Separation and Purification Technology 306 (2023) 122690

Contents lists available at ScienceDirect

Separation and Purification Technology

journal homepage: www.elsevier.com/locate/seppur

Review

A critical review on the techno-economic feasibility of nutrients recovery from anaerobic digestate in the agricultural sector

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ARTICLE INFO

Keywords:
Agricultural digestate
Nutrient recovery
Anaerobic digestion
Biobased fertilizers
Ammonia stripping
Pressure driven membrane
Struvite precipitation

ABSTRACT

The unsustainable environmental impact of fossil derived fertilizers and the depletion of raw materials for their production make the research of alternative sources for fertilizers production one of the main priorities of the international agenda. Agricultural digestate, especially if derived from livestock manure, is considered a potential candidate for the recovery of bio-based fertilizers: 180 M tonnes of digestate, which contain high concentrations of nitrogen (2–5 kg/m^3) and phosphorous (0.5–1.5 kg/m^3), are produced annually in the EU. This paper overviews different technologies, their robustness and yields in terms of nutrients recovery for different bio-based fertilizers. Ammonia stripping and struvite precipitation are the technologies more developed at full scale allowing an average removal and recovery yield of 80–90 % for nitrogen and phosphorous, respectively. Due to their high degree of development, costs for ammonia stripping and struvite precipitation are relatively low, ranging from 2 to 7 € per kg of nutrient recovered. However, other technologies are rapidly developing and spreading. Pressure-driven membrane technologies (ultrafiltration and reverse osmosis) are emerging as important processes for nutrient recovery, although not selective. Nitrogen and phosphorous removal efficiencies for these technologies are around 75–95 % and 85–99 %, respectively. The main drawback of membrane processes is represented by the membrane fouling which prevents their large diffusion at full scales because of management difficulties. Moreover, the operational cost of this process is higher compared to other available technologies, ranging from 4 to 12 € per m^3 of digestate. Full-scale applications for membrane technologies are available, but still limited, while several studies at laboratory and pilot scale are currently under development. Membrane contactors are receiving attention for the possibility to strip ammonia directly in the membrane with high ammonia recovery (over the 95 %). But this technology is currently developed at low TRL in the EU with several studies at laboratory and pilot scale. Ion exchange, adsorption, evaporation, and electrodialysis are also discussed in this review paper. The difficulty to have solid matrices able to assure a selective nitrogen/phosphorous compound adsorption and the need for regeneration has limited the ion exchange process at full TRL for treatment of agricultural digestate. However, some pilot scale applications of this technology have been investigated for wastewater treatment. Similarly, the high economic and energetic costs for evaporation and electrodialysis have prevented further applications of these techniques for pilot and full scale operation.

1. Introduction

World population will reach 9.7 and 11.2 billion by 2050 and 2100 respectively, 34 and 53 percent more than the current level [1,2]. At the same time the most recent demographic forecasts emphasized that population boost will mainly involve the developing Countries, and in particular their urban areas: about 70 percent of the world population will live in cities in 2050. This migration from rural to urban areas, and

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Abbreviations: AD, anaerobic Digestion; CHP, Combined Heat and Power; CMC, Component Material Categories; COD, Chemical Oxygen Demand; EBA, European Biogas Association; EC, European Council; ED, Electrodialysis; EU, European Union; FAO, Food and Agriculture Organization; HFMC, Hollow-Fibre Membrane Contactors; HTC, Hydrothermal Carbonization; IEX, Ion EXchange; MAP, Magnesium Ammonium Sulphate; MF, Microfiltration; NVZs, Nitrate Vulnerable Zones; OECD, Organisation for Economic Co-operation and Development; OFMSW, Organic Fraction of Municipal Solid Waste; OUR, Oxygen Uptake Rate; PP, Polypropylene; PTFE, Polytetrafluoroethylene; RO, Reverse Osmosis; TAN, Total Ammoniacal Nitrogen; TRL, Technology Readiness Level; TS, Total Solids; UF, Ultrafiltration; VFAs, Volatile Fatty Acids; vS, Voltile solids; WWTP, Wastewater Treatment Plant.

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https://doi.org/10.1016/j.seppur.2022.122690
Received 29 August 2022; Received in revised form 7 November 2022; Accepted 14 November 2022
Available online 17 November 2022
1383-5866/

the subsequent difference of working and living conditions, will significantly affect the dietary requirements and food demand of the population [2].

In order to feed this larger, more urban and richer population, crops production yield is expected to increase by around 30 % in 2050 [2]. The cereal production increase is projected to 336 Mt in the next decade [3]. The request for animal proteins will increase too [4]: according to the OECD-FAO Agricultural Outlook 2021 – 2030 [3], meat consumption will increase by 14 % by 2030, reaching 35.4 kg/y in retail weight equivalent. Therefore, the production of crops for livestock feeding will increase as well, reaching almost 2 billion tonnes by 2030. As a consequence, the current global demand for fertilizers is 200 million tonnes, with a progressive reduction of supply and demand balances over the years [5].

To overcome this situation several activities should be undertaken: to enlarge the availability of arable land, to improve agricultural efficiency, and, importantly, to increase the availability of fertilizers. In addition, all these actions have to take into consideration the environmental impact, implementing a sustainable intensification of agriculture, reducing nutrient losses, crop protection chemicals and greenhouse gases emissions [2,6].

Nitrogen (N), Phosphorus (P) and Potassium (K) are the main components of fossil-based, mineral fertilizers [7]. Because of their fossil-based and non-renewable origin, the production of conventional fertilizers is unsustainable, with concerns over long-term availability [8]. The conventional, and most used, ammonia-based fertilizer production process is represented by the Haber-Bosch process, which was developed more than one century ago, in 1913. Atmospheric molecular nitrogen (N_2) is converted to ammonia by reacting with hydrogen (H_2), at pressures and temperatures up to 100 bar and 500 °C, respectively, at the presence of a heterogeneous catalyst, usually iron. N_2 is extracted from air, while H_2 is produced by steam-methane reforming, from fossil natural gas, with temperatures up to 1,000 °C. The Haber-Bosch process produces 1.5 tonnes CO_2 per tonne of produced NH_3, with an energy consumption of around 6.4×10^{12} MJ/year, 2 % of worldwide total energy consumption [9–11], and accounting for 0.93 % of worldwide greenhouse gas emission [12]. With specific reference to conventional phosphorus-based fertilizers, these are produced by extraction of mineral phosphate rocks, generally as impure calcium phosphate. The extracted rock is then treated with sulphuric acid to produce raw phosphoric acid (H_3PO_4) and gypsum as a waste. The raw phosphoric acid, which can contain pollutants and heavy metals, is refined in order to remove such pollutants, and to obtain more soluble and thus bioavailable phosphate fertilizers [13]. Phosphate rocks are non-renewable and high requested sources. According to Mehta et al. [8], by 2033 the demand of phosphoric fertilizers will be higher than the availability, causing a rapid depletion of reserves. Moreover, 90 % of phosphate rock reserves are located in few countries, especially Morocco, Iraq, China, Algeria, and Syria. Regarding the greenhouse gas emissions, superphosphate ($Ca(H_2PO_4)_2$) and ammonium phosphate ($(NH_4)_3PO_4$) fertilizers produce 0.4–1.6 and 1.3–8.9 $kgCO_2/kgP_2O_5$ respectively [14]. The energetic consumption corresponds to 7700 kJ/kg phosphate [15]. In addition, phosphorus is considered strategically and critical raw material for the European Union (EU) [16].

The EU accounts for 133.9 million hectares of fertilized agricultural land [17], mainly cultivated intensively, as illustrated in Fig. 1. Europe consumed 11.2 million tonnes of mineral fertilizers in 2020, with a consumption increase of 6.9 and 21.9 % for N and P respectively compared to 2010 [18]. Moreover, 30 %, 68 % and 85 % of N, P and K respectively of the total nutrient consumption was imported, mainly from Russia and Belarus [19]. The recent Ukrainian-Russian war remarked how EU is not independent in the supplying of resources. The Russian Federation, economically sanctioned by the EU, is the largest natural gas and fertilizers exporter to the EU, with over 155 billion cubic

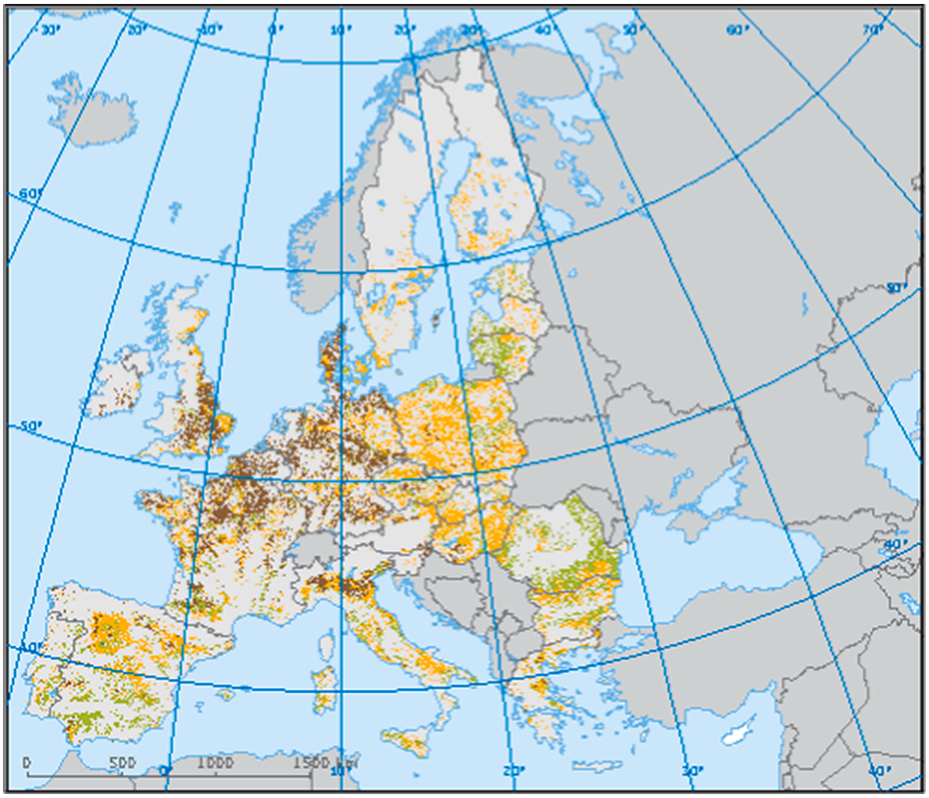

Fig. 1. Agricultural land use intensity [21]. Green: extensive used arable land; Yellow: moderately intensive used arable land; Brown: intensive used arable land; Light gray: non agricultural land; White: no data; Dark gray: outside coverage. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

meters (bcm) of natural gas and 1.1 billion € of fertilizers exports [19,20].

The unsustainability of the classic fertilizers production, the depletion of the conventional P-based fertilizer reserves, combined with the increasing instability of the geopolitical scenario, highlight that the research of new biobased alternatives for fertilizers production is now urgent and necessary. The intensive agriculture and livestock concentration across Europe could offer a valid source of nutrients and an alternative to classic synthetic fertilizers. In 2020, the EU crops production reached over 285 and 61 million tonnes of cereals and fresh vegetables respectively, with a consequent generation of over 4 million tonnes of vegetal wastes [22,23]. Regarding the EU livestock production, in 2021, there were over 1.5 billion poultries, 141 million pigs, 75 million bovines and 75 million sheep and goats [24,25], with a global production exceeding 1.4 billion tonnes per year of manure between 2016 and 2019 [26]. The organic wastes derived from these agricultural and farming activities are often treated by Anaerobic Digestion (AD) for biogas production, whose main byproduct is represented by digestate rich in nutrient compounds. This review article analyzed different technologies for the extractions of nitrogen and phosphorous compounds, discussing on their removal yields and their Technology Readiness Level (TRL) in the EU.

1.1. *Agricultural residues and manure characterization*

The typical physical–chemical characteristics of crop residues and

Table 1
Agricultural residues and manure physical–chemical characterization. n.d.: not determined.

Source	Total Solids	Volatile Solids	Total Nitrogen	Ammonia	Total Phosphorus	Reference
Crop Residues						
Barley	26.31 %w/w	25.44 %w/w	12.31 mg/g	0.53 mg/g	n.d.	[29]
	25.8–66.3 %w/w	25.1–59.1 %w/w	7.0–19.9 g/kg	n.d.	0.8–3.9 g/kg	[30]
Barley Straw	86.83 ± 0.13 %w/w	81.37 %w/w	0.76 %TS	n.d.	n.d.	[31]
Corn	20.1–40.4 %w/w	18.3–36.6 %w/w	4.0–5.8 g/kg	n.d.	0.3–0.6 g/kg	[30]
Corn Straw	90.0 ± 0.2 %w/w	81.3 ± 0.1 %w/w	1.3 ± 0 %TS	n.d.	n.d.	[32]
	66.91 ± 0,95 %w/w	76.22 ± 0.54 %TS	0.78 ± 0.03 %TS	n.d.	n.d.	[33]
Fresh Vinegar	32.56 ± 1.32 %w/w	31.01 ± 0.97 %w/w	1.92 ± 0.05 %TS	n.d.	n.d.	[34]
Rice Husks	90.7 %w/w	74 %w/w	25.5 g/kg	n.d.	3.5 g/kg	[30]
Triticale	30–30.8 %w/w	27.9 %w/w	13.5 g/kg	n.d.	0.7 g/kg	[30]
Livestock Effluents						
Cows	15.9 ± 0.2 %w/w	78.4 ± 0.2 %TS	25.9 mg/gVS	n.d.	n.d.	[35]
	9.50 %w/w	80 %TS	0.351 %w/w	n.d.	0.082 %w/w	[36]
	208.4 ± 1.9 g/kg	166.4 ± 1.8 g/kg	6.1 ± 0.3 g/kg	2.0 ± 0.0 g/kg	1.9 ± 0.3 g/kg	[37]
	15.6–47.7 %w/w	13.5–32.1 %w/w	3.2–7.1 g/kg	n.d.	0.2–1.5 g/kg	[27]
	19.97 ± 0.12 %w/w	11.95 ± 0.25 %w/w	4.02 ± 0.02 %TS	n.d.	n.d.	[38]
Horses	34 %w/w	n.d.	0.82 %TS	n.d.	1.76 g/kg	[39]
Pigs	39.2 g/L	27.4 g/L	12.5 g/L	5.0 gN/L	0.2 g/L	[40]
	27.5 ± 0.7 %w/w	22.0 ± 0.3 %w/w	3.9 ± 0.3 %TS	n.d.	n.d.	[32]
	27.13 ± 0.27 %w/w	19.89 ± 0.20 %w/w	5.32 ± 0.01 %TS	n.d.	n.d.	[38]
	25.95 ± 1.17 %w/w	20.18 ± 0.84 %w/w	2.27 ± 0.03 %TS	n.d.	n.d.	[34]
	36.1 %w/w	35.9 %w/w	n.d.	n.d.	n.d.	[30]
Pigs slurry	1.9 ± 0.3 %w/w	61.1 ± 2.8 %w/w	0.28 %w/w	0.22 %w/w	0.061 %w/w	[41]
Poultry	20 %w/w	75 %TS	1.032 %w/w	n.d.	0.413 %w/w	[36]
	25 ± 0.4 %w/w	66 ± 3.3 %TS	1.4 ± 0.6 %w/w	8.0 ± 1.3 g/kg	8.7 ± 1.0 g/kg	[42]
	37.76 ± 0.62 %w/w	26.45 ± 0.24 %w/w	2.36 ± 0.12 %w/w	0.55 ± 0.15 %w/w	n.d.	[43]
	31.5–78.3 %w/w	21.3–51.7 %w/w	2.3–38.9 g/kg	n.d.	5.2–15.3 g/kg	[27]
	23.00 ± 0.53 %w/w	67.52 ± 0.89 %TS	4.84 ± 0.37 %TS	1.11 ± 0.05 g/kg	n.d.	[33]
	48.3 %w/w	69.5 %TS	4.7 %TS	0.8 %TS	n.d.	[44]
Rabbits	21.8 %w/w	18.1 %w/w	1.7 g/kg	n.d.	n.d.	[30]

livestock manure are reported in Table 1. The vegetal residues, like straw of different origin, are rich in Total Solids (TS), thus carbon, but show limited levels of nutrients, while livestock effluents generally show high content of nitrogen and phosphorus [27]. The chemical composition of livestock manure heavily depends on the animal feed used and on the quality of feed application, thus enhancing the characterization variability of livestock effluents [28].

Typically, vegetal residues and manure are directly applied on fields as a soil improver [45]. However, the direct application of manure for soil amending and fertilization is regulated and can be applied on specific seasonal windows, due to odor and environmental pollution issues [46]. As an alternative, vegetal residues and livestock effluents can be used as substrates for biogas production, through AD processes [47]. This process could assume a valid and strategic role for biogas and fertilizers production [48].

In the AD process, organic matter is degraded under anaerobic conditions, to produce biogas, a mix of methane (CH_4) and carbon dioxide (CO_2), and digestate as a by-product, which can be further valorized for biobased fertilizer production. In Europe in 2019 were present a total of 18,943 biogas plants, according to the European Biogas Association (EBA) [49], producing 167 TWh of biogas and 180 million tonnes of digestate annually [50]. The AD plants are mainly located in Germany (11,269), followed by Italy (1,710), United Kingdom (1,233) and France (890) [49]. Regarding the feedstock composition, over 70 % (13,477) of biogas plants in Europe used agricultural substrates, mainly from energy crops, manure and food crops residues (i.e., maize triticale, wheat straw and rice husk) [49]. The main byproduct from AD is represented by digestate, which includes the stabilized organic matter, nitrogen, and phosphorous compounds, potentially exploitable for biobased fertilizer production.

1.2. Characteristics of typical agricultural digestates

As discussed above, during the AD process all the easily accessible organic carbon is converted into methane (CH_4) and carbon dioxide (CO_2). Thus, the digestate is the remaining part of the substrate and is primally composed by recalcitrant lignocellulosic residues, and nutrients, especially N, P and K.

Digestate characteristics are variable, depending on the origin and characteristics of manure fed to the digester and as well as the operational parameters applied to the AD reactor [57]. Table 2 reports the characterization of digestates from the most common agricultural substrates. The pH is usually slightly basic, ranging from 7 to 8.5. Overall, pH increases when ammonia is present, Volatile Fatty Acids (VFAs) are degraded and when basic cations are released; pH typically decreases when carbonate and phosphate precipitation reaction occurs [7,58] or VFA accumulate in the system. The Total Solids (TS) content varies between 1 and 25 %, due to the variability of input substrates biodegradability. Generally, higher TS content are due to feeding of high levels of lignocellulosic substrates and low digestibility, typical of agricultural residues [7]. When the AD substrate is rich in easily biodegradable substances, the digestate will exhibit a lower TS content and VS/TS ratio [52]. The degradation of organic nitrogen, mostly derived from the protein and aminoacidic content of the substrate, by the AD process leads to an accumulation of ammonium, which represents a major part of the total nitrogen content. Digestates from manure and protein rich substrates are expected to have a higher ammonium content compared to digestates from vegetal residues. The phosphorus content is not altered by the AD [7]. The environment inside an AD bioreactor favors the formation of phosphates, that can precipitate as magnesium or calcium salts [59]. During the AD, the organic P is converted in orthophosphate. However, 90 % of phosphate interacts and precipitates with Ca^{2+} and Mg^{2+} cations, thus increasing P concentration in the solid fraction of the digestate [60].

1.3. The European legal framework for digestate management

The conventional use of digestate is the direct application on farmland soils as conditioner/fertilizer. This use, if not properly managed, can lead to several pollution issues, mainly nutrient surplus leaching, with contamination and eutrophication of surface and ground-waters [61] and greenhouse gas emissions, especially ammonia (NH_3) and nitrous oxide (N_2O) [62]. Ammonia gas in atmosphere can then lead to particulate formation, mainly as ammonium sulfate, one of the major issue for atmospheric pollution (micro-particulate) in both urbanized and rural areas [63].

These pollution issues are particularly concentrated in the European Union's agricultural areas, which accounts for around 47 % of total EU land use (EU27 + UK) [64]. According to the EU report 2021/1000 [65], the nutrient balance of an agricultural area is the difference between nutrient input, usually with the use of fertilizers or manure, and nutrient output, represented by crops and fodder. A shifting from neutrality of the nutrient balance could lead to two scenarios: (i) nutrients deficit, when the balance is negative; (ii) nutrients surplus, with a positive balance. These two conditions lead to soil infertility, nutrient leaching, and dispersion in the environment, respectively. Regarding the EU, the nutrients balance on agricultural land is dramatically positive: 47.1 kg/ha of N surplus and 1.1 kg/ha for P surplus in 2014 [64]. Moreover, EU has passed the limits for N and P cycles, which are 2.1 and 0.07 Tg/y (teragrams per year) respectively, by a factor 3.3 for N (6.8 Tg/y) and 2 for P (0.14 Tg/y) [66].

The EU started to regulate digestate application from 1991, with the European Council (EC) directive concerning the protection of waters against pollution caused by nitrates from agricultural sources, otherwise named "Nitrate's Directive" [67]. This directive imposed to all Member States to monitor the welfare of surface and ground waters and the concentration of nitrates, in order to designate "Nitrate Vulnerable Zones" (NVZs), where there is high risk of nitrate pollution. This monitoring must be periodic, with a maximum of 4 years between each control. Moreover, this directive requires Member States to implement

Table 2
Characterization of various feedstock digestates. n.d.: not determined.

Substrate	pH	Total Solids (TS)	Volatile Solids (VS)	Total N	Ammonia (NH_3)	Total P	Total K	Ref.
Cattle manure + pig slurry	7.9	6.20 %	81.50 %TS	3.7 kg/m^3	1.6 kg/m^3	0.7 kg/m^3	2.7 kg/m^3	[51]
Cattle manure	8.3	7.10 %	81.30 %TS	3.8 kg/m^3	1.8 kg/m^3	0.6 kg/m^3	3.3 kg/m^3	[51]
Manure (unspecified)	7.3–8.6	2.2–9.2 %	67.8–75.0 %TS	0.05–0.62 %TS	0.255–1.01 %TS	0.034–0.221 %TS	0.03–0.43 %TS	[52]
Diary cows digestate	n.d.	70 ± 3 g/kg	49 ± 2 g/kg	3.35 ± 0.3 gN/kg	1.73 ± 0.1 gN/kg	1.64 gP/kg	n.d.	[53]
Cow dung slurry	8.89	n.d.	n.d.	38.4 mg/g	23.58 mg/g	6.16 mg/g	12.73 mg/g	[54]
Pig digestate	n.d.	32 ± 3 g/kg	21 ± 2 g/kg	2.25 ± 0.4 gN/kg	1.16 ± 0.3 gN/kg	0.36 ± 0.01 gP/kg	n.d.	[53]
Pig Slurry	8.4	4.80 %	63.90 %TS	5.2 kg/m^3	1.6 kg/m^3	1.5 kg/m^3	2.1 kg/m^3	[51]
Energy Crops + Manure	7.7–8.1	6.1–8.3 %	4.4–6.3 %	7.6–9.6 kgN/t	4.9–6.1 kgN/t	n.d.	n.d.	[7]
Energy Crops	7.6–8.0	6.6–9.3 %	4.8–6.9 %	3.6–4.9 kgN/t	1.3–2.4 kgN/t	n.d.	n.d.	[7]
Agricultural Feedstock	7.5–8.4	7.4–24.0 %	69–74 %	22–88 gN/kg	6–45 gN/kg	2–66 gP/kg	9–100 gK/kg	[55]
Agricultural Feedstock	7.5–8.4	6.41–24 %	69–77 %	0.14–2.1 % TS	0.04–1.71 %TS	0.058–2.400 %TS	0.324–0.392 %TS	[52]
Digestate (unspecified)	7.5–9	1.5–45.7 %	38–77 %TS	0.005–5.04 %TS	0.052–2.75 %TS	0.002–2.400 %TS	0.001–2.52 %TS	[52]
Digestate (unspecified)	8.1–8.6	4.98–12.0 %	2.8–7.6 %	0.17–0.75 %	0.52–3.41 g/L	0.14–0.65 %	0.20–0.50 %	[56]

measures to prevent and reduce nitrates pollution, with an annual limit of 170 kg/ha of manure-derived nitrogen. Fig. 2 illustrates the actual NVZs for EU.

The NVZs are coincident with European plains, flat areas, and river drainage basins. Consequently, a majority of livestock production sites and farms are concentrated in NVZ in Europe, leading to an excess of digestate that cannot be directly applied on farmland. Moreover, even if the manure and digestate application follows the Nitrate Directive regulations, another issue is represented by P accumulation. The stoichiometric N/P ratios documented for soil microorganisms and plants (around 6 – 8 [69]) are higher than the N/P ratios of most types of manure (typically < 5 gN/gP, Table 1 and 2). This indicates that even manure applied to land in line with the Nitrates Directive contributes significantly to the observed P accumulations in agricultural ecosystems that receive high manure loads.

Therefore, it turns out evident that digestate needs to be treated both to favor a further stabilization of the organic matter and to recover the nitrogen and phosphorous compounds, leading to a proper fertilization product to be applied on non-vulnerable or nutrient deficient areas. However, until 2019 the EU failed to issue a legislation act that covers nutrient recovery, biobased fertilizers production and their market. The EU Regulation 2003/2003 [70] only covered inorganic fertilizers, mined or chemically produced. Thus, this regulation ignored fertilizers derived from organic waste materials or organic–inorganic mixed products, as well as soil improver or conditioner. For this reason, the organic fertilizers were regulated and commercialized, inside and outside the origin country, in accordance with the National Legislation, thus discouraging the whole process of bio-fertilizers production at EU level. To overcome all the gaps in the old regulation, the European Parliament and the EU Council approved in 2019 the new Fertilizing Product Regulation 1009/2019 [71] that has entered into effect on 16 July 2022. This new regulation opened the access to the EU Single Market for biobased fertilizers, with a harmonized legislation that remove all the issues and costs regarding the mutual certification of the national rules. The biobased fertilizers can receive the "CE mark", making it easier to be commercialized and, consequently, promoting the production. This promotion is needed to foster the circular economy development and allowing a more efficient resources usage, while reducing the EU dependence from other countries.

Digestates are classified in two different Component Material Categories (CMC): CMC 4 for digestates from crops growth exclusively for biogas production (i.e., energy crops); CMC 5 for digestate derived from (i) bio-waste according to the directive 2008/98/EC [72] and (ii) derived products referring to the EU Regulation 1069/2009 [73], which includes manure and digestion residues from transformation into biogas. To be considered as organic fertilizer, the final product shall have a minimum quantity of N, P (as phosphorus pentoxide, P_2O_5) and K (as potassium oxide, K_2O). For solid, single-component fertilizers, this threshold is 2.5, 2 and 1 % w/w of N, P and K respectively. For liquid, single-component fertilizers, the threshold is 2, 1 and 2 % w/w for N, P and K respectively. In case of multi-component fertilizers, both solid and liquid fertilizers shall have at least 1 % w/w for each element. The process line for digestate nutrient recovery must be isolated and, in addition, is forbidden to physical mix the input and output streams of the processing line. Regarding the chemical-physical properties of the digestate, the regulation sets an Oxygen Uptake Rate (OUR) limit of 25 mmol O_2/kg vS per hour; with a maximum biogas production potential of 0.25 L/g vS [71].

2. Main technologies for the valorization of digestate

For decades, digestate has been used only as a soil improver, however a valorization is required before soil application, due to the above-mentioned environmental issues.

In this framework, the new European directive will facilitate digestate valorization development.

Indeed, there are several technologies, developed and exploited at industrial level, for nutrient recovery from digestate (ammonia stripping, adsorption, membrane filtration/concentration, struvite crystallization / precipitation, evaporation). These were typically developed and

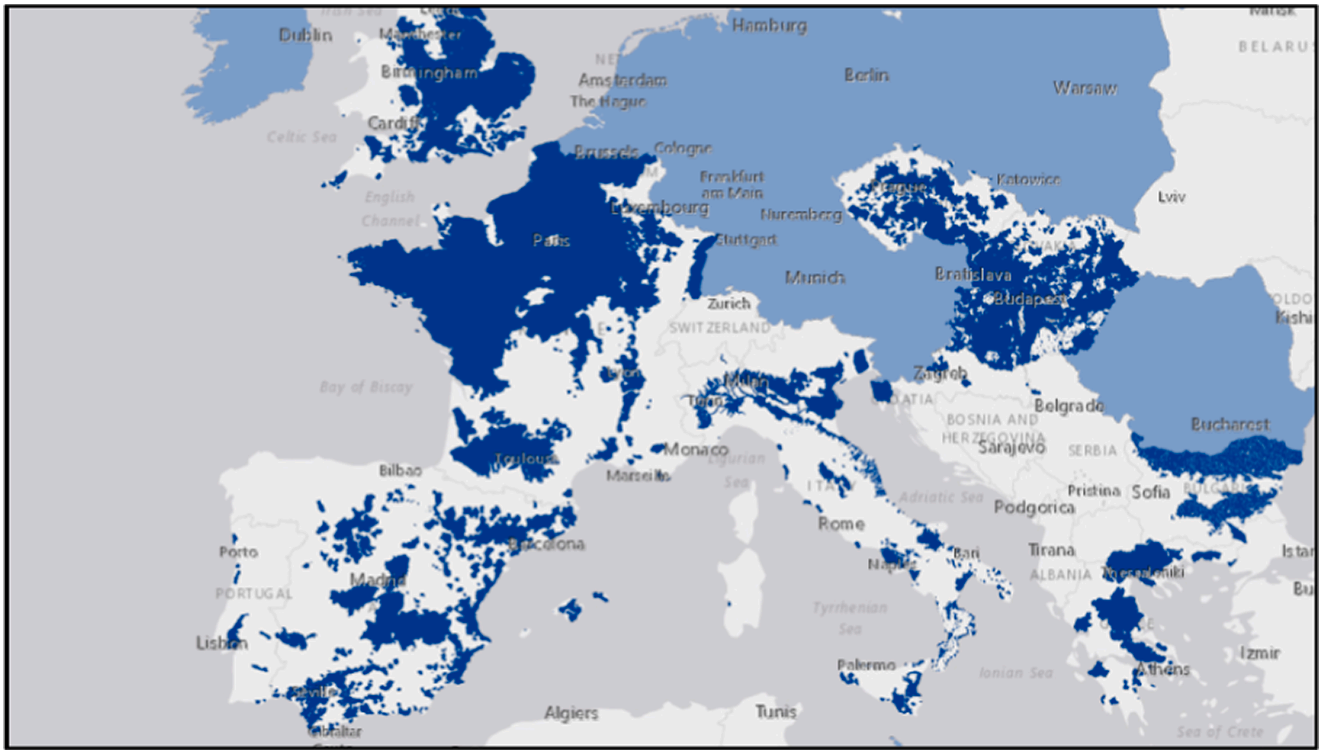

Fig. 2. Nitrate Vulnerable Zones in Europe [68]. Blue: NVZs Designated Areas; Light Blue: Countries exempted to designate specific areas (art 3.5 of EU Nitrate Directive). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

applied in the industrial sectors of wastewater, sludge and organic waste management but can be easily transferred to the agricultural sector.

Ascertained that nutrient recovery from digestate is a feasible option, this process has to be: (i) efficient as the conventional technologies for fertilizer and soil improver production; (ii) economically convenient, and (iii) a guarantee that the produced fertilizer is marketable [56]. All these three conditions could be met: the high nutrient content of the digestate, especially in N and P, combined with the new European regulations, give a high economical potential to digestate and, consequently, to the new biobased fertilizer market.

The first step of almost every nutrient recovery process is the solid–liquid separation of digestate. This separation is accomplished by screw-presses, centrifuges, and belt filters. To improve the solid removal, additives, such as precipitant or flocculant salts, can be added in this step (if allowed by local regulations). The solid part is typically characterized by TS ranging between 20 and 25 % w/w, and high amount of organic N and P [74]. Due to the reduction of water content, the solid part is more stable, transportable, and storable, thus can be used directly as soil conditioner or can undergo further treatments, such as drying or composting, to produce a marketable fertilizing product [7]. The liquid part, with TS content of 3 – 7 % w/w, is rich in ammoniacal nitrogen [52].

The different technologies employed for the digestate valorization, the conventional and the more innovative ones, will be shortly described along the following paragraphs focusing the attention on their main advantages, drawbacks, development potential and costs. By this way, it will be possible to discuss the technologies with the lower environmental impact and economic costs and, consequently, with the highest possibilities to be developed at full scale levels.

2.1. Ammonia stripping

Ammonium (NH_4^+) represents the major nitrogen compound of liquid digestate: depending on pH and temperature it can be found in ammonia form (NH_3) (~10 % of soluble N).

The most adopted technology for its recovery is the ammonia stripping. In this process, nitrogen is removed from the digestate liquid fraction by a gas stream, normally air, then the volatized nutrient compound is selectively collected. This process is usually done in packed-bed stripping towers to increase the available area for mass transfer [75]. Regarding the ammonia stripping process, the digestate is firstly pretreated to enhance NH_3 volatilization, usually by basification with strong bases, commonly CaO, $Ca(OH)_2$ and NaOH to a pH value of around 10 [76]. To enhance this step, the stripping is usually conducted at high temperatures, typically 50 – 85 °C, and with negative pressure [77]. After the pretreatment, the stripped ammonia is recovered by an acid scrubber, commonly using sulfuric (H_2SO_4) acid to produce ammonium sulphate. The ammonium sulphate can be used as a biobased fertilizer of N and S, thus representing a valid alternative to fossil-based fertilizers [78]. However, ammonium sulphate has a low N content in comparison with other fertilizers such as urea ($CO(NH_2)_2$), 21 and 45 % w/w respectively, representing only 4 % of the worldwide *N*-fertilizer application. As alternatives: (i) nitric acid (NHO_3) can be used instead of sulfuric acid to produce ammonium nitrate (NH_4NO_3) [79]; (ii) vapor stripping can produce ammonia water, removing the acid scrubber but providing more energetic cost for vapor production [7].

Clearly, one main issue of this process is the necessity to operate with liquids without solid particles. Therefore, after the initial solid/liquid separation, a further step for the removal of fine particles in the liquid is requested [53].

The separated solid fraction, that is the ammonia-depleted digestate, can be either used as soil improver or can be recirculated in the biogas reactor to reduce ammonia content, preventing the inhibition of the AD process [77]. The performance of ammonia stripping process depends mainly on temperature and pH, and, less importantly, on NH_3 concentration in the substrate as well as physical parameter, i.e. air/liquid flow and transfer area of the packing bed [75,80,81]. The ammonia stripping yield can reach a theoretical level of up to 99 % [76], but to reduce operational, is acceptable an ammonia removal of 80–90 % [56]. Economical costs for the N recovery varies between 2 and 7 €/kgN removed, depending mainly on the strategy for pH management: type of base used to control pH and temperature. These costs include acid (1.5 L H_2SO_4/kg NH_3) and energy (0.057Wh/m^3 air) consumption too [56,82]. Moreover, the packed-bed stripping towers are prone to clogging due to calcium carbonate ($CaCO_3$) scales formation [83], and corrosion due to the low pH caused by sulfuric acid on the scrubbing unit.

Overall, the total cost for both solid–liquid separation and ammonia stripping was evaluated between 3 and 6 €/m^3 of digestate [74].

Several scientific studies on ammonia stripping and recovery were conducted at full, pilot, and laboratory scales in last years. The distribution of some experience in the EU is shown in Fig. 3. The color indicates the TRL of the process: Red, Green and Gray stay for high (8–9), medium (5–7) and low (<4) TRL, respectively. As evident from Fig. 3 there are different ammonia stripping plants working at the highest TRL of 9 [74]. Laureni et al. [84] evaluated the overall performance of an ammonia stripping process, treating pig slurry digestate from Catalonia region (Spain). Overall results find an efficiency up to 80 % in ammonia removal, depending on Chemical Oxygen Demand (COD) content of the organic matter, obtaining an ammonium sulphate concentration higher than 40 gN/L. In a study from Bolzonella et al. [53], the liquid fraction from cow manure, pig effluents and energy crop digestate from full-scale AD digester located in the province of Brescia (Italy) were treated by stripping and allowed a recovery lower than 40 % of the N originally present in the unprocessed digestate, due to N dispersion between the solid/liquid pretreatment before the stripping process. In a farm site located in Ottersberg (Germany), Brienza et al. [85] studied the process implemented at full-scale as a strategy to prevent the methanogenic inhibition by ammonia. In particular, the liquid fraction of digestate was recirculated in the AD bioreactor after the ammonia stripping step. Another peculiarity of this study was the gypsum use as alternative to sulfuric acid, obtaining both ammonium sulphate and fertilizing liming substrate. N was recovered for the 57 % w/w as ammonium sulphate and for the 7.5 % w/w as liming substrate. Capital and operational cost accounted to 5.8 €/t digestate processed.

A commercially available stripping technology is represented by the AMFER® stripping system of Colsen industry (Netherlands). The input can be either whole digestate or its liquid fraction, with ammonium sulphate or nitrate production depending on the use of sulfuric or nitric acid. The removal efficiency is typically of 50 % w/w on mineral nitrogen, with a capacity from 1 to 500 ton/hour [86]. In the Biogas Bree system, located in Bree (Belgium), the agricultural digestate of a full-scale biogas plant is dried with an evaporation process. Then, the ammonia-rich air in the exhaust pipe of the evaporation system is treated with an acid scrubber, producing about 30 L of ammonia sulphate per kg of ammonia recuperated and an annual production of 600 tonnes of ammonium sulphate. [87]. Another full-scale nutrient recovery system is the "Detricon" process, which produces ammonium nitrate from agricultural digestate by scrubbing with nitric acid [88] near Gistel (Belgium). The ammonium nitrate concentration on the final product is 52 % w/w, corresponding to 18 % w/w of the initial N content in digestate. The Italian company BTS Biogas Srl/Gmbh developed "NITROStripp", an ammonia stripping system on a full-scale agricultural biogas AD plant near Chiari (Italy), which allowed the removal of up to 70 % w/w of the N present in the input digestate [89].

2.2. Struvite precipitation and its alternatives

Struvite is a Magnesium (Mg) Ammonium (NH_4^+) Phosphate (PO_4^{3-}) (MAP) hexahydrate mineral ($MgNH_4PO_4{\cdot}6H_2O$), composed by equimolar concentration of Mg, NH_4^+ and PO_4^{3-} [81]. Struvite is an eco-friendly fertilizer, that reduce soil and surface water pollution by releasing nutrient with a slower rate than conventional mineral

Fig. 3. Ammonia Stripping plants considered in this study. Red: High TRL (8–9) plants; Green: Medium TRL (5–7) plants. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

fertilizer, allowing a major assimilation of the nutrients by the agricultural crops [90]. Struvite formation requires a NH_3 and PO_4^{3-} soluble rich medium, such as digestate, and is induced by addition of Mg (as MgO/$MgCl_2$) and NaOH to increase the pH to 8.3–10 [52]. The struvite crystals precipitate and are collected for direct fertilization application. This process has high P-removal capacity, with up to 80–90 % of removal efficiency, but lacks of NH4-N removal, with only 10–40 % [91]. The crystals obtained have dimensions ranging between 0.5 and 5 mm. This process then requires a large quantity of chemicals, especially alkali to raise the pH, due to the presence of buffer ions, such as H_2CO_3/HCO_3^-/CO_3^- and NH_3/NH_4^+ [92]. Sometimes, Mg presence is below the requested stoichiometric limit for struvite formation and this mineral should be added as MgO [91].

The economic cost for struvite precipitation process varies from 270 to 2,000 €/t P removed, depending mainly for the high reagent cost for pH managing [92]. The struvite precipitation has been implemented in more than 40 world-wide full-scale plants, however, this process requires a minimum P concentration of 100 mg/L, for this reason is often applied for wastewater and municipal sludge digestate, and only a few are developed for agricultural digestate, due to low P content [74].

Not only ammonium can react with bivalent cations and phosphate to form struvite or similar salts.

K^+ ions can be involved in the reaction instead of NH_4^+ ions in the precipitation of magnesium potassium phosphate hexahydrate formation ($KMgPO_4{\cdot}6H_2O$), an isomorphous analogue of struvite. This precipitation can achieve the recovery of K and P [93]. However, precipitation can occur only when K^+ ions are in excess [74]. In order to remove ammonium ions, ammonia stripping can be implemented before the precipitation step. K-struvite precipitation allowed a recovery of 72 % P and 73 % K, with 99 % of N removed in the stripping step [93]. P can also be precipitate as calcium phosphate, in form of hydroxyapatite ($Ca_5(PO_4)_3OH$) or brushite ($CaHPO_4{\cdot}2H_2O$), by addition of calcium hydroxide (Ca(OH)2) when pH and temperature are above 10 and 70 °C, respectively [74]. The calcium phosphate precipitation has a P removal efficiency of 50–60 %, with a daily capital cost of 2300 – 2900 €/kg P [52].

As mentioned above, phosphorus precipitation by struvite formation is a nutrient recovery system mainly applied in municipal wastewater treatment plant (WWTP) [74]. Its application in WWTP is often needed to avoid pipe clogging rather than P recovery as a resource [91,94,95].

The recovery of phosphorus in the agricultural sector is rarely applied as there is not a specific regulation aiming at the control of this nutrient like it happens with nitrogen through the Nitrate Directive.

However, being a critical raw material, its recovery is attracting increasing interest.

Few examples at medium and large scale are available, according to a recent review by Lorick et al. [96]. On the other hand, technologies are now commercially available with several case studies in the municipal sector treating anaerobic sewage sludge digestates, which also have application potential for the agricultural sector.

One of the first high TRL scale plant was developed by Battistoni et al. [97] in the Northern Italy. In particular, a demonstrative plant was built to recover phosphorus compounds and produce struvite from municipal wastewater treatment plant in northern Italy. The system was based on the auto-nucleation of struvite from anaerobic supernatant by a fluidised bed reactor (FBR) operating in *meta*-stable conditions and to reach optimum performances reducing the managing costs. The results were encouraging: the treating of about 650 m^3 of anaerobic supernatants led to the formation of 0.28 tonnes of granulated crystalline stuvite. The chemical analysis showed its possible use in agriculture as fertilizer.

Larriba et al. [98] applied P-struvite precipitation on a pilot-scale plant near Barcelona (Spain), treating wastewater, with 45 % – 60 % w/w of input P recovered as struvite. One established commercial P-recovery system is “NuReSys” [99], usually applied on the effluent of wastewater treatment plants or UASB reactor to recover P as struvite, thus preventing tubing clogging due to unwanted struvite formation. The obtained struvite pellet is characterized by a nutrient content of 5.6 % w/w N and 12.6 % w/w P. Another industrial commercial application is the AirPrex™ process. AirPrex™ is a struvite precipitation system, operating with sewage sludge in full scale plants of Berlin-Wassmannsdorf and Mönchengladbach (Germany), with 90 % w/w of precipitation and 50 % w/w of harvesting efficiency of formed struvite crystals [100]. Another commercial system is Ostara’s Pearl® technology from the Spanish company “Canal de Isabel II” [101]. This process treats effluents from WWTP in Madrid (Spain) and is dimensioned to produce 2 tonnes per day of struvite granules. Saerens et al. [102] investigated a full-scale struvite precipitation plant operated by Aquafin in Leuven region (Belgium). In this plant, the municipal wastewater digestate is treated with a fiber cutter in order to remove the biggest fibers, and then it is crystallized with $MgCl_2$ addition (Mg/P ratio 2), forming struvite. The MAP precipitate was separated from the remaining sludge by a hydrocyclone. The weekly production of struvite was 500 kg and the overall P recovery as struvite was estimated at 5 % w/w of the input P.

Focusing on struvite formation from agricultural digestate, there are some examples at advance development stage (TRL 7). REVAWASTE® system, developed by CARTIF company (Spain), treats agricultural residues by AD process, at demonstrative scale. The system includes a crystallization unit, allowing up to 95.4 % of nutrients recovery [103]. In Olburgen (Hollande) an agricultural digestate from a potato processing wastewater AD plant, struvite precipitation system Phosphaq paired ™ [104] was combined with one-step Anammox system to remove residual N [105]. Removal efficiencies were 76 % w/w for P, consistent with another similar application where digestate derived from a food waste AD plant in Lomm (Netherlands) [106].

Regarding the recovery phosphorus as calcium phosphate, the most representative European example is the Ash2Phos system, developed by Swedish company EasyMining. This process extracts phosphorus from incinerated sewage sludge with a recovery of over 90 % w/w. This process will enter the market in 2023, with a production estimation of 13,000 tonnes of precipitated calcium phosphate per year [107].

2.3. Ion exchange and adsorption

In adsorption and ion-exchange (IEX) processes, nutrient compounds or ions are adsorbed into a solid matrix, such as zeolites, resins and clays [52], usually suspended in a packed-bed column, in order to increase the surface/volume ratio [8]. The absorption and IEX processes are regulated by intramolecular and ionic bonds respectively. Once the adsorption column is saturated, it can be eluted and regenerated. Zeolites, which are a slow releaser of ammonium, can be directly used as a fertilizer after the adsorption process [52]. IEX and adsorption are suitable for waste streams with low solid concentration (<2 g/L) [8]. When solids concentration is higher than 2 g/L, red mud, metal oxide, and zirconium sorbents could be used to recover P (maximum recovery of 58 gP/kg), while N and K could be recovered using zeolites and clinoptilolite (maximum recovery of 21.5 gN/kg) [8]. These methods can achieve high P accumulation, without pH corrections and additional sludge production. However, the main drawbacks, especially in case of high solid content, are fouling of the packed-bed column, ion competition, and the reduction over time of adsorption capacity [56,108].

According to Vaneeckhaute et al. [56], adsorption and ion-exchange as nutrient recovery processes are currently not applied on advanced digestate treatment processes. However, studies, conducted at laboratory scale, achieved a nutrient removal efficiency up to 89, 78 and 80 % w/w for ammonium, potassium and orthophosphate respectively, using clinoptiolite as adsorption matrix from animal manure digestate [109]. Another interesting study by Rodríguez Alberto et al. [110] used as solid matrix the biochar from the pyrolysis of agricultural digestate solid fraction. It allowed the adsorption of around 20 % w/w of phosphate in the fraction liquid of digestate. Pinelli et al. [111] tested the P removal performance of an hybrid anion exchanger resin (Layne^{RT}) on demonstrative scale at Cranfield (UK) using municipal wastewaters. The study found a maximum adsorption capacity of 41 $mgP/g_{dry\ resin}$, with a recovery of 51 % of adsorbed P.

2.4. Evaporation

If excess heat is available, e.g., produced by the biogas burning in a Combined Heat and Power (CHP) unit, this process can be applied to evaporate liquid fraction volume and, consequently, to concentrate and recover a high ammonia content digestate [52,112]. This is a two-step process, in the first part, after solid–liquid separation, the liquid part is acidified to pH 4.5, usually with sulfuric acid, and CO_2 is stripped. Due to the acidification, the nitrogen is in the form of ammonium ion (NH_4^+), and it remains in the concentrate part after the evaporation process. Then, the digestate is concentrated by a multistage evaporation system, applying low pressure in order to exploit low-grade excess heat at 90 °C (or lower temperatures when operating under vacuum conditions). The process vapor, after being condensate, still contains low amount of nitrogen and volatile acids; depending on the use it could be necessary to further treat it. This process allows a volume reduction of 50 % of the digestate. The main drawback of evaporation is the high thermal energy demand of 300 – 350 kWh per ton of water evaporated which prevent its adoption at large scale [7]. A work by Chiumenti et al. [113] obtained, with a vacuum evaporation process, a concentrate with a nitrogen content of 55 g/kg.

2.5. Freeze concentration

In the freeze concentration process, solutes are separated from the solution by selectively freeze, crystallize, and remove solvent molecules [114]. The main advantage of this process is the lower energy requirements compared to evaporation (2500 kJ/g vs 335 kJ/g) [28]. This process is under development regarding the agricultural digestate, and the few works are present in literature: Uald-lamkaddam et al. [114], on a laboratory-scale system in Vic (Spain), obtained a N recovery of 40 – 71 % w/w and P recovery of 46 – 90 % w/w. Overall, the main drawbacks regards the ice crystal formation and their separation from the concentrated solute.

2.6. Membrane separation technologies

2.6.1. Pressure-driven membrane filtration

Membrane filtration is a physical based process, where the digestate is separated through the application of a membrane in a solid fraction, also named concentrated or retentate, and in a liquid fraction that passes through the membrane, called permeate. Commonly, membrane technologies involve several filtration steps. The first steps are rough solid–liquid separations, common in all digestate treatments as mentioned above. Since the TS content of the liquid part effluent a screw press is still high and can damage the membrane module, another step of solid/liquid separation with a centrifuge or a vibrating screen is needed.

The pre-treated liquid fraction can enter in a microfiltration (MF) step, represented by membrane with pore size > 0.1 μm under a pressure 0.1–3 bar. MF is followed by ultrafiltration (UF, pore size > 0.001 μm, pressure 2–10 bar) step able to remove all the suspended solids and microorganisms. The remaining small molecules and ions are then removed by a reverse osmosis (RO, pore size < 1 nm, pressure 10–100 bar) step, to obtain a nutrient rich retentate and pure, particle and pathogen free, water as permeate. As final products, a solid fertilizer, N and P rich (8.2–12.0 kg t^{-1} TN; 5.6–10.4 kg t^{-1} P_2O_5), can be obtained from the solid fraction, while a liquid fertilizer, rich in ammonium and K (2.9–5.6 kg t^{-1} NH_4^+; 6.2–9.2 kg t^{-1} K^+), can be obtained from UF and RO concentrates [115]. Membrane fouling is the main disadvantage of these technologies. Periodic maintenance or substitutions in order to maintain adequate separation performance are required [116]. To reduce maintenance costs, ceramic membranes can be used instead of less expensive organo-polymeric membranes, as they can be easily cleaned and are more resistant to pressure and chemicals [74]. According to Gienau et al. [115], the UF step is the most energetically expensive, with a consumption of 10 – 15 out of 20 – 30 kWh/m^3 of digestate. In order to reduce the energy cost, they suggested the adoption of an enzymatic pretreatments (1 g/L of amylase, pectinase, cellulase and protease) able to decrease the viscosity of the digestate. With the same purposes, ozone treatment can be applied [117]. The application of membrane technologies to a digestate from the AD of animal manure, recovery yields of 75 – 96 % of Total Ammoniacal Nitrogen (TAN) and of 100 % TAN were achieved at pH 8 and at pH 4, respectively [118]. Regarding the phosphorus removal yields, 87 – 98 % w/w of P removal were achieved [111,119]. The membrane technologies have a TRL level of 9, with the total cost for a full-scale plant between 4 and 12 €/m^3 [74].

2.6.2. Electrodialysis

An alternative to pressure-driven membrane filtration can be the electrodialysis (ED), which is based on the application of electric-driven forces. ED process involves the creation of an electric field where anions move towards the anode, while cations towards the cathode. Using ion-exchange membranes, ED allows ions separation from liquid digestate, transferring them to the output solution with higher ammonium concentration than RO (16–21 g/L) [81]. ED system with a mono-selective membrane for PO_4^{3-}, can promote a successive struvite precipitation step [81]. Studies on ammonia concentration with ED lead to a maximum theoretical concentration of 16 gTAN/L [120]. Coupling ED with ammonia stripping allowed the reaching of 21.356 gTAN/L [121]. The high energetic and economical costs are considered the major bottleneck for the development of ED nutrient recovery processes [56]. In particular, similarly to pressure-driven membrane processes, the membrane fouling is unpredictable and has a major impact on the operational costs [122]. Due to this reason, there are no evidence in literature of full-scale ED nutrient recovery processes.

2.6.3. Gas-permeable membrane

This process uses a hydrophobic microporous membrane that can guarantee the selective passage of NH_3 from manure or digestate liquid fraction, under low pressures to facilitate NH_3 volatilization. The gaseous ammonia, once it has permeated through the membrane, reach an acidic solution for his conversion on non-volatile ammonium ion, similar to the classic acidic scrubbing of ammonia stripping process [123].

2.6.4. Membrane contactors

Membrane contactors allows a non-dispersive contact between two phases, that can be either gas or liquid. In particular, membrane contactor technology is composed by hydrophobic membranes, typically disposed in a hollow fiber configuration across which gaseous species such as ammonia gas can transfer. An acid, such as sulfuric acid or nitric acid, is flowed counter-currently in order to react with ammonia gas to create ammonium sulphate or nitrate, respectively [124]. The high porous membranes determines a large contact area, improving the mass-transfer coefficient [125]. Membrane contactors can be applied for water treatments, such as desalinization, but also for nutrient recovery, especially ammonia, from agricultural digestate [10]. At this scope, Hollow-Fibre Membrane Contactors (HFMC), composed by polypropylene (PP) or polytetrafluoroethylene (PTFE) gas-permeable membrane, can be applied for nitrogen recovery through ammonia stripping: NH_3 is stripped from the origin solution, passing through the gas-filled porous membrane. On the other side of the membrane, ammonia is then recovered by sulfuric acid to obtain ammonium sulphate. In this membrane technology, the driving force is the difference of free ammonia concentration between the two sides, promoting ammonia diffusion [10]. Ammonia removal was found to be 96 – 98 % [10,126]. As the liquid phase does not pass through the membranes, but only the gaseous ammonia, the membrane fouling was negligible [126].

Being rich in ammonia, agricultural digestate was exploited for ammonium sulphate recovery by means of membrane contactors. Garcia-González and Vanotti [123] developed a lab scale hydrophobic membrane contactor to recover of ammonia from animal manure. Firstly, they increased the pH through NaOH addition in order to favor the ammonia's passage from liquid to gaseous phase. Then a series of PTFE membranes were submerged in the solution. The ammonia gas was subsequently treated with a circulated acid solution in the lumen. By this way, they achieved an ammonia removal of 98 % from manure. The process was further improved by the injection of air in the manure in the increasing of the pH: air reacted with the bicarbonate alkalinity producing carbon dioxide and hydroxyl species. It led to a saving of NaOH usage and mainly to a decreasing of the 50 % of the operational costs, even considering the additional cost of the aeration stage.

Wäeger-Baumann and Fuchs [127] studied the ammonia removal from an anaerobic digester effluent, putting the membrane contactor directly in the digester effluent without any shell casing present. By this way, they avoided the fouling of the membrane due to the particulate compounds present in the digestate. The acid stripping solution was passed through the lumen side. It is fundamental to emphasize that to reach a good ammonia removal efficiency the specific membrane area was required to be relatively high (32 m^2/m^3). Another lab-scale study recovered ammonia from chicken manure digestate. In particular, a membrane contactor, operating in sweep gas mode, with air under different gas flowrates and ammonia concentration value, was submerged in the chicken manure digestate. The TAN removal fluxes obtained were in the range of 1.22 and 1.48 g/m^2/day [128]. Another work tested an external PTFE membrane contactor for ammonia recovery form chicken manure digestate. The removal yield was high of about 93.6 % [129].

Even if the laboratory studies were promising, in term of ammonia removal, the membrane fouling, which is one of the main drawbacks of this technology, prevented its large application at higher TRL.

A pilot plant membrane contactor was tested in Switzerland by Boehler et al. [130] to remove ammonia from wastewater treatment plant effluent and to convert it into ammonium sulphate. They set up three hollow fiber membrane contactors in series and sulphuric acid solution was passed through the lumen side. The process required a first pH adjustment of the pH to 9.5. The ammonia removal yield was of the 95 %. Another interesting example of pilot scale membrane contactor was the one located in Denmark [131], where an anaerobic digester which was firstly processed in an ultrafiltration step before to be sent to a polypropylene hollow fiber membrane system. The ammonia removal was in the range of 85–90 % w/w.

Although examples of the application of this technology can be found at full scale, only few works are reported in the scientific literature on nutrients recovery from digestate through membranes technologies. The distribution of the main applications and relative studies on the valorization of digestate by membrane processes in the EU is shown in Fig. 4. The color indicates the TRL of the process: Red, Green and Gray stay for high (8–9), medium (5–7) and low (<4) TRL, respectively. Chiumenti et al. [132] monitored the performance of an animal manure and digestate full-scale plant located in Lastrup (Germany), adopting UF and RO. Overall, this process recovered 99 % w/w of N and P in the two corresponding retentates. Moreover, pure water in the RO permeate was obtained. It met the legislative criteria for environment discharge. A similar recovery yield was found by Bolzonella et al. [53], that evaluated the nutrient recovery through membrane separation from two full-scale AD plants near Brescia (Italy), treating pig effluents, cow manure and energy crops. The two digestates were filtered through sequential unit operations constituted by screw-press, UF and RO steps. 50 % w/w of the digestate mass was recovered as almost pure water. In the RO

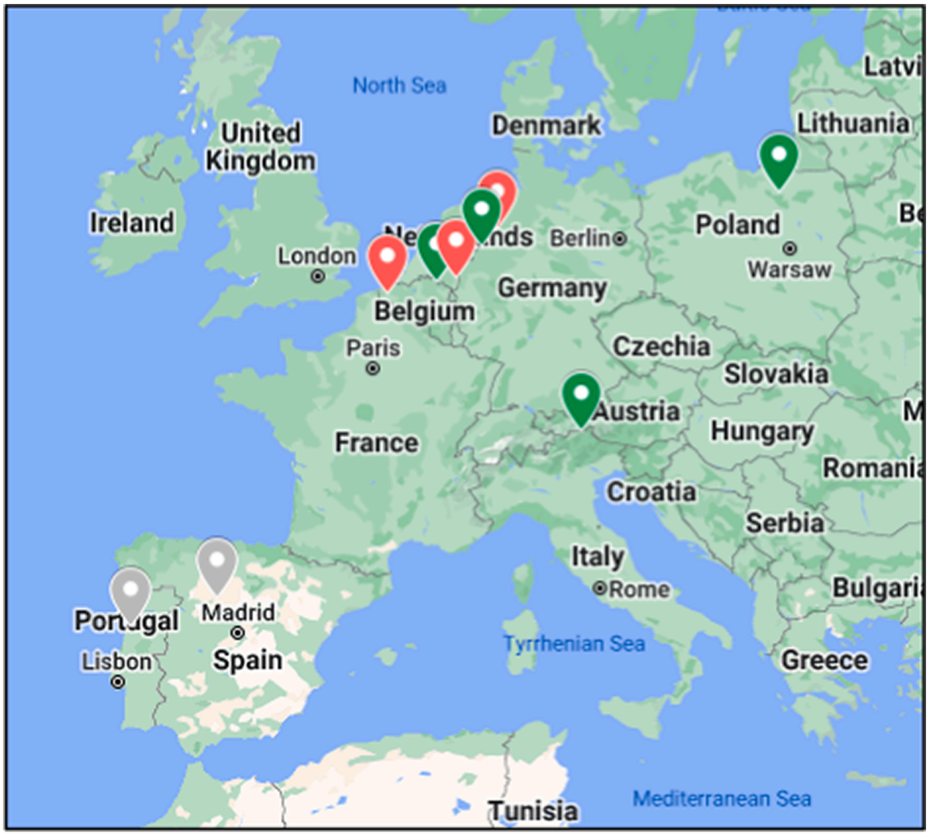

Fig. 4. Membrane filtration systems reported in this study. Red: High TRL (8–9); Green: medium TRL (5–7); Gray: low TRL (<4). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

concentrate, a concentration of 4.8 and 0.36 g/kg of N and P, respectively, has been achieved for dairy cows digestate. For piggery digestate, the concentration was of 5.27 gN/kg and 0.26 gP/kg. A pilot test, carried out by Gienau et al. [133] in Lower Saxony (Germany), reported an almost complete nutrient removal through the RO step. A pilot-scale study carried out in Poland, conducted by Zielińska and Mikucka [119] achieved a rejection of 81 and 87 % w/w of N and P respectively, using UF as the final step of the valorization process. An new and interesting study of Van Puffelen et al. [134] implemented a full-scale cascaded membrane filtration system in Beltrum (Netherlands) on agricultural digestate, with the purpose of recover and separate P and ammoniacal nitrogen. Results showed a recovery of 98 % w/w of initial P before the RO step and a recovery in the RO concentrate of 48 % w/w of digestate's ammoniacal nitrogen.

The Dutch company Vlako B.V. operates full-scale plants treating digestate from the treatment of pig and cattle slurry, producing a mineral concentrate, using belt press sieve and RO processing [135]. Another industrial-scale nutrient recovery system is represented by "VP-Hobe" where membrane separation is combined with other nutrient recovery technologies from manure and agricultural digestate [136]. In particular, RO is coupled with evaporation and ammonia stripping. By this way, the stripping/scrubbing unit operation allows for the recovery of ammonium sulphate by stripping/scrubbing from the liquid fraction of digestate, while P recovery derived from RO concentrate after evaporation. In Italy, the company Biogas Wippital from Bolzano produces a solid fertilizer from animal manure digestate through a RO system "OB Slurless 100" [137]. Another promising application of membrane filtration is within the EU Interreg project ALG-AD [138]. In this project, the liquid part of the digestate, suitably refined and sterilized by membrane processes, is used as nutrient source for biomass production, in particular microalgae [139]. This biomass can then be used as animal feedstock to foster a circular economy approach, and also representing a valid alternative to common feed supplies [140]. This project has 3 pilot-scale facilities installed in Devon (UK), Ghent (Belgium), and Brittany (France).

Regarding the non-pressure driven membrane separation, the technologies are on early development stage. Some examples are represented by the adoption of gas-permeable membranes for swine manure digestate upgrade at laboratory scale near Vallaloid (Spain), which achieved up to 81 % w/w of ammonia recovery [123]. Oliveira et al. [141], at Aveiro (Portugal), combined electrodialysis and gas permeable membranes at laboratory scale to recycle 81 % w/w of the P present in municipal solid waste digestate. At the best authors' knowledge, no higher TRL examples of non-pressure driven membrane were tested for agricultural digestate valorization in Europe.

2.7. Thermal treatments

Thermal treatments are usually applied to the solid phase of digestate, after preliminary solid/liquid separation. They include pyrolysis and Hydrothermal Carbonization (HTC). In the pyrolysis process, the solid fraction is mainly converted in biochar by an endothermic process with high temperatures (400 – 600 °C), and anaerobic atmosphere, usually using N_2 as replacement gas [142,143]. The side products of pyrolysis, syngas and oil, can be used as a fuel to foster the preliminary drying operations by providing thermal energy. The high energy demand for the reduction of the water content is the main drawback of the pyrolysis. The water content of the inlet substrate has to be under 15 % w/w in order to reduce the overall energy output [144].

Due to the lower energy and pretreatment requirements, HTC is an innovative process and can be a valid alternative to pyrolysis. In this wet thermochemical process, the biomass is treated at temperatures between 180 and 280 °C, in autogenous saturated vapor pressure of 10 – 80 bars, for 1 to 12 h [145,146]. During this process, water acts as an organic solvent towards the substrate molecules, promoting hydrolysis, dehydration and decarboxylation reactions [144,145]. HTC solubilize nutrients, such as N and P, in liquid phase. The final product is constituted by hydrochar, similar to the pyrolysis biochar, and process-water. Hydrochar presents different applications: i) as soil improver and fertilizer, due to N and P content; ii) as adsorber matrix in adsorption processes, capable of removing ammonia, phosphate and pollutants, such heavy metals and polycyclic aromatic hydrocarbons (PAHs) [142]; iii) biochar can also be used as an active carbon filter for treatment of wastewater and drinking water [147]. HTC process-water, containing N and P from the solubilization reactions, can be used as fertilizers or recycled in the AD reactor to enhance biogas production [148]. Drawbacks of this process are related to the water content of the substrate, that lead to high substrate volumes, impacting on the transportation cost and storage [149]. Energy consumption for hydrochar is up to 27 kWh/m^3 solid digestate, with an estimation of the cost of 157 €/t [52].

Organic material can also be combusted in a Thermal Oxidation process, obtaining ash that can be used directly as fertilizer. However, this process can hinder the P-solubility and plant availability. Moreover, ash can contain high quantity of potentially toxic heavy metals, depending on the origin substrate. Thus, ash is not suitable for land application, and needs to be differently treated in order to recover nutrients, especially P [147]. These processes, that can recover up to 90 % w/w of P [150], are divided in i) wet-chemical and ii) thermal. Wet-chemical processes are based on the addition of chemicals to induce a complete acidic dissolution of P at low pH values (<2), rendering the P bio-available. The P will be recovered by wet-chemical extraction techniques such precipitation, liquid–liquid extraction and adsorption. Nutrients can be recovered also by high-temperature thermal processes, separating P from volatile heavy metals in the gas phase.

The possibility to save more energy than pyrolysis is making HTC the most studied and promising digestate thermal treatment [55]. Lucian et al. [151] found a final P-recovery yield of 91 % w/w on an industrial-scale HTC plant at Mezzocorona (Italy), treating agro-industrial digested sludge. A German company, Terranova, realized a HTC process at full scale in the city of Jining (China). The plant treats 14,000 tonnes per year of sewage sludge. A laboratory-scale study from Roy et al. [152], at Leicester (UK), applied HTC of sewage sludge digestate to recover hydrochar and a high nutrient process water. The latter was used as microalgae growth medium, with a concentration 11 and 50 times higher, for sulfur (S) and P respectively, than artificial microalgae growth medium. Numviyimana et al. [153] processed, at laboratory-scale near Limerick (Ireland), diary wastewater using HTC followed by struvite precipitation of the HTC liquor, obtaining 99.9 % w/w of overall P recovery. Another study by Gerner et al. [154], carried out at lab-scale at Wädenswil (Switzerland), applied HTC followed by ammonia stripping and leaching. Results showed a nutrient recovery of 84 and 71 % w/w of phosphorus with stripping and leaching respectively, as well as 53 and 54 % w/w respectively of nitrogen from the liquid phase. This study also conducted an economic evaluation for a possible industrial-scale application, confirming that HTC can be economically feasible.

3. Conclusions

This review investigated the maturity level of different technologies applied for nutrient recovery from anaerobic digestate produced in the rural context. The amount of this stream accounts for 180 Mt per year. Different technologies were discussed along the review, emphasizing the TRL level, the capital and operational costs (when available). Ammonia stripping is the most developed technology in EU: its main application is the recovery of nitrogen from the liquid fraction of digestate with a N recovery yield of 80 – 90 %w/w and total economic cost of 3 – 6 € per m^3 of digestate. In recent years, pressure-driven membrane processes, i.e. reverse osmosis, saw a considerable increase in its application due to its efficiency in nutrient recovery with yields up to 99 %w/w for P and N and total costs in the range 4 – 12 €/m^3 per year of digestate.

Struvite precipitation and HTC, although have a recovery rate of up to 99 %w/w of P and total cost of 270 – 2,000 €/t P removed, are rarely applied on agricultural digestate due to relatively low P content. Other technologies, such as adsorption and non-pressure driven membrane separation, are still at initial development stages, with a recovery yield of around 80 %w/w of N and P, with total economic cost not yet evaluated.

The practices for digestate valorization need to accelerate as the request for fertilizers is becoming more and more urgent as consequence of the depletion of mineral nitrogen and phosphorous sources. In the past years the potential of digestate was largely not exploited. A likely reason is due to the lack of legislation on biobased fertilizer, which hampered the research. The new Fertilizing Product Regulation of the EU (1009/2022) allows to fill up the lack of normative by the harmonization of the regulation between Member States, by the promotion of the biobased fertilizer market and, consequently, by the boosting of the research development.

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.

Data availability

No data was used for the research described in the article.

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