ammonium, phosphorous and potassium in the RO-RET were 12.17 ± 0.85 g kg^{-1}, 1.32 ± 0.08 g kg^{-1} and 15.43 ± 0.14 g kg^{-1}, respectively. While for POL tests were 12.76 ± 0.92 g kg^{-1}, 1.36 ± 0.08 g kg^{-1} and 16.04 ± 0.25 g kg^{-1}, respectively (Supplementary Materials, Table S1). This minor reduction is attributed not to chemical removal but to indirect physical entrapment and adsorption of these ions within the flocs formed during flocculation and their subsequent removal in the centrifugation step [23,44]. Similar behavior has been reported for agricultural digestates and manure slurries, where polymer-assisted solid–liquid separation improves clarity and solid removal efficiency without substantially altering the overall nutrient composition of the liquid fraction entering downstream membrane units [24].

Finally, considering the previous discussed low changes compared the CTR test, especially in terms of nutrients recovery in the RO-RET, COA, FLO and POL did not achieve the two required RENURE criteria of a NH_4^+/TKN over 90% w/w and TOC/TKN lower than 3, being not suitable to obtain a final product which can be classified as bio-fertilizer. Regarding the water recovery, FLO and POL tests increased a little bit the amount of RO-PER, passing from 27 to 29% w/w.

3.3. *Performance of pH reduction on AGRD valorization (ACD3, 4, 5, 7 tests)*

Among the pretreatment strategies summarized in Table 2, acidification consisted of adjusting the pH of the SUR phase to values between 3 and 7 by addition of sulfuric acid (1 M) after the CF step and before UF. This treatment was introduced to modify the physicochemical characteristics of the digestate liquid fraction, with potential effects on nutrient partitioning, colloidal stability, inorganic precipitation tendency and membrane processability. The acidification demands of the SUR corresponded to sulfuric acid consumptions of approximately 0.80, 1.01, 1.13 and 1.23 kg H_2SO_4 per tonne of SUR to reach pH 7, 5, 4 and 3, respectively. These values indicate a moderate buffering capacity of the digestate liquid fraction and provide a first quantitative indication of the acid demand associated with the acidification pretreatment.

ACD3, 4, 5 and 7 tests, respectively performed at pH 3, 4, 5 and 7, showed that the effect of acidification on nutrients concentration in the final RO-RET was markedly pH-dependent. In particular, ammonium concentration in the RO-RET progressively increased from 12.80 ± 1.01 g kg^{-1} in the CTR to 13.54 ± 0.08, 15.54 ± 0.17, 16.65 ± 1.52 and 16.09 ± 0.26 g kg^{-1} for the ACD7, ACD5, ACD4 and ACD3 tests, respectively (Supplementary Materials, Table S1). These results indicate that acidification significantly improved the overall efficiency of ammonium concentration in the integrated UF–RO chain. However, no substantial further benefit was observed when decreasing the pH from 4 to 3, despite the higher acid consumption required. For this reason, the subsequent discussion focuses on the ACD4 condition, which represented the best compromise between nutrient recovery improvement, membrane performance and acid demand.

The acidification of the SUR phase prior to UF did not produce any significant change in the subsequent mass distribution among the process streams during UF and RO. The proportion of the UF-RET slightly increased from 15.97 ± 1.19 % w/w in the CTR to 16.91 ± 0.38 % w/w in the ACD4 test, while the TS and COD concentrations in the UF-RET were 6.70 ± 0.03 % w/w and 47.31 ± 4.36 g kg^{-1}, respectively, values very close to those obtained under control conditions (6.50 ± 0.06 % w/w and 46.35 ± 5.10 g kg^{-1}) (Figs. 3 and 4). These differences were non-significant ($p \geq 0.05$) and therefore indicate that acidification did not substantially affect the bulk solids and COD distribution along the treatment chain, in agreement with previous observations reported in the literature [11,19].

In contrast, acidification produced a very highly significant increase in ammonium recovery in the RO-RET, which rose from 38.45 ± 1.74 % w/w in the CTR to 50.14 ± 0.85 % w/w in the ACD4 test ($p < 0.001$) (Fig. 5). At the same time, phosphorus and potassium recoveries remained almost unchanged and non-significant ($p \geq 0.05$), varying from 15.05 ± 0.60 % to 14.91 ± 0.15 % w/w and from 41.81 ± 1.80 % to 42.26 ± 0.58 % w/w, respectively (Figs. 6 and 7). These findings indicate that the beneficial effect of acidification was selective and mainly concerned ammonium recovery. This result was consistent with some previous works on scientific literature, which demonstrated that reducing the pH of the digestate leads to the stabilization of NH_4-N, reducing its potential to be volatilized and, consequently, improving the possibility to be recovered [22,45].

It is important to underline that the superiority of ACD4 cannot be explained by NH_3/NH_4^+ equilibrium alone. Although acidification contributes to maintaining nitrogen in the protonated NH_4^+ form, the fraction of free ammonia is already very low under mildly acidic conditions. Therefore, the choice of pH 4 as the best pretreatment cannot be attributed solely to acid–base equilibrium considerations. Rather, the results suggest that pH 4 represents the best operational compromise for the integrated UF–RO treatment chain, reflecting the combined effect of several favorable phenomena occurring simultaneously in the digestate–membrane system.

In fact, acidic conditions can reduce the tendency to form carbonate- and phosphate-based precipitates, which are known to occur in digestates under neutral or slightly alkaline conditions, reducing the risk of the fouling of the membrane, even underline by some previous works [19,46–48]. A further contribution is likely associated with modified colloidal interactions in the liquid digestate, which can affect the structure and compactness of the fouling layer and, consequently, the membrane processability. Specifically, acidification may have contributed to fouling mitigation by altering the electrostatic stability of colloidal and biopolymeric fractions. This interpretation is supported by studies showing that pH reduction can shift zeta potential, promote protein–polysaccharide aggregation, increase particle size, and improve UF flux in anaerobic sludge supernatants, while acidification of slurry has been associated with larger particles and reduced electrical charges during solid–liquid separation [49–51]. This interpretation is supported by the permeability results discussed in Section 3.5. In fact, the acidification tests performed at different pH values clearly showed that the highest UF permeability was obtained at pH 4, whereas significantly lower values were observed at pH 7 and 5, and no substantial additional improvement was achieved at pH 3. Therefore, the selection of ACD4 as the best pretreatment was not based on a single isolated mechanism, but on the combined evidence of: (i) the highest ammonium recovery and concentration in the RO-RET, (ii) the best membrane permeability, and (iii) the absence of a substantial further advantage at pH 3 despite the higher acid demand.

Phosphorus and potassium, on the contrary, exhibited conservative behavior. Phosphorus in the liquid digestate is largely present as orthophosphate ($H_2PO_4^-/HPO_4^{2-}$) and as weakly bound forms associated with colloidal matter. Within the investigated pH range, phosphate speciation changes only moderately, without major precipitation or dissolution phenomena, except under more extreme acidic conditions [32]. Similarly, potassium behaves as a conservative cation in digestates, existing predominantly as K^+ in solution and not forming stable precipitates or complexes under the investigated acidification conditions. For this reason, its distribution remained mainly governed by physical separation and membrane concentration effects [24].

Under these conditions, the RO-RET reached NH_4^+/TKN and TOC/TKN values of 91.69% w/w and 2.06, respectively, thus complying with the mineral N/total N and TOC/TKN thresholds considered in this study. Water recovery was not significantly affected by the ACD4 pretreatment, with RO-PER values remaining close to those of the CTR test (27.0–27.5% w/w). In a practical implementation scenario, a final conditioning step, such as controlled neutralization or dilution within irrigation water, may be integrated downstream of RO-RET depending on the intended application.

The role of acidification in enhancing nutrient availability prior to downstream recovery has also been highlighted in other integrated manure-processing schemes. For instance, the ManureEcoMine approach [52], combining anaerobic digestion with ammonia stripping, SLS, UF and struvite precipitation, demonstrated that acidification can increase soluble nitrogen and phosphorus concentrations. Although the recovery pathways investigated in that study differ from the membrane-based concentration strategy adopted here, the results are consistent with the interpretation that acidification can improve nutrient solubilization and influence nutrient partitioning before membrane separation.

3.4. Performance of thermal conditioning on AGRD valorization (HEAT test)

As reported in Table 2, the HEAT test consisted of maintaining the SUR obtained after CF step at 50°C during the UF, with the aim of reducing its viscosity and thereby improving the filtration performance. The first interesting result was that, in contrast to the other pretreatments, the overall mass balance obtained for this test showed a noticeable deviation, with a deficit of approximately 5 % of the total initial AGRD mass. Up to the CF step, the process mass distribution remained almost identical to that of the CTR test, as the pretreatment was not applied to the SLS and CF steps. Differences became evident only during the UF stage. The proportion of the UF-PER and UF-RET with respect to the initial AGRD mass decreased from 38.18 ± 3.03% w/w to 35.32 ± 1.14% w/w and from 15.97 ± 1.19% w/w to 14.03 ± 0.32% w/w, respectively, when comparing the HEAT test with the CTR. These significant reductions ($p < 0.001$) are attributed to evaporation losses occurring during the heating of the SUR at 50°C, since no other phase transformations or chemical reactions could account for such discrepancies. The temperature rise likely caused partial water evaporation from the open system, thereby reducing the total mass available for downstream membrane filtration. Apart from this evaporation-related mass loss, the overall distribution of solids and solutes across the UF and RO steps remained comparable to that of the CTR test, indicating that the applied thermal conditioning mainly affected the physical properties, particularly viscosity and density, rather than the chemical composition of the SUR phase.

The mass balance analysis applied to nutrients provided additional insights into their fractioning across the process streams. As shown by the mass distribution data, the fractions of potassium (Fig. 7) and phosphorus (Fig. 6) recovered in the RO retentate (RO-RET) remained non-significant ($p \geq 0.05$) compared to the CTR test, accounting for 38.72 ± 0.47% w/w and 15.28 ± 0.20% w/w of the initial mass, respectively. In contrast, the fraction of ammonium recovered in the RO-RET markedly decreased from 38.45 ± 1.74% in the CTR test to 16.43 ± 0.16% in the HEAT test ($p < 0.001$), as derived from the mass distribution reported in Fig. 5. These yields corresponded to the following ammonium, phosphorous and potassium concentrations in the RO-RET of 6.42 ± 0.78 g kg^{-1}, 1.99 ± 0.13 g kg^{-1} and 18.94 ± 1.63 g kg^{-1}, respectively (Supplementary Materials, Table S1). The main result was represented by the sharp decline of ammonium recovery yield and concentration in the RO-RET compared to the CTR test. It can be attributed to ammonia volatilization during the heating stage. Increasing the temperature to 50°C shifts the NH_3/NH_4^+ equilibrium toward the gaseous form, since the pKa of the conjugate acid decreases with temperature, thereby enhancing the fraction of free ammonia and its loss to the atmosphere if the system is not hermetically closed [53, 54]. As a result, part of the total ammoniacal nitrogen was removed before entering the membrane stages, leading to the reduced recovery observed in the RO-RET. In contrast, potassium and phosphorus species are thermally stable and non-volatile under the applied conditions. Potassium remains as K^+ in solution and does not form temperature-dependent complexes, whereas orthophosphate ions maintain their solubility between 20 and 50°C, showing no evidence of precipitation or release [24,32].

As a consequence of the significant ammonium loss observed during the heating stage, the HEAT test failed to comply with the key criteria established by the RENURE guidance. In particular, the NH_4^+/TKN ratio in the RO retentate dropped well below the 90 % threshold required for RENURE-compliant fertilizers, while the TOC/TKN ratio exceeded the limit value of 3. These deviations are directly linked to the volatilization of ammonia during thermal conditioning, which reduced the mineral nitrogen content and increased the relative proportion of organic matter. Therefore, although heating at 50°C may reduce viscosity and slightly improve filtration behavior, it cannot be considered an appropriate pretreatment when the objective is to produce a RENURE-compliant recovered fertilizer.

3.5. Analysis of the performance of permeability

Table 3 shows the average values of permeability at 1, 5 and 10 h since the beginning of the UF step for the CTR test and the different pretreatments on AGRD.

All configurations show a monotonic decline from 1 to 10 h, probably justified by progressive fouling and concentration polarization. The CTR tests dropped from 2.52 after 1 h, to 2.11 after 5 h until 1.71 kg m^{-2} bar^{-1} after 10 h, which means a decline of 32% of the permeability performances. This a quite typical trend for UF-PER as colloids and macromolecules accumulate on the membrane, forming a cake which caused the fouling of porosity of the layer. Comparable flux decline profiles on digestate are widely reported for polymeric UF modules, with

Table 3
Permeability values of AGRD along the different pretreatments.

(kg m^{-2} bar)	1 h	5 h	10 h
CTR	2.52 ± 0.28	2.11 ± 0.19	1.71 ± 0.09
COA	2.64 ± 0.31	2.23 ± 0.23	1.80 ± 0.08
FLO	2.26 ± 0.36	1.89 ± 0.24	1.52 ± 0.17
POL	3.30 ± 0.45	1.96 ± 0.11	1.59 ± 0.12
ACD4	4.55 ± 0.41	3.18 ± 0.17	2.04 ± 0.14
ACD7	2.77 ± 0.21	2.02 ± 0.04	1.73 ± 0.10
ACD5	2.82 ± 0.14	1.91 ± 0.07	1.63 ± 0.03
ACD3	4.01 ± 0.30	2.34 ± 0.15	1.95 ± 0.09
HEAT	4.25 ± 0.35	2.07 ± 0.14	1.70 ± 0.23

the exact magnitude depending on Molecular Weight Cutoff (MWCO), Crossflow Velocity (CFV) and pretreatment intensity [55].

COA test maintained slightly higher permeability than CTR throughout, moving from 2.64 to 2.23 and 1.80 kg m^{-2} bar^{-1} after 1, 5 and 10h, respectively ($p < 0.05$). It was consistent with the idea that charge neutralization/sweep floc remove colloids that dominate irreversible fouling. Some previous studies confirm how coagulation/flocculation upstream of low-pressure membranes reduces fouling rates and cake resistance [56,57].

The performance of the flocculant-based tests (FLO and POL) followed a different pattern compared with the COA sample. In the FLO test, the permeability was relatively low from the beginning (2.26 kg m^{-2} bar^{-1} after 1 h) and continued to decline steadily over time, reaching only 1.52 kg m^{-2} bar^{-1} after 10 h. Although the difference is non-significant, this behavior suggests that the flocs formed by the polymeric flocculant were probably soft and highly compressible, leading to the development of a dense and resistant cake layer on the membrane surface. These flocs, typically generated through polymer bridging, are large but fragile: while they initially help clarify the liquid phase, they collapse under filtration pressure, reducing porosity and increasing hydraulic resistance. Furthermore, part of the polymer may have adsorbed directly onto the membrane surface, contributing to pore blocking and an additional loss of permeability over time [58]. The trend of the POL test, which also involved a polymer-based pretreatment, was slightly different ($p < 0.05$). Here, the initial permeability was the highest among all tests (3.30 kg m^{-2} bar^{-1} after 1 h), indicating that the treatment temporarily reduced the viscosity of the feed and improved the hydrodynamic conditions for filtration. However, the permeability dropped sharply after 5 h (1.96 kg m^{-2} bar^{-1}) and continued to decline to 1.59 kg m^{-2} bar^{-1} after 10 h. This rapid flux decay suggests that the initial improvement was short-lived, and that polymer residues may have accelerated fouling by increasing the deposition of organic matter on the membrane and by forming a more compact fouling layer as filtration progressed. Therefore, although polymer-based pretreatments can initially enhance permeability, they tend to promote faster fouling in the long term, particularly when the flocs are unstable or the polymer interacts with the membrane surface [58,59].

Acidification to pH 4 (ACD4 test) significantly ($p < 0.01$) outperforms all others across the window, moving from a value of 4.55 ± 0.41 after 1 h to 3.18 ± 0.17 and 2.04 ± 0.14 kg m^{-2} bar^{-1} after 5 and 10 h, respectively. Two mechanisms align with the literature: (i) protonation of organic/colloidal surfaces reduces electrostatic interactions and cohesion within the fouling layer, improving permeability; (ii) reduced inorganic scaling propensity (e.g., carbonate) at low pH.

With specific reference to acidification tests at different pH values, it was observed that reduced permeability values were found at pH 7 and 5 ($p < 0.05$), while at pH 3 permeability was very close to the one at pH 4 ($p < 0.01$) (Table 3). Consequently, the choice to consider the ACD4 test as the best pretreatment to AGRD is not only due to NH_3/NH_4^+ equilibrium, which depends on the pH reduction, as the NH_3 fraction at pH 6 is already negligible. Rather, the benefit of stronger acidification is likely associated with improved processability of the AGRD as reflected by the higher UF permeability values, together with reduced formation of carbonate and phosphate precipitates and modified colloidal interactions under more acidic conditions. These effects can enhance nutrients retention in the liquid phase and improve the overall efficiency of the ammonium recovery in the RO-RET, as described in the previous sections.

Reviews and experimental studies on digestate and related feeds show that suitable pretreatment (including pH adjustment) stabilizes flux and mitigates fouling; studies on pH-controlled coagulation also report lower UF fouling under acidic conditions [57,60]. It is important to underline that ACD4 resulted in a larger relative decrease in membrane permeability over the investigated operating period (−55% after 10 h) compared to the CTR test (−32%). Anyway, as previously reported in Table 3, it should be noted that the initial permeability under acidified conditions was substantially higher. As a consequence, the absolute permeability values remained consistently higher for the acidified digestate throughout the entire filtration test. Therefore, the relative decline in permeability alone does not fully describe fouling severity, and absolute permeability values must be considered to properly assess membrane performance.

HEAT test began high (4.25 ± 0.35 kg m^{-2} bar^{-1} at 1 h), probably as effect of the viscosity decrease, but converged to CTR by 10 h (1.70 ± 0.23 kg m^{-2} bar^{-1}), indicating the thermal benefit is transient and overshadowed by faster foulant transport/aggregation at elevated temperature ($p < 0.05$). Temperature both lowers viscosity allowing the raising of the initial flux, but also modifies fouling behavior, accelerating organic deposition on the UF membrane [61].

In conclusion, over 10 h, ACD4 is the only pretreatment that sustains a meaningfully higher permeability, while COA offers a modest, steady benefit; FLO/POL suffer from polymer-related fouling dynamics; HEAT gives a short-lived gain that vanishes with time.

3.6. Validation of the best pretreatment at TRL 7

DEMO was designed to treat approximately 20 tonnes per day of AGRD. Table 4 shows the daily flow and the concentrations of all the streams in presence of the best pretreatment from lab scale (ACD4-DEMO test) and without it (CTR-DEMO test).

3.6.1. CTR-DEMO test

DEMO-SLS step generated about 4 tonnes per day of DEMO-SLS-SF, with TS content of 16.50% w/w, and 16 tonnes per day of DEMO-SLS-LF with TS content of 4.50% w/w. The DEMO-SLS-SF consisted mainly of fibrous material and coarse particles with equivalent diameters above 250–500 μm. Nutrient partitioning at this stage was strongly size- and solids-driven: more than 60% w/w of the phosphorus originally present in the AGRD accumulated in the DEMO-SLS-SF, whereas only 17.00%, 16.00% and 20.50% w/w of TKN, ammonium and potassium, respectively, were recovered in this fraction. This confirms that nitrogen- and potassium-based species were largely solubilized and therefore transferred to the DEMO-SLS-LF, in agreement with the laboratory-scale observations. Then, DEMO-SLS-LF was treated by DEMO-UF yielding 3.2 tonnes per day of a retentate (DEMO-UF-RET) with 13.5% w/w TS and 12.8 tonnes per day of permeate (DEMO-UF-PER) with 2.20% w/w TS. Colloidal material, microorganisms, organic nitrogen and non-soluble phosphorus species were mostly retained in DEMO-UF-RET, whereas ammonium, soluble phosphorus and potassium permeated the membrane and accumulated in DEMO-UF-PER. As a consequence, the ammonium/TKN ratio, which was close to 70% in the AGRD and in all previous streams, increased to 77.90% in DEMO-UF-RET and decreased to 54.55% in DEMO-UF-PER. The recovery yields of TKN, ammonium, phosphorus and potassium in DEMO-UF-RET were 28.80%, 22.40%, 21.30% and 17.95% w/w, respectively. The DEMO-UF-PER stream was then processed through RO, producing 2.8 tonnes per day of RO retentate (DEMO-RO-RET) with a TS content of 9.00% w/w, enriched in ammonium, potassium and the remaining soluble phosphate. The permeate (DEMO-RO-PER), consisting of approximately 10

Table 4
Mass flow rates and chemical composition of the main process streams from DEMO plant operated without pretreatment (CTR) and with ACD4 pretreatment. AGRD: raw agricultural digestate fed to the demonstrative plant; DEMO-SLS-LF: liquid fraction obtained after the primary solid–liquid separation; DEMO-SLS-SF: solid fraction obtained after SLS; DEMO-UF-PER: ultrafiltration permeate; DEMO-UF-RET: ultrafiltration retentate; DEMO-RO-PER: reverse osmosis permeate; DEMO-RO-RET: reverse osmosis retentate.

		Flow rates (tonne d^{-1})	TS % w/w)	TS tonne d^{-1})	COD (g kg^{-1})	COD (tonne d^{-1})	TKN (g kg^{-1})	TKN (kg d^{-1})	Ammonium (g kg^{-1})	Ammonium (kg d^{-1})	TP (g kg^{-1})	TP (kg d^{-1})	K_2O (g kg^{-1})	K_2O (kg d^{-1})
CTR-DEMO	AGRD	20.0	7.0	1.4	780.0	15.6	5.0	100.0	3.5	70.0	1.5	30.0	4.1	82.0
	DEMO- SLS -LF	16.0	4.5	0.7	500.0	8.0	5.2	83.2	3.7	59.2	0.7	11.2	4.1	65.6
	DEMO- SLS -SF	4.0	16.5	0.7	1,850.0	7.4	4.2	16.6	2.8	11.0	4.6	18.4	4.0	16.0
	DEMO-UF -PER	12.8	2.2	0.3	245.0	3.1	4.3	55.0	3.4	43.5	0.4	5.2	4.0	50.6
	DEMO-UF-RET	3.2	13.5	0.4	1,450.0	4.6	8.9	28.5	4.8	15.4	1.9	5.9	4.7	15.0
	DEMO-RO-PER	10.0	0.2	0.0	5.0	0.0	0.1	0.9	0.1	0.5	0.0	0.0	0.1	0.5
	DEMO-RO-RET	2.8	9.0	0.3	1,100.0	3.1	19.1	53.8	15.2	42.8	1.8	5.1	17.6	49.6
ACD4-DEMO	AGRD	20.0	7.0	1.4	780.0	15.6	5.0	100.0	3.5	70.0	1.5	30.0	4.1	82.0
	DEMO- SLS -LF	16.0	4.5	0.7	500.0	8.0	5.2	83.2	3.7	59.2	0.7	11.2	4.0	64.0
	DEMO- SLS -SF	4.0	16.5	0.7	1,850.0	7.4	4.2	16.6	2.8	11.0	4.6	18.4	4.2	16.8
	DEMO-UF -PER	13.1	2.2	0.3	245.0	3.2	3.7	48.5	3.6	47.2	0.4	5.2	3.9	50.4
	DEMO-UF-RET	2.9	13.5	0.4	1,450.0	4.2	11.9	34.5	4.2	12.2	2.0	5.8	4.6	13.3
	DEMO-RO-PER	10.5	0.2	0.0	5.0	0.1	0.1	1.0	0.1	1.0	0.0	0.0	0.1	0.5
	DEMO-RO-RET	2.6	9.0	0.2	1,100.0	2.9	18.1	47.4	17.5	45.9	1.8	4.7	17.3	45.3

tonnes per day, was essentially purified water with a TS content of only 0.20% w/w. Recovery yields of TKN, ammonium, phosphorus and potassium in DEMO-RO-RET were 53.22%, 60.75%, 16.90% and 49.80% w/w, respectively, demonstrating a clear fractionation of nutrients across the DEMO outstreams. Regarding the RENURE criteria, DEMO-RO-RET met the TOC/TKN requirement, with a value of 1.35 (<3), but did not satisfy the NH_4^+/TKN threshold, reaching 79.60%, which is lower than the required 90%. These findings indicate that an upstream pretreatment of the AGRD is necessary to achieve a RENURE-compliant fertilizer product.

3.6.2. ACD4-DEMO test

The most effective pretreatment identified at laboratory scale, namely ACD4, was subsequently implemented at demonstrative scale by applying it to the DEMO-SLS-LF after the SLS. In line with the laboratory-scale results, ACD4 did not significantly affect the mass distribution during the DEMO-UF step, with the DEMO-UF-PER and DEMO-UF-RET fractions remaining close to 80% and 20% w/w, respectively. Likewise, the recovery yields of total phosphorus and potassium were not substantially influenced by the acidification step, confirming the conservative behavior of these nutrients under the applied conditions. In contrast, ACD4-DEMO exerted a clear positive effect on ammonium concentration in the final RO retentate. Specifically, the ammonium concentration increased from 15.2 g kg^{-1} in the CTR-DEMO test to 17.5 g kg^{-1} in the ACD4-DEMO test, while the NH_4^+/TKN ratio increased from 79.60% to 96.65% w/w. These results confirm, also at demonstrative scale, that acidification at pH 4 improves the effectiveness of the integrated membrane treatment chain for ammonium concentration. As already discussed for laboratory-scale trials, this effect should not be interpreted as the consequence of a single isolated mechanism. Rather, the demonstrative-scale results are consistent with the combined action of several favorable effects induced by stronger acidification, including improved retention of ammonium in the liquid phase, reduced tendency to precipitation phenomena, and improved processability of the feed stream entering the membrane units. In this sense, the DEMO results validate the choice of ACD4 as the best operational compromise previously identified at laboratory scale. Direct comparison of this work with previous demonstrative- or full-scale results remains challenging, as only limited and heterogeneous data are available in the literature for AGRD treatment chains. Nevertheless, comparable full-scale field studies have evaluated different SLS systems in agricultural biogas plants. In such studies, nitrogen diversion to the solid fraction ranged from approximately 13–14% for screw and roller separators up to about 38% for centrifuges operated as part of multi-stage processes, while phosphorus diversion values as high as 80% were reported in the solid fraction. These results highlight the strong influence of separation technology and operating conditions on nutrient partitioning and are consistent with the trends observed at demonstrative scale in the present study. It should be noted, however, that these studies primarily focused on mechanical separation performance, whereas the present work further investigates the integration with membrane-based concentration steps [62].

4. Conclusions

This work demonstrates that pretreatment is a fundamental design variable in membrane-based processing of AGRD. Among the investigated strategies, acidification at pH 4 emerged as the most effective approach, as it provided the best overall compromise between ammonium recovery, UF permeability and process stability. Coagulation provided only limited improvements in solids removal and membrane operability, whereas polymeric flocculation promoted faster fouling. Moderate heating, although initially reducing viscosity, proved detrimental to nitrogen recovery due to significant ammonium losses and therefore was not suitable for RENURE-oriented applications. Phosphorus and potassium exhibited a more conservative trend across the different pretreatments, indicating that their recovery was mainly governed by physical separation rather than chemical conditioning under the investigated conditions.

The validation of ACD4 at TRL 7 confirmed the transferability of the laboratory findings to real operating conditions and highlighted its potential for producing RENURE-oriented nutrient concentrates. Further work should address final product conditioning and the optimization of acid consumption at larger scale.

CRediT authorship contribution statement

Fabio Rizzioli: Writing – original draft, Methodology, Investigation, Data curation, Conceptualization. **Tommaso Della Gatta:** Investigation. **David Bolzonella:** Validation, Supervision. **Federico Battista:** Writing – original draft, Supervision, Funding acquisition, Conceptualization.

Declaration of competing interest

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

Acknowledgement

This research was financially funded by the European "Life Dimitra" project, LIFE22-CCM-EL-DIMITRA (project number: 101113253).

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.rineng.2026.111355.

Data availability

Data will be made available on request.

References

[1] L. Wester-Larsen, D.S. Müller-Stöver, T. Salo, L.S. Jensen, Potential ammonia volatilization from 39 different novel biobased fertilizers on the European market – A laboratory study using 5 European soils, J. Environ. Manage. 323 (2022) 116249, https://doi.org/10.1016/j.jenvman.2022.116249.

[2] G. Ferretti, G. Galamini, V. Medoro, B. Faccini, Amount and speciation of N leached from a sandy soil fertilized with urea, liquid digestate, struvite and <scp>NH 4 </scp>-enriched chabazite zeolite-tuff, Soil Use Manag. 39 (2023) 456–473, https://doi.org/10.1111/sum.12855.

[3] F. Rizzioli, M. Cirilli, N. Frison, D. Bolzonella, F. Battista, Nutrient recovery from anaerobic digestate by different combination of pressure driven membranes, J. Clean. Prod. 494 (2025) 144958, https://doi.org/10.1016/j.jclepro.2025.144958.

[4] Phosphorus Platform, Commission 'Renure' Nitrates Directive proposal moves towards adoption, Phosphorsus Platform (2025), accessed November 17, https://www.phosphorusplatform.eu/scope-in-print/enews/2713-espp-enews-no-100-september-2025#_Toc209707087, 2025. accessed November 17.

[5] European Council, Protection of waters against pollution caused by nitrates from agricultural sources, Off. J. Eur. Union 34 (1991) 1–8.

[6] European Commission, European Commission amending Council Directive 91/676/EEC as regards the use of certain fertilising materials from livestock manure, European Commission, 2025. https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=PI_COM:Ares(2024)2885619. accessed November 18, 2025.

[7] F. Rizzioli, D. Bertasini, D. Bolzonella, N. Frison, F. Battista, A critical review on the techno-economic feasibility of nutrients recovery from anaerobic digestate in the agricultural sector, Sep. Purif. Technol. 306 (2023), https://doi.org/10.1016/j.seppur.2022.122690.

[8] J. Jamadi-Torab, A. Malekzadeh, M. Rahbari-Sisakht, A.F. Ismail, State-of-the-art in forward osmosis membrane process: materials, fabrication, and applications, Chem. Eng. Process. - Process Intensif. 216 (2025) 110385, https://doi.org/10.1016/j.cep.2025.110385.

[9] C. Vaneeckhaute, V. Lebuf, E. Michels, E. Belia, P.A. Vanrolleghem, F.M.G. Tack, E. Meers, Nutrient recovery from Digestate: systematic technology review and product classification, Waste Biomass Valorization 8 (2017) 21–40, https://doi.org/10.1007/s12649-016-9642-x.

[10] E.M. Barampouti, S. Mai, D. Malamis, K. Moustakas, M. Loizidou, Exploring technological alternatives of nutrient recovery from digestate as a secondary resource, Renew. Sustain. Energy Rev. 134 (2020) 110379, https://doi.org/10.1016/j.rser.2020.110379.
[11] F. Waeger, T. Delhaye, W. Fuchs, The use of ceramic microfiltration and ultrafiltration membranes for particle removal from anaerobic digester effluents, Sep. Purif. Technol. 73 (2010) 271–278, https://doi.org/10.1016/j.seppur.2010.04.013.
[12] T. Gienau, U. Brüß, M. Kraume, S. Rosenberger, Nutrient recovery from Biogas digestate by optimised membrane treatment, Waste Biomass Valorization 9 (2018) 2337–2347, https://doi.org/10.1007/s12649-018-0231-z.
[13] M. Zielińska, W. Mikucka, Membrane filtration for valorization of digestate from the anaerobitreatment of distillery stillage, Desalin. Water Treat 215 (2021) 60–68, https://doi.org/10.5004/dwt.2021.26772.
[14] E. Monfet, G. Aubry, A.A. Ramirez, Nutrient removal and recovery from digestate: a review of the technology, Biofuels 9 (2018) 247–262, https://doi.org/10.1080/17597269.2017.1336348.
[15] V. Proskynitopoulou, I. Garagounis, A. Vourros, P.Dimopoulos Toursidis, S. Lorentzou, A. Zouboulis, K. Panopoulos, Nutrient recovery from digestate: pilot test experiments, J. Environ. Manage. 353 (2024) 120166, https://doi.org/10.1016/j.jenvman.2024.120166.
[16] B. Qi, X. Jiang, H. Wang, J. Li, Q. Zhao, R. Li, W. Wang, Resource recovery from liquid digestate of swine wastewater by an ultrafiltration membrane bioreactor (UF-MBR) and reverse osmosis (RO) process, Environ. Technol. Innov. 24 (2021) 101830, https://doi.org/10.1016/j.eti.2021.101830.
[17] M. Konucu, D. Tekdal, E.Eker Develi, E. Meers, M. Fernandes de Souza, Moringa oleifera Lam. As a bioflocculant for harvesting microalgae grown on agricultural wastewaters for feed production, Appl. Sci. 12 (2022) 12968, https://doi.org/10.3390/app122412968.
[18] K. Meixner, W. Fuchs, T. Valkova, K. Svardal, C. Loderer, M. Neureiter, G. Bochmann, B. Drosg, Effect of precipitating agents on centrifugation and ultrafiltration performance of thin stillage digestate, Sep. Purif. Technol. 145 (2015) 154–160, https://doi.org/10.1016/j.seppur.2015.03.003.
[19] J.L. Van Puffelen, C. Brienza, I.C. Regelink, I. Sigurnjak, F. Adani, E. Meers, O. F. Schoumans, Performance of a full-scale processing cascade that separates agricultural digestate and its nutrients for agronomic reuse, Sep. Purif. Technol. 297 (2022), https://doi.org/10.1016/j.seppur.2022.121501.
[20] A. Pantelopoulos, J. Magid, L.S. Jensen, D. Fangueiro, Nutrient uptake efficiency in ryegrass fertilized with dried digestate solids as affected by acidification and drying temperature, Plant Soil 421 (2017) 401–416, https://doi.org/10.1007/s11104-017-3463-y.
[21] J. Liu, D.S. Müller-Stöver, L.S. Jensen, Acidification prior to drying of digestate solids affects nutrient uptake and fertilizer value when applied to maize, Sustain. Mater. Technol. 41 (2024) e01020, https://doi.org/10.1016/j.susmat.2024.e01020.
[22] J. O'Connor, B.S. Mickan, S.K. Gurung, K.H.M. Siddique, M. Leopold, N.S. Bolan, Enhancing nutrient recovery from food waste anaerobic digestate, Bioresour. Technol. 390 (2023) 129869, https://doi.org/10.1016/j.biortech.2023.129869.
[23] G. Beggio, W. Peng, F. Lü, A. Cerasaro, T. Bonato, A. Pivato, Chemically enhanced solid–Liquid separation of digestate: suspended solids removal and effects on environmental quality of separated fractions, Waste Biomass Valorization 13 (2022) 1029–1041, https://doi.org/10.1007/s12649-021-01591-y.
[24] M.S. Camilleri-Rumbau, K. Briceño, L. Fjerbæk Søtoft, K.V. Christensen, M.C. Roda-Serrat, M. Errico, B. Norddahl, Treatment of manure and digestate liquid fractions using membranes: opportunities and challenges, Int. J. Environ. Res. Public Health 18 (2021) 3107, https://doi.org/10.3390/ijerph18063107.
[25] APHA, Standard Methods for Examination of Water and Wastewater,, American Public Health Association, 1998, 20th ed.
[26] B. Van der Bruggen, Microfiltration, ultrafiltration, nanofiltration, reverse osmosis, and forward osmosis. Fundamental Modelling of Membrane Systems, Elsevier, 2018, pp. 25–70, https://doi.org/10.1016/B978-0-12-813483-2.00002-2.
[27] W.R. Bowen, F. Jenner, Theoretical descriptions of membrane filtration of colloids and fine particles: an assessment and review, Adv. Colloid Interface Sci. 56 (1995) 141–200, https://doi.org/10.1016/0001-8686(94)00232-2.
[28] M. Fechter, I.P. Petrova, M. Kraume, Balance of total mass and nitrogen fluxes through consecutive digestate processing steps: two application cases, J. Environ. Manage. 326 (2023) 116791, https://doi.org/10.1016/j.jenvman.2022.116791.
[29] G.P.S. Ibrahim, A.M. Isloor, R. Farnood, Fundamentals and basics of reverse osmosis. Current Trends and Future Developments on (Bio-) Membranes, Elsevier, 2020, pp. 141–163, https://doi.org/10.1016/B978-0-12-816777-9.00006-X.
[30] J. Schaep, Modelling the retention of ionic components for different nanofiltration membranes, Sep. Purif. Technol. 22–23 (2001) 169–179, https://doi.org/10.1016/S1383-5866(00)00163-5.
[31] M. Fechter, I.P. Petrova, M. Kraume, Balance of total mass and nitrogen fluxes through consecutive digestate processing steps: two application cases, J. Environ. Manage. 326 (2023) 116791, https://doi.org/10.1016/j.jenvman.2022.116791.
[32] S. Mazzini, G. Borgonovo, L. Scaglioni, F. Bedussi, G. D'Imporzano, F. Tambone, F. Adani, Phosphorus speciation during anaerobic digestion and subsequent solid/liquid separation, Sci. Total Environ. 734 (2020) 139284, https://doi.org/10.1016/j.scitotenv.2020.139284.
[33] J.A. Molina-Bolívar, J.L. Ortega-Vinuesa, How proteins stabilize colloidal particles by means of hydration forces, Langmuir 15 (1999) 2644–2653, https://doi.org/10.1021/la981445s.
[34] E. Monfet, G. Aubry, A.A. Ramirez, Nutrient removal and recovery from digestate: a review of the technology, Biofuels 9 (2018) 247–262, https://doi.org/10.1080/17597269.2017.1336348.
[35] K.A.S. Meraz, S.M.P. Vargas, J.T.L. Maldonado, J.M.C. Bravo, M.T.O. Guzman, E.A. L. Maldonado, Eco-friendly innovation for nejayote coagulation–flocculation process using chitosan: evaluation through zeta potential measurements, Chem. Eng. J. 284 (2016) 536–542, https://doi.org/10.1016/j.cej.2015.09.026.
[36] H. Xu, S. Wei, G. Li, B. Guo, Advanced removal of phosphorus from urban sewage using chemical precipitation by Fe-Al composite coagulants, Sci. Rep 14 (2024) 4918, https://doi.org/10.1038/s41598-024-55713-2.
[37] A.A. Owodunni, S. Ismail, S.B. Kurniawan, A. Ahmad, M.F. Imron, S.R.S. Abdullah, A review on revolutionary technique for phosphate removal in wastewater using green coagulant, J. Water Process Eng. 52 (2023) 103573, https://doi.org/10.1016/j.jwpe.2023.103573.
[38] N.P. Kocatürk-Schumacher, S. Bruun, K. Zwart, L.S. Jensen, Nutrient recovery from the liquid fraction of digestate by clinoptilolite, Clean (Weinh) (2017) 45, https://doi.org/10.1002/clen.201500153.
[39] M. Farghali, Z. Chen, A.I. Osman, I.M. Ali, D. Hassan, I. Ihara, D.W. Rooney, P.-S. Yap, Strategies for ammonia recovery from wastewater: a review, Environ. Chem. Lett. 22 (2024) 2699–2751, https://doi.org/10.1007/s10311-024-01768-6.
[40] L. Zhang, S. Wang, L. Jiao, Y. Li, J. Yang, R. Zhang, S. Feng, J. Wang, Effects of organic matter content and composition on ammonium adsorption in lake sediments, Environ. Sci. Pollut. Res. 23 (2016) 6179–6187, https://doi.org/10.1007/s11356-015-5820-9.
[41] O. Popovic, M. Hjorth, L.Stoumann Jensen, Phosphorus, copper and zinc in solid and liquid fractions from full-scale and laboratory-separated pig slurry, Environ. Technol. 33 (2012) 2119–2131, https://doi.org/10.1080/09593330.2012.660649.
[42] R.I.S. Gill, T.M. Herrington, The effect of surface charge on the flocculation of Kaolin suspensions with cationic polyacrylamides of varying molar mass but similar cationic character, Colloids Surf. 25 (1987) 297–310, https://doi.org/10.1016/0166-6622(87)80310-4.
[43] M. Hjorth, M.L. Christensen, P.V. Christensen, Flocculation, coagulation, and precipitation of manure affecting three separation techniques, Bioresour. Technol. 99 (2008) 8598–8604, https://doi.org/10.1016/j.biortech.2008.04.009.
[44] F. Tambone, V. Orzi, M. Zilio, F. Adani, Measuring the organic amendment properties of the liquid fraction of digestate, Waste Manage. 88 (2019) 21–27, https://doi.org/10.1016/j.wasman.2019.03.024.
[45] A. Pantelopoulos, J. Magid, L.S. Jensen, Thermal drying of the solid fraction from biogas digestate: effects of acidification, temperature and ventilation on nitrogen content, Waste Manag. 48 (2016) 218–226, https://doi.org/10.1016/j.wasman.2015.10.008.
[46] Z. Deng, N. van Linden, E. Guillen, H. Spanjers, J.B. van Lier, Recovery and applications of ammoniacal nitrogen from nitrogen-loaded residual streams: A review, J. Environ. Manage. 295 (2021) 113096, https://doi.org/10.1016/j.jenvman.2021.113096.
[47] A. Serna-Maza, S. Heaven, C.J. Banks, Biogas stripping of ammonia from fresh digestate from a food waste digester, Bioresour. Technol. 190 (2015) 66–75, https://doi.org/10.1016/j.biortech.2015.04.041.
[48] L. Shi, S. Xie, Z. Hu, G. Wu, L. Morrison, P. Croot, H. Hu, X. Zhan, Nutrient recovery from pig manure digestate using electrodialysis reversal: membrane fouling and feasibility of long-term operation, J. Memb. Sci. 573 (2019) 560–569, https://doi.org/10.1016/j.memsci.2018.12.037.
[49] T. Gienau, M. Kraume, S. Rosenberger, Biopolymer interactions of anaerobic sludge and their influence on membrane performance, J. Memb. Sci. 564 (2018) 634–642, https://doi.org/10.1016/j.memsci.2018.07.066.
[50] A. Urbanowska, I. Polowczyk, M. Kabsch-Korbutowicz, P. Seruga, Characteristics of changes in particle size and zeta potential of the digestate fraction from the municipal waste biogas plant treated with the use of chemical coagulation/precipitation processes, Energies (Basel) 13 (2020) 5861, https://doi.org/10.3390/en13225861.
[51] G. Cocolo, M. Hjorth, A. Zarebska, G. Provolo, Effect of acidification on solid–liquid separation of pig slurry, Biosyst. Eng. 143 (2016) 20–27, https://doi.org/10.1016/j.biosystemseng.2015.11.004.
[52] C. Pintucci, M. Carballa, S. Varga, J. Sarli, L. Peng, J. Bousek, C. Pedizzi, M. Ruscalleda, E. Tarragó, D. Prat, G. Colica, M. Picavet, J. Colsen, O. Benito, M. Balaguer, S. Puig, J.M. Lema, J. Colprim, W. Fuchs, S.E. Vlaeminck, The ManureEcoMine pilot installation: advanced integration of technologies for the management of organics and nutrients in livestock waste, Water Sci. Technol. 75 (2017) 1281–1293, https://doi.org/10.2166/wst.2016.559.
[53] S.G. Sommer, L.S. Jensen, S.B. Clausen, H.T. Sogaard, Ammonia volatilization from surface-applied livestock slurry as affected by slurry composition and slurry infiltration depth, J. Agric. Sci. 144 (2006) 229–235, https://doi.org/10.1017/S0021859606006022.
[54] N.C.T. Ellersiek, H.-W. Olfs, An incubation system for the simulation of ammonia emissions from soil surface-applied slurry: effect of pH and acid type, Agronomy 14 (2024) 1078, https://doi.org/10.3390/agronomy14051078.
[55] C. Yue, Y. Chen, W. Zhang, Y. Zheng, X. Hu, B. Shang, Direct purification of digestate using polymeric ultrafiltration membranes: influence of materials on filtration behavior and fouling characteristics, Membranes (Basel) 12 (2022) 882, https://doi.org/10.3390/membranes12090882.
[56] M. Xu, Y. Luo, X. Wang, L. Zhou, Coagulation-ultrafiltration efficiency of polymeric Al-, Fe-, and Ti- coagulant with or without polyacrylamide composition, Sep. Purif. Technol. 280 (2022) 119957, https://doi.org/10.1016/j.seppur.2021.119957.
[57] A. Chuda, K. Ziemiński, Ultrafiltration of digestate liquid fraction by hollow-fiber membranes: influence of digestate pre-treatment on hydraulic capacity and nutrient removal efficiency, Chem. Eng. J. 473 (2023), https://doi.org/10.1016/j.cej.2023.145426.
[58] Y. Long, X. You, Y. Chen, H. Hong, B.-Q. Liao, H. Lin, Filtration behaviors and fouling mechanisms of ultrafiltration process with polyacrylamide flocculation for

water treatment, Sci. Total Environ. 703 (2020) 135540, https://doi.org/10.1016/j.scitotenv.2019.135540.
[59] J. Ji, J. Li, J. Qiu, X. Li, Polyacrylamide–starch composite flocculant as a membrane fouling reducer: key factors of fouling reduction, Sep. Purif. Technol. 131 (2014) 1–7, https://doi.org/10.1016/j.seppur.2014.04.013.
[60] H. Dong, B. Gao, Q. Yue, Y. Wang, Q. Li, Effect of pH on floc properties and membrane fouling in coagulation – Ultrafiltration process with ferric chloride and polyferric chloride, Chemosphere 130 (2015) 90–97, https://doi.org/10.1016/j.chemosphere.2015.03.049.
[61] G.-J. Liu, Y. Liu, Z.-Y. Wang, Y.-H. Lei, Z.-A. Chen, L.-W. Deng, The effects of temperature, organic matter and time-dependency on rheological properties of dry anaerobic digested swine manure, Waste Manag. 38 (2015) 449–454, https://doi.org/10.1016/j.wasman.2014.12.015.
[62] A. Chiumenti, F. da Borso, S. Limina, B. Piaia, Single-stage and multi-stage liquid/solid separation of digestate in full scale biogas plants. 2024 Anaheim, California July 28-31, 2024, American Society of Agricultural and Biological Engineers, 2024, https://doi.org/10.13031/aim.202400461.