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Research article

Nutrients recovery from anaerobic digestate of agro-waste: Techno-economic assessment of full scale applications

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ABSTRACT

The sustainable production of fertilizers, especially those based on phosphorus, will be one of the challenges of this century. Organic wastes produced by the agriculture, urban and industrial sectors are rich in nutrients which can be conveniently recovered and used as fertilizers. In this study five full scale systems for the recovery of nutrients from anaerobic digestate produced in farm-scale plants were studied. Monitored technologies were: drying with acidic recovery, stripping with acidic recovery and membrane separation. Results showed good performances in terms of nutrients recovery with average yields always over 50% for both nitrogen and phosphorus. The techno-economic assessment showed how the specificity of the monitored systems played a major role: in particular, membranes were able to produce a stream of virtually pure water (up to 50% of the treated digestate) reducing the digestate volume, while drying, because of the limitation on recoverable heat, could treat only a limited portion (lower than 50%) of produced digestate while stripping suffered some problems because of the presence of suspended solids in the liquid fraction treated. Specific capital and operational costs for the three systems were comparable ranging between 5.40 and 6.97 € per m^3 of digestate treated and followed the order stripping > drying > membranes. Costs determined in this study were similar to those observed in other European experiences reported in literature.

1. Introduction

Nitrogen (N), phosphorus (P), and potassium (K) are critical to intensive agriculture and there are concerns over long-term availability and costs of production of these nutrients. This is particularly true for P and K which are predominantly sourced from mineral deposits which are concentrated in defined geographical Regions (Mehta et al., 2015). These issues are of major concern especially when considering a possible 9 billion population at 2050 and the necessity to sustainably produce more food through agriculture intensification on a global scale (Buckwell et al., 2015).

Nutrients, however, are present in abundance in waste streams: nitrogen and phosphorus contents are typically 1 kgN/ton and 0.25 kgP/ton in food waste (Micolucci et al., 2016), and 2 kgN/ton and 0.5 kgP in waste activated sludge at 5% dry matter (Leite et al., 2016). On the other hand, typical nitrogen and phosphorus concentrations range between 5 and 15 kgN/ton and 0.1 and 1 kgP/ton respectively in cattle and chicken manure (Giuliano et al., 2013). These nutrients remain in digestate after anaerobic digestion and, after a proper treatment, can be recovered in a concentrated form which can be conveniently transported.

Among the different agricultural, urban and industrial waste streams, livestock effluents because of their abundance, ubiquitous presence and characteristics are of primary interest for nutrients recovery. In fact, the number of heads in EU28 can be estimated in 100 million dairy cows and cattle, 100 million pigs and 1.5 billion poultries (European Commission – DG Environment, 2014; Flotats et al., 2013). The resulting annual production of manure is estimated in some 150 million tons for pigs, 450 million tons for cattle slurry, 300 million tons for cattle dung, and 110 million tons of chicken manure, for a total of 1380 million tons per annum (European Commission – DG Environment, 2014). Part of this material is used directly on fields after open-air stabilization but a considerable portion is stabilized through anaerobic digestion. Anaerobic digestate can be therefore considered as a new mine for fertilisers recovery (Flotats et al., 2013). Noticeably, according to

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data of the European Biogas Association (European Biogas Association EBA: Biogas and Biomethane Report, 2015), over 14,000 anaerobic digestion plants are currently running in Europe, 80% of which are operating in the agricultural sector and are farm based. During the anaerobic process normally used for the stabilization of livestock effluents part of the organic matter is transformed into biogas, a mix of carbon dioxide and methane, while the residual complex organic matter, such as lignin, and the inorganic part, including N, P and K, remain in digestate: during the digestion process the major part of nitrogen bound to organic matter will be released and then found in the soluble fraction as ammonium (NH_4^+) while the remaining part will be in the particulate fraction. The same process is valid for potassium, while phosphorous will be mainly present in the particulate fraction. In fact, also P released in the soluble fraction in the form of phosphate during the anaerobic digestion process will be largely precipitated because of the immediate reaction with soluble cations (i.e., calcium, magnesium, iron ….).

Digestate, which is rich in nutrients, can be therefore directly used as a renewable fertilizer because of its contents of stable organic carbon and nutrients (Möller and Müller, 2012; Vaneeckhaute et al., 2013) or, when the nutrients loads are in excess in a given area, can be further treated for nutrients recovery in concentrated forms to be then translocated at sustainable prices in different agricultural areas (Fuchs and Drosg, 2013; IEA Bioenergy, 1015). The excessive presence of nutrients loads and the necessity to control their presence in specific areas is a well-known problem in some European Countries and Regions, both in north (Denmark, Belgium, Netherlands, northern Germany, Brittany) and southern (Catalonia and Aragon, Spain, and the Po valley, Italy) Europe (Bernet and Béline, 2009).

Digestate can be therefore the mine for fertilizers production in a circular economy vision: this virtuous process is however hindered by legislative constrains for the time being. In fact, the use of bio-based fertilizers is not established yet, and the legislative framework is not encouraging this opportunity: most of these digestate-derived products, despite their characteristics, similar to those of commercial fertilizers, are not classified in any way (Möller and Müller, 2012; Vaneeckhaute et al., 2013).

Among the different commercial options for digestate treatment and nutrients recovery the most relevant are drying, stripping, evaporation and membranes technology which have been applied in recent years with alternate success for the treatment of anaerobic digestate or its solid or liquid fraction (Fuchs and Drosg, 2013; IEA Bioenergy, 1015; Bernet and Béline, 2009; Arbor project; Monfet et al., 2017; Sheets et al., 2015).

Here, we have considered the full-scale applications of technologies including stripping, drying, and membranes, the most common technologies for the treating of digestates originated from farm anaerobic digestion plants treating different livestock effluents and energy crops in the Po valley, northern Italy.

Drying consists in removing water in digestate and concentrate the residual fraction by using hot air. In fact, anaerobic digestion plants with a combined unit for heat and power (CHP) generation often have the availability of a considerable amount of heat after digester warming e.g., (Fuchs and Drosg, 2013; IEA Bioenergy, 1015; Arbor project; Sheets et al., 2015; Pöschl et al., 2010). Part of this heat can be used to treat digestate so to obtain a dried solid (powder) material which is strongly reduced in volume and stable in biological terms. Ammonia nitrogen can be removed with vapor or kept in the digestate if it is acidified through the addition of mineral acids. If removed with vapor, nitrogen can be then recovered by means of acidic scrubbing or reverse osmosis as ammonium sulfate, when H_2SO_4 is used, ammonium nitrate, when HNO_3 is used, or as concentrated ammonium solution (in water). In general, because of the diluted feedstock used in the digester, the heat amount recovered from the CHP unit is not sufficient for the complete drying of all produced digestate which is normally characterized by a water content of around 90% (Monfet et al., 2017; Sheets et al., 2015; Vaneeckhaute et al., 2017). Additional heat can be however recovered from the CHP off gas by means of dedicated gas-water heat exchangers. This will allow for further removing of some water from digestate.

In the stripping systems, digestate undergoes to one or more pre-treatments for solid/liquid separation and the liquid stream is then sent to a packed bed tower where ammonia (NH_3) is stripped and physically transferred from the aqueous to the gas phase. This gas stream passes then in a second system (typically another packed column system) where NH_3 is absorbed in an acidic media, normally sulfuric acid, producing ammonium sulfate at 25–35% (Fuchs and Drosg, 2013; IEA Bioenergy, 1015; Sheets et al., 2015; Bonmati and Flotats, 2003; Adani, 2011). The advantage here is that nitrogen is recovered in a pure form while other nutrients like K, P and stable C, remain in the treated liquid phase which can be used on fields. In case NaOH or other alkali solutions are used to increase pH, relatively high levels of Na^+ can be found in the liquid phase thus altering the salinity of this stream. This aspect should be considered when reusing this stream for agricultural purposes as the high salinity level can then influence the cationic exchange capacity (CEC) of soils (Tao et al., 2016).

In pressure-driven membrane filtration the liquid phase of digestate (after solid/liquid separation and further solids removal by means of centrifuge or cartridge) is treated in ultrafiltration (UF) and reverse osmosis (RO) systems. The produced concentrate from RO is rich in both macro and micro nutrients and has characteristics similar to those of vinasses obtained from distillation, a recognized fertilizer (Fuchs and Drosg, 2013; IEA Bioenergy, 1015; Masse et al., 2007; Ledda et al., 2013).

In this study, we considered the full-scale application of drying, stripping and membranes systems for nutrients recovery and concentration in livestock digestate. Beside the characteristics of obtained outputs (fertilizers), mass balances and efficiencies of the monitored technologies were determined. Finally, a techno-economic analysis was carried out to verify the effective sustainability of the process.

2. Materials and methods

2.1. Experimental set up and studied plants

Five farm anaerobic digestion plants using different techniques for post-treatment of digestate were considered in this study: two using a drying belt, one using a stripping column and two adopting membrane technologies. While the drying system can treat the solid fraction or digestate as a whole, stripping and membrane systems operate only on the liquid fraction of digestate after a proper solid/liquid separation step. Both drying belt and the stripping column were coupled with a scrubber to clean up the exhausted air and to recover ammonium sulfate in an acidic solution. These plants were monitored for a period of at least six months (on average, a period equivalent to at least 3 HRTs of the anaerobic digester) and the main relevant parameters were determined for feedstock, biogas, digestate and processed streams. Table 1 reports a resume of the main features of the studied plants: the main feedstock composition, the potential of biogas electrical power and the digestate treatment technology for each plant are reported. Beside the determination of chemical-physical characteristics, also economic data related to capital and operation costs were collected to define the economics of the studied techniques.

Table 1
Main features of the monitored plants.

Plant	Feedstock	Plant Size, kW	Treatment system	Digestate fraction treated	Products
A1	Pigs effluents, chicken manure, energy crops	999	Dryer	Solid fraction + portion of the liquid fraction	Ammonium sulfate and dried organic digestate
A2	Cow manure, energy crops, slaughterhouse residues (blood), food waste	999	Dryer	Solid fraction + portion of the liquid fraction	Ammonium sulfate and dried organic digestate
B1	Pigs effluents, energy crops	999	Membrane separation	Liquid fraction	Reverse osmosis centrate, ammonium sulfate, water
B2	Cow manure, energy crops	190	Membrane separation	Liquid fraction	Reverse osmosis centrate, ammonium sulfate, water
C1	Cow manure, pigs effluents, energy crops	600	Stripping	Liquid fraction	Ammonium sulfate

2.2. *Drying system*

In this study, we considered two farm scale drying installations where digestate derived from the anaerobic digestion of cow or pigs manure plus energy crops was treated. In one case, also slaughterhouse (mostly blood) effluents were treated thus increasing the nitrogen load (see plants A1 and A2 features in Table 1). Digestate passed a solid/liquid separator and the liquid part was then mixed with part of the produced dried material so to reach a 10–12% dry matter content (Fuchs and Drosg, 2013; IEA Bioenergy, 1015; Arbor project). This material was distributed on the stainless-steel belt by means of a screw conveyer. The dryer belt was 18 m long, constituted by a rolling single layer with holes where hot air at 70–80 °C passes through the digestate (bottom-up flow). Further heat to produce warm air was recovered from a heat exchanger treating the CHP off gas. The produced vapor phase, rich in ammonia, passed through an acidic scrubber where sulfuric acid was used to recover ammonia in the form of ammonium sulfate.

2.3. *Stripping system*

In this system digestate underwent to a pre-treatment for solid/liquid separation (screw press type) and then the liquid stream was further treated for suspended solids removal in lamella settlers and sent to the stripping column (Fuchs and Drosg, 2013; IEA Bioenergy, 1015; Arbor project). The studied system considered a double column process: the first column was dedicated to ammonia stripping from the liquid fraction of digestate while the second column was dedicated to the recovery of nitrogen as ammonium sulfate. Both columns were 4 m high. In particular, the liquid stream of digestate was basified with $Ca(OH)_2$ to pH values over 9 and then injected (up – down) in countercurrent to hot air (down – up) in the first column. Air from environment was put in contact with the cooling system of the CHP unit so to produce air at 60–70 °C to be used for the stripping process. In the first column, because of the combined effect of pH and temperature, ammonia leaves the liquid phase to pass in the gas phase. This gas phase passes then in the second packed column (down-up) where it reacts with sulfuric acid to form ammonium sulfate. Both columns are filled with filling material with a high specific surface so to facilitate the mass transfer phenomena. The residual liquid phase, with a low nitrogen content, but with the same amount of P and K of digestate, was used on fields.

2.4. *Membrane separation systems*

The studied systems were characterized by a series of physical treatments which allowed for the separation of the particulate and liquid fractions of digestate (Fuchs and Drosg, 2013; IEA Bioenergy, 1015; Arbor project): the first solid/liquid separation was achieved by means of a screw press separator (FAN, Germany). The liquid fraction obtained was added of polymer and treated in a horizontal centrifuge (decanter, Mammoth, Pieralisi) for the further removal of particulate solids. The effluent liquid was then treated in an ultrafiltration (UF) system with plate and frame membrane (Pleiade, Orelis Environment) with a molecular weight cut off at 40 kDa and operating at maximum pressure of 3.5 atm. The filtered liquid effluent from the UF unit was then ready for the treatment in a double cartridge reverse osmosis (RO) unit operating at 30 atm and able to recovery up to 70–80% of water from the treated stream (50% of the initial digestate mass). The system operated in batch mode, 14 m^3 each cycle up to a maximal treatment capacity of 100 m^3 per day. In this study two digestates of different feeding compositions, one deriving from the anaerobic digestion of dairy cows manure and energy crops and the other one coming from the anaerobic digestion of piggery effluents and energy crops, were tested. The system can be further implemented with a stripping tower to recovery ammonium sulfate from the reverse osmosis centrate (Ledda et al., 2013).

2.5. *Sampling and chemical analysis*

Anaerobic digestate, and the liquid and solid fractions originated from the treatment systems were all weekly sampled at least three times during a period of two months (the typical hydraulic retention time of farm scale anaerobic digesters monitored in this study) and the monitoring was repeated three times, covering a total period of six months. The chemical-physical characteristics of collected samples were determined according to the Standard Methods for Water and Wastewater Analysis (APHA et al., 2012). In particular, dry and volatile matter, organic matter (as COD), nitrogen and phosphorus in their soluble and particulate phases, were the main targets of the monitoring activity.

2.6. *Techno-economic analysis*

The basics for the techno-economic analysis were the mass and energy balances of the systems and their performances as well as the capital costs (CAPEX) and operational costs (OPEX). Data on investments, labor, energy and chemicals consumption were determined so to define a techno-economical assessment of the studied technologies. Revenues from fertilizers or nutrients selling were not taken into account in the economic balance so to define the worst scenario. The Capex were amortized according with the following equation:

$$Q = C \cdot \frac{r(1+r)^n}{(1+r)^n - 1}$$

where, Q is the periodic amortization payment, C is total investment cost for the plant installation, r is the interest rate fixed at 3% while n is the lifetime of the equipment (10 years). Opex considered the items: energy, chemicals, labor. These were communicated by the farmers adopting the monitored systems or the commercial firm commercializing the specific techniques. Both capex and opex were referred to the amount of treated digestate so to obtain a comparison in Euros per cubic meter of digestate treated (€/m^3) and facilitate the comparison among different treatment systems (IEA Bioenergy, 1015).

3. Results and discussion

3.1. Drying process

Drying aims at reducing the volume of digestate (or its solid fraction) in systems like belt dryer, drum dryer, fluidized bed dryer. In all cases, the liquid or semi-liquid stream or the solid fraction of digestate are put in contact with hot air warmed using the heat coming from the CHP unit for biogas combustion. Available hot air is sometime implemented with the thermal energy recovered by a heat exchanger on the off-gas pipe. With specific reference to the application of drying process in plants A1 and A2, the thermal energy available in each CHP units of 999 kW of electrical power is around 22–24 MWh per day (Fig. 1). Around 1.5–2.5 MWh/d of this thermal energy are used to keep the digester at 40–42 °C of temperature, while the rest is used to evaporate water in the dryer. Considering a typical specific heat request of around 1.1 MWh per ton of water evaporated, some 26–28 m^3/d of water were eliminated (case A1). On the contrary, plant A2 had lower thermal energy available for the drying system due to the thermal energy required (11.5 MWh/d) for the pasteurization of slaughterhouse wastes (Figs. 1b and 2b). The residual 10 MWh/d allowed for the evaporation of around 10 m^3 per day of water.

It should be emphasized here that, in general, the excess heat of the CHP system is not sufficient for the complete drying of digestate, especially when liquid feedstocks are used (e.g., liquid manure). As a rule of thumb, available heat is sufficient to evaporate a portion of water ranging between 25 and 50% of the total water in the digestate. Because of this limitation on recoverable heat, the digestate effectively treated is less than 50% of the total amount produced. Therefore, also recovered nitrogen is only a portion of the total quantity of nitrogen originally present in the feedstock. This is clearly a limitation of this technology if nitrogen recovery is the main aim.

Organic nitrogen and the totality of phosphorus and potassium originally present in the digestate remained in the dried fraction together with stabilized organic matter and other inorganic nutrients. This dried fraction can be eventually pelletized to facilitate transportation and use. The characteristics of digestates considered in this study are reported in Table 2: digestate originated from piggery effluents (A1) presented a lower dry matter and nitrogen and phosphorus contents compared to the digestate produced in the digester treating cow manure, energy crops and slaughterhouse residues (A2). Dry matter concentration in the digestate from cow manure was 78 g/kg versus 63 g/kg of piggery effluents while nitrogen showed an average concentration of 6.8 gN/L, a value 4 times higher than the one observed in the case of piggery effluents. Also phosphorus was more abundant in the case of cow manure digestate (A2): total phosphorus was 0.8 gP/kg versus 0.3 gP/kg. In both cases digestate passed through a drum thickener and was slightly dewatered increasing the total solids concentration to levels around 10–12%. This material is squeezed on the drying belt and then dried. Obtained dried solids (Table 2) showed a dry matter concentration greater than 90% (Fuchs and Drosg, 2013; IEA Bioenergy, 1015) and 65% volatile matter, while N and P concentrations where 21 and 27 g/kg for N and 4 and 12 g/kg for P. These characteristics are in good agreement with values reported in literature for these systems: average dry matter contents around 90% and N concentrations of 24 gN/kg are reported also in other studies (IEA Bioenergy, 1015; Arbor project; Vaneeckhaute et al., 2017).

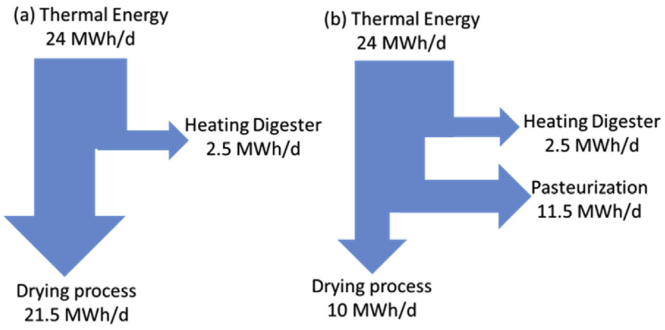

Fig. 1. Thermal Energy balances for plants A1(a) and A2(b).

Ammonium sulfate can be recirculated in the scrubber reaching a final concentration above 25% with an N concentration normally greater than 4% and up to 6%. However, in this study, the recovered ammonium sulfate was in both cases diluted: nitrogen reached levels of 2.4 and 3.5% insufficient for the definition of fertilizers (N at 6%) but however sufficiently concentrated for a convenient transportation.

A typical mass balance for the applied process for the two monitored plants is shown in Fig. 2. Clearly, because of heat limitation, only part of digestate could be treated: this fraction was between 20% and 30% of total produced digestate in our study. After the treatment on the belt drier half of soluble nitrogen was recovered as ammonium sulfate and half remained in the dried fraction. The nitrogen recovered in ammonium sulfate was 38% and 47% of the nitrogen present in the treated fraction of digestate in the two cases, respectively. Because only part of digestate was treated in the drying system in the two case studies reported here nitrogen recovered in ammonium sulfate was 15% and 20% of the whole nitrogen load in anaerobic digestate in plants A1 and A2 respectively.

Carbon, phosphorus and other inorganic nutrients were completely recovered in the solid dried material: reported mass balances (Fig. 2) calculated around the drying system closed with errors below 10%.

3.2. Membranes technology

In membrane processes digestate is pre-treated by means of one or more solid/liquid separation systems like screw press and centrifuge so to obtain a stream with a low suspended solids content to be then treated in ultrafiltration and reverse osmosis systems. This will allow for the production of water of good quality (up to 50% of the treated digestate) and a RO centrate phase rich in nutrients (Fuchs and Drosg, 2013; IEA Bioenergy, 1015; Arbor project).

In the studied systems digestate was primarily treated in a screw press separator, the liquid stream generated was added with flocculants and treated in a centrifuge decanter to obtain a liquid characterized by a solid content <2%. This liquid is then refined in an ultrafiltration membrane and then treated in a reverse osmosis unit where nutrients were concentrated. Tables 3 and 4 reports the typical concentrations observed in the different steps when dairy cows (Table 3) and piggery (Table 4) digestates were treated.

Most of the soluble nutrients originally present in the digestate are retained in the centrate of the reverse osmosis unit. In this

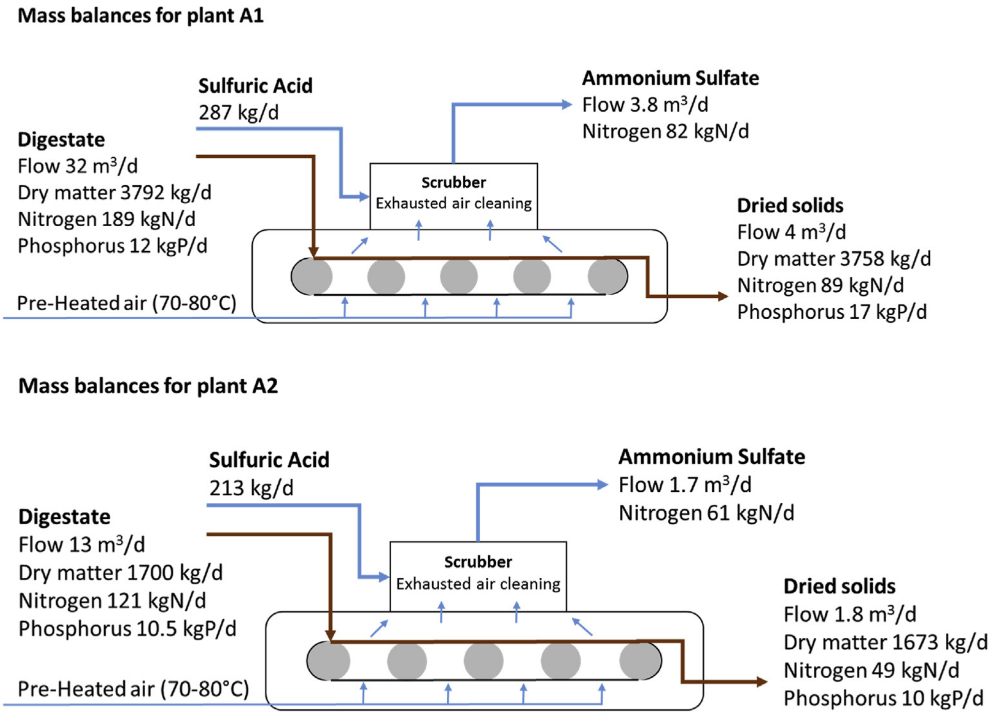

Fig. 2. Mass balance for a typical drying belt system.

Table 2
Characteristics of influent (digestate) and effluents (dried solids and ammonium sulfate) of the drying system.

	Dry matter, g/kg	Volatile matter, g/kg	Total nitrogen, gN/kg	Ammonium, gN/kg	Total Phosphorus, gP/kg	Phosphate, gP/kg
Plant A1						
Digestate (influent)	63 ± 5	44 ± 4	3.2 ± 0.3	1.6 ± 0.2	0.3 ± 0.1	0.1 ± 0.1
Dried solids (effluent)	939 ± 43	709 ± 31	21 ± 21.5	nd	4.0 ± 0.5	Nd
Ammonium sulfate (effluent)	nd	nd	25 ± 2	24 ± 3	nd	Nd
Plant A2						
Digestate (influent)	78 ± 7	56 ± 6	9.5 ± 1.8	6.8 ± 0.3	0.8 ± 0.1	0.2 ± 0.2
Dried solids (effluent)	936 ± 38	625 ± 27	27 ± 2	nd	6.0 ± 1	Nd
Ammonium sulfate (effluent)	nd	nd	36 ± 7	35 ± 6	nd	nd

Nd: not determined.

Table 3
Characteristics of dairy cows digestate, filtrated and centrate streams in a membrane process.

	Dry matter, g/kg	Volatile matter, g/kg	Total nitrogen, gN/kg	Ammonia, gN/kg	Total Phosphorus, gP/kg	Phosphate, gP/kg
Digestate	70 ± 3	49 ± 2	3.35 ± 0.3	1.73 ± 0.1	1.64 ± 0.3	0.069 ± 0.03
Screw press solid fraction	220 ± 27	198 ± 15	3.25 ± 0.3	nd	4.00 ± 0.9	nd
Screw press liquid fraction	55 ± 2	37 ± 2	3.23 ± 0.2	1.61 ± 0.2	1.29 ± 0.1	0.067 ± 0.03
Centrifuge solid fraction	201 ± 13	140 ± 12	7.25 ± 1.0	nd	5.25 ± 1.2	nd
Centrifuge liquid fraction	18 ± 1	10 ± 1	1.7 ± 0.1	1.5 ± 0.1	0.13 ± 0.05	0.060 ± 0.01
Ultrafiltration centrate	38 ± 2	27 ± 1	2.9 ± 0.2	1.25 ± 0.2	0.25 ± 0.03	0.066 ± 0.01
Ultrafiltration filtrate	8 ± 1	3.5 ± 1	1.3 ± 0.1	1.3 ± 0.1	0.092 ± 0.01	0.060 ± 0.01
Reverse osmosis centrate	36 ± 2	15 ± 2	4.8 ± 0.3	4.8 ± 0.2	0.36 ± 0.03	0.086 ± 0.02
Reverse osmosis permeate	<1	<1	<0.1	<0.1	<0.01	<0.01

Nd: not determined.

process 50% of the digestate mass is recovered as virtually pure water which can be reused in the AD process, on fields or discharged into water bodies (when available). This technology allows for important savings for the transportation costs of the concentrated nutrients but clogging and fouling are major problems while energy costs for reverse osmosis are still high.

The dairy cows digestate showed on average a 7% dry matter content, a total nitrogen concentration of 3.35 gN/L, half of which being ammonia, and a total phosphorus concentration of 1.64 g/L, 40% soluble (see Table 3). With specific reference to data of Table 3, it turned out clear that the soluble forms of N, and P, ammonia and phosphate, passed all the solid/liquid separation steps and were then concentrated in the reverse osmosis centrate, where TN and TP concentration were 4.8 mgN/L and 0.36 mgP/L, respectively.

Noticeably, nitrogen was present in the soluble form (ammonium) while most soluble P (phosphate) was only 25% of TP.

On the other hand, particulate forms remained in the “solids” fraction: in this sense, it is of interest observing that most of N and P

Table 4
Characteristics of piggery digestate, filtrated and centrate streams in a membrane process.

	Dry matter, g/kg	Volatile matter, g/kg	Total nitrogen, gN/kg	Ammonia, gN/kg	Total Phosphorus, gP/kg	Phosphate, gP/kg
Digestate	32 ± 3	21 ± 2	2.25 ± 0.4	1.61 ± 0.3	0.36 ± 0.01	0.079 ± 0.01
Screw press solid fraction	231 ± 12	198 ± 10	4.62 ± 0.8	nd	1.92 ± 0.01	nd
Screw press liquid fraction	21 ± 5	11 ± 3	2.20 ± 0.3	1.60 ± 0.3	0.25 ± 0.02	0.077 ± 0.01
Centrifuge solid fraction	187 ± 12	130 ± 11	3.74 ± 0.3	nd	1.54 ± 0.02	nd
Centrifuge liquid fraction	9.4 ± 1.2	3.6 ± 0.6	1.66 ± 0.2	1.46 ± 0.2	0.15 ± 0.01	0.076 ± 0.02
Ultrafiltration centrate	21 ± 2.2	9.5 ± 1.1	1.52 ± 0.5	1.31 ± 0.1	0.19 ± 0.02	0.078 ± 0.01
Ultrafiltration filtrate	7.1 ± 0.5	3.5 ± 0.3	1.56 ± 0.3	1.42 ± 0.1	0.08 ± 0.02	0.078 ± 0.03
Reverse osmosis centrate	25 ± 2	12 ± 0.9	5.27 ± 0.8	4.37 ± 0.6	0.26 ± 0.01	0.1 ± 0.03
Reverse osmosis permeate	<0.1	<0.1	0.08 ± 0.05	0.07 ± 0.06	<0.01	<0.01

Nd: not determined.

particulates forms remained in the solid fraction separated by the decanter. In this case, the use of polymers enables to capture most of the suspended solids and colloids, so the major part of nutrients remained in this stream.

In a second plant monitored in this study piggery digestate was treated. Here the level of total solids is clearly lower than the one observed in the case of dairy cows digestate. The nutrients concentrations were also lower: these were 2.25 gN/L for TN and 0.36 gP/L for TP, mostly in the soluble forms.

Also in these cases we observed that the particulate fractions were mainly recovered at the level of the centrifuge while soluble forms remained in the reverse osmosis centrate where final concentrations for N and P reached average levels of 5.27 gN/L and 0.26 gP/L, respectively.

The mass balances of the process for the two treatment systems are reported in Figs. 3 and 4.

These put under light that some 50% of water originally present in digestate can be recovered (43% for cows effluent and 46% for piggery effluent) while, considering the final fate of nutrients, 41% of nitrogen is recovered in the reverse osmosis centrate when treating piggery effluents but only 17% when treating dairy cows digestate.

On the other hand, phosphorus is recovered in the solid streams originated from screw press and centrifuge: more than 80% of P is present in those streams for both the monitored case study.

If nitrogen is the major target, this can be further separated by means of a (cold) stripping process adding alkali and varying the pH in the range > 10 as it was the case in these plants (Ledda et al., 2013).

3.3. Stripping process

In this process digestate undergoes to a preliminary solid-liquid separation system, typically a screw press, and the liquid fraction is then further treated for the removal of residual suspended solids. The remaining liquid is then treated in a stripping column. Here, ammonia is transferred from the liquid phase to the gas phase due to the action of hot (60–70 °C) air recovered from the CHP unit. Moreover, also the liquid can be warmed to favorite the mass transfer from the liquid to the gas phase. This process can be further improved by adjusting liquid pH to levels above 9.5 adding soda (Limoli et al., 2015) but this increases opex on one hand and leave Na^+ in the liquid phase increasing the salinity on the other hand. The gas phase, rich in ammonia, enter then in a second packed column where sulfuric acid is spread to form ammonium sulfate. Both columns are normally organized with filling media so to increase the specific surface available for the mass transfer.

The removal of organic and inorganic fine particles together with the use of alkali for pH control are fundamental features of this technology, influencing problems of scaling, fouling and clogging of the packed columns thus strongly influencing the operational costs of this process.

The characteristics of digestate treated are reported in Table 5: it is a typical digestate originated from the anaerobic digestion of piggery and cow manure with energy crops addition. The dry matter content was around 5% and the nitrogen concentration was 3.6 gN/L where 75% as ammonium. Total phosphorus was 0.5 gP/L. Digestate was then separated in a liquid and solid phase by means of a screw press and the liquid phase is then settled for further solid removal by means of a lamella clarifier. Considering the mass balance of the process (Fig. 5) around 17% of the nitrogen originally present in digestate is recovered in the form of ammonium sulfate while the remaining part of nutrients remain in the liquid phase and are not concentrated. This liquid part, which still contain the residual soluble part of nitrogen and phosphorus, is generally use directly on fields.

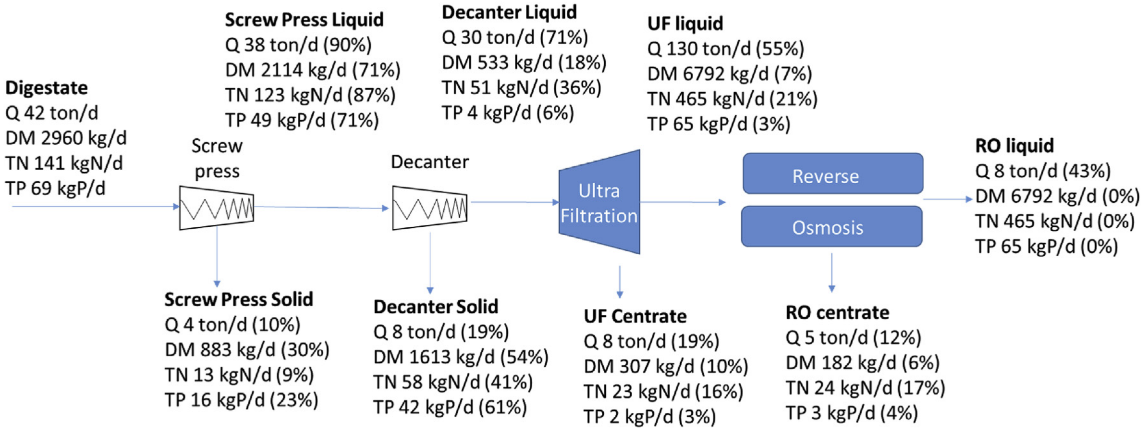

Fig. 3. Mass balance for the system treating digestate of dairy cows effluents (Q, flowrate; DM, dry matter; TN, total nitrogen: TP, total phosphorus).

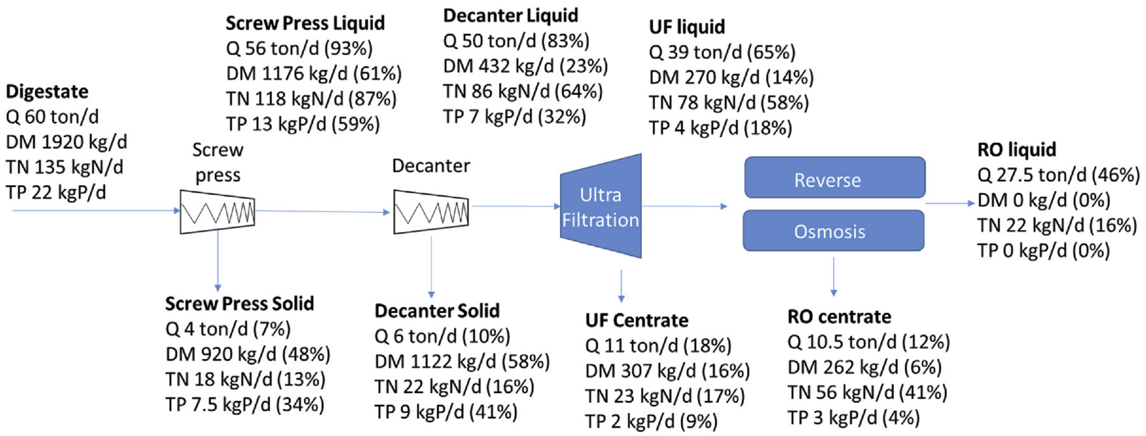

Fig. 4. Mass balance for the system treating digestate of piggery effluents (Q, flowrate; DM, dry matter; TN, total nitrogen: TP, total phosphorus).

Table 5
Digestate, liquid effluent and ammonium sulfate characteristics.

	Dry matter, g/kg	Volatile matter, g/kg	Total nitrogen, gN/kg	Ammonium, gN/kg	Total Phosphorus, gP/kg	Phosphate, gP/kg
Digestate	52 ± 4	42 ± 4	3.6 ± 0.5	2.6 ± 0.2	0.5 ± 0.2	0.2 ± 0.2
Liquid effluent	42 ± 9	29 ± 6	2.7 ± 0.7	1.8 ± 0.3	0.2 ± 0.4	0.1 ± 0.2
Ammonium sulfate	nd	nd	26 ± 5	24 ± 4	nd	nd

Nd: not determined.

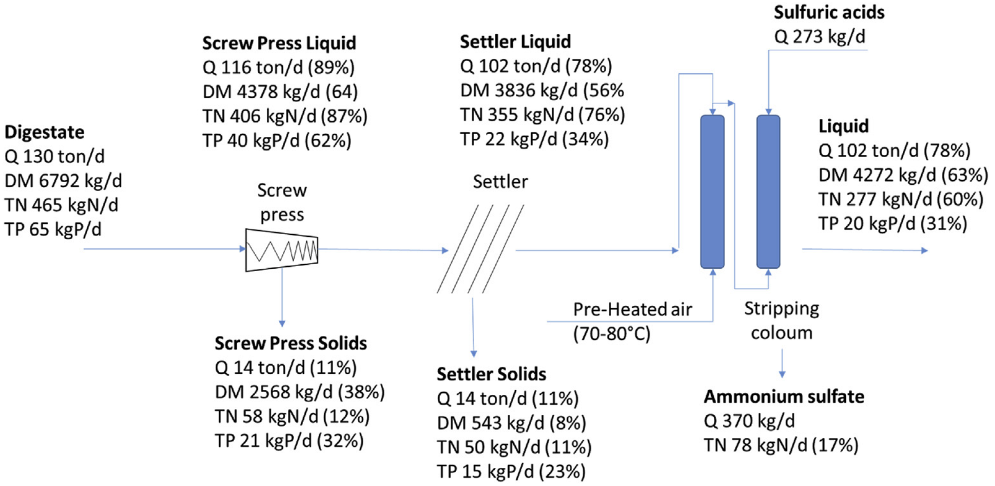

Fig. 5. Mass balance for the stripping process.

3.4. *Techno-economic assessment*

The techno-economic assessment of the considered technologies was based on both capital (capex) and operational (opex) costs. As described in the materials and methods section information derived from selling firms, operators (farms) and technical literature (e.g., Navarotto, 2017) have been considered to put together a reliable economic analysis. Moreover, direct interviews with technicians were organized to discuss both technical and economic aspects.

The capital costs for the dryer, stripping and membrane systems were amortized over a period of 10 years considering an interest of 3%. The flowrate treated in the same period was considered so to determine a specific value referred to a single cubic meter of digestate produced by the biogas plant. In the same manner, operational costs for energy, chemicals, labor/service were considered and referred to the treated digestate so to obtain a specific value to be added to the capex (€ per m^3 of treated digestate).

Table 6 reports a summary of the calculated costs collected during this experimentation. Clearly, costs estimation is a difficult exercise since there are great uncertainties on industrial investments and running costs especially when a limited time span is considered. Moreover, opex are very often site-specific, being strongly influenced by boundary conditions. However, a first estimate, at least for the considered case studies, can be defined and compared with other similar researches.

The capital cost of a dryer treating 30 m^3 per day of digestate was estimated in 300,000 €: with an amortization span of 10 years the corresponding capex was calculated in 2.74 €/m^3. The specific costs for energy were calculated in some 1.00 € per m^3. Requested chemical is sulfuric acid, for a corresponding cost of 1.00 €/m^3. The

Table 6
Costs analysis for the three technologies considered. All costs are in € per m^3 of digestate treated (*the cost of electrical energy was considered 0.1 €/kWh).

Cost item	Dryer (€/m^3)	Stripping (€/m^3)	Membrane (€/m^3)
Capital cost (amortization)	2.51	1.58	2.74
Energy (power)*	1.00	1.06	1.85
Chemicals	1.00	1.50	0.33
Labor/service	1.3	0.3	1.05
Total estimated costs	5.81	5.44	6.97

costs for personnel/service were estimated in 1.30 €/m^3. The corresponding total specific cost was some 6.04 € per m^3 of digestate. This cost is similar to costs reported by IEA – bioenergy (IEA Bioenergy, 1015).

The investment cost for a stripping system treating up to 100 m^3/d of digestate was estimated in 750,000 €. The corresponding specific amortization is 1.58 €/m^3. The installed power in our study was around 40 kW working 24 h per day. The specific cost is therefore 1.06 €/m^3. Chemicals used in the stripping system are soda or $Ca(OH)_2$ for pH correction above 9, and sulfuric acid for ammonium sulfate recovery. Costs for chemicals were estimated in 1.5 € per m^3 of digestate. As for labor and service we considered 1 person per year fully dedicated to the system and some additional costs for a total of 1.3 €/m^3. The total specific cost was calculated in some 5.44 € per m^3 of digestate treated. Vaneeckhaute et al. (2017) reported costs around 8 € per m^3 of digestate treated for a 90% N recovery from leachate at a temperature of 70 °C and pH 11 for a treated flowrate of 70 m^3/h. on the other hand, reported costs are as low as 2 € per m^3 of digestate treated when operating at lower temperatures (Vaneeckhaute et al., 2017). Interestingly, the same authors calculated costs of 4.5–8.6 € per m^3 of digestate treated in case of NaOH addition. Overall, these data are comparable with those obtained in our study.

When considering the membrane system, the investment cost is particularly high (up to 1 million € for a system with a treatment capacity of 100 m^3 of digestate per day) but the same apply for the treated digestate: therefore, the specific cost is only 2.74 €/m^3, a value similar to the one determined for drying systems. The installed power for the two membrane systems considered in this study were around 50 kW. The corresponding specific cost for energy consumption was 1.85 €/m^3. Costs for used chemicals were associated with flocculant for the decanter and the solutions for membranes cleaning. Specific costs were estimated in 0.33 €/m^3 while costs for personnel/service were high because of the need for a skilled person. These were some 2.05 €/m^3 for a total specific cost of 6.97 € per m^3 of digestate treated. The costs reported in Dutch and Flemish studies were as high as 12 € per m^3 of digestate treated for flowrate as low as 2 m^3 per day and in any case in the range 11–12 € per m^3 when treating manure, while lower costs, down to 4.22 € per m^3 of digestate treated, were reported for a Canadian application where piggery effluent was treated (Vaneeckhaute et al., 2017).

Beside the costs, the specific peculiarities of the systems should be mentioned: a membrane system can recover half of the water content of digestate as pure osmotized water. Therefore, half of the volume is reduced while water of very good quality is recovered. On the other hand, the dryer system can treat only part of the digestate (unless external heat is added) while the stripping system maintain unaltered the volumes treated. In particular, as shown above, in a dryer system treating the digestate produced form a biogas plant with size of 1 MW, the availability of thermal energy is around 1000 kWh per day. Considering that the thermal request for keeping the digester at a temperature of 37 °C is around 250 kWh in winter and 100 kWh in summer (Italian latitude), the thermal energy available for the dryer is 800 kWh on average. This is sufficient for removing 670 kg of water. This is clearly an intrinsic limitation of the system which will remove around 12–15 m^3/d of water in the given conditions.

Table 7
Expected concentrations of nitrogen and phosphorus in recovered dried digestate, ammonium sulfate and osmosis centrate found in the present survey.

	Nitrogen content, %	Phosphorus content, %
Dried solid fraction of digestate	2.5–3.5	0.3–0.6
Ammonium sulfate	5–6	–
Reverse Osmosis centrate	0.5–0.6	0.1

Despite all these uncertainties and boundaries conditions it is important to emphasize how determined costs are in line with those reported for German case studies by the IEA (IEA Bioenergy, 1015) as well as in Dutch and Flemish studies (Arbor project; Vaneeckhaute et al., 2017).

3.5. Unlocking the fertilizer market for recovered products

The recovered materials from the applied technologies reported in this study show interesting quality and levels of nutrients. Table 7 summarizes the expected concentrations of nutrients in these materials.

However, recovered fertilizers, like ammonium sulfate or osmosis centrate, are generally diluted if compared with urea or ammonium nitrate commercially available: in the first case nitrogen content is around 5–6% while in commercial products nitrogen concentration passes 20%. Clearly, the product quality for these products should be guaranteed for commercialization: in this sense the recovery of ammonium sulfate through stripping and acidic scrubbing is of primary interest since the product purity is related to the sulfuric acid used in the process and can be therefore controlled (Vaneeckhaute et al., 2017). For ammonium sulfate of good quality (6% nitrogen, 30% ammonium sulfate) the expected corresponding value is around 30 € per m^3.

Despite this, Vaneeckhaute et al. (2017) reported that although ammonium sulfate is a recognized inorganic fertilizer for Flanders and the Netherlands, marketing is still hindered due to N and S variable concentrations, low pH, and high salinity. The same situation applies to Italy and normally the same companies commercializing sulfuric acid for the scrubbing towers buy and then commercialize the produced ammonium sulfate, without any extra benefit.

It is however important to emphasize that today most of these products are often under the definition of waste and despite their technological, economic and environmental sustainability a real market is not established yet.

In this sense, the adoption by the European Commission of the Circular Economy package in December 2015 and the proposed modification of Regulation No 2003/2003 on fertilisers (EC,), are fundamental instruments to open a new market for these recycled materials.

4. Conclusions

Five full scale treatment systems for nutrients recovery from anaerobic digestate of livestock effluents were monitored. The studied systems were drying, membranes and stripping. The systems worked properly and gave good results which are however quite different: membranes systems can recover water of good quality while reducing the digestate volume, while drying systems, because of limitation on heat availability, can treat only part of the digestate although very effectively. The stripping system allowed for the recovery of less than 40% of the influent nitrogen and do not change the volume of the residual part. Costs for operations, including amortization of capital costs are in the range 5.40–6.97 € per treated m^3 of digestate and in the order stripping $<$ drying $<$ membranes. These results are in line with similar studies carried out around Europe. There is still need for harmonized legislation favoring the commercialization of renewable fertilizers.

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