Deliverable

Project Acronym: FERTIMANURE

Project full name: Innovative nutrient recovery from secondary sources – Production of high-added value FERTIlisers from animal MANURE

Grant Agreement No. 862849

D2.6. Mass and energy balance of the on-farm pilots to WP5

Project start date	January 1st, 2020
Duration in months	54
Deliverable due date	December 31st, 2023
Actual submission date	December 22nd, 2023
Work package concerned	WP2
Author(s) and Co-author(s)	F. Adani, E. Clagnan, X. You, R. Rodríguez, N. Guerra, M. Mora, L. Llenas, I. Sigurnjak, R. Vingerhoets, K. van Dijk, O. Schoumans, N. Thevenin, L. Ruidavets, S. Schönfeld
Contributor(s)	UMIL, LEITAT, CPV, ALGE, UGENT, WENR, RITTMO, UVIC, FHR

Document History

Date	Author	Action	Status
01/06/23	**F. Adani; E. Clagnan,**	**First request**	**Starting preparing deliverable.**
25/10/2023	**F. Adani**	**Second request**	**Deliverable on going. First draft**
08/11/2023	**F. Adani, E. Clagnan, X. You, N. Guerra, M. Mora, L. Llenas, I. Sigurnjak, R. Vingerhoets, K. van Dijk, O. Schoumans, N. Thevenin, L. Ruidavets, S. Schönfeld**	**Deliverables uploaded on Microsoft Teams platform**	**Deliverables first draft: continuous updating**
22/12/2023	**F. Adani, E. Clagnan, X. You, N. Guerra, M. Mora, L. Llenas, I. Sigurnjak, R. Vingerhoets, K. van Dijk, O. Schoumans, N. Thevenin, L. Ruidavets, S. Schönfeld**	**Final draft**	**Final draft ready to be submitted**
15/10/2024	**F. Adani, E. Clagnan, X. You, N. Guerra, M. Mora, L. Llenas, C. Fabregas, I. Sigurnjak, R. Vingerhoets, K. van Dijk, O. Schoumans, N. Thevenin, L. Ruidavets, S. Schönfeld**	**Revision of the deliverable following the comments received from reviewer**	**Revised deliverable**
18/10/2024	**F. Adani**	**Final revised draft**	**Final revised version**

Preface

This deliverable is part of the European project FERTIMANURE funded by the H2020 programme (project number 862849). The project FERTIMANURE focuses on the formulation and evaluation of bio-based fertilisers (BBFs) produced at five pilot nutrient recovery installations and tailor-made fertilisers (TMFs) as blends of BBFs and (synthetic) mineral fertilisers to meet the soil-crop specific requirements.

In FERTIMANURE work package (WP) 2 (Nutrient recovery from animal manure), five different on-farm pilots have been implemented, and they will be operated and optimized from month (M) 13 to M48, to reach the completion of the following deliverables:

D2.1. Processes and technologies specification and set up to produce BBFs from animal manure (TRL 6-7) (M12, M26)
D2.2. BBFs production and characterization vs. time (list, average composition and composition variability) (M12, M25)
D2.3. Mass and energy flows and balances of the demonstration on-farm pilots (TRL 6-7) (M30)
D2.4. BBFs production and characterization vs. time (list, average composition and composition variability) (TRL 6-7) (M36, currently being finalized)
D2.5. BBFs production and characterization vs. time (list, average composition and composition variability) (to be completed for M48)
D2.6. Mass and energy balance of the on-farm pilots to WP5

Deliverable D2.6 (Mass and energy balance of the on-farm pilots to WP5) reports final data on mass and energy flow balances of the different on-farm pilots.

Summary

The FERTIMANURE concept integrates a set of innovative treatment schemes to efficiently valorise animal manure and to obtain fertilising products with high added value. FERTIMANURE pursues the improvement of several technologies that are either currently under development or that have been successfully used for similar applications and proposes innovative integrated solutions to finally reach a zero-waste manure management approach. In work package (WP) 2 (*Nutrient recovery from animal manure*), five different on-farm pilots were implemented during the first year of the project, and they will be operated and optimised from M13 to M48. WP2 activities are implemented by partners having different profiles: scientific partners (UVIC, LEITAT, WENR, RITTMO, UGENT and FHR), farmers (APF, CPV, APCA), public bodies (DARP, APCA) and technology providers (DORSET).

During the demonstration period, the pilots were closely monitored for operational performances at the level of processes, as well as for the stability and quality of raw products/fertilizers produced, getting both mass and energy balance. These balances provide a deeper understanding on the overall system (Task 2.3 - Mass and energy flows and balances of the demonstration on-farm pilots). Data collected in this FERTIMANURE task coupled with those coming from constructors and partners involved in this project, will be useful to update and optimize processes by a continuous feedback approach, to make them sustainable from both the economic and environmental point of view. Doing so, complete mass and energy flows and balances has been performed to frame a picture as far as possible close to reality. At the same time, environmental data was recovered according to a list delivered by WP5.

Deliverable 2.6 (Mass and energy balance of the on-farm pilots to WP5) reports final mass and energy flow balances of the different five on-farm of FERTIMANURE pilots, therefore updating and finalising the D2.3.

Content

List of Tables

List of Figures

List of Abbreviations

BBF	Bio-based Fertiliser
TMF	Tailor-made Fertiliser
ALGE	ALGAENERGY
APF	Arjan Prinsen Farm
CPV	Cooperativa Plana de Vic
D	Deliverable
DARP	Catalan Department of Agriculture
DM	Dry matter
DORSET	Dorset Group
FHR	Fraunhofer UMSICHT
LEITAT	LEITAT Technological Center
LF	Liquid fraction
MAP	Mono-ammonium phosphate
MC	Membrane contactor
MF	Microfiltration
OM	Organic matter
RITTMO	RITTMO Agroenvironment
RO	Reverse osmosis
S/L	Solid/liquid
SF	Solid fraction
TCR	Thermo-catalytic reforming
TK	Total K
TN	Total N
TP	Total P
UGENT	Ghent University
UMIL	University of Milano
UVIC	University of Vic – Central University of Catalonia
VS	Volatile solids
WENR	Wageningen Environmental Research
WP	Work package

1. Introduction

World's population continues to rise expecting a global population increase above 10 billion inhabitants by 2050 (United Nations, 2020). As a result, there is a need to increase food production to meet both the world's expanding population and the Sustainable Development Goals, i.e., Zero Hunger, which was defined by the United Nations in 2015 (United Nations, 2020). Since agriculture is a primary source of food, improving crop yields is a key concern. Agriculture productivity has mostly been improved via the use of fertilizers in recent decades, and global demand for nitrogen, phosphate, and potassium for fertiliser usage was predicted to grow by nearly 10% from 2016 to 2022 (FAO, 2020). Food production and fertilizers use raise major concerns in terms of their production, use and environmental impacts. Among the most used synthetic fertilizers, N and P are obtained from non-renewable resources that use high-cost methods (Cherkasov et al., 2015; Günther et al., 2018). In addition, environmental concerns associated with fertilizers use are well documented (e.g., eutrophication, gaseous emissions) (Khan et al., 2014). Bio-based fertilizers (BBFs) have improved the sustainability of agriculture by reducing the use of non-renewable resources and the impacts of agriculture on the environment (Wang et al., 2018). Animal manure might provide a sustainable supply of BBFs, which would be low-cost and ecologically beneficial.

In this context, the activities of the FERTIMANURE project are of particular relevance in order to extend the knowledge in the field of innovative recovery of nutrients and production of high-added value fertilizers from animal manure. Indeed, results of WP1 (*FERTIMANURE framework*) showed that animal manure may represent a valuable source of nutrients and that the transformation of animal manure in concentrated BBFs is mandatory to maximise their benefits. To this aim, in WP2 (*Nutrient recovery from animal manure*), five different pilots were implemented to test innovative technologies for nutrients recovery from animal manure (Table 1, Figure 1).

Table *1*. The five pilot plants of FERTIMANURE

On-farm pilot	Integrated Technologies	Main feedstock	Bio-based fertilisers obtained
Spain	Membrane contractors Freeze concentration. Micro-algae reactor Thermo-enzymatic reactor Biodrying + thermal treatment	Pig slurry and poultry manure	Nutrient-rich concentrate (ES-NC), Biodried solid fraction (ES-DSC), Phosphorous (ashes or phosphoric acid) (ES-PA), Ammonium sulphate (ES-AS), AA-based biostimulants (ES-AA)
Netherlands	Biological acidification Phosphorus precipitation Nitrogen stripping Acid scrubbing	Liquid cattle slurry	Ammonium sulphate solution (NL-AS), Liquid potassium fertilizer (NL-LK), Organic soil conditioner (NL-SC), Wet organic phosphorus rich fertilizer (NL-WP), Dried organic P-rich fertilizer (NL-DP)
Germany	Thermo-catalytic reforming Selective NH_3-removal reaction	Solid cattle manure	Biochar (DE-BC), Ammonium phosphate on perlite (DE-AP)
Belgium	N-stripping Acid scrubbing Vacuum dryer + condenser	Pig slurry	Ammonium nitrate (BE-AN), Ammonium sulphate (BE-AS), Ammonium water (BE-AW)
France	Mobile pyrolysis Mobile N-stripping	All type of manure (pig, cattle, poultry)	Biochar (FR-BC), Ammonium sulphate (FR-AS), Liquid K-fertilizer (FR-LK)

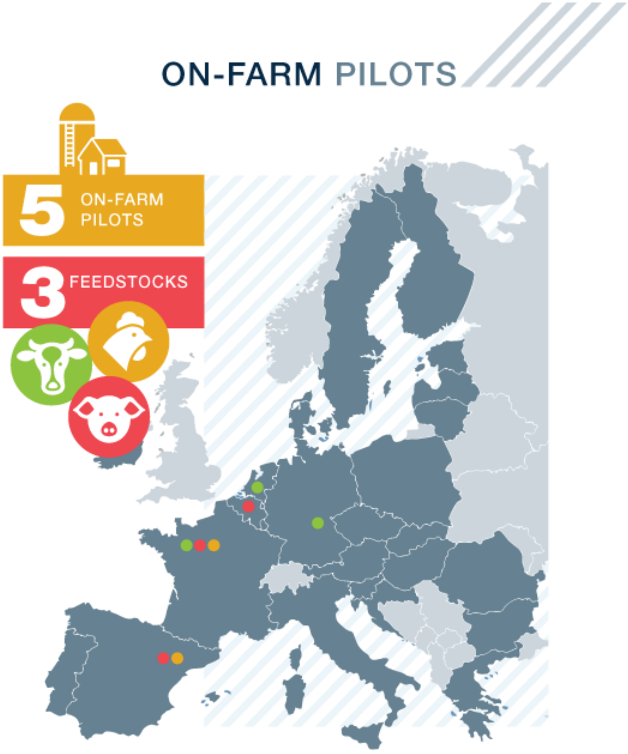


Figure 1. Location of on-farm pilots in the FERTIMANURE project

Pilots’ implementation and demonstration, as well as first data of BBFs characterization and the results have been previously reported in Deliverables D2.1, D2.2, D2.3 and D2.4.

The aim of Task 2.3 (Mass and energy flows and balances of the demonstration on-farm pilots) is to monitor both operational performances of proposed processes, and the stability and quality of raw products/fertilizers produced. Indeed, mass and energy balance provide a deeper understanding of the entire processes. Moreover, mass and energy balances help to identify the bottleneck of a process at-scale, which is uncommon in laboratory works and literature (Gowd et al., 2022). In addition, the comparison of the different processes, proposed for nutrient recovery and biofertilizers production, by using mass and energy balance, helps to find the most feasible process from a resource recovery perspective, i.e., mass and energy recovery (Kehrein et al., 2020).

Data collected in this FERTIMANURE task, coupled with those coming from constructor and partners involved in this project, were useful to update and optimize processes by a continuous feedback approach and to achieve sustainability from both the economic and environmental point of view. Complete mass and energy flows and balances were therefore performed leading to the creation of a picture as far as possible close to reality. At the same time, environmental data were recovered according to WP5.

This deliverable reports final mass and energy balances across all the M30-M48 of the five FERTIMANUREpilots.

2. FERTIMANURE on-farm pilots: brief description

A short description of the FERTIMANURE on-farm pilots and their BBFs is reported below in order to better understand mass and energy balances. For a complete description of pilots and BBFs please refer to Deliverables D2.1, D2.2 and D2.4.

2.1. Spanish pilot

The Spanish pilot combines two separate treatment trains either for the treatment of solid or liquid streams derived from raw pig slurry (Figure 2). As a first pre-treatment step, a solid/liquid separation unit is installed to obtain different flows from the raw material. From this unit, a solid fraction is obtained and valorised either as organic amendment or as phosphorous while the liquid fraction is valorised as nutrient-rich concentrate and/or biostimulant. Reclaimed water and ammonium sulphate/nitrate solution are also generated as high added value by-products.

The solid fraction of pig slurry feeds the biodrying reactor or trench to remove part of the moisture contained in the stream. A dried solid fraction (ES-DSC) leaves the biodrying trench to be applied as an organic amendment to the soil or to be used as a biofuel in a biomass boiler. After the combustion of the biodried solid fraction in the boiler, phosphorous-rich ashes (ES-PA) are generated and valorised to produce phosphoric acid.

The liquid fraction of pig slurry is firstly treated through three subsequent membrane systems which are successively fed with the permeate of the previous system. Membrane contactor is supplied with the permeate of the previous membrane filtration system. As a result, it produces ammonium sulphate (ES-AS) solution as product. The last filtration is a reverse osmosis. The retentates or waste by-products from membrane filtration and reverse osmosis are further concentrated through freeze concentration technology. The crystalliser produces ice crystals, which are then washed and separated to obtain reclaimed water and nutrient-rich concentrate (ES-NC) as a product. Finally, the permeate received from the membrane systems is used to cultivate microalgae, which are then dewatered and enzymatically hydrolysed to obtain aminoacid- based biostimulants (ES-AA) as product.

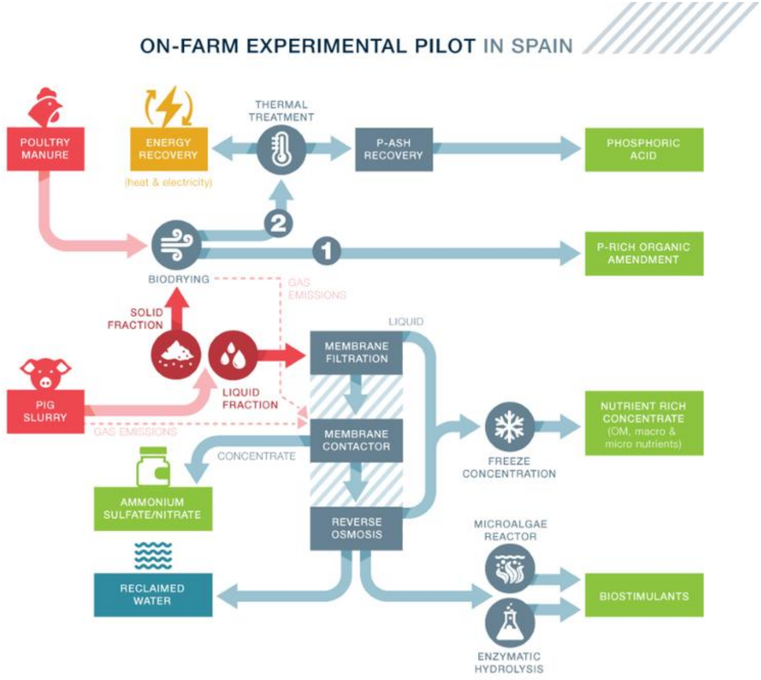


Figure 2. Spanish pilot infographic

2.2. Dutch pilot

The process flow diagram of the Dutch pilot plant Arjan Prinsen Farm (APF) is shown in Figure 3. Cattle slurry produced in the stable is collected in a continuously mixed cellar with a storage capacity of about 400 m^3.

At APF a digester is part of the manure processing approach in order to valorise cattle slurry into valuable products. The anaerobic digester is fed from this cellar of about 500 m3 by pumps every 140 minutes. The digester has a total capacity of about 800 m^3, but the effective (used) capacity is about 650 m^3. The digestion operates at a temperature of about 35 – 37 ^oC (mesophilic digestion). The residence time varies between 40 and 98 days during the year. The residence time is low in the spring, when large amounts of digestate are required for the fertilisation of farmer's land, while in the autumn the residence time is rather long.

The added co-substrates are solid farmyard manure and feed residues like hay, maize, remaining feed (and beet tips only in 2020). These products are mixed in a separate biological acidification tank together with a part of the digestate, and at a later stage with a part of the separated liquid fraction. The acidified mixture (after fermentation) is pumped to the anaerobic digester in order to increase the biogas production due the easily decomposable material of co-products.

Table 2 shows the amounts of different type of biomass used as feedstock for the digester. In 2021 APF became part of dairy cooperative Friesland Campina's program called "Jumpstart" which demands that only farm's own biomass residues may be used as feedstock for the digester. From that time onward no sugar beet tips from other farms were used anymore, and the total biogas production (± 55% CH_4) dropped down from 27.1 Nm^3/tonne to 21.3 Nm^3/tonne. The total amount of produced biogas was on average 42,863 Nm^3/year, ranging from 54,318 in 2020 to 30,663 in 2022, which equals to respectively about 70 and 39 tonnes biogas per year. This is the amount of mass of feedstock that is transferred to the air as biogas, with reduction of the mass of digestate compared to mass of feedstock.

Table 2. Annual feedstocks of the anaerobic digester in the years 2020-2022 at the Dutch pilot Arjan Prinsen Farm (APF) in tonnes fresh matter per year.

Feedstock types	**2020**	**2021**	**2022**	**Average 2020-2022**
Dairy slurry	1,828	1,993	1,450	1,757
Farmyard manure	25	47	20	31
Sugar beet tips	122	-	-	122
Feed residues (maize, straw, etc.)	30	10	5	15
Total biomass	**2,005**	**2,050**	**1,475**	**1,843**

The produced digestate flows into a buffer tank with a storage capacity of 0.2 m^3. From there it is fed to a screw press with a 500 µm pore size filter to separate the digestate in a solid and liquid fraction. The solid fraction is rich in fibres and is therefore used as an organic soil conditioner (NL-SC), which is mainly sold to customers (hobby gardeners) but can also be sold to farmers in the region. The liquid fraction of digestate, which contain still large amounts of fine particles, is pumped to a settling tank, with an effective volume of 17 m^3, to remove part of the remaining particles before it is treated in the stripper. The settling tank operates in batches and alkaline material can be added to raise the pH in order to facilitate the precipitation and settling of organic phosphorus (P) rich fertiliser (NL-WP) to be collected at the bottom of its conus. This P rich sludge contains mainly P as calciumphosphate or struvite and drying of the product would be an option for better characteristics as fertiliser. The remaining liquid fraction flows to the stripper that operates at 35-40 ^oC in batches of 1 m3 for six hours, where ammonia in the liquid fraction is stripped to the gas phase and subsequently absorbed in the attached scrubber containing (96%) sulphuric acid solution. Consequently, a concentrated ammonium sulphate solution (NL-AS) and a liquid potassium (K) fertiliser (NL-LK) are produced. In fact, the NL-LK solution also contains substantial amounts of N and P and meso and micronutrients, because it is a N-stripped solution of the separated liquid fraction of the digestate. Furthermore, at APF not all the ammonia has to be stripped since products are used on own farm to optimise the fertilisation plan, and consequently the liquid potassium fertiliser still contains nitrogen.

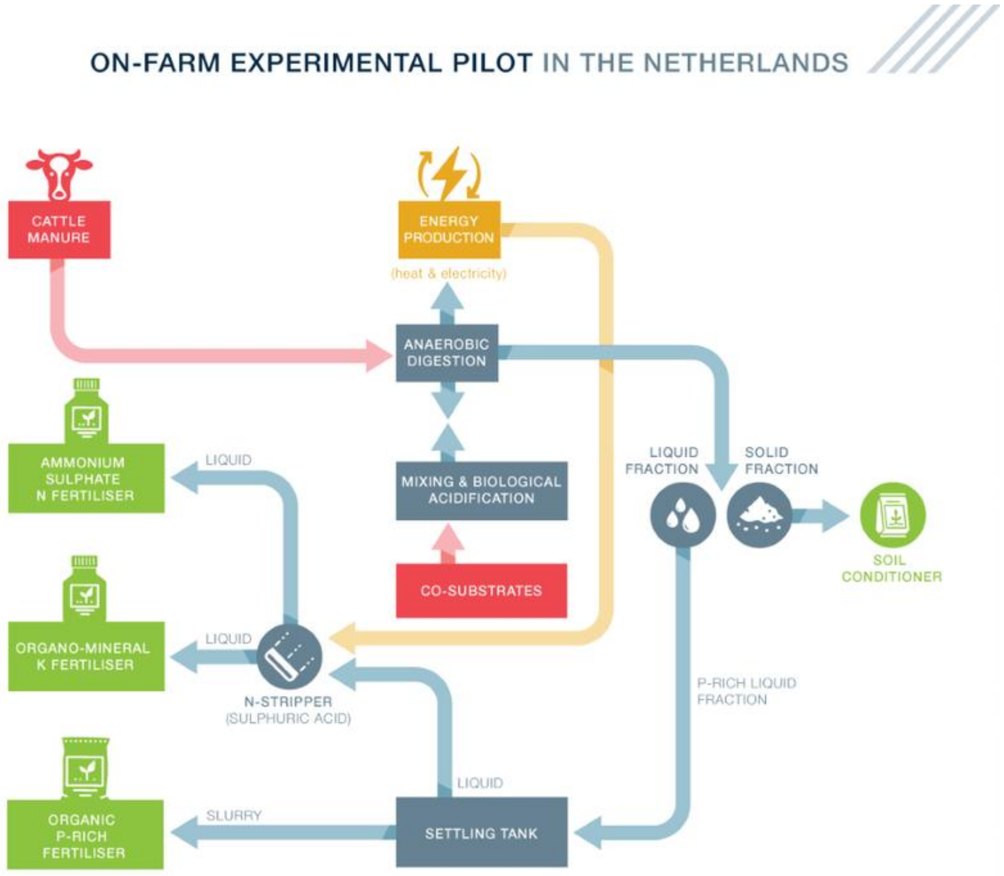


Figure 3. Dutch pilot infographic

2.3. German pilot

Figure 4 schematically reports the pilot plant process for BBF production from cattle dung in the German pilot. The process can be divided in three main steps: cattle dung pre-treatment, thermochemical conversion and mono-ammonium phosphate production.

The innovative key components are the (i) application of additives for binding NH_3 in the solid dung, (ii) the Thermo-Catalytic Reforming (TCR) unit for the conversion of the cattle dung, (iii) the mono-ammonium phosphate reactor.

The targeted BBFs of the pilot plant are:

1. Phosphorous rich biochar (DE-BC) produced from Thermo-Catalytic Reforming of dried cattle dung.
2. Mono-ammonium phosphate on perlite (DE-AP) obtained from the MAP reactor.

After all the preliminary work done in the German pilot, it has been agreed that ammonium sulphate solution from the German pilot will not be further considered as part of the BBFs produced in the FERTIMANURE project. Detailed justification to this decision is reported in Section 3.3. Consequently, the FERTIMANURE BBF list and pilot infographic, will be adapted and changed accordingly in the following deliverables and in the project promotional material.

After on-site mixing of cattle manure with citric acid the mixture is dried up to a water content of less than 20 % and pelletized afterwards. The pellets are processed within the German pilot plant TCR30 which processes 30 kg of Feedstock per hour. The originally designed MAP reactor was based on a packed bed reactor filled with perlite which was impregnated with phosphoric acid including active charcoal filters to prevent side reactions. During trials with the fully commissioned system, it became evident that due to side reactions and adsorption effects the desired product monoammonium phosphate on perlite could not be obtained. Tests regarding the recovery of ammonia from TCR-gas were performed on a smaller system with 2 kg/h of throughput to stay flexible and be cost effective. Mass balances regarding the nitrogen flow were adapted using the results from these optimized trials where the desired product DE-AP could be recovered through a scrubbing process followed by crystallization.

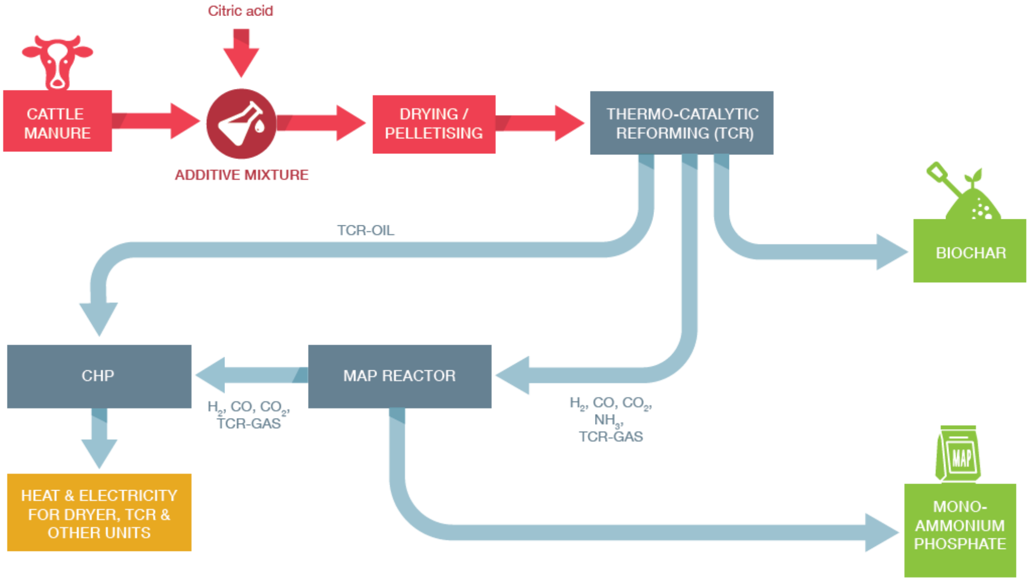


Figure 4. German pilot infographic

2.4. Belgian pilot

From the beginning of 2022, it was decided to switch to a new stripping-scrubbing pilot plant because the initial pilot plant considered in FERTIMANURE was outdated and encountered many operational issues. The initial pilot (located at IVACO pig farm in Gistel) was built in October 2015 by a constructor (DETRICON) without experience in the field of nutrient recovery technologies. Because of the lack of experience, this installation had many construction errors, and the pilot was often not operational and a continuous and stable monitoring was thus impossible. Also, the pilot had a very high electricity consumption (e.g., heating and pumping) because of the poor isolation and over dimensioned pumps. Furthermore, the nutrient recovery rates were limited because it was not feasible to reach high temperatures (max. 30°C) in the stripping tank which is the main driving force for the mass transfer rate of ammonia from liquid to air phase.

Due to the fast deterioration of the machinery in combination with the unsatisfying operational efficiency, DETRICON and IVACO decided to halt the investments in the initial pilot installation which eventually led to its retirement (end of 2021). Therefore, University of Ghent (UGENT), together with DETRICON, have been looking for an alternative installation which uses a similar technological approach. The new Belgian pilot, which is now located in the pig farm Bio Sterco in Hooglede, is an ammonia (NH_3) stripping -scrubbing unit and is one of the several manure treatments steps that are implemented at the Bio Sterco (Figure 5). Adjustments to the pilot infographic following the change of installation are expected to be available in the next months and surely for the end of the Reporting Period. The ammonia stripping-scrubbing unit receives the nitrogen (N)-rich liquid fraction (LF) of manure after mechanical separation of raw manure and strips a part of the ammonia enclosed in the LF by an air ventilation flow of 1000–1800 m^3 h^{-1} with an air speed of 0.2-0.8 m s^{-1}. By increasing pH and temperature of the LF of manure, NH_3 volatilization is induced. During the monitoring period, the stripping unit was operated at a temperature of 51.2 ± 1.1 °C and a pH of 8.4 ± 0.2. The stripped NH_3 is sent to a scrubber column where nitric or sulphuric acid are added as a sorbent, resulting in the production of ammonium nitrate or ammonium sulphate, respectively. As both counter acids were tested as sorbent during the monitoring period, two mass balances can be differentiated according to the used counter acid (Section 3.4).

A comparison between the two pilots (IVACO in Gistel and Bio Sterco in Hooglede) is available in the Section 3.4 of this deliverable.

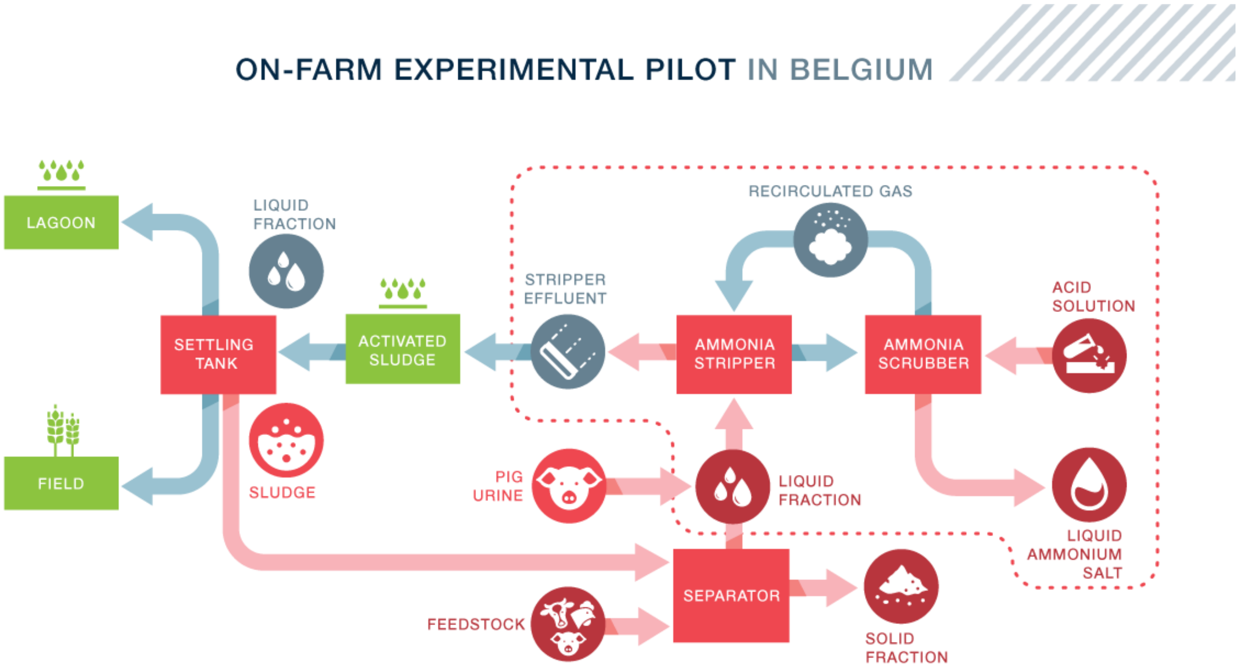


Figure 5. Belgian pilot infographic

2.5. French pilot

The French consists of mobile pilots dedicated to experiment processes on different sites with different inputs (Figure 6). The pilots focus on technologies (pyrolysis and N-stripping) that are able to produce secondary products from pig/cattle/poultry manures. The main technology innovations are (i) that French pilots are adapted to various substrates (ii) pilots are mobile and move on different French farms. The objective is to treat manure sources directly on their production sites, with the possibility of using bio-based fertilisers (BBFs) on the same site. To use the equipment on site, the pilots are able to be loaded in two trucks.

The priority tested substrates are:

- Liquid pig slurry which are pressed (phase separation) for nitrogen stripping.
- Poultry manure that will be turned into biochar due to a slow pyrolysis process.

The nitrogen from liquid phase is recovered by a stripping process, producing ammonium sulphate (FR-AS). Potassium from the liquid phase is also recovered from the K-containing sludge residue (FR-LK) after the striping process. The solid phase could be utilized in the anaerobic digestion process and/or the pyrolysis process and/or the composting process.

The pyrolysis unit converts solid manure at a temperature between 550°C and 700°C and under anaerobic conditions produced due to an N_2 flow. Final products are biochar (FR-BC) and pyrolysis gases. Gases are burned in a flare and biochar is collected to evaluate their agronomic properties.

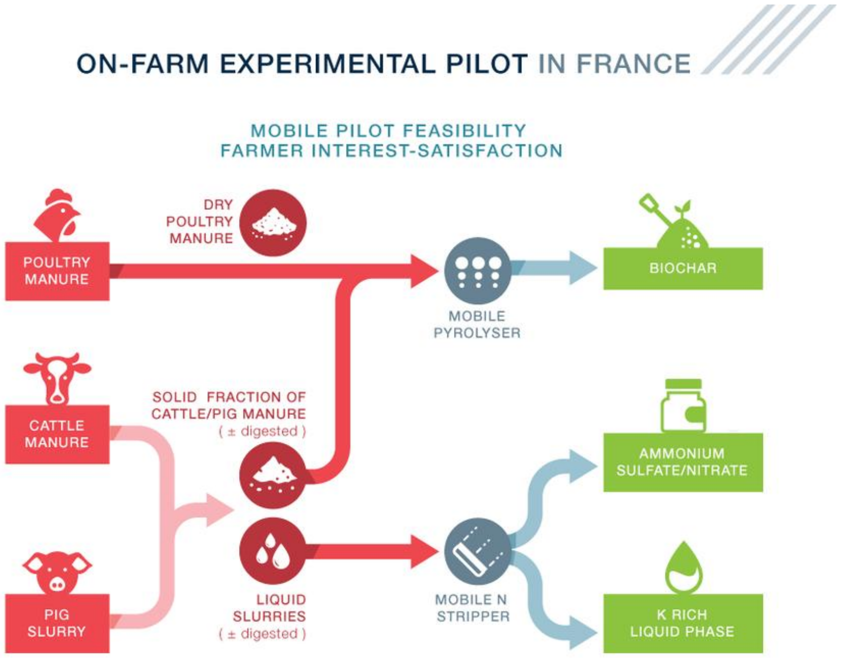


Figure 6. French pilot infographic

In the following paragraphs, detailed mass and nutrient balances are illustrated for each on-farm pilots.

3. Mass and energy balance of the on-farm pilots

3.1. Spanish pilot

3.1.1. Spanish pilot: mass and nutrients balance

The separation unit recovers, in average, 91.1% of the inlet slurry mass as liquid fraction, which contains a major part of the slurry nutrients. For instance, 88%, 91.7% and 91.4% of the inlet nitrogen, phosphorus and potassium, respectively, are recovered in the liquid fraction. It is remarkable that the high variability in the characteristics of pig slurry is reflected mainly in the highly variable characteristics of liquid fraction. Additionally, according to the mass balance of liquid fraction in membranes system coupled with freeze concentration (shown in Annex Table A2), between 4 and 46% of variation was found in the nutrient mass balances, being particularly significant the variability of phosphorus distribution. This variability can be attributed to the seasonality and the age of slurry and therefore separation efficiency, characteristics of LF being highly affected by that. First, raw slurry highly varied along the year probably due to the animal habit to drinking water depending on the season. During hot season, pigs tend to drink more and therefore slurry is more liquid, leading to a better and significantly faster separation performance. Also, along the storage time in the basin, slurry is continuously degraded leading to an overall mineralisation of C and N, modifying the distribution of nitrogen species and partly leading to N most via volatilization.

Related to all the mentioned, the high variability in the characteristics of liquid fraction affects greatly to the operation of membrane units, while solid fraction produced shows more stable characteristics.

Table 3 shows the summary of the general mass and nutrient balances in the main treatment scheme (MF and RO retentates are concentrated via freeze concentration and solid fraction is valorised as organic amendment). Additionally, the mass (Figure 7), nutrient (Figure 8) and Carbon (Figure 9) balances are visually shown.

Table 3. Summary of mass and nutrients balances in the Spanish pilot (starting material = 100; n = number of observations)

	Mass (kg)	C (kg)	N (kg)	P (kg)	K (kg)
INPUTS					
Pig slurry	100.0 ± 20.9 (N=7)	2.87±0.87 (N=7)	0.45±0.04 (N=7)	0.10±0.03 (N=7)	0.23±0.06 (N=7)
Sulphuric Acid	0.15 ± 0.04 (n=4)	-	-	-	-
Sodium Hydroxide	0.53±0.05 (n=4)	-	-	-	-
Inoculum	13.44 (N=1)		0.017 (N=1)	0.002 (N=1)	0.022 (N=1)
CO_2 supply for microalgae culture		0.38 (N=1)			
Total	115.17 ± 4.2	3.25	0.46	0.10	0.26
OUTPUTS					
Ammonium Sulphate	1.25±0.02 (n=4)	-	0.07 ± 0.1 (n=4)	-	0.06± 0.2 (n=2)
Nutrient rich concentrate	37.6 ± 15.1 (n=4)	0.52±0.22 (n=4)	0.14 ± 0.06(n=4)	0.022±0.010(n=4)	0.083±0.042 (n=4)
Melted ice	32.1±11.4(n=4)	0.25±0.10 (n=4)	0.070±0.027 (n=4)	0.012±0.006 (n=4)	0.041±0.024(n=4)
Biostimulant	0.89±0.003 (n=2)	0.022±0.000 (n=2)	0.0004±0.000 (n=2)	0.001±0.000 (n=2)	0.001±0.000 (n=2)
Residual biomass from algae	0.102±0.01 (n=1)	0.003±0.001 (n=1)	0.0011±0.001 (n=1)	0.002±0.000 (n=1)	0.001±0.000 (n=1)
Permeate excess from microalgae	40.7±1.7(n=2)	0.000	0.000	0.000	0.000
Biodried organic amendment	3.82±2.0 (n=5)	0.93±0.59 (n=5)	0.04±0.03 (n=5)	0.010±0.006 (n=5)	0.019±0.012 (n=5)
Biodegradation/ volatilization BD	4.11±1.58 (n=5)	0.32±0.14 (n=5)	0.014±0.008 (n=5)	-	-
Total	120.6±4.2	2.07±0.18	0.34±0.03	0.047±0.01	0.206±0.06
Balance (% initial mass)	+5.64	-36.18	-26.47	-53.99	-19.83

Due to the mentioned variability, a huge variability was observed in all outputs of membrane technologies unit (Microfiltration (MF) retentate, Reverse osmosis (RO) permeate and Ammonium Sulphate), although they also reflected the process optimization performed during operation. Membrane filtration and reverse osmosis retentates are subsequently concentrated via freeze concentration and therefore, nutrient-rich concentrate and melted ice also report quite high variability in their characteristics.

Microfiltration retentate's quality reported the highest variability in the total phosphorus content as depending on the metal content of the pig slurry, because phosphorus can be found as free phosphate (which trespasses the microfiltration membrane) or as metallic complex (which remains in the microfiltration retentate). Microfiltration retentate retains a major part of the nutrients to be recovered in the biorefinery, namely 23% of the inlet carbon as slurry, 27% of the nitrogen, 40% of the phosphorus and 51% of the potassium are retained in the microfiltration retentate. Apart from the nutrients, this flow also retains a major part of zinc and copper. Considering the aim of a zero-waste approach in the biorefinery, microfiltration retentate is aimed to be valorised through freeze concentration, however, the zinc and copper concentrations might be a constrain when producing a regulation compliant fertilising product.

The high variability mentioned as well as the process optimization assessed led to a high variability in the nitrogen content and the obtained product mass in ammonium sulphate solution obtained in the membrane contactor (MC) unit. During the first runs of the pilot, it was observed that the obtained BFF contained low concentration of ammonium. After optimization, an important increase in nitrogen concentration in the ammonium sulphate was achieved (up to 44 g TN/kg in the most optimized case) by reducing the acid volume used in the membrane contactor (up to 8 L of stripping acid per cubic meter of liquid fraction treated) as well as the sodium hydroxide used to force the volatilization of ammonia (5.8kg of NaOH per cubic meter of liquid fraction treated). In this regard, according to the mass balance performed, 15% of the inlet nitrogen is being recovered as ammonium sulphate in the membrane contactor. The percentage of the recovered nitrogen is equal to the previous stage, but optimization allowed the enhanced performance of the contactor by reducing the inlet of chemicals, therefore obtaining a more concentrated product.

Finally, Reverse Osmosis permeate quality variability, especially in terms of Total Carbon (TC) is directly related to microfiltration permeate quality as reverse osmosis systems are designed for retaining up to 90% of total COD. Thus, the microfiltration cutoff modification showed a significant impact in reverse osmosis permeate quality as higher loaded influents reports higher TC in reverse osmosis permeate.

Different combinations of membrane units were tried so different characteristics on the retentate of reverse osmosis would lead to nutrient rich concentrates of different characteristics. These combinations assessed do not affect the satisfactory characteristics of the permeate which was subsequently used for microalgae cultivation.

Considering the combination of the three membrane systems (MF, MC, and RO), the nutrient rich concentrate obtained contains almost 36% of the inlet N in a 38% of inlet mass. Regarding phosphorus, almost 24% of the inlet phosphorus is recovered in the nutrient rich concentrate, probably because the retentate of MF retains a major part of the phosphorus and it is then diluted when the RO retentate is added into the freeze concentration unit. Finally, almost 39% of the inlet K is recovered in the nutrient rich concentrate. In this regard, it is worth mentioning that from performance point of view, the combination of both retentates valorised through freeze concentration was the combination obtaining the best yields. Although MF retentate alone could obtain a more attractive product richer in nutrients, results obtained during optimization of freeze concentration (results shown in the section devoted to optimization of pilot) showed that the nutrient distribution was roughly equal between concentrate and melted ice. Therefore, co-valorisation of both retentates is regarded as the best configuration, reaching also the aimed zero-waste approach. Finally, although nutrient recovery efficiencies are quite high in freeze concentration (up to 60.5% of N, 55% of P and 63.8% of K of the retentates treated is recovered in the nutrient rich concentrate), still the water content of nutrient rich concentrate is too high (volume reduction was in average only of 48.4% in average). Therefore, alternative concentration technologies enabling better mass efficiency would be needed in this case, vacuum evaporation for instance. However, this option would probably lead to a higher energy demand and volume reduction capacity even with this kind of technology is limited. Moreover, volatilization of some of the elements of interest and uncontrolled change in the chemical composition of the concentrate might also occur, which is avoided with freeze concentration. The other outlet flow from freeze concentration unit is the melted ice consisting of 43.9% of the inlet mass. It still retains some nitrogen (31%), phosphorus (21%) and potassium (32%).

Biostimulant (liquid hydrolysate with 5-6% of total solids) is produced by enzymatic hydrolysis of the microalgae cultivated on permeate (from RO). Optimisation and lab scale tests of enzymatic hydrolysis (10L) were assessed using the actual microalgae biomass cultivated on RO permeate. However, due to the required volume of microalgae biomass, the testing of the pilot reactor was performed using commercial biomass.

The hydrolytic unit has a total capacity of about 100 L, but the used capacity is about 65 L. The main input flow is the microalgae (Scenedesmus obliquus) which is enzymatically hydrolysate into protein hydrolysate (biostimulant). In this process a residual biomass from algae (residual solid fraction) is also produced.

As mentioned, the pilot hydrolytic unit has been operated with commercial Scenedesmus sp biomass due to the impossibility to achieve the required Scenedesmus paste cultivated over RO permeate to operate it. However, to make mass balance and nutrient flows more representative for the actaul biorefinery, balances are calculated considering the average data of biostimulants produced during pilot optimisation phase (10L reactor, see D2.5), where Scenedesmus paste is the main input. Data from hydrolytic unit operation is also considered to make comparison among them.

When microalgae paste is used as raw material, over 50% of the inlet microalgae paste is recovered as biostimulant (protein hydrolysate). So, considering that from 100Kg of pig slurry, 0,2kg of *Scenedesmus* paste is produced, from 100 kg of pig slurry, 0.09 kg of biostimulant (5-6% total solids) is recovered. During the process a residual solid fraction is also obtained, representing 50% (50-60% total solids) of the inlet microalgae paste. Low part of the total solids (18%) is recovered in biostimulant whereas the 82% of total solids remains in the residual solid fraction. The nutrient efficiencies were calculated for all measured nutrients from microalgae that are distributed between biostimulant and residual solid fraction. 30 % of the inlet nitrogen is recovered in this biostimulant. 70% of nitrogen remains in the solid fraction. In addition, 48% of the inlet K as well as 33% of the inlet P are recovered in the biostimulant whereas only 6% of the inlet C is recovered in the biostimulant. Residual solid fraction is still rich in nutrients with the 52%, 67% and 94% of the inlet K, P and

C, respectively. It is important to headline that this nutrient recovery results are in line with literature (see below). The presence of robust cell wall in microalgae directly affect nutrient solubilisation yield.

Differences are observed when lyophilised commercial biomass of Scenedesmus sp is used. In this case, lyophilised microalgae have been diluted to 10% w/w solution to simulate Scenedesmus sp paste. 73% of this inlet biomass solution is recovered as biostimulant (6% total solids). It can be observed that the recovery of liquid biostimulant increases 20% in comparison with process with Scenedesmus sp paste. Using lyophilised microalgae could contribute to enhance cell wall disruption and, in consequence, improve nutrient recovery in biostimulant. Using commercial lyophilised biomass, the hydrolytic unit recovers 38% of the inlet total solids in the biostimulant. In this case, the recovery of the inlet total solid increase 20% in comparison with process whit Scenedesmus paste. In addition, the inlet nitrogen recovering in the biostimulant increase to 70%. Carbon, phosphorus, and potassium balance was not calculated in this case.

For 100 kg of slurry treated, 2.3 kg of organic amendment are produced from the valorisation of the solid fraction through biodrying technology (Table 3). The product also contains the major part of the inlet carbon in slurry (39%) while it contains also a considerable part of the inlet nitrogen (11%), phosphorus (12%) and potassium (9.6%). During this biological process, part of the carbon is biodegraded (21%) while part of the nitrogen is lost through volatilization (3% of initial nitrogen in slurry or 27.7% of initial nitrogen in solid fraction). However, biodegradation also leads to a concentration effect of other macronutrients and micronutrients. For instance, compared to their content in the solid fraction, phosphorus, potassium, zinc and copper increased between 2.1- and 2.8- fold. It is remarkable also that, as average, 61.2 of the moisture content in the initial solid fraction is removed in 14 days via evaporation mainly by biological action.

One of the options for the biodried product obtained is its use as an organic amendment. However, since it still contains a considerable content of organic carbon, its calorific value might be high enough to be used as a non-conventional biomass fuel in a biomass boiler. In fact, in average 18MJ/kg DM were determined for the organic fraction being translated in a lower heating value of between 6 and 8 MJ/kg. Several combustion trials of biodried products were performed after its pelletisation. 4% of the pelletised biodried fraction is recovered as ash. Flue gases were monitored during the combustion process of the biodried product (N=1). According to combustion results, 33% of the N and 96% of the S in the biodried pellets are partially oxidized into NOx and SO2 emissions. These values were significantly higher than the values obtained for wooden pellets in combustion (2.8- and 24-fold higher NOx and SO2 emissions in biodried pellets vs. wooden pellets). The ash contains high amounts of nutrients, being particularly interesting phosphorus and potassium. According to the average values calculated in the mass balances, ash retains 12.8% and 8.4% of the inlet phosphorus and potassium respectively. However, it should be considered also that combustion process also concentrates the unwanted zinc and copper elements in the ash.

As a further step, combustion ashes were used to produce phosphoric acid, a fertilising product with very high value in the market. Combustion ashes were pre-treated with water according to literature and preliminary trials demonstrated to lead to better phosphorus extraction afterwards. In the upscaled trials, only 22% of phosphorus and 14% of potassium was extracted with an ash to acid (H_2SO_4 1.2M) ratio of 1 to 5. First, ash to acid contact time should be optimized to maximize extraction and second, ash to acid ratio should be also adjusted as 1 to 5 ratio led to a partial gelation of the supernatant after extraction, probably leading to not appropriate analysis. Table 4 shows the mass and nutrient balances of the mentioned strategy for the organic amendment in which it is combusted and then phosphorus is recovered from the ashes. The streams monitored from the exhaust gases in combustion are also included in the balance.

Table 4. Summary of mass and nutrients balances of the downstream in biomass boiler and acidic extraction of biodried product in the Spanish pilot (starting material = 100; n = number of observations).

	Mass (kg)	C (kg)	N (kg)	P (kg)	K (kg)
INPUTS					
Biodried organic amendment	2.34(n=5)	1.12(n=5)	0.051(n=5)	0.012(n=5)	0.023(n=5)
Water	0.933	-	-	-	-
Sulphuric Acid (1.2M)	0.47	-	-	-	-
Total	3.74	1.12	0.05	0.012	0.023
OUTPUTS					
Flue gases in combustion	2.25	-	0.009	-	-
Supernatant (recycled water)	0.93	-	-	0.00001	0,00311
Exhaust ash	0.085	-	-	0.0028	0.0017
Phosphoric acid	0.140	-	-	0.0016	0.0011
Total	1.16	-	0.009	0.004	0.006
Balance (% initial mass)	-69.03	-	-82.05	-63.91	-73.96

In the case of the biodrying process of poultry manure, 2 trials were performed. The overall mass and nutrient balances of this case are shown in Table 5. Poultry manure is presented in solid form and therefore, its content in nutrients and carbon per kilogram of manure is higher than in pig slurry or even in the solid fraction of pig slurry (229%, 168% and 400% higher nitrogen, phosphorus and potassium in poultry manure when compared to the SF of pig slurry). When biodrying is applied to poultry manure, over 55% of the initial mass is recovered as organic amendment, which is rich in nitrogen and potassium. C conservation during biodrying process is achieved as only 12.6% of the volatile solids are degraded along the process. According to the mass balances only 14% of the carbon is lost while 67.4% of the initial moisture content is removed in the process.

Only 12.4% of the nitrogen is lost via volatilization even though the C/N was rather low (approximately C/N of 18 vs. the optimal 20-25 for aerobic processes) that could lead to important N loses as ammonia emissions. Although the nitrogen content is higher and the C/N lower when treating poultry manure, nitrogen losses are more relevant when biodrying is performed with the solid fraction of pig slurry. This fact can be related to the nitrogen species in each of the matrixes, as almost 53% of the nitrogen in the solid fraction of pig slurry is present in form of ammonium nitrogen which can be easily lost as ammonia whereas it is around 20% in the case of poultry manure.

Table *5*. Summary of mass and nutrients balances of the biodrying process of poultry manure in the Spanish pilot (starting material = 100; n = number of observations).

	Mass (kg)	C (kg)	N (kg)	P (kg)	K (kg)
INPUTS					
Poultry manure	100 (n=2)	20.05 ± 1.98	1.45 ± 0.04	0.2±0.1	0.89±0.19
Total	100	20.05 ± 1.98	1.45 ± 0.04	0.2±0.1	0.89±0.19
OUTPUTS					
Biodried organic amendment	55.3±6.6	17.2±2.1	1.22±0.15	0.2±0.0	0.89±0.11
Biodegradation/volatilization BD	45.4± 4.2	2.76±0.27	0.2±0.0	-	-
Total	100.7±5.4	20±1	1.40±0.08	0.20±0.02	0.89±0.11
Balance	100.74±5	99.7±5.8	96.5±5.2	97.3±5.8	100±6

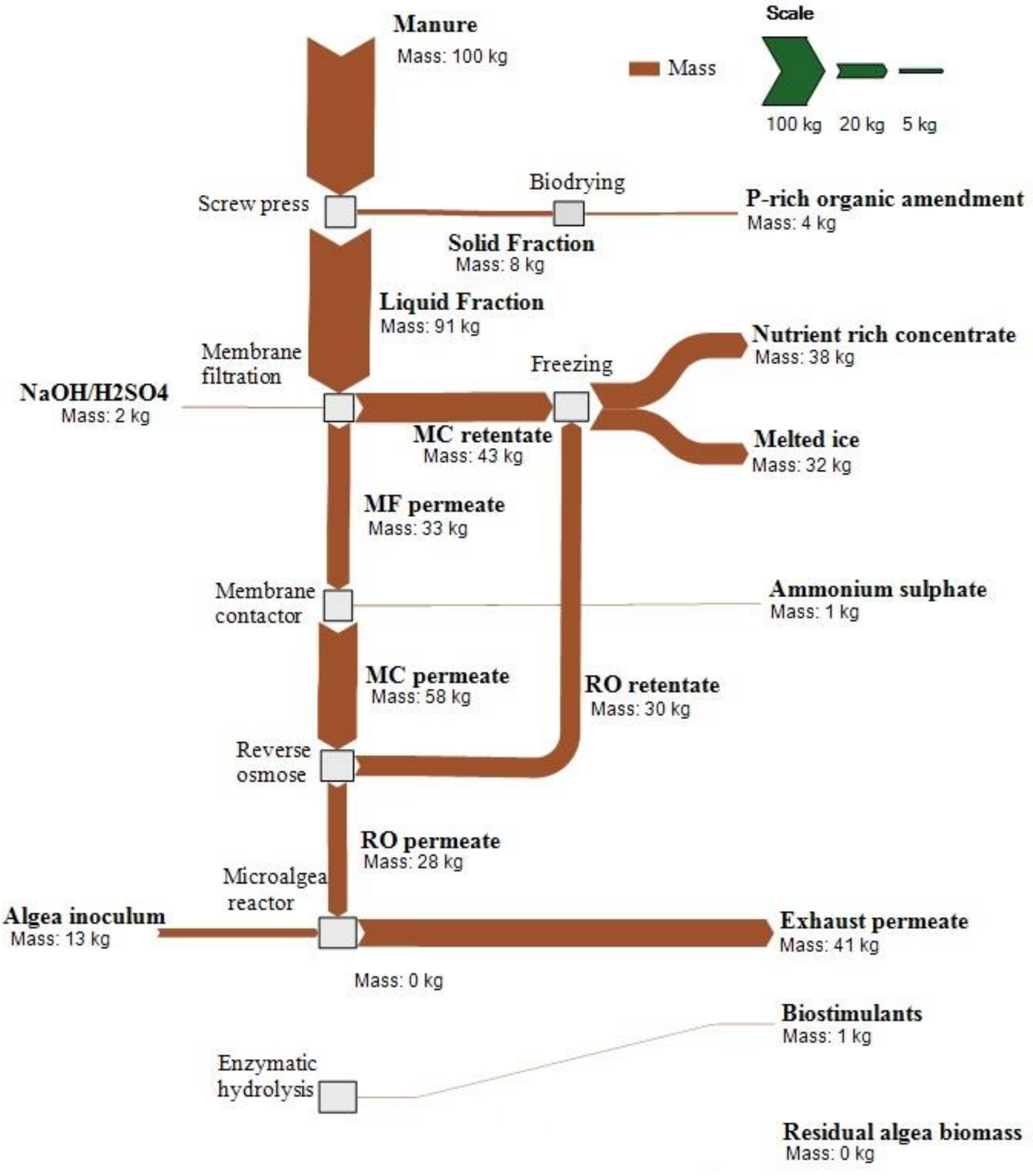


Figure 7. Mass balance in the Spanish pilot

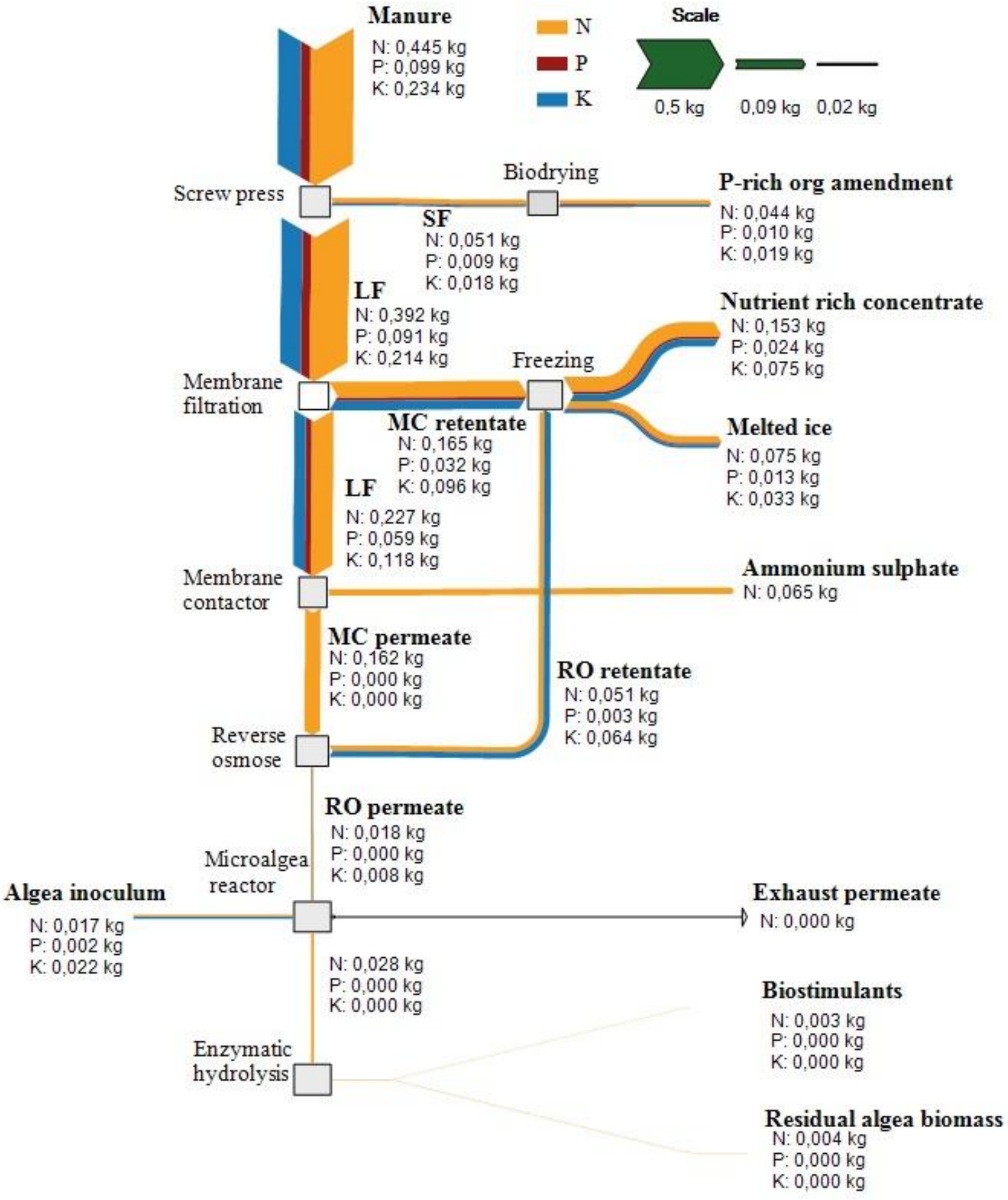


Figure 8. Nutrients balance in the Spanish pilot

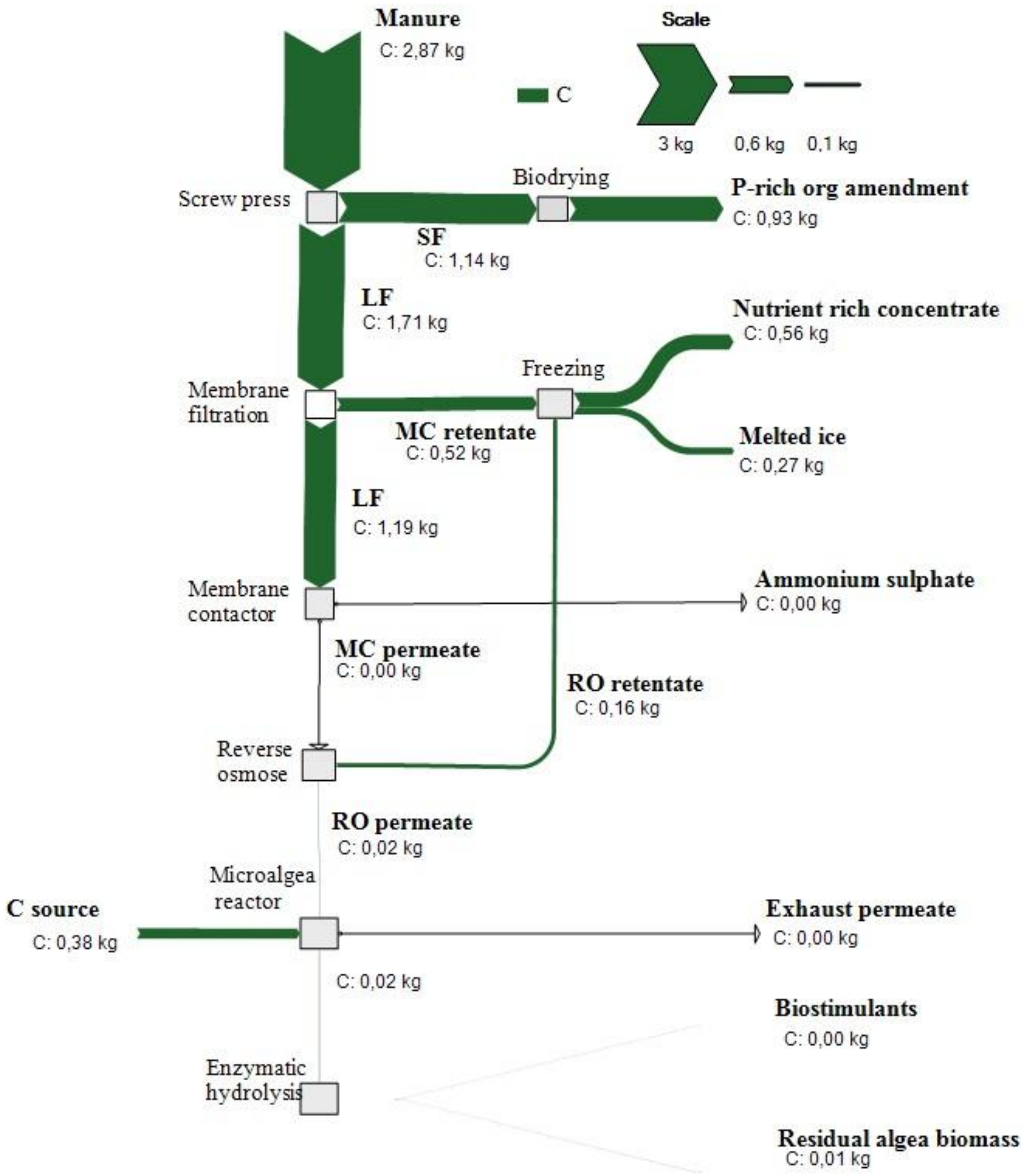


Figure 9. Carbon balance in the Spanish pilot

During pilot plant operation, some equipment specifications have been modified so some bottlenecks could be solved. The mesh size of the solid-liquid separation unit was modified from 520 microns to 260 microns to obtain a liquid fraction suitable for microfiltration in terms of particle size and distribution. After the first runs after the mentioned modification, the liquid fraction seemed to show appropriate characteristics for subsequent treatment stages.

Additionally, the microfiltration membrane was modified to have a cut-off from 130 nm to 400 nm. This modification resulted in MF retentate with less total solid content together with a lower content in phosphorus, potassium, zinc, and copper, compared to the analogous fractions obtained before the modification of the separation unit. It seems that given the less retention of total solids in the MF retentate, a major part of phosphorus and potassium in the liquid fraction of pig slurry will be able to reach the retentate from reverse osmosis which will the then concentrated into the nutrient rich concentrate via freeze concentration. Additionally, the content in zinc and copper of the MF retentate seems to be suitable for its use in freeze concentration unit, being able recycling it in the biorefinery and in recovering the major part of the nutrients contained in the pig slurry in the 5 BBFs obtained in the process. Moreover, with recent modifications, a higher content in nutrients is expected to be obtained in the nutrient rich concentrate, increasing significantly its quality as fertilizer.

Finally, the melted ice obtained from the freeze-concentration still contains some nitrogen and potassium. Therefore, during the optimization period, this flow will be recycled back into the reverse osmosis unit so a higher production rate of clean permeate is expected to be produced while recovering a bigger part of nitrogen and potassium also.

3.1.2. Spanish pilot: energy balance

After the optimization work done and assessment of the pilot for more than a year of operation, the energy consumption was 0.5 kWh/kg slurry treated although high variation along the year was found (64% variation coefficient was found in overall energy consumption). Seasonality affects greatly to the characteristics of raw slurry as well as the performance yields and energy consumption of units. The detailed energy consumption of technological units is reported in Table 6.

The most energy consuming unit is the photobioreactor (55% of the energy consumption), mainly due to the long algae cultivation period as well as low RO permeate production efficiency.

In the membrane's unit, the microfiltration has shown the highest specific energy consumption (32% of overall energy consumption) as it is the unit which treats the raw material and aims to remove suspended solids. Membrane contactors consumption can be almost considered as zero as it uses only two peristaltic pumps which provides only flowrate but not pressure. Finally, for the reverse osmosis, despite traditionally has been reported to be the most energy-consuming unit, in the case of FERTIMANURE pilot the equipment is over dimensioned, and so it operates at low pressure (15 – 18 bar) and low recovery (60 – 65%).

Separation unit shows a high variability in energy consumption due to the variable production rate of solid and liquid fractions along the year which was adjusted to the characteristics of slurry. Reverse osmosis and freeze concentration show also relevant variability. They were highly affected by the environmental temperature, being able to work more efficiently during the cold season. Similarly, since biodrying performance efficiency in terms of moisture removal was greatly affected also by environmental conditions, estimated energy production shows also variability, being more promising during hot season. Still, on average, combustion of biodried product would be able to cover almost 17% of the overall energy demand of the biorefinery.

Table 6. Energy balance of the Spanish pilot (kWh kg^{-1} of fresh treated material) (n = number of observations).

	Energy (kWh kg^{-1} raw slurry)
Consumption 1 (S/L separation)	4.6 x10^{-3} ± 3.1 x10^{-3} (n=8)
Consumption 2 (biodrying)	0.008 ± 0.009 (n=4)
Consumption 3 (microfiltration)	0.22 ± 0.09 (n=2)
Consumption 4 (membrane contactors)	4.86x10^{-3} ± 1.96x10^{-3} (n=2)
Consumption 5 (reverse osmosis)	15.70x10^{-3} ± 2.20x10^{-3} (n=2)
Consumption 6 (freeze concentration)	6.0 x10^{-3} ± 0.1 x10^{-3} (n=3)
Consumption 7 (microalgae cultivation)	0.28 (n=1)
Consumption 8 (enzymatic hydrolysis)	0.02 (n=1)
Total consumption	0.55 ± 0.32
Production 1 (energy recovery biomass boiler)	0.07 ± 0.04 (n=4*)
Total production	0.07 ± 0.04
Net energy	-0.48

*LHV of 3 of the 4 samples was theoretically estimated according to its elemental analysis following the calculation procedure suggested in Koppejan et al. (2012).

The current energy balance of the Spanish pilot is graphically shown in Figure 10.

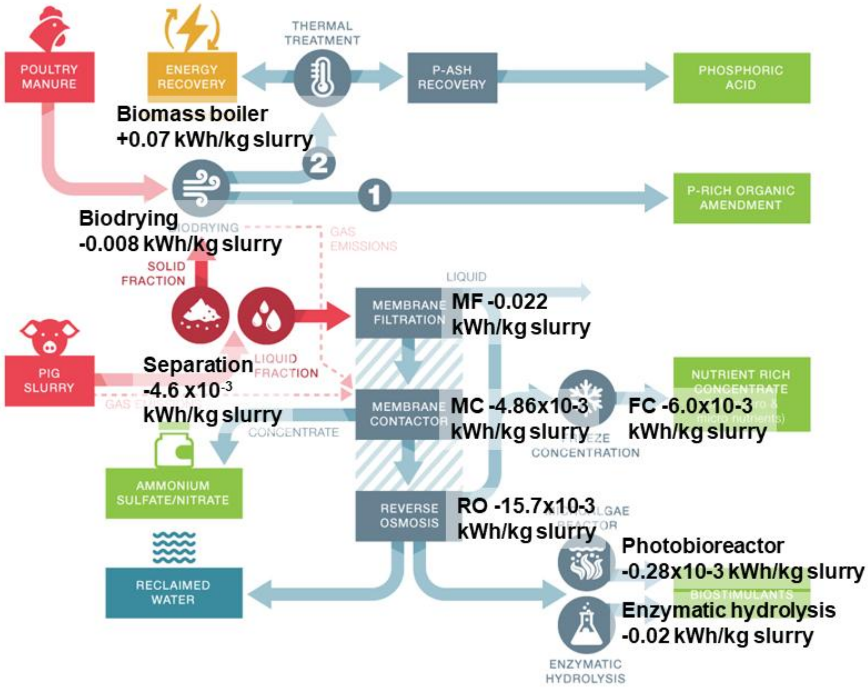


Figure 10. Energy balance of the Spanish pilot. Energy consumption is marked with a negative value while energy production with a positive value.

3.1.3. Spanish pilot: comparison with literature

a) Membrane-based systems: focus on membrane contactor to obtain ammonium sulphate.

Membrane-based technologies are very sensitive to suspended solid presence and it might cause pore (and membrane channels or spacers) blockage. Therefore, a proper pre-treatment is essential for assuring a good performance of nutrient recovery technologies – Membrane Contactor (MC) in this case. The most assessed pre-treatment technologies include flocculation, centrifugation of microfiltration/ultrafiltration (MF/UF). Flocculation and centrifugation reported significant improvement in effluent quality, although it cannot achieve total suspended solids removal, especially with the smaller ones (< 5 microns) (Rico et al., 2012). MF and/or UF reported a total removal of suspended solids and have been implemented successfully as pre-treatment for membrane-based technologies such as forward osmosis or reverse osmosis (Gripa et al., 2021).

Regarding nitrogen recovery technologies, MC has become a promising technology as it is capable of selectively recovering ammonium from wastewater and other liquid streams, resulting in low pollutant content ammonium salts solution in the stripping acid side. The technology has been successfully validated at laboratory scale for ammonium recovery from urban wastewater, cow manure, and swine manure. In the following table are shown the performance of MC reported in literature in terms of Total Ammonium Nitrogen (TAN) recovery rates.

Table 7. Comparison of the nitrogen (N) recovery rates in membrane contactors applied to different residual streams.

Reference	Feedstock	N recovery rate
Spanish pilot	Pig slurry liquid fraction	76 – 98% TAN
(Sheikh et al., 2022)	Wastewater	>95% TAN
(Aguilar-Moreno et al., 2022)	Wastewater	67% TAN
(He et al., 2020)	Wastewater	75.9% TAN
(Noriega-Hevia et al., 2020)	Wastewater	>99% TAN
(Ding et al., 2020)	Cow manure	>90% TAN
(Shi et al., 2022)	Swine manure	>90% TAN
(Vecino et al., 2019)	Swine manure	76% TAN

Regarding specific energy consumption, membrane-assisted stripping reported a specific energy consumption of $9.372 \times 10^{-3} \pm 3.780 \times 10^{-3}$ kWh/kg AS while conventional air stripping-scrubbing processes reports SEC of 0.199 ± 0.064 kWh/kg AS (Kar et al., 2020. Thus, it can be concluded that membrane-assisted stripping by using membrane contactors reports significant reduction in terms of energy consumption, mainly due to the lack of heating and pressurizing, using energy only for pumping liquids.

In addition, it was observed that more than 80% of total phosphorus content is retained during MF, resulting in difficult valorization as it is blended with particulate organic matter and bonded to metals as complex. Therefore, it is necessary to perform further research on alternative technologies that would allow the valorization of manure as bio-based fertilizer (Rodríguez-Alegre et al., 2023).

Finally, it was observed seasonality in the manure composition, which had affected the proper performance of the membrane-based treatments in terms of process efficiency (especially in MF step) and process kinetics (in the MC) as has been reported and discussed extensively elsewhere (Rodríguez-Alegre et al., 2024). For mitigating the effect of seasonality in the proposed train of technologies, an additional pre-treatment step (i.e., centrifugation, sieving) should be assessed.

b) Membrane-based systems- coupled with freeze concentration.

Freeze concentration is not an extensively used technology for nutrient recycling, although it has been conventionally used in food production systems or desalination plants (Dadrasnia et al., 2021). Application of freeze concentration for nutrient recycling is quite innovative and only few scientific papers explore this kind of application.

Although freeze concentration was successfully applied to concentrate the nutrients (up to 60-65%) present in the retentates obtained from the different membranes, the concentrated fraction of freeze concentration is still too diluted to be regarded as a fertilising product. Nutrients were slightly concentrated after freeze concentration for nitrogen (1.2-fold), phosphorus (1.1- fold) and potassium (1.3- fold). In literature, 4-to-6-fold concentration of nutrients has been reported for urine-based side streams via freeze concentration (Gulyas et al., 2004; Noe-Hays et al., 2021). Table 8 shows a comparative analysis of masa and nutrient recovery efficiencies found in literature from different sources.

Analysing more specifically the nutrient recovery rates achieved, Nutrient Rich Concentrate retains, in a 40% of the inlet slurry volume, the 34%, 24%, 32% and almost 20% of the inlet nitrogen, phosphorus potassium and carbon. When assessment is done compared to the liquid fraction, the concentrated product recovers almost 40% of the nitrogen and potassium, although only 26% of the phosphorus from the liquid fraction is retained in the product.

When working with membrane retentates, Uald-Lamkaddam et al. (2021) achieved 42%, 46% and 29% of nitrogen, phosphorus and potassium recovery of the nutrients in the RO retentate when applying suspension freeze concentration in two stages, although higher recovery rates were achieved in first stage. Regarding the concentration effect of the retentates, nitrogen, phosphorus and potassium of nutrient rich concentrate obtained in the mentioned study were concentrated up to 1.9-, 2- and 1,3-fold, respectively.

Compared to other analogous concentration technologies such as vacuum evaporation, Samanta et al., (2022) concentrated 12 times the ammonium nitrogen content of a MF permeate when treating pig manure. Vacuum evaporation (VE) is regarded to be a technology able to significantly reduce (often by 50%) the inlet volume of a concentrated stream (Herbes et al.,. 2020). 50% volume reduction seems to be achievable also with freeze concentration, although the nutrient distribution efficiency does not seem to be as comparable. When applying VE technology to digestate, 70% of the ammonia nitrogen can be concentrated obtaining a nutrient rich concentrate doubling the nitrogen content while concentrating by 3 the soluble phosphorus content (Li et al., 2016). Even though freeze concentration seems to be able to reduce the working volume and concentrate nutrients up to 70%, the higher viscosity and particulate material in the resulting concentrated fraction could be difficult to be further concentrated in an additional stage of freeze concentration (Uald-Lamkaddam et al., 2021). However, the acidic treatment of the MF retentate could help improving the nutrient recovery in the concentrated fraction of freeze concentration.

Table 8. Comparison of the nutrient recovery rates in Freeze Concentration applied to different residual streams for carbon (C), nitrogen (N), phosphorus (P) and potassium (K).

Reference	**Feedstock**	**Recovery (%)**			
		C	**N**	**P**	**K**
Spanish pilot	Retentates from microfiltration and reverse osmosis (1.5:1, v:v) from the treatment of the liquid fraction of pig slurry	19.7% (of slurry); 32.9% (of LF) 83.2% (of membrane retentates)	34.4% (of slurry); 39.0% (of LF); 70.6% (of membrane retentates)	23.7 % (of slurry); 25.9% (of LF); 68.9%% (of membrane retentates)	31.8% (of slurry); 34.9%(of LF); 50.0% (of membrane retentates)
Uald-Lamkaddam et al., 2021	Reverse osmosis retentate from the treatment of the liquid fraction of digestate of agro-industrial waste	-	42% (in two stages); 62% (in first stage)	46% (in two stages); 88% (in first stage)	48% (in two stages); 60% (in first stage)
Noe-Hays et al., 2021	Urine	-	91.51%	91.36%	91.58%
Hidayat et al., 2023	Permeate of the ultrafiltration of digested Cheese Wey			70% (in two stages); 40% (in first stage)	

Melted ice still retains an important part of the nutrients (about 30% of inlet nutrients from retentates) and carbon. Although it would be the desirable scenario, direct application of the melted ice in the reverse osmosis membrane to recover a higher volume of water would not be possible considering those characteristics and the particulate material. Therefore, to maximise nutrient and water recovery, melted ice should be directed again to the MF unit. To maximise both, concentrate and ice recoveries, the simultaneous concentrated fraction recovery by applying fine sieving together with vacuum could help enhancing nutrient recovery as well as obtaining a cleaner ice.

Regarding energy consumption, freeze concentration of membrane retentates consumed significantly less (0.6kWh/ kg water removed) than the values reported in the literature (14 kWh/kg water removed in Noe-Hays et al., 2021). The higher energy consumption reported is probably related to the freezing temperatures set by the author mentioned (-6.5ºC and -13ºC during the first and second stages, versus -5ºC in the tests performed in the Spanish pilot). Moreover, the freezing periods in the mentioned study were significantly longer than the ones in the pilot (27.3- 109 h versus approximately 4h, respectively). Optimisation of the energy consumption during freeze concentration could be achieved evaluating the timeframe-dependent nutrient recovery efficiencies. To do so, Noe-Hays et a., (2021) suggested a conductivity monitoring protocol. Finally, compared to vacuum evaporation technology, freeze concentration was demonstrated to be significantly less energy demanding technology for the equivalent water removal. Freeze concentration of membrane retentates consumed approximately 30kWh/m3 of retentate treated. Vondra et al., (2017) compared different evaporator configurations in which energy demand was similar, 26-30kWh/m3 for forced circulation evaporators, 14-18kWh/m3 in multistage flash evaporator and 12-24kWh/m3 in falling film evaporators. However, the values reported do not consider the additional energy consumption of cooling system. Therefore, freeze concentration shows an energetic advantage when compared to evaporation technologies. Nevertheless, scale dependency should also be considered as the energy efficiency in full scale evaporation plants could be improved significantly and freeze concentration might not be comparable at that scale. For instance, Chiumenti et al., (2013) reported estimated energy consumptions of 5-8 kWh/m3 of digestate or 350 kWh/m3 of evaporated water at full-scale evaporator plant being those values about four times lower of what was monitored for freeze concentration (considering water removed).

c) Microalgae cultivation coupled with enzymatic hydrolysis to obtain biostimulants

Recovering nutrients from microalgae biomass is difficult due to the structure and composition of the microalgal cell wall (Elvira Navarro-López, 2020). Wijffels et al. (2010) highlighted the importance, challenges and need for research in cell disruption. In addition, microalgae concentration can affect the nutrient recovery yield. To get the higher extraction and to overcome this difficulty, mechanical cell disruption methods, such as high-pressure homogenization (HPH), ultrasounds (UAE), microwave (MAE) or enzymatic hydrolysis can be applied to facilitate the extraction of the compounds of interest. Even though HPH, UAE, and MAE are effective, these are not easily practicable due to the high costs involved in the form of specialized instruments, time consumption, and the skills required. Enzymatic hydrolysis has been reported as one of the most promising processes. Several studies have been carried out evaluating enzymatic hydrolysis of Scenedesmus sp. biomass grown in piggery wastewater (Rojo et al., 2021; Martin Juarez et al., 2021) (Table 9). Nitrogen recovery in final hydrolysates of above 10-33% using proteases or combination of proteases and cellulases are reported.

Table 9. Comparison of the nitrogen (N) recovery rates in hydrolysis processes of Scenedesmus sp cultivated in swine effluents (wastewater, slurry and manure).

Reference	Culture medium	Hydrolytic conditions	N recovery rate
Spanish pilot	Permeate from RO	Protease + endopeptidase	30%
Romero-Garcia et al. 2013	Slurry wastewater	Proteases and cellulases	30%
Rojo et al. 2021	Swine wastewater	Protease	19.80%
Rojo et al. 2021	Swine wastewater	Protease +cellulase	32.80%
Juarez et al., 2021	Pig manure	Cellulase+protease	10%

The average of the nitrogen recovery in the hydrolytic unit of Spanish pilot (30%) is comparable with the range what other authors reported (10-34%). Some studies (Elena M. Rojo, 2021); (Garcia-Moscoso, Obeid, Kumar, & Hatcher, 2013)) reported that the combination of proteases and cellulases increased protein recovery yield from 10% to 30%, even to 80% when the protease Protamex® and cellulase Celluclast ® were used, in comparison with process with only proteases. Spanish pilot hydrolytic unit only operated with proteases since no improvement were detected when combination with cellulases were evaluated (see D2.5). In addition, the nutrient recovery yield can be also affected by microalgae conditioning. In this sense, it was observed difference between using fresh or paste biomass or lyophilised biomass. Kroger et al, (2019) (Kröger, Klemm, & Nelles, 2019) reported that freeze drying of the fresh *Scenedesmus sp* gave a higher yield of extraction than the direct extraction of the fresh ones. These results are in line with those obtained within the project where higher nutrient recovery yield have been observed in lyophilised microalgae (70%) in comparison with fresh microalgae (30%).

b) Biodrying to obtain organic amendment.

According to the "Inventory of manure processing activities in Europe" (Foged et al., 2011), in 2011 there were 77 biodrying installations in Europe for manure processing, of which 80% were considered farm size installations. According to the data reported, after composting, biodrying seemed to be the preferred management alternative for solid manures in small size farms (5% of farm size installations).

Although there is extensive literature assessing biodrying of different feedstocks (sewage and paper mill sludges, municipal solid waste and manures), in general, the aim of the biodried product is recovered solid fuels rather than fertilizing products.

In summary, the average moisture removal efficiency in the biodrying trench of the Spanish pilot (62%) is in the high range of what other authors reported (53-79%) for biodrying of manure at comparable scale (swine, poultry and cow manure in Choi et al., 2001; Sharara et al., 2012 and Sadaka and Ahn, 2012) (Table 11) The removal values reported at similar scale for sludge (46-53%) (Frei et al., 2004; Cai et al., 2012) are in general lower, although higher efficiencies were reported for studies at industrial scale for sludge and municipal solid waste (66%-90%) (Tambone et al., 2011; Winkler et al., 2013). It is noteworthy also that the average moisture removal value represents biodrying trials performed in spring (N=2), summer (N=2) and autumn (N=1) showing the robustness of the technology, along the year at a farm size scale.

Regarding nutrient mass balances, only few publications report nutrient evolution along biodrying processes. In general, a partial loss of nitrogen is usually reported (Choi et al, 2001) as the increase of C/N ratio due to nitrogen loss in form of ammonia (Sharara et al., 2012; Sadaka and Ahn, 2012; González et al., 2019; Guerra-Gorostegui et al., 2021). Nevertheless, a concentration effect of nitrogen, phosphorus and potassium can also be expected by the end of biodrying due to organic matter consumption (Sharara et al., 2012; Sadaka and Ahn, 2012). According to the mass balances performed, nitrogen loss during biodrying was calculated to be 20% of initial nitrogen in the trials done in May-June (N=2) while 40% of initial nitrogen the trial performed in autumn (N=1). In literature, higher nitrogen loss was reported (up to 51% in Choi et al., 2001 for poultry manure biodrying). Sadaka and Ahn (2012) reported 32% and around 11% increase in C/N values along the biodrying processes of poultry and swine manure, respectively, reportedly mainly due to nitrogen loss rather than organic matter loss (only 6-8% of volatile matter was lost during biodrying processes).

Table 10. Comparison of the nutrient recovery rates during biodrying of different animal manures for carbon (C), nitrogen (N), phosphorus (P) and potassium (K).

Reference	Feedstock	Recovery (%)			
		C	N	P	K
Spanish pilot	Solid fraction of pig manure	32.3% of C in slurry and 81.2% of the C in solid fraction	9.8% of N in slurry and 86% of the N in solid fraction	8.2% of the P in slurry, 108% of the P in the solid fraction	8.2% of the K in the slurry and 104.3% of the K in the solid fraction
Spanish pilot	Poultry manure	86% of C in poultry manure	83.8% of N in poultry manure	97.3% of P in poultry manure	100.1% of K in poultry manure
Sadaka and Ahn, 2012	Beef manure	68.9%*	63%*	96%*	98.3%*
	Swine manure	66.1%*	59.6%*	102.7%*	95.1%*
	Poultry manure	61.1%*	46.3%*	91.3%*	105.5%*
Choi et al., 2001	Poultry manure	16% of VS estimated to be approximately 26% of C	49%	-	-
Sharara et al., 2012	Dairy manure	58.1-63.7% of C estimated from VS content	-	-	-

*Estimated from the values reported in the paper

Regarding energy consumption, the biodrying trench consumed 110 kWh per ton of solid fraction. When the consumption is reported as energy consumed per tons of moisture removed, the values are highly variable (392 ± 249 kWh/t water). Despite variability, the consumption values resulted in a better energy performance than the previously reported values. At bench scale, biodrying of animal manures consumed 2.2 - 2.5 kWh per each kilogram of water removed (Sharara et al., 2012), whereas biodrying of sewage sludge were found to be more like our values in the Spanish pilot. 0.4 - 0.9 kWh per each kg of water removed was reported by Guerra-Gorostegui et al., 2022.

Moreover, when biodrying is compared to convective and conductive drying technologies (700 - 1400 kWh/t sludge), a significant reduction of energy consumption can be achieved (Bennamoun et al., 2013), significantly lower than the 110kWh measured for the Spanish pilot.

c) Biodrying coupled to biomass boiler: obtaining P rich ashes and phosphoric acid.

A first screening of operational parameters was conducted for the acidic extraction of phosphorus from the ashes obtained from the biodried fraction of the solid fraction of pig slurry. During this first screening different mineral and organic acids (H_2SO_4, HNO_3, HCl and citric acid), concentrations (1.2M and 2.4M HNO_3 and HCl; 0.6M and 1.2M for H_2SO_4 and 2.5M for citric acid) for and solid to liquid ratios (1 to 10 and 1 to 5) were assessed as well as pre-treatment of the ashes with water to remove salts (Oshita et al., 2016).

The best phosphorus efficiencies at lab scale were obtained when using HCl and H_2SO_4 at highest concentration and lowest solid to liquid ratio. From the lab scale screening and considering its comparatively lower cost in the market, sulphuric acid was selected as extractant. Besides, the extraction liquid to solid ratio selected was that one allowing the lowest acid volume use and highest potential concentration in the extracted solution. That way, the phosphoric acid extracted could have a higher commercial value. Thus, for scaling up after pre-treatment with water (1 to 10 solid to liquid ratio) sulphuric acid 1.2M was used in a 1 to 5 solid to liquid ratio. Scaling up was done in 2L reactors in which 0.365g of pre-treated and dried ash was treated. Extraction was done at ambient temperature for one hour of continuous shaking.

Table 11 shows a comparison of the upscales acid P recovery yields with the data reported in literature.

Table *11*. Comparison of the nutrient recovery rates after acidic treatment of ashes from different origin for carbon (C), nitrogen (N), phosphorus (P) and potassium (K).

Reference	**Feedstock**	**Recovery (%)**			
		C	**N**	**P**	**K**
Spanish pilot	Ash from the combustion of biodried solid fraction of pig manure			21.87%	14.18%
Rivera et al., 2022	Poultry manure ash			Up to 95%	
Kleemann et al., 2017	Sewage sludge ash			Up to 93%	
Fang et al., 2018	Sewage sludge ash			94%	
Donatello et al., 2010	Sewage sludge ash			72-91%	

Extracted supernatant resulted in a low concentration of phosphorus and potassium reaching very low extraction efficiencies (21.9% for P and 14.2% for K) compared to the extraction efficiencies found in literature for acidic phosphorus extraction of different origin ashes (72-95% in Rivera et al., 2022; Kleemann et al., 2017; Fang et al., 2018 and Donatello et al., 2010), even when compared to the high efficiencies obtained at lab scale (up to 93% of extraction). Under these circumstances, assessment of the extraction timeframe should be done. Additionally, probably due to the low solid to liquid ratio employed, jellification of the extracted supernatant was noticed and therefore the representativity of the concentration of the elements of interest in the extracted product cannot be guaranteed. Although it would lead to lower concentration of phosphorus in the extracted product, a 1 to 10 solid to liquid ratio seems to be operatively more feasible. Afterwards, when the concentration of phosphoric acid is low in the solution, other authors demonstrated several satisfactory precipitation strategies by modifying the pH of the solution (Kaikake et al., 2009). That strategy should be also explored to evaluate its feasibility as well as the quality and purity of the precipitated product obtained.

3.2. Dutch pilot

3.2.1. Dutch pilot: mass and nutrients balance

The APF pilot installation has been monitored on a regular basis in the period March 2020 till February 2023, with in total 14 sampling rounds. In almost half of the monitoring rounds also micronutrients/heavy metals were analysed. The configuration of the installation had some small changes and optimizations during this period and the stripper with ammonium sulphate and liquid potassium fertilisers was ready from March 2021 onwards.

In Table 12 three years are considered; the input mass flows are presented for the nutrient recovery and reuse (NRR) system at APF. The main input flow is the produced digestate, on average about 1,780 tons per year. Because the N stripper was not functioning all the time and there was sometimes a malfunctioning of the system or in data registration, the annual produced amounts of BBFs are related to the annual produced amount of digestate. The amounts of produced soil conditioner are based on the measurement of the mass separation efficiency of the screw press, which is on average 9.3% becoming solid fraction and 91.7% liquid fraction. The remaining amount of digestate liquid fraction is stripped and for the N-stripping process on average 3.34 kg 96% H_2SO_4 is used per m^3 (or per tonne) liquid digestate. As a result, 22.8 kg ammonium sulphate solution with an average concentration of 65-gram N and 61-gram ammonium per litre is produced per m^3 of separated liquid digestate. This residual flow of liquid fraction that remains after N-stripping is called the liquid potassium (K) fertiliser, a product that still contains some nitrogen and is the largest amount of product produced out of digestate at APF. All BBFs, except ammonium sulphate solution, are mainly used on the farmers own agricultural land to produce grass and maize used as feed for his cattle. Since the RENURE criteria for fertilisers out of manure are not legally valid yet, the ammonium sulphate is partly used on own land and partly sold on the regional market. Almost all organic soil conditioner (solid fraction of digestate) is sold to individual consumers for gardening but could also be used by regional farmers to maintain organic matter in soils. The wet organic rich phosphorus sludge is formed by passive precipitation from the liquid fraction in a settling tank. This processed could be optimized by addition of calcium of magnesium base for active precipitation into calciumphosphate and magnesiumammoniumphosphate (struvite) but became not part of the overall manure processing. Some separated experiments and tests were carried out.

Table 12 Inputs and output mass flows [tonnes/year] of the total nutrient recovery and reuse (NRR) system after anaerobic digestion at the Dutch pilot Arjan Prinsen Farm (APF) for the year 2020-2022, including % of the total input of feed stocks and/or output as biobased fertilisers, and for comparison also the total amount of liquid fraction is provided produced out of digestate within the system.

Year	2020	2021	2022	Average 2020-2022	% of input
Digestate	1,925	1,986	1,430	1,780	99.7%
H_2SO_4	5.8	6.0	4.3	5.4	0.3%
NRR total in	**1,931**	**1,992**	**1,434**	**1,786**	**100%**
Produced BBFs					**% of output**
Organic soil conditioner	179	184	133	179	9.3%
Wet organic rich phosphorus sludge	10	10	7	10	0.5%
Liquid potassium fertiliser	1,703	1,757	1,265	1,703	88.2%
Ammonium sulphate solution	40	41	30	40	2.1%
NRR total out	**1,931**	**1,992**	**1,434**	**1,931**	**100%**
Liquid fraction (in system)	1,746	1,801	1,297	1,615	-

Based on data of the average composition of the BBF products and the mass flow within the NRR system, the nutrient flows were calculated (Table 13). The average values of the APF nutrient flows are visualised in a Sankey diagram for mass, C, N, P and K (Figure 11 and Figure 12). For the carbon balance the measured TOC is used for ammonium sulphate and K rich fertiliser, but for digestate, soil conditioner and P rich sludge TOC was not measured, and organic matter is used as a proxy for the amount of TOC (TOC = 0.58 x OM).

Although the produced amount of the soil conditioner is limited, a substantial amount of carbon / organic matter (OM) is accumulated in the soil conditioner leading to a high average content of about 119 kg C/t (≈ 205 kg OM/t ≈ 20.5% OM). Overall, about 55% of total P and 61% of total N which was available in the digestate remains in the liquid K fertiliser meaning that this BBF is an organic NPK fertiliser rather than a liquid K fertiliser. About 29% of total N in digestate is recovered as ammonium sulphate.

Table 13. Summary of mass and nutrients balances in the Dutch pilot APF (starting material = 100; n = number of observations) for carbon (C), nitrogen (N), ammonium (N-NH4), phosphorus (P) and potassium (K), including the balance difference as % of initial mass.

	Mass (kg)	C (kg)	N (kg)	N-NH_4 (kg)	P (kg)	K (kg)
			INPUT			
Digestate	100	3.15 ± 0.71 (n=14)	0.45 ± 0.07 (n=14)	0.26 ± 0.05 (n=14)	0.07 ± 0.018 (n=14)	0.5 ± 0.04 (n=14)
H_2SO_4	0.3	-	-	-	-	-
Total	100.3	3.15	0.45	0.26	0.07	0.5
			OUTPUT			
Organic soil conditioner	9.3	1.11 ± 0.33 (n=14)	0.06 ± 0.01 (n=14)	0.02 ± 0.01 (n=14)	0.022 ± 0.005 (n=13)	0.05 ± 0.005 (n=14)
Wet org rich phosphorus sludge	0.5	0.018 ± 0.003 (n=12)	0.0031 ± 0.0004 (n=12)	0.0018 ± 0.0002 (n=12)	0.002 ± 0.0005 (n=12)	0.0023 ± 0.0004 (n=12)
Liquid potassium fertiliser	88.3	1.39 ± 0.34 (n=14)	0.27 ± 0.08 (n=15)	0.15 ± 0.06 (n=15)	0.04 ± 0.01 (n=15)	0.45 ± 0.03 (n=15)
Ammonium sulphate solution	2.2	0.0018 ± 0.0003 (n=9)	0.13 ± 0.02 (n=13)	0.13 ± 0.02 (n=13)	0.000059 ± 0.000021 (n=10)	0.00072 ± 0.00028 (n=10)
Total	100.3	2.51	0.47	0.30	0.063	0.50
Balance (% initial mass)	+0.3	-20	+5	+17	-12	-1

In Figure 14. the nutrient separation efficiencies (SEs) are shown for all the measured nutrients (and heavy metals) in digestate that are distributed over the solid versus the liquid fraction by separation by the screw press at APF. Figure 15 SEs of the settling tank representing the nutrient recovery of nutrients in the end-product wet organic P rich sludge within the passive precipitation process as is.

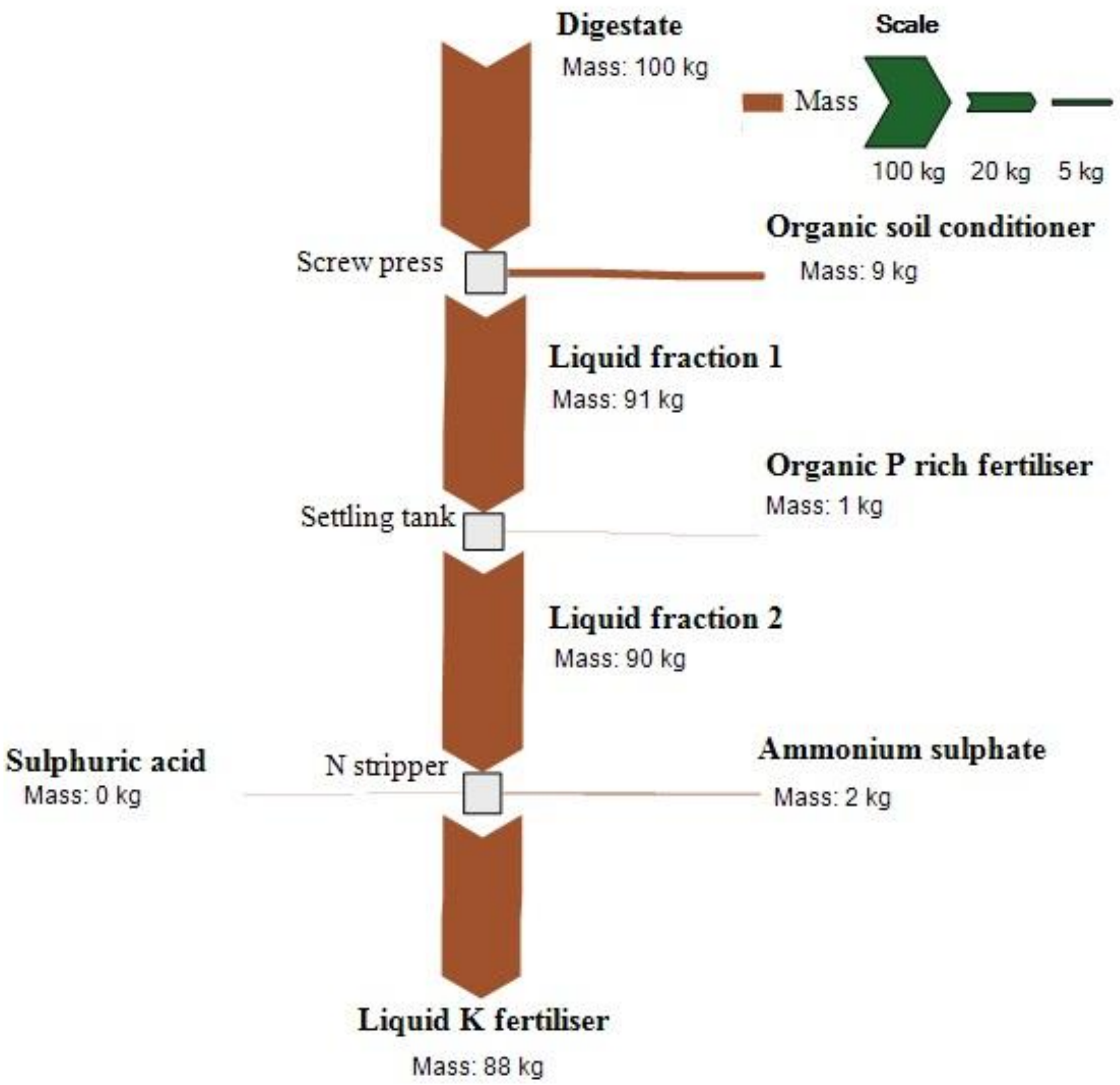


Figure 11. Mass balance in the Dutch pilot

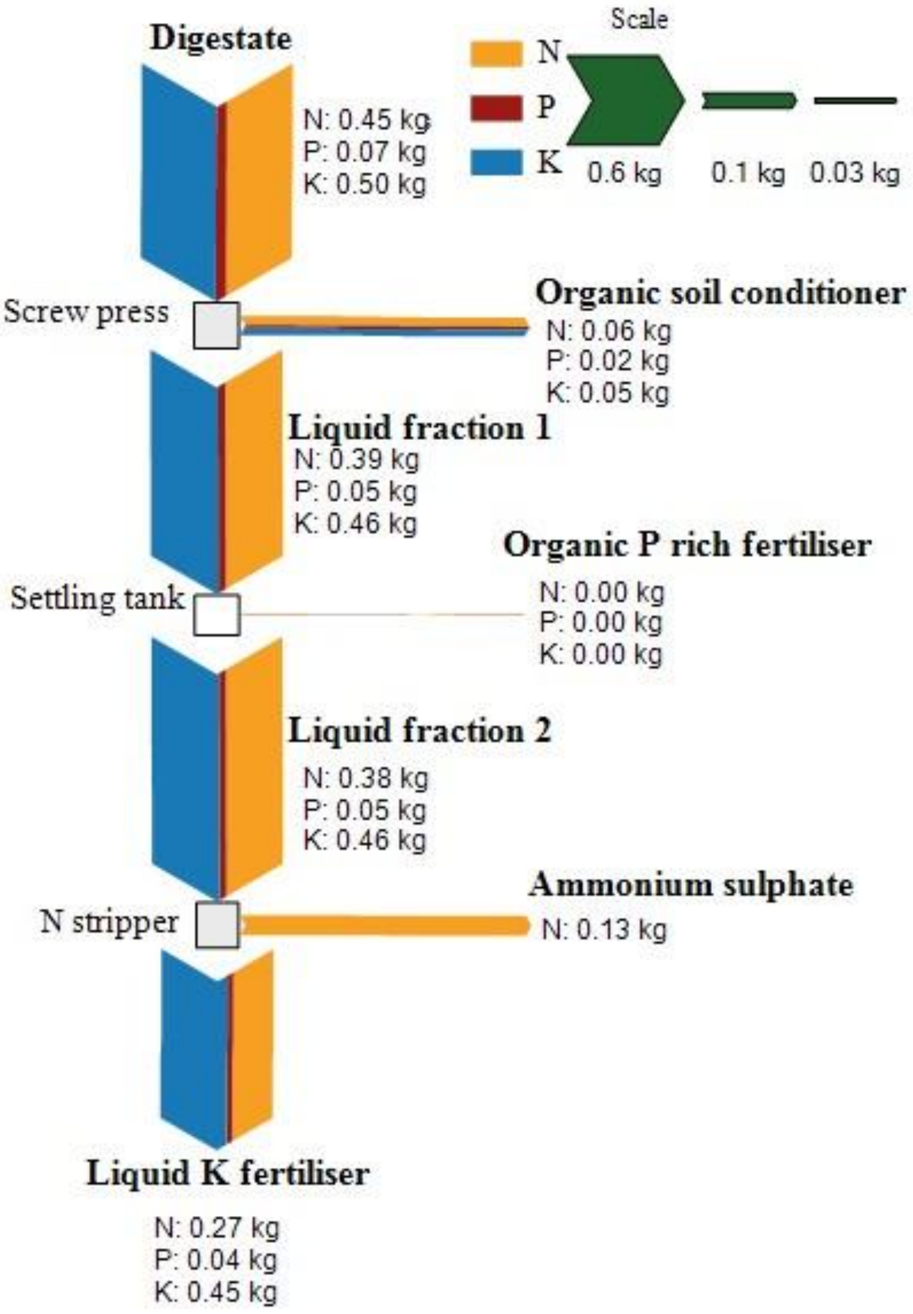


Figure 12. Nutrients balance in the Dutch pilot

A substantial part of the carbon/organic matter flows with the large amount of liquid fraction of digestate with an average concentration of 22.5 kg C/t (≈ 38.9 kg OM/t ≈ 3.9% OM). About 91% of the ammonium and 90% of the potassium in the digestate flows towards the liquid fraction of the digestate since these compounds are highly soluble. A relative high amount of 86% of total nitrogen (N_{tot}) also accumulates in the liquid fraction of digestate. Approximate 31% of the amount of phosphorus (P) in the digestate accumulates in the soil conditioner and the remaining part in the separated liquid fraction, which is relative high indicating that a large part of P is associated to fine and colloid particles within the digestate. The settling tank filters approx. 3.1% of the P from the separated liquid fraction of digestate, which has to be improved by installing the acidification and precipitation equipment. Since the settling tank to recover P in terms of wet organic rich P sludge was a scientific test and not part of the overall monitoring system, the produced amount (tonnes/year) is very low (estimated by the farmer who operates the system) and only small parts of the nutrients were collected in the sludge.

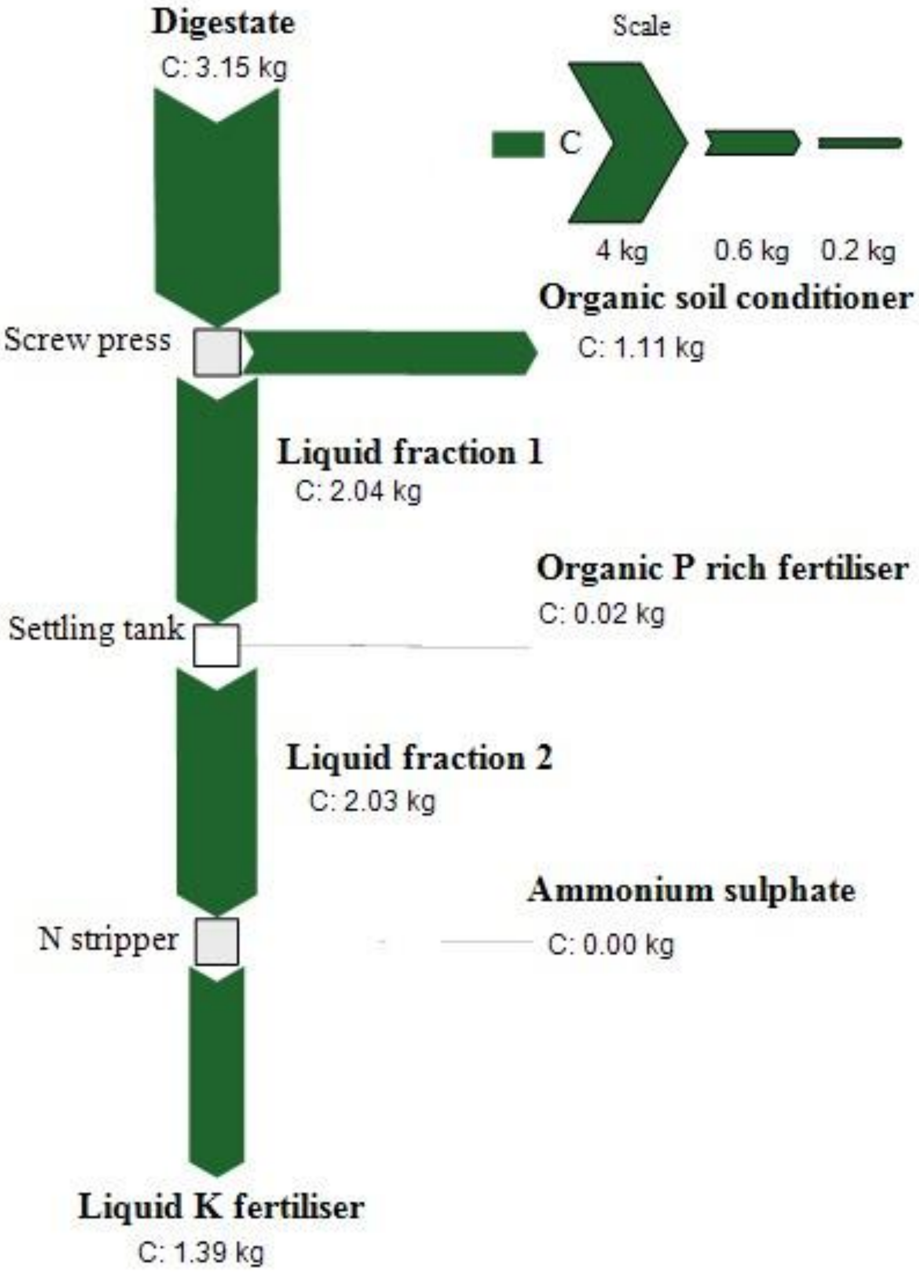


Figure 13. Carbon balance in the Dutch pilot

The separation efficiencies and the standard deviation for Zinc (Zn) and Copper (Cu) and some other micronutrients/heavy metals are relatively low/poor for both the screw press (Figure 14.) and in the settling tank (Figure 15). For these elements also the calculated balance is mostly higher than 100%. This overestimation can be explained by the fact that often the concentrations of those elements in the large amount of liquid fraction are at below the detection limit, and consequently set at the detection limit value. In practice the concentration values are probably lower but unknown. Both the error in the mass balance becomes increases as well as the percentage standard error.

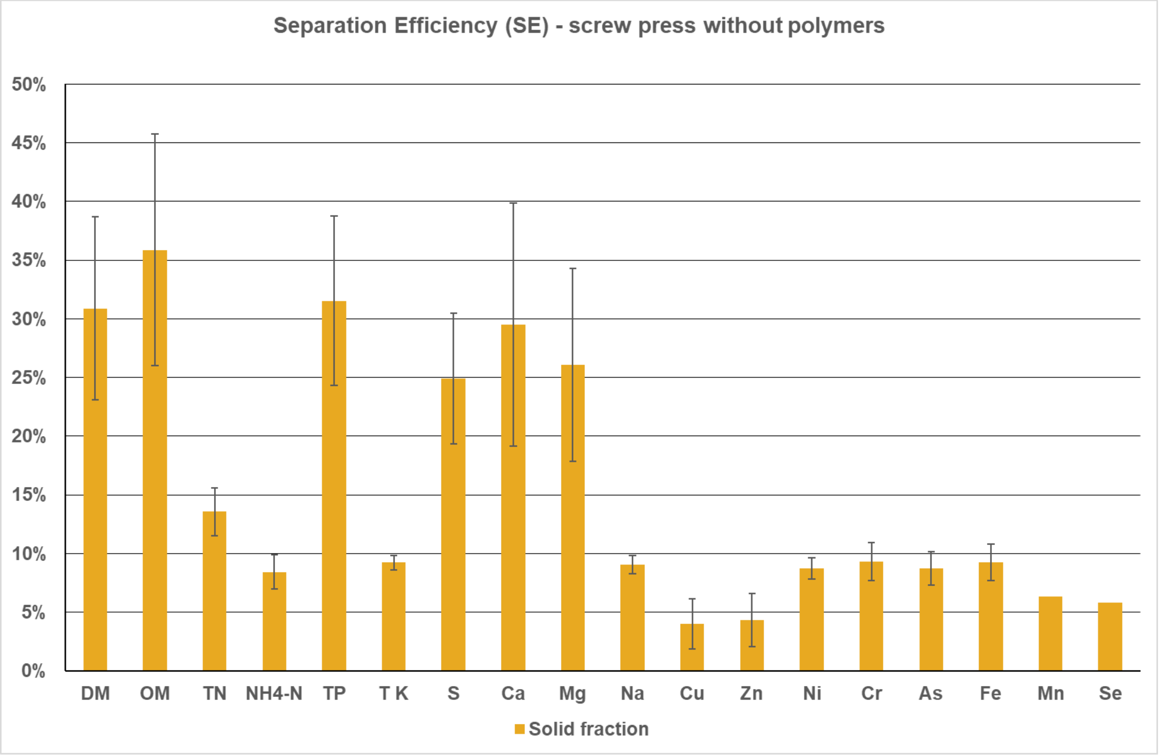


Figure 14. Separation efficiencies (%) of the solid fraction of digestate separated by a screw press without polymers used expressed as % of 100% (including liquid fraction) for compounds in digestate, including DM = dry matter, OM = organic matter, TN = total nitrogen, NH4-N = N as ammonium, TP = total P, TK = total K, and other macro, meso and micronutrients, and standard deviation.

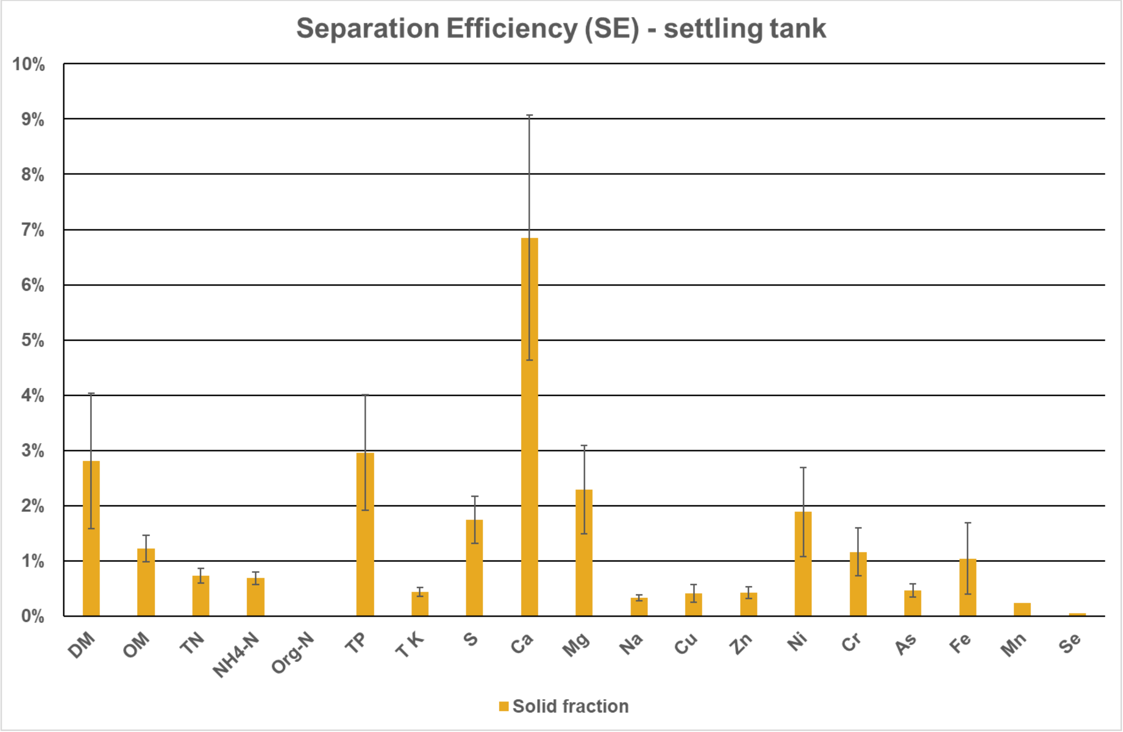


Figure 15. Separation efficiencies (%) of the solid fraction of digestate separated by the settling tank expressed as % of 100% (including liquid fraction) for compounds in digestate, including DM = dry matter, OM = organic matter, TN = total nitrogen, NH4-N = N as ammonium, TP = total P, TK = total K, and other macro, meso and micronutrients, and standard deviation.

3.2.2. Dutch pilot: energy balance

In 2020, 2021 and 2022 on average the total amount of produced biogas is estimated 42.9 thousand Nm^3/year (= 55 t/year), ranging between 54 to 31 thousand Nm^3/year containing about 55% methane (Table 12). The caloric value of biogas is about 21.1 MJ/Nm^3 and consequently on average 904 GJ (= 251 MWh) is potentially available to produce electricity and heat in the Combined Heat Power (CHP) installation. More details per year can be found in Table 12. In Table 15 and Table 16, the conversion of produced energy (biogas) into electricity, heat and loss (mainly heat) of the APF plant is shown. From the amount of potential available energy in biogas (average 251 MWh/year), on average 27% is made available as effective electric energy (average 69 MWh/year) of which a part is sold to the grid and at the same time part is received from the grid. In addition, on average 657 GJ (≈183 MWh) heat is produced per year by the CHP, which partly used functionally for the digester (34%), and the nitrogen stripper and the warming of the house (48%). However, a substantial part of the heat is lost to the environment (19%). These are average values of three years, but within a year it will fluctuates especially regarding heat consumption / distribution (summer/winter).

Measured heat consumption of each of the units of the NRR is currently not available because the NRR installation at APF was implemented as a whole system in practise at an existing farm, so rather rough estimates were made. The use of heat is made available on an annual base because of seasonal effects and the stripper is not always operational. The distribution of the use of energy of different equipment related to electricity consumption (consumption 1) in is shown in Table 16. These are the aggregated results of a detailed survey and measurements of about 27 specifics on farm pilot equipment in place at APF. Electricity uses were measured as specific as possible per part of the system, including subsystems as the dairy farm itself (buildings, dairy equipment, etc), the manure collection and pre-treatment system, the co-substrates handling, the digester and CHP, the screw press, the N stripper-scrubber, and overall the pumps, mixers and shredders together, houses, etc. Additionally, the energy balance is represented in the Dutch pilot infographic (Figure 16).

Table 14. Energy production from the combined heat power installation running on biogas from the digester, and consumption of electricity and heat by the Dutch pilot AFP in the 2020-2022 period.

		2020	**2021**	**2022**	**2020-2022 average**
Biogas production	Nm3/year	54,318	43,610	30,663	42,863
Biogas production	t/year	69.7	55.9	39.3	55.0
Methane (CH4) content	Nm3/year	29,875	23,985	16,864	23,575
Biogas - total caloric value	**MWh**	**318**	**256**	**180**	**251**
Biogas - total caloric value	GJ	1,146	920	647	904
CHP yield electricity	%	26.6	29.8	25.3	27.2
CHP yield heat	%	59.8	57.2	60.8	59.3
CHP total loss	%	13.6	13.0	13.8	13.5
Electricity (measured)	**MWh**	**84.6**	**76.2**	**45.5**	**68.8**
- sold to grid	MWh	16.9	15.2	0.0	10.7
- received from grid	MWh	0.0	25.0	43.7	22.9
- own use plant (farm, digester, screw press, N stripper, house, etc.)	MWh	67.7	86.0	99.2	84.3
Heat (partly measured)	**MWh**	**233.8**	**179.4**	**134.2**	**182.5**
	GJ	841.6	645.7	483.2	656.8
- digester	MWh	78.5	60.2	45.1	61.3
- total stripping & house (measured, about 50% : 50%)	MWh	112.0	85.9	64.3	87.4
- loss of heat	MWh	43.3	33.2	24.9	33.8
Other loss (residual)	**MWh**	**0**	**0**	**0**	**0**

Table 15. Energy consumption, production, and balance for the Dutch pilot as kWh kg^{-1} of fresh treated material, for APF the feed stock of the digester being mainly cattle slurry.

	Energy (average 2020-2022) (kWh kg^{-1} digester feed stock)
Consumption 1: electricity - own use plant (e.d. farm, digester, screw press, total N stripping, house)	0.046
Consumption 2: heat – digester	0.033
Consumption 3: heat - total stripping & house measured	0.047
Consumption 4: loss of heat	0.018
Total energy consumption	0.145
Production 1 (biogas in CHP)	0.136
Total energy production	0.136
Net energy balance	-0.008

Table 16. Percentage of energy consumption of the equipment related to energy consumption 1 (as mentioned in 15, total measured consumption in 2022 was about 99 MWh per year.

Estimated percentage of use of consumption 1	
Combined Heat Power internal electricity consumption	13.2%
Three houses	9.1%
Other buildings & equipment (stables, milking robot, etc.)	27.3%
Manure collection and pre-treatment	1.9%
Co-substrate handling	8.9%
Digester mixer	2.2%
Screw press	3.1%
Nitrogen stripper/scrubber	18.2%
All pumps	6.9%
Additional / others	9.1%
TOTAL	100%

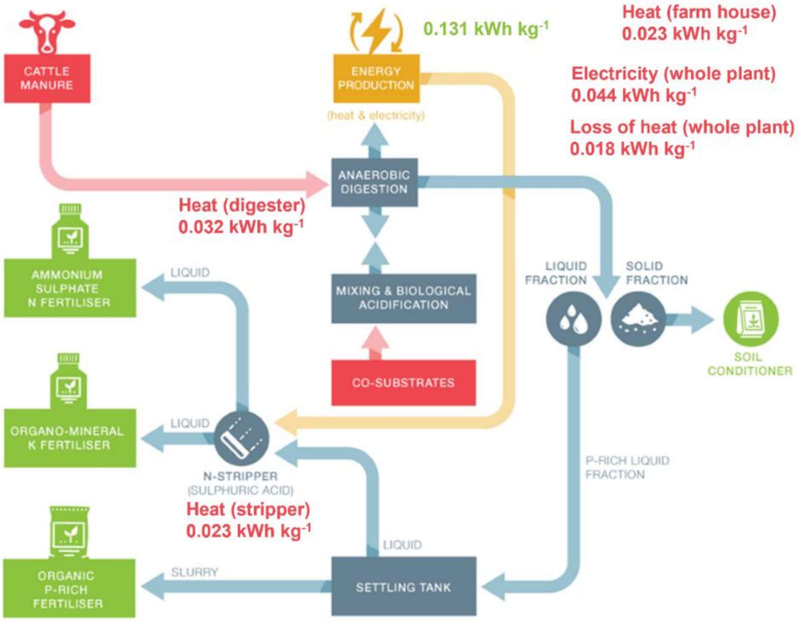


Figure *16*. Energy balance of the Dutch pilot APF; green numbers refer to energy production; red numbers refer to energy consumption which can be heat or electricity.

3.2.3. Dutch pilot: comparison with literature

The production of ammonium sulphate (AS) solution by the nitrogen scrubbing-stripping process is the most advanced technology in this Dutch pilot at APF. A detailed comparison with other systems and the literature is presented in section 3.4.3 for the Belgium pilot which operates a similar process. The APF AS contains on average 20.2% N dry matte basis (322 g dry matter/kg) and nihil amount C, P and K of about 0.1%. The recovery efficiency percentage for total N is 35% and 54% for NH4-N on fresh matter basis. This is in line with the recovery performance of the Belgium pilot plant with 32 - 36% of TN and 56 – 67 % of NH_4^+-N, and the presented comparable installations from literature. The energy consumption of the scrubber-pilot installation is partly measured and estimated at about 18.06 MWh per year for about 35% of the total throughput treated (about 2,000 tonnes). Assuming a 40 tonnes of AS production per year containing 6.52% N fresh matter, results in 2.61 tonnes N in the AS. Consequently, the energy consumption is 19 kW per kg N recovered, assuming only part of the digestate flow treated. This energy consumption is on the high side compared to the of Belgium (6.5 kW) and other installations presented. One explanation is that the stripper is in the end of the process train at APF with a potential reduction of 10 ºC since pipes and installations are not well isolated, which causes additional need of heat to get at 35-40 ºC for treatment in the stripper. See the tables of the Belgium pilot in this report for more information and specific comparison.

The other important separation process at APF is solid-liquid separation by the used screw press, commonly in use in the dairy livestock sector in the Netherlands. Like in the French pilot, the effect of the screw press is a distribution of elements according to their affinity to water or organic matter. Consequently, organic matter and phosphorus are relatively concentrating in the solid phase, while nitrogen and potassium concentrates in the liquid phase since soluble (Table 17). This table shows that separation efficiencies of C, N, P and K for screw press can vary largely between type of screw press and process configurations. The APF separation efficiencies for C, N, P and K are in line with the calculated average of the values based on the studies mentioned, for C fully similar, for N 13% lower, for P 36% higher and for K 21% higher than the average.

The energy use by the APF screw press is in higher than the energy uses presented by Cathcart et al. (2023). The APF energy use by the screw press separator for 1.5 m3/h treatment capacity (incl. conveyor belt and 2 pumps) is about 4.3 kW compared to on average 9.25 kWh (range 7-11) mentioned by Cathcart et al. (2023) for a separator with a capacity of 12.5 m3/h. Consequently, the APF electricity use per m^3 digestate is about 2.9 kWh/ton compared to on average 0.37 kWh/m^3 (range 0.28 - 0.44). The electricity uses per ton of solid fraction recovered for APF is about 16.8 kWh/ton compared to on average 9.46 kWh/ton (range 6.14 – 12.79). The alternative option of a decanter centrifuge can make higher separation efficiencies, but also higher investment and running costs, especially because of higher electricity uses (Cathcart et al. 2023). The higher energy use could maybe be explained by the fact that the APF screw press is relatively small with a low treatment capacity.

The Dutch pilot shows a unique combination (train) of manure digestion, separation and valorisation technologies, at this moment not often studied and especially not published in scientific literature. So further comparison with scientific literature is limited. Recently in the Netherlands digestion in combination with stripping got more attention and there are comparable set up rolled out and tested. The performance of APF in terms of nutrient separation and energy use efficiencies is in line with those examples.

Table 17. Comparison of the mass and nutrients balances of the screw press solid-liquid separation process at the Dutch pilot with literature as % recovery in solid fraction compared to ingoing material.

	C	N	P	K
Dutch pilot	35.1	13.6	30.9	9.2
Cathcart et al. 2023	25.6 - 27.1	8.3 (7.9 - 8.6)	9.6 (9.1 - 10.1)	2.8 - 4.1
Tambone et al. 2017	-	5.5 - 23.9	28.4 (17 - 54)	-
Fournel et al. 2019	55.1	21.0	30.9	15.3
Hjorth et al. 2010	-	18.6 (4 - 60)[1]	13.7 (3 – 28)[2]	-
Average	**35.1**	**15.5**	**22.7**	**7.6**

*Review of 7 studies/installations

**Review of 6 studies/installations

3.3. German pilot

3.3.1. German pilot: mass and nutrients balance

For the operation of the German pilot fresh cattle manure was mixed with citric acid (c = 50 wt%). The resulting mixture was dried and pelletized resulting in cattle manure pellets. Up until now the German pilot plant was operated three times under experimental conditions. Within these experimental campaigns 1500 kg of cattle manure pellets were processed. Due to unsatisfactory results in the area of ammonia recovery we had to take a step back from the pilot plant (process volume 30 kg/h) to a smaller plant (process volume 2 kg/h) in order to optimize the recovery of ammonia. For balance see Figure 17, 18 and 19.

Within a process of several experiments the medium perlite as a carrier material was abandoned since it absorbed not only ammonia but other compounds present within the TCR-gas. Another strategy was the scrubbing of the ammonia with phosphoric acid (85%). During this process we have learned that the phosphoric acid reacts with carbohydrates and tends to polymerize. We constantly reduced the concentration of the acid in order to find a reaction optimum. The optimum was found to be 4 mol/L.

In Table 18 all nutrients based on 100 kg of fresh cattle manure are given along the process chain. The values of the fresh manure originate from the analysis of 10 samples. Since 30 tons from two different farms of the manure were dried and pelletized the samples are only representative for a part of the overall sample. Since the values from the manure pellets deviate strongly in nitrogen, phosphorus and potassium content indicates that the overall manure sample had higher heterogeneity than initially expected. Therefore, for a meaningful evaluation of the BBF production process, the main focus is on the values of the cattle manure pellets as input.

Table 18. Summary of mass and nutrients balances in the German pilot (starting material = 100; n = number of observations).

	Mass (kg)	**C (kg)**	**N (kg)**	**P (kg)**	**K (kg)**
INPUT					
Cattle manure fresh	100 (n = 10)	11.80 ± 0.31	0.59 ± 0.10	0.03 ± 0.02	0.58 ± 0.12
Citric acid (c = 50 wt%)	2	0.04	0	0	0
Phosphoric acid (c = 100 wt%)	1.01	0	0	0.32	0
Total	103.01	11.84	0.59	0.35	0.58
OUTPUT					
Water	62.02	-	-	-	-
Cattle manure pellets	*36.48 (n = 8)*	*13.86 ± 0.17*	*0.84 ± 0.02*	*0.23 ± 0.03*	*1.17 ± 0.08*
Biochar	14.74 (n = 8)	7.67 ± 0.14	0.21 ± 0.02	0.22 ± 0.03	1.09 ± 0.23
Monoammonium-phosphate (MAP)	1.00 (n = 8)	0.00 ± 0.00	0.12 ± 0.002	0.27 ± 0.03	0.00 ± 0.00
TCR-Water	9.02 (n = 8)	0.22 ± 0.04	0.26 ± 0.04	-	-
TCR-Oil	1.46 (n = 8)	1.51 ± 0.03	0.12 ± 0.02	-	-
TCR-Gas	10.28 (n = 8)	2.26 ± 0.49	0.14 ± 0.11	-	-
Total (except of pellets)	101.01**	11.66	0.85	0.49	1.09
Balance (% of inital mass)	-1.9	-1.5 (-15.87)*	+44.0 (+1.2)*	+40.0 (-10.9)*	+87.0 (-6.8)*

*considering the pellets as input (please, see the text).

**pellets included.

A closer look at the nutrient flows reveals a discrepancy in the carbon contents between the fresh cattle manure and the cattle manure pellets (Figure 18). This may be due to the challenging process of sampling fresh cattle manure. Overall, 10 samples of the fresh manure were analysed. Compared to the 30 tons of manure that were processed this is a rather small amount and can only represent a section of the real conditions. The product distribution of the products from the catalytic reforming (TCR®) process is within the expected range and comparable with similar products from digestate (Neumann et al., 2015, Conti et al., 2017). The highest amount of carbon can be found in the biochar. Furthermore, the biochar contains all potassium and phosphorus introduced into the process by the feedstock. A closer look at the material flows associated with the contained nitrogen shows that 22 % can be found in the biochar and a non-negligible 31% is found in the condensed TCR water. A rather small amount of the input nitrogen is contained in the TCR-Oil. As expected, however, the TCR gas contains the largest proportion of nitrogen with 63%, which is used in the downstream process of the MAP reactor.

$$NH_3 + H_3PO_4 \rightarrow (NH_4)H_2PO_4 \quad \text{(Equation 1)}$$

The production of ammonium dihydrogen phosphate in industry is usually performed by passing dry ammonia gas through an 80 wt% phosphoric acid. (Charles Wittmann 70769 Ascension Parish La. Weston, patent DE3148423A1). Campaigns where the TCR-gas was scrubbed with concentrated phosphoric acid (85 wt%) showed that side reactions of the phosphoric acid with carbohydrates within the gas took place. Previous work, in which the pyrolysis gases passed through phosphoric acid impregnated perlite and silica gel showed other organic impurities like acetonitrile, pyrrole and acetone were detected on the perlite. In the presence of acid, the polymerisation of pyrrole can occur leading to the formation of various compounds such as porphyrin, which explains the red or rust-brown coloration of the obtained reacting solution. The side-reaction of pyrrole with acetone was performed. The emergence of the rust-brown product had confirmed the mentioned conjecture. Also, the acid solution would promote side reactions with the gaseous short-chain hydrocarbons, which mostly contained in the pyrolysis gases. (Bekker et al. 2009; de Klerk 2011) Their oligomerized products can be an explanation for the characteristic red-brown coloration and the fuel smell of the obtained reacting solution. A range of different compounds can be formed through the oligomerization of light olefins.

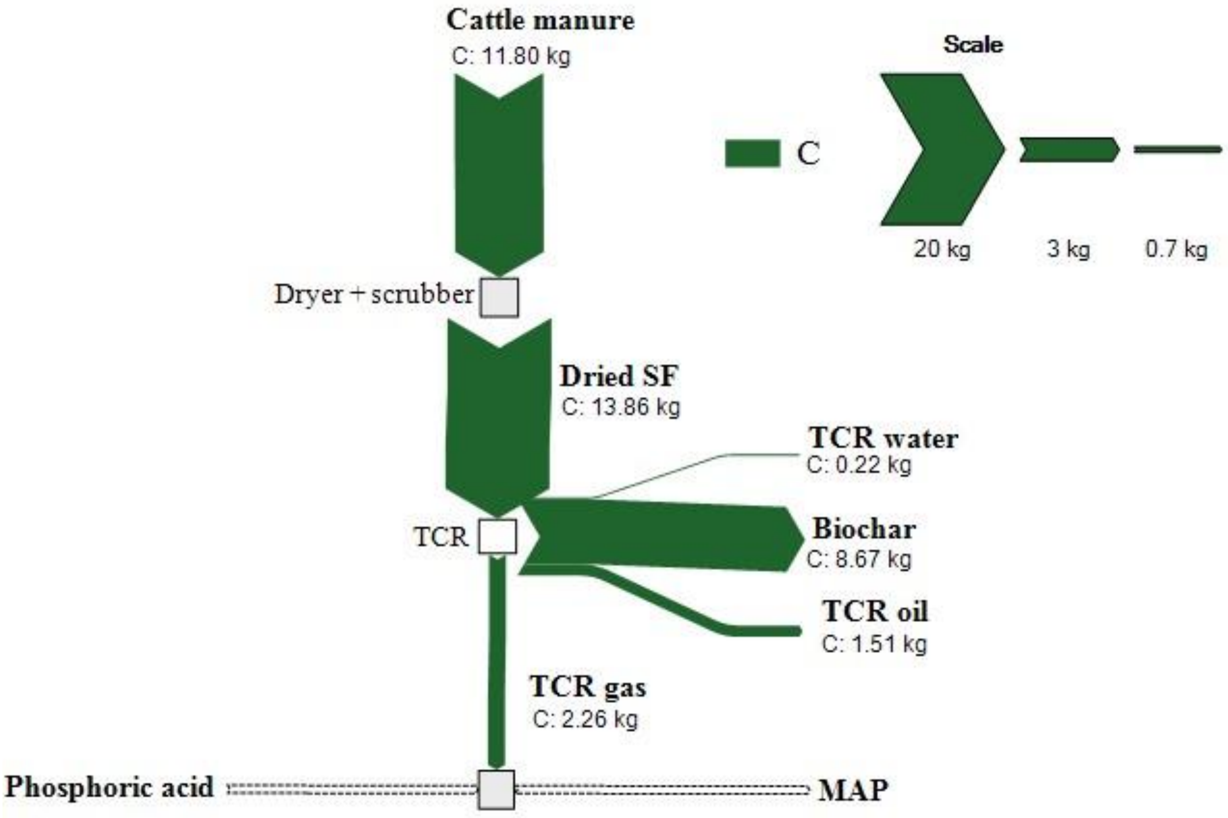


Figure 17. Mass balance in the German pilot

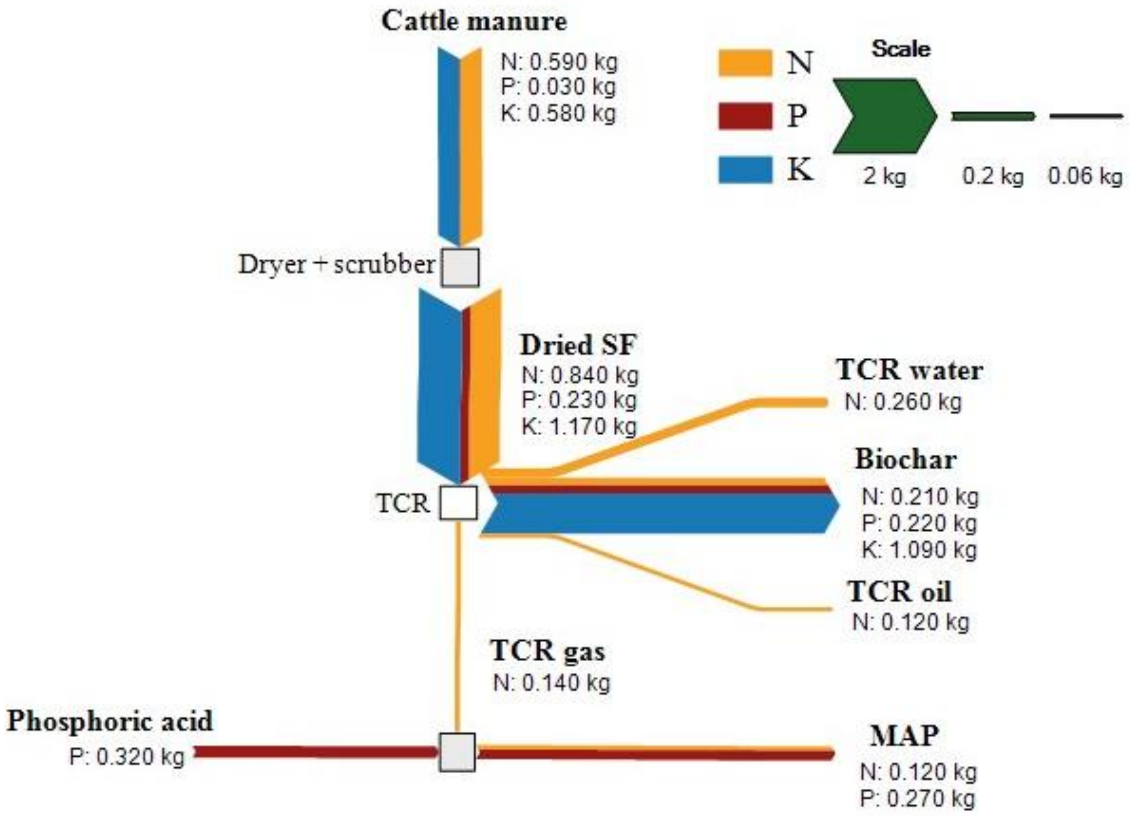


Figure 18. Nutrients balance in the German pilot

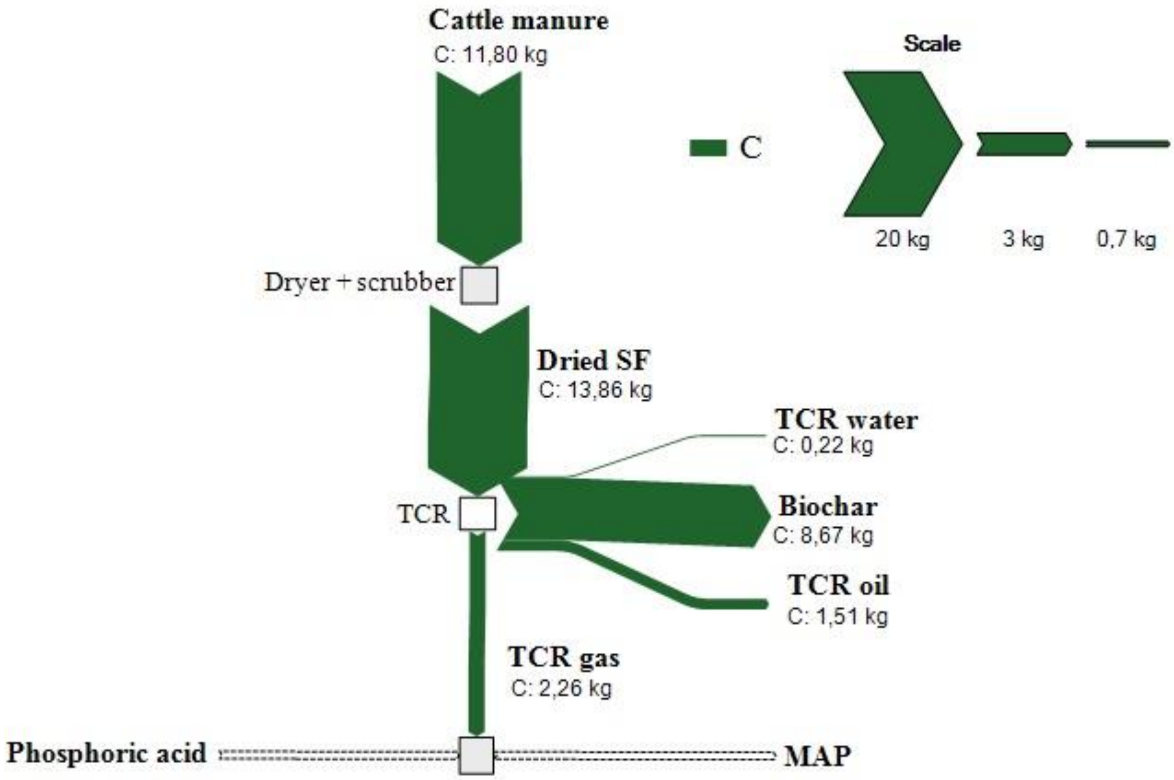


Figure 19. Carbon balance in the German pilot

3.3.2. German pilot: energy balance

The data of the energy balance is adjusted to the pilot scale although scrubbing experiments were performed on laboratory scale plant with a processing capacity of 2 kg/h. In terms of energy consumption, the scrubbing process can be easily transferred to the pilot scale since there is no large energy consumption within this step. Drying and pelletizing of the fresh manure was carried out in advance in a large scale with an external drying and pelletizing company. Therefore, these values are presented without standard deviation as reference values of the respective company were used for the calculations (Table 19, Figure 20).

Since the fresh cattle manure has, as average, a water content of 77% the drying step consumes most of the energy during the whole process. Furthermore, the energetic expenditure of pelletizing through the resulting values becomes apparent. The data given in Table 21 regarding drying was calculated with the help of general numbers from the Board of Trustees for Technology and Construction in Agriculture e.V. Since this is only a guideline, data was obtained from the agitator dryer manufacturer RHS. The use of advanced drying technology with higher efficiency allows the process to be more effective and lower in energy consumption. Pelletizing of the manure was carried out as a precaution to avoid possible blocking of the cattle manure in the reactor during the TCR® process. However, a variety of tests with other feedstocks shows that this energy-intensive step can be dispensed with in future for comparable projects. With that the process can be
Part of the whole process chain is a CHP (combined heat and power unit), which converts part of the resulting products from the TCR® process into electricity, generating heat at the same time. This energy can compensate for the heat required for the pretreatment of the cattle manure and thus has a positive effect on the overall process. Nevertheless, processing 1 ton of fresh manure requires 155.2 kWh of energy when standard values for drying and pelletizing are considered. Due to optimization within the drying process the number of needed energies would drop, and 284.3 kWh of energy could be gained. There is still potential of optimization within the TCR process regarding the optimization of the heat usage and losses from heat dissociation. A major advantage would be the elimination of the pelletizing process. This was done within the German pilot as a precaution to avoid blockage within the pyrolysis reactor. Future work should investigate if processing of the dried raw material is possible.

Table 19. Energy balance of the German pilot (kWh kg^{-1} of fresh treated material).

	Electrical Energy (kWh kg^{-1} treated material)	**Heat (kWh kg^{-1} treated material)**
Consumption 1 (drying)	0.0278* 0.0114 (RHS)	0.620* 0.1969 (RHS)
Consumption 2 (pelletizing)	0.0374	0.273
Consumption 3 (TCR-Process)	0.0374	0.1496
Consumption 4 (MAP-Reactor)	0.011**	0
Total consumption	0.1136/0.0972 (RHS)	1.043/0.6199 (RHS)
Production 1 (MAP-Reactor)	0	0
Production 3 (TCR-Reactor)	0	0.1818
Production 2 (CHP enginge)	0.2288	0.5316
Total production	0.288	0.7134
Net energy	-0.1552/+0.2843 (RHS)	

*theoretical data for evaporation of amount of water that was removed with data from The Board of Trustees for Technology and Construction in Agriculture e.V..
**energy refers to the evaporation of all water from the scrubbing solution (2.57 L)
RHS = provider of agitator dryer (data from sample calculation of digestate with Rhino 7000, RHINO® Industrie – RHS Tech (rhs-tech.de)

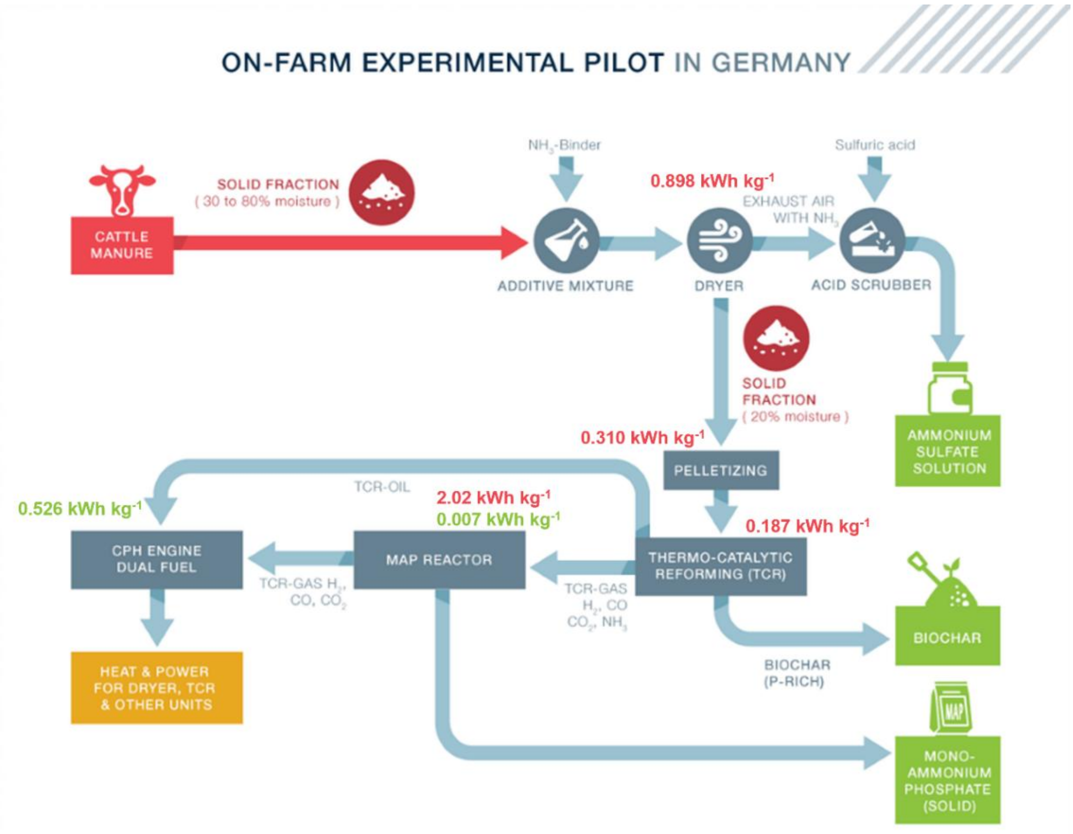


Figure 20. Energy balance of the German pilot. Green numbers refer to energy production; Red numbers refer to energy consumption.

3.3.3. German pilot: comparison with literature

The German pilot plant utilizes intermediate pyrolysis for the treatment of cattle manure, a process designed to balance both the advantages of fast and slow pyrolysis. Pyrolysis is a thermochemical decomposition of organic material at elevated temperatures in the absence of oxygen, resulting in the production of biochar, bio-oil, and syngas. The distinctiveness of the German pilot lies in its intermediate pyrolysis mode, operating at moderate heating rates and temperatures between 500-600°C, designed to optimize the yield of both biochar and syngas while maintaining energy efficiency. This contrasts with slow pyrolysis, which operates at lower temperatures (~300-500°C) with long residence times, primarily maximizing biochar yield, and fast pyrolysis, which uses higher temperatures (~600-1000°C) with short residence times to primarily produce bio-oil.

In comparison, slow pyrolysis produces large amounts of biochar (30-50% of biomass input) but limited syngas and bio-oil. This method is useful when the primary goal is carbon sequestration or soil amendment. Fast pyrolysis, on the other hand, rapidly heats the biomass to generate a higher yield of bio-oil (~60-75%) while sacrificing biochar production. This method is more suitable for liquid fuel generation but is more energy-intensive due to the required high temperatures and rapid quenching of bio-oil. Studies have emphasized the use of pyrolysis to decarbonize gas infrastructures and manage livestock waste, providing a broader understanding of manure-to-energy conversion technologies (Mehta et al., 2022).

Intermediate pyrolysis in the German pilot achieves a balance by optimizing the distribution of biochar, bio-oil, and syngas production. The biochar produced has high nutrient content, particularly phosphorus and potassium, making it suitable for use as a bio-based fertilizer (Rout et al., 2022). Additionally, the syngas generated is reformed in a unique post-reforming stage that distinguishes this pilot from other pyrolysis plants. The post-reforming unit further treats pyrolysis gases, converting them into a cleaner syngas suitable for energy production and for the recovery of ammonia through a monoammonium phosphate (MAP) crystallization process. This added step increases the process efficiency by enhancing the quality of the syngas and recovering valuable nitrogen-based products, a method consistent with other manure conversion studies, such as hydrothermal carbonization combined with digestion for nutrient recovery (González et al., 2021).

Overall, the intermediate pyrolysis employed in the German pilot, with its focus on syngas reformation, offers a more versatile and efficient approach than traditional slow or fast pyrolysis methods. By balancing energy recovery, biochar production, and nutrient recovery, the German pilot demonstrates a sustainable path for biomass conversion technologies (Song et al., 2022).

The best option to compare the German pilot plant within literature is a rotary kiln system (Figure 21). The German pilot plant other than that system has an auger reactor and was extended by a post-reforming module and a scrubbing system to treat the pyrolysis gases. The base of the system has been described in literature before (Santos, 2019). Prior processing the feedstock needs to be dried to avoid blocking of the reactor and ensure sufficient conversion of the biomass. This is common for pyrolysis plants of this kind since the biochar from biomass that was dried may have higher nutrient content then the one from solid-liquid separation which is also an option for producing biomass with the right water content (Rathnayake, 2023).

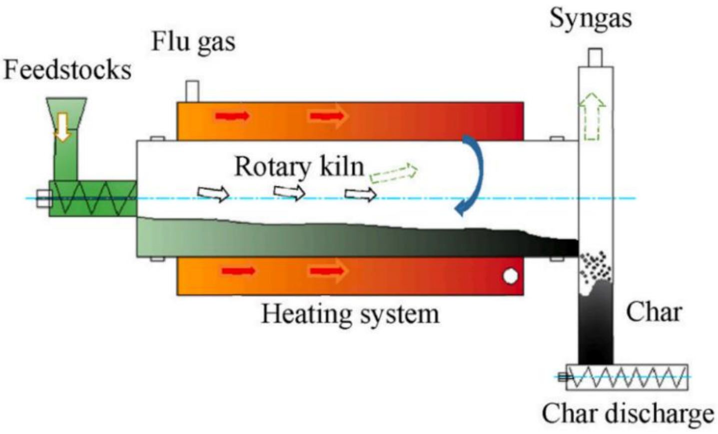


Figure 21. Schematic diagram of a rotary kiln slow pyrolysis system (Cong et al., 2022).

The mass- and energy balance strongly depends on the feedstock and the process parameters. For a rotary kiln system Cong et al. give an expected char yield of 30 wt%, 5 wt% of oil and 340 m^3 of gas by kg of raw material (Cong et al., 2022) For the treatment of wheat husk the system used within the German pilot plant produced 21.7 wt% biochar, 5.8 wt% of oil and 29.6 wt% of gas (Santos, 2019). The treatment of cattle manure produced 40 wt% biochar, 5.6 wt% Oil and 28 wt% of gas. In comparison with the chosen process parameters and the given feedstock the biochar production of the German pilot plant is higher but within a range expected for biochar from cattle manure (Rathnayake, 2023). Given the high diversity of manure-based feedstocks and pyrolysis parameters a comparison of the nutrient flows of the German pilot plant with other systems is challenging. As mentioned before the char production is within the expected range for this type of pyrolysis. Also, the elemental composition with regards to the main nutrients C, N, K and P is within the expected range (Table 20). The biochar from the German plant sticks out with high potassium contents. This can be explained by different diets of the animals or composition of the manure itself. For a stable production in char quality other feedstocks low in nutrients could be co-fed to produce a char with a constant concentration of nutrients.

Table 20. Comparison of the nutrient contribution within the received biochar from the German pilot plant.

	Recovery (g kg^{-1} d.m.; ‰ d.m.)*			
	C	N	P	K
DE-BC	520	14.3	14.7	74.2
Cantrell et al. (700°C)	567	15.1	16.9	23.1
Rathnayake et al. 2023 (700°C)	400-600	11.0	5-20	5-30
Qin et al. 2019 (600°C)	385	14.5	-	-
Yue et al. 2017(700°C)	528	10.6	0.16	4.4

*d.m.: dry matter

Unique about the German pilot plant is the integrated recovery of nitrogen from the gas stream. All other systems within the literature directly process the gas from pyrolysis for energy production. Within the German plant the gas is scrubbed with a solution of phosphoric acid. At the beginning of the project a packed bed reactor was designed for the recovery of nitrogen from the gas. Since side reactions with the phosphoric acid impregnated perlite took place, this approach had to be discarded. Similar side reactions were observed in solution when scrubbing with concentrated phosphoric acid (85 wt%) (Figure 22).

Figure 22. Brown colour and precipitate formed during scrubbing with concentrated phosphoric acid (85 wt%).

According to laboratory experiments of Dong et al. (2018) the highest absorption efficiency within a stripping process (98.7%) was at a phosphoric acid concentration of 4.2 M. The improvement of the ammonia absorption has been shown by increasing the reacting (absorbing) volume instead of increasing its concentration. Therefore, a concentration of 4 mol/L for the scrubbing was chosen within the MAP reactor. From the nitrogen present within the TCR-Gas about 85% can be recovered as mono ammonium phosphate (MAP) using scrubbing followed by crystallization (Figure 23).

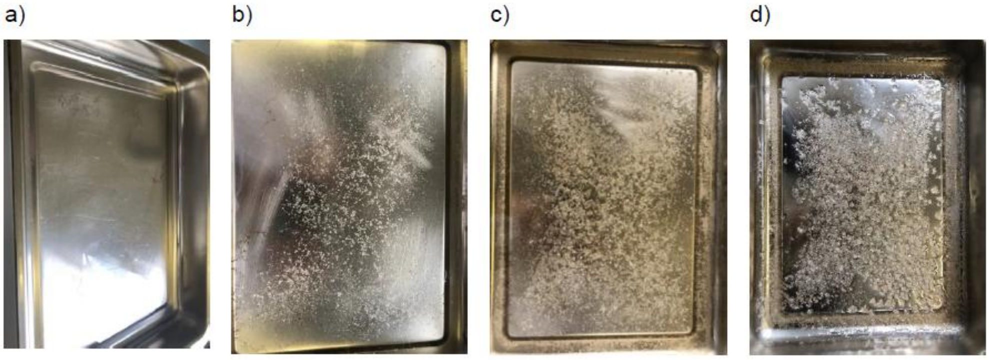


Figure 23. Crystal growth (a) initial, (b) after 5 min (c) after 10 min and (d) overnight.

With the optimised recovery of MAP through scrubbing with diluted phosphoric acid (4 mol/L) the German pilot can not only produce biochar as biobased fertiliser but also MAP within one process. With the results from the small plant, we are now able to adapt the scrubbing process to the initial pilot scale. For a bigger gas flow, a combination of a spray absorber and a packed column would be ideal to insure effective recovery (Figure 24, 25) (Santoleri, 2003). Within such an installation continuous contact between the gas and the scrubbing agent is ensured. A unit with a sufficient scrubbing capacity was designed to be integrated in the initial pilot plant with higher capacity. The proposed scrubber will be produced and integrated into the pilot plant to efficiently recover ammonia from pyrolysis gases in the future. Incorporated within the scrubber will be a pH measurement system and a storage reservoir for acids so that continuous operation is possible.

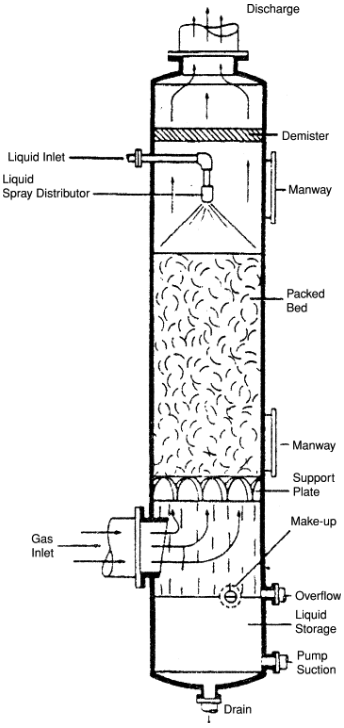


Figure 24. Schematic drawing of a packed-bed scrubber (Santoleri 2003)

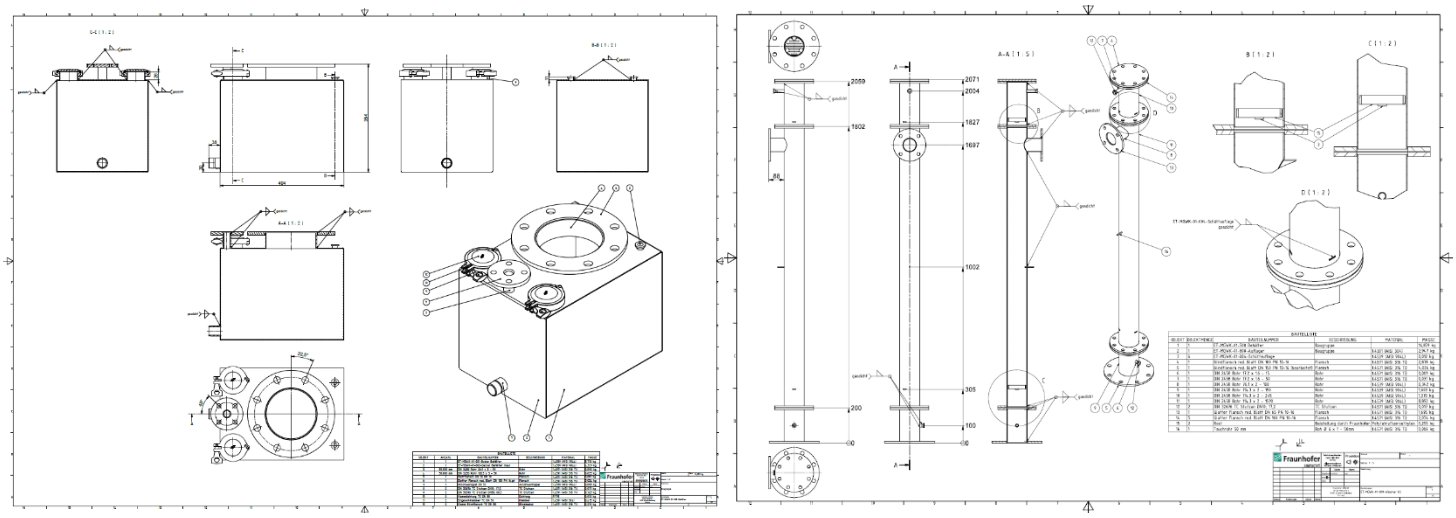


Figure *25*. Technical drawing for a possible pilot scale scrubber.

Comparison of the energy balance with literature

Since there is no specific system that brings together all the parts from the German plant a comparison of the pyrolysis process with other systems will be done. PYREG is a German manufacturer of carbonization plants for upcycling organic waste into high-quality CO_2-sequestering biochar (vegetable carbon) and regenerative heat. In Literature (Brokmeier, 2022) and on their company page there are numbers regarding the energy consumption and production given (Pyreg 2023). Biomacon is a manufacturer of CO_2 negative biomass boilers. Their pyrolysis-based boilers produce heat and bio coal. In Table 21 specific data provided by the companies regarding energy usage and production is listed.

Table 21. Specific data of the pyrolysis process provided by Pyreg and Biomacon.

	Annual throughput (t)	Annual operation (h)	Power consumption kW	Annual excess heat energy kWh	Excess heat power (kW)
Pyreg 500	1070	7.500	16	1.125.000	150
Pyreg PX system data best case	3.300.000	7.500	48	20.000.000	600
Pyreg PX system data worst case	3.300.000	7.500	48	4.800.000	170
Biomacon C500-I	23.600	8.000	16	No data	No data

Based on the given data the consumption and production of energy for the systems was calculated (Table 22). The Pyreg data set is based on wood chips. For the Pyreg PX system which can processes sludge two cases were calculated based on their information that an amount of 170 kW_{th} (worst case) up to 600 kW_{th} (best case) can be used.

Table 22. Comparison of the energy balance of the TCR-unit of the German pilot with literature.

	Energy (kWh/kg raw slurry)		
	Total consumption	**Total production**	**Net energy**
German pilot (cattle manure)	0.1874	0.94	0.76
Pyreg 500	0.1121	1.05	0.94
Pyreg PX system data worst case	0.1090	0.3863	0.28
Pyreg PX system data best case	0.1090	1.3636	1.25
Biomacon C500-I	0.054	No data	No data

The German TCR (thermo catalytic reforming) unit produces 0.76 $kWh \cdot kg^{-1}$ raw slurry. When compared with the installation of Pyreg and Biomacon it is apparent, that the total consumption of energy in the beginning is higher. It must be considered that the amount of energy is dependent on the used feedstock and the respective water content. For the Pyreg and Biomacon system data wood chips with a dry matter content of 80% were used. Compared to the cattle manure pellets wood chips have a higher calorific value then residue-based feedstocks like cattle manure. Therefore, the data can only be compared with each other to a limited extent. Nevertheless, are the values coming from the German system within a range expected for a pyrolysis plant. The optimized production with wood chips generates more energy than the German pilot. On the website it is stated that from 170 kW_{th} up to 600 kW_{th} of the excess heat can be used for drying, heating or power generation. With an excess heat production of 170 kW_{th} the energy production titled as worst case is with 0.28 kWh/kg raw slurry lower than the production within the German system. In fact, the German production is exactly between the best and worst production of the Pyreg PX system with 0.76 $kWh \cdot kg^{-1}$ raw slurry. The TCR process of the German system allows the recovery of ammonia from the pyrolysis gas and the condensation of biooil that can be either used within a CHP engine or used within the petrochemical industry.

3.4. Belgian pilot

3.4.1. Belgian pilot: mass and nutrients balance

As mentioned in section 2.4, the new Belgian pilot is located at Bio Sterco pig farm. Prior to pilot's installation at Bio Sterco, UGENT and DETRICON performed optimization tests with nitric acid as scrubber sorbent (reported in the deliverable 2.3). After finalization of the optimization tests, the pilot unit was delivered and installed at Bio Sterco pig farm in February 2022. The pilot was monitored in two phases from February 2022 till March 2023. During the months from March 2022 till May 2022, nitric acid was used as scrubbing sorbent, while during the other months sulphuric acid was used as a scrubber sorbent.

The Belgian pilot was monitored for approximately 1 year, allowing to draw a mass balance for the mentioned sorbents. The stripping was performed by pH between 8-9 and a temperature ranging from 45 to 52 °C. An overview of the overall nutrient balance can be seen in Table *23*, while Table 24, Table *25* and Figure *27* show the summary of mass and nutrients balances of the Belgian pilot with sulphuric acid and nitric acid as a scrubber sorbent, respectively.

Raw manure was first separated in a liquid fraction (LF) and solid fraction (SF) by the centrifuge unit, where the majority of P was recovered in the SF with a separation efficiency above 85 %. The high separation efficiencies as compared to literature found in our study were due to polymer addition (cationic polyacrylamide) which induces coagulation between the solids in combination with the most efficient swine slurry separation technology (i.e. centrifugation) yields elevated separation efficiencies (Hjorth et al., 2010). The majority of N was found in the LF as separation efficiencies for N did not exceed 35%. After separation, the solid fraction was exported to nutrient-poor regions in France.

The LF treatment was more complex, on average 25% of the total input was processed in the NH_3 stripping-scrubbing unit, where a counter-airflow captures the NH_3 of the liquid phase. The recirculation gas - rich in NH_3 was sent over a NH_3 absorber containing 0.4 t HNO_3 diluted in water to produce 0.6 t ammonium nitrate (AN) solution (15% of N) in the configuration with nitric acid as sorbent or 0.2 t H_2SO_4 diluted in water to produce 0.7 t d^{-1} of ammonium sulphate (AS) solution (7% of N) in the configuration with sulphuric acid as sorbent. Between 32% (nitric acid) and 36% (sulphuric acid) of total nitrogen (TN) contained in the stripper influent was recovered as fertilizer suspension depending on the counter acid used, corresponding to an NH_4–N recovery efficiency of 56 % (nitric acid) and 57 % (sulphuric acid). The N-poor stripped LF (circa 25 kg) is then mixed with about 55 kg of non-stripped LF and biologically treated through a nitrification-denitrification (NDN) system.

Table *23*. Summary of mass and nutrients balances of the Belgian pilot with sulphuric acid as a scrubber solution (starting material = 100 kg, n= number of observations).

	Mass (kg)	C (kg)	N (kg)	P (kg)	K (kg)
			INPUT		
Pig manure	100 (n=12)	3.24 (n=12)	0.71 (n=12)	0.17 (n=12)	0.45 (n=12)
Sulphuric acid	0.4 (n=13)	0.00	0.00	0.00	0.00
Water	0.4 (n=12)	0.00	0.00	0.00	0.00
$FeCl_3$	0.3	0.00	0.00	0.00	0.00
Total	101.1	3.24	0.71	0.17	0.45
			OUTPUT		
SF of manure	19,1	1.18	0.26	0.15	0,09
Discharge effluent	77.8 (n=12)	0.00 (n=12)	0.00 (n=12)	0.00 (n=12)	0.27 (n=12)
Ammonium sulphate	0.7 (n=13)	0.00 (n=4)	0.05 (n=13)	0.00 (n=13)	0.00 (n=13)
P sludge	3,1	0.0	0.00	0.03	0,01
Total	100.7	1.19	0.31	0.18	0.37
Balance (% initial mass)	100.4	36.7	43.6	105.9	82.2

Table 24. Summary of mass and nutrients balances of the Belgian pilot with nitric acid as a scrubber solution (starting material = 100 kg; n= number of observations).

	Mass (kg)	C (kg)	N (kg)	P (kg)	K (kg)
INPUT					
Pig manure	100 (n=12)	3.24 (n=4)	0.71 (n=12)	0.17 (n=12)	0.45 (n=12)
Sulphuric acid	0.2	0.00	0.00	0.00	0.00
Nitric acid	0.4 (n=3)	0.00	0.05	0.00	0.00
Water	0.4 (n=3)	0.00	0.00	0.00	0.00
$FeCl_3$	0.3 (n=12)	0.00	0.00	0.00	0.00
Total	101.1	3.24	0.76	0.17	0.45
OUTPUT					
SF of manure	19,1 (n=12)	1.18	0.26	0.15	0,09
Discharge effluent	77.8 (n=12)	0.00 (n=2)	0.00 (n=12)	0.00 (n=12)	0.27 (n=12)
Ammonium nitrate	0.6 (n=12)	0.00 (n=2)	0.11 (n=12)	0.00 (n=12)	0.00 (n=12)
P sludge	3,1 (n=12)	0.00 (n=4)	0.00 (n=12)	0.03 (n=12)	0,01 (n=12)
Total	100,7	1.19	0.37	0.18	0.37
Balance (% initial mass)	100.4	36.7	48.7	105.9	82.2

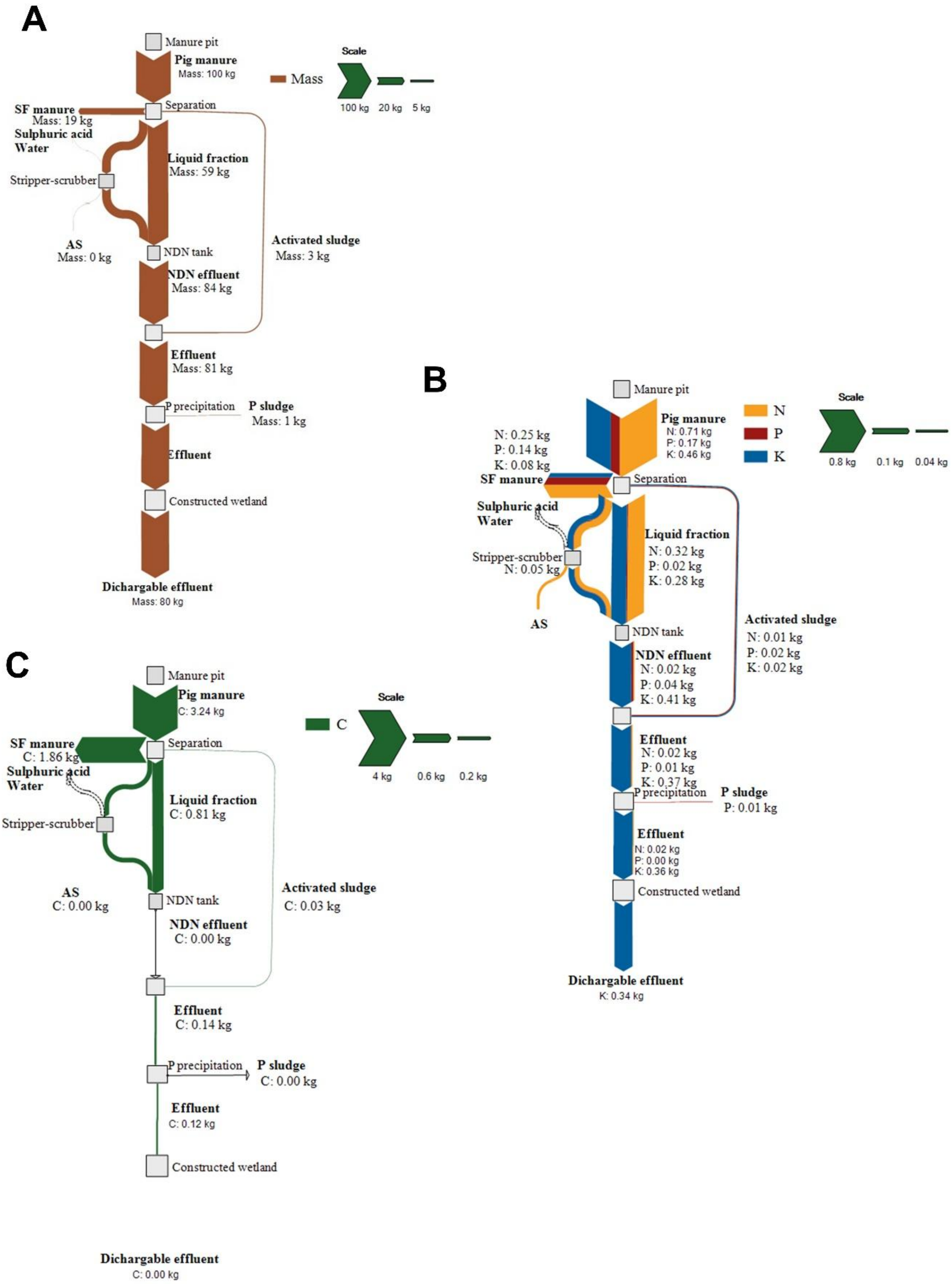


Figure 26. Mass and nutrients balance in the Belgian pilot with sulphuric acid

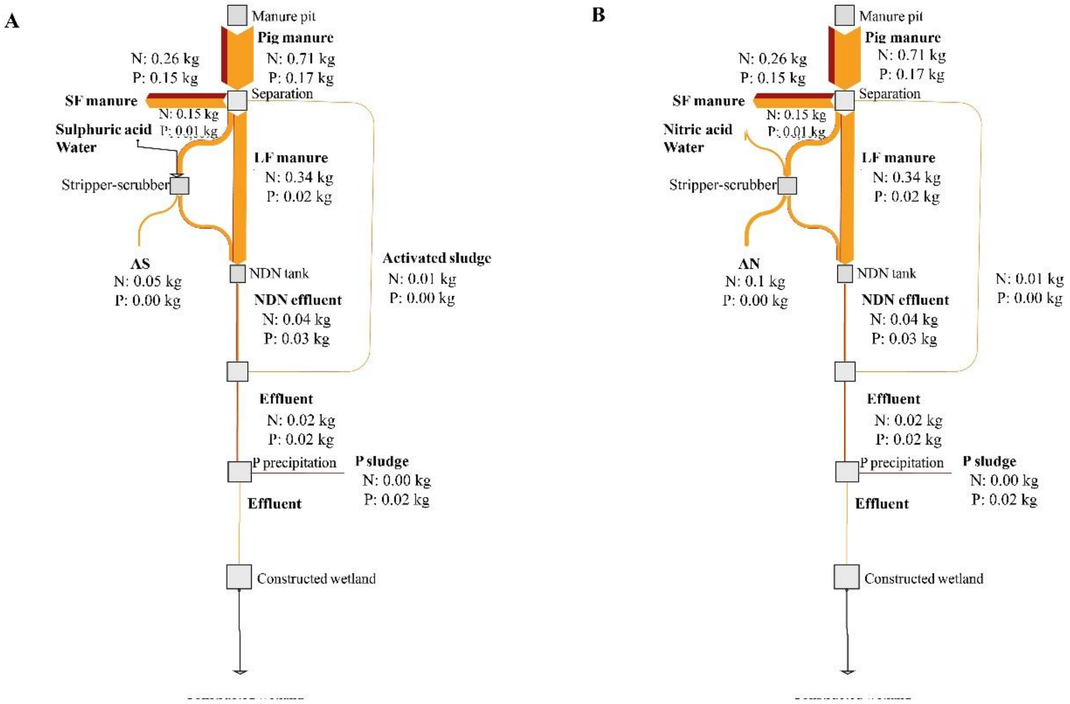


Figure *27*. N and P balance in the Belgian pilot with sulphuric acid (A) and nitric acid (B) as scrubber solution.

The NDN unit was fed with the remaining N of the LF which equaled on average 508 kg N d^{-1} of TN, of which 59 % was present in the form of NH4–N and 41 % in the form of organic nitrogen (org-N) and achieved a N removal efficiency of 92% resulting in an effluent containing $4*10^{-5}$ kg N and $3*10^{-5}$ kg. The NDN effluent was sent to the settling tank where 4 m-1 d-1 activated sludge is removed from the effluent and mixed brought back to the centrifuge. The effluent was further polished. In the polishing step, phosphorus precipitation is induced in the effluent by adding 4.5 H_2SO_4 (55%) and 3.5 l of $FeCl_3$ (40%) per ton effluent before being fed to the constructed wetland. Phosphorus was mostly recovered in the P sludge (0.03 kg P), while TN, mainly in the form of NH_4–N and NO_3-N, was associated with effluent proceeding to the constructed wetland (0.03 kg N). The constructed wetland further removed N, P, BOD and COD by plant uptake, microbiological degradation, and sedimentation, resulting in the effluent meeting surface water discharge criteria (15 mg l^{-1} for N, 1 mg l^{-1} for P, for 250 mg l^{-1} COD, 25 mg l^{-1} for BOD and 35 mg l^{-1} for SS) (VCM, 2021).

3.4.2. Belgian pilot: energy balance

The energy consumption rate of the pilot installation was measured during the monitoring period at the Bio Sterco farm (Table 25).

Table *25*. Energy balance of Belgian pilot (kWh t^{-1} of fresh treated material).

	Energy (kWh $tonne^{-1}$ treated material)
Separation	1.9
NH_3 stripping-scrubbing	3.1
Thermal	1.7
Non-thermal	1.4
NDN tank and settler	11.4
P precipitation and constructed wetland	0.3
Total consumption	16.7
Total production	0
Net energy	16.7

The electricity required by the manure processing equaled 16.7 kWh t^{-1} raw manure depending on the configuration used. Manure separation required an energy input of about 2 kWh t^{-1} raw manure treated. When installing a stripper-scrubber before the NDN treatment, the costs by oxidation requirement decreased to 11.4 kWh t^{-1} raw manure. However, N recovery by the NH_3 stripping-scrubbing unit has an electrical energy requirement of 12.4 kWh t^{-1} LF manure stripped, which equals 3.0 kWh t^{-1} raw manure treated. Phosphorus precipitation and constructed wetlands consumed about 0.3 kWh t^{-1} raw manure treated.

3.4.3. Belgium pilot: comparison with literature

A) Separation

The centrifuge unit first separated raw manure into LF and SF, recovering the majority of P in the SF with a separation efficiency above 85% for all configurations. A review of 11 studies evaluating mechanical S/L separation technologies used in manure treatment by Lyons et al. (2021) found that the P separation efficiencies for a decanter centrifuge without chemical addition varied between 30–91%, while screw presses recorded a P separation efficiency of only 4–34%. Polymer addition (cationic polyacrylamide), inducing coagulation between solids, along with the most efficient swine slurry S/L separation technology (i.e. centrifugation), resulted in elevated separation efficiencies compared to the literature findings in our study (Hjorth et al., 2010).

B) NH_3 Stripping-scrubbing

NH_4^+–N recovery efficiencies were found to be between 56 and 57% depending on counter acid used. The decrease in NH_4^+ content and NH_4^+-N:TN ratio was less significant for the configuration with HNO_3 as scrubbing agent. Because of the lower density of HNO_3 compared to H_2SO_4, the rate of ventilation flow had to be reduced to prevent HNO_3 from being carried with the ventilation air from the scrubbing unit to the stripping unit which would acidify the LF of manure and reduce stripping efficiency. However, the reduced air flow rate hampers the stripping efficiency because it has a strong impact on mass transfer coefficient, mixing, and gas-liquid interfacial area, which are important parameters determining gas-liquid transfer rate of NH_3. For example, Liu et al. (2015) showed that increasing the air flow rate from 60 m^3 h^{-1} m^{-3} to 840 m^3 h^{-1} m^{-3} enhances the hourly stripping efficiency from 8.6 to 86.4% when performing a stripping experiment on pig urine at a temperature of 50°C and an increased pH of 10. However, further increasing the air flow rate above 600 m^3 h^{-1} m^3 showed reduced benefits on NH_3 removal rates.

A considerable number of experiments to assess the efficacy of the implementation of NH_3 stripping-scrubbing technology to recover N from (digested) LF of pig manure has been performed at laboratory, pilot and full-scale over the last decades. Brienza et al. (2023) recovered on average 22% of N in the form of AN (81 g kg^{-1} TN) when stripping LF of digestate at ambient temperature and low pH (8.0) in a pilot-scale installation for 2 hours. A range of temperatures (55-65°C) were tested in series of trials by Pintucci et al. (2017) for a low pH (7.8) and found NH3 removal rates ranging from 28 to 46%. Baldi et al. (2018) achieved an high removal efficiency of 62% NH_4^+ in a stripping experiment on digestate by subjecting it to a temperature of 48°C and pH of 9.5 over a period of 2 hours, while Bolzonella et al. (2018) recovered 22% of TN in the form of $(NH_4)_2SO_4$ solution (26 g kg^{-1} TN) when stripping a mixture of digested swine and cow manure at pilot scale. As compared to other studies, the efficiency found in our study (32 - 36% of TN and 56 – 67 % of NH_4^+-N) was at the higher end of the spectrum as it was performed at both high pH (8.5 – 8.6) and temperature (50 °C). Also, high N concentrations in the NH_4^+ solutions were achieved in our study compared to literature results.

Despite the NH_4^+ solutions recovered less than 1% of the COD embedded in the influent LF, a reduction in of 8% in the COD content of LF was found during the stripping-scrubbing process. This trend was also described by Brienza et al. (2023) who found a loss in COD content of 13% when HNO_3 was used as absorbent, and Bonmati and Flotats (2003) who recorded COD losses above 5% when using H_2SO_4 as scrubber acid. Therefore, it can be assumed that HNO_3 nor H_2SO_4 are not able to absorb volatile organics which stress the need for additional air treatment to reduce impact on the environment.

C) Nitrification/denitrification tank

Corbala-Robles et al. (2018) reported on a typical wastewater treatment plant treating the LF of pig manure after centrifugation in Flanders with a tank volume of 2846 m^3 which could process on average 58 m^3 LF manure per day with an associated N loading rate of 0.09 kg N m^3 d^{-1}. Smet et al. (2003) evaluated 14 different biological manure treatment installations according to the Trevi concept with a total treatment capacity of 300,000 m^3 y^{-1} and found that the operation of the biological treatment installations allows a loading rate of 0.13 kg N m^3 d^{-1}. The average loading rate sustained by the NDN tank during configuration 1 in our study equalled 0.14 kg N m^{3}d^{-1} and is thus similar as the values found in literature. A review of 25 studies by Skouteris et al. (2020) showed that the replacement of ambient air by PO as aeration agent could enhance the treatment capacity by increasing the oxygen transfer rate through an elevated partial pressure of oxygen, especially for high strength wastewaters with high Mixed Liquor Suspended Solids (MLSS). Similar conclusions were yielded by Rodríguez et al. (2012) who compared the use of PO and air on the nitrification rate in a pilot-scale MBR system used for wastewater treatment and found that aeration by PO could enhance the nitrification rate with 8-13%. As the nitrification rate increases, it effectively diminishes the NH_4^+ concentration within the NDN tank, preventing the accumulation of NH_4^+ and thereby mitigating the inhibitory effects of FA. This inhibition generally impedes the entirety of the nitrification process when FA concentrations exceed 150 mg N l^{-1} (Elawwad, A. 2018). Conversely, the inhibition of NO_2^- oxidation commences at FA levels surpassing 2.8 mg N l^{-1} (Jubany et al. 2008). Hawkins et al. (2010) conducted a comprehensive review of 15 studies, revealing that the FA concentration threshold at which NO_2^- oxidation begins to be inhibited varies significantly across studies, yet predominantly initiates at FA concentrations of 2-3 mg N l^{-1}. This finding is consistent with observations of approximately 160 mg l^{-1}of total NH_4^+-N present in the NDN tank under standard operational conditions.

These findings support the increased processing capacity found for configuration 2. When the LF was partially pretreated by NH_3 stripping-scrubbing, an increased volume of LF could be processed by the NDN tank. However, only a slight difference was found in processed N load between configuration 2 and 3, which could be due to the more favorable COD:N ratio (Phanwilai et al., 2020). Lower COD concentrations were found for configurations 1 and 2 as compared to configuration 3 due to the higher COD loading rates of configuration 3. In addition to augmenting the treatment capacity of the NDN system, N recovery by stripping-scrubbing in configuration 3 has the potential to decrease N_2O emissions per m^3 of LF treated. Specifically, the N_2O emissions are estimated to be 0.057 kg N_2O-N in configuration 3, compared to 0.063 kg N_2O-N per m^3 of LF treated in configuration 1, assuming N_2O emissions account for 1.1% of the N load in the NDN (de Haas and Andrews, 2022), The conventional N_2O emission factors are applicable on concentrated piggery wastewaters (Ravi et al., 2023). However, the reduction in NO_2^- accumulation in configuration 3 is expected to contribute to an even more pronounced decrease in N_2O emissions, given the strong association between NO_2^- accumulation and N_2O emission rates (Van Hulle et al., 2012).

D) P precipitation and constructed wetland

Through the acidification of the NDN effluent in our study, high P removal efficiencies could be achieved for low FeCl3 dosages which is crucial to meet the stringent local Cl discharge limits of 1000 mg Cl- l-1 (VCM, 2021). As Meers et al. (2006) only achieved a removal efficiency of 39, 88 and 95% of P when applying 1, 3 and 5 l of FeCl3 to LF of pig manure treated by NDN with a P content of 332 mg P l-1, decreasing pH below 8 by the addition of H2SO4 is crucial to increase the P removal rates for similar FeCl3 dosage.

The removal rates found in the CW were in accordance with literature. Meers et al. (2006) conducted bench experiment with a corresponding loading rate of 0.23 g N m−2 d−1, 0.27 g P m−2 d−1 and 3.2 g COD m−2 d−1 and achieved removal efficiencies between 73%–83% for N, 71% - 98% for P and 64 – 75% for COD, while Meers et al. (2008) reported on a CW of 4500 ha that could sustain a nutrient load of 0.75-1.22 g N m−2 d−1 and 0.04 g P m−2 d−1 with a removal efficiency of 96% and 99%, respectively. Lee et al. (2014) loaded 0.84 g N m−2 d−1 into a CW (4492 ha) in the form of piggery effluent and achieved a removal efficiency of 55% N.

The plant uptake was estimated by the crop cut method (Sapkota et al. 2016) to account for 17 % of N and 26% of P removal, which is considerably lower than the values found by Meers et al. (2006), Meers et al. (2008) and Lee et al. (2014) as the biomass yield remained under expectations. However, COD, BOD and N were mainly removed by microbiological degradation and denitrification processes resulting in C and N losses to the atmosphere as CO2 and N2, while the remainder of the P removal is attributed to sorption by substrate and sedimentation. Due to the finite capacity of these P removal mechanisms, sorption and sedimentation will not sustain long-term P removal (Meers et al., 2008). Therefore, it is crucial to establish a correlation between the loading rates of P and its uptake by the biomass that can be harvested to maintain the long-term effectiveness of a system in removing P. By reducing the P load through the introduction of a P precipitation before the effluent enters the CW, an elevated amount of effluent can be treated without compromising the sustainable operation of the CW.

E) Energy requirement

Similar energy requirements for centrifugal S/L separation (2 kwh t^{-1}) were reported by Willeghems et al. (2016), which are considerably higher than S/L separation by a screw press. Tampio et al. (2016) provided a review study reporting an energy requirement between 0.8 and 28 kWh kg^{-1} N for N recovery through NH_3 stripping-scrubbing of a wide range of substrates including manure, digestate and urine. As circa 2 kg N t^{-1} LF manure is recovered by the stripping-scrubbing unit in our study, the energy requirement for AS and AN production are in accordance with data provided by literature. According to Brienza et al. (2023), the energy consumption of the stripping-scrubbing unit ranged between 6.4-13.6 kWh kg^{-1} N recovered from digestate, while Bolzonella et al. (2018) and Brienza et al. (2021) reported 12.0 and 3.8-5.0 kWh kg^{-1} N recovered, respectively. Different values for energy consumption of NDN treatment are stated in literature. Corbala-Robles et al. (2018) reported 17 kWh t^{-1} LF of swine manure for aeration and mixing, whereas Willeghems et al. (2016) mentioned two different energy consumption for the two systems used in Flanders, namely 16 kWh t^{-1} LF of manure (Bio Armor system) and 17 kWh t^{-1} of LF manure (Trevi). The reduced energy consumption when using PO aeration can be explained by the increased oxygen transfer efficiency for PO aeration which increases the aeration efficiency (kg O_2 kWh^{-1}). As nitrification demands an oxidizing power of 4.57 g O_2 per g of N oxidized, the reduction in N content in configuration 3 through the stripping-scrubbing unit further reduces the energy demand per t LF treated.

Table 26. Comparison of the mass and nutrients balances of the Belgian pilot with literature (% removal dry material).

	Recovery (g kg^{-1} dry matter; ‰ d.m.)*			
	C	N	P	K
Belgian pilot	0	36	0	0
Brienza et al. (2023)	0	22	0	0
Pintucci et al. (2017)	0	28-46	0	0
Bolzonella et al. (2018)	0	22	0	0
Baldi et al. (2018)	0	62	0	0

*d.m.: dry matter

Table 27. Comparison of the energy balance of the Belgian pilot with literature.

	Energy consumtion (kWh kg^{-1} N recovered)
Belgian pilot	6.5
Tampio et al. (2016)	0.8 - 28
Brienza et al. (2023)	6.4 - 13.6
Bolzonella et al. (2018)	12.0
Brienza et al. (2021)	3.8 - 5.0

3.5. French pilot

3.5.1. French pilot: mass and nutrients balance

Presented results were obtained during the 2023 production campaign. They are resulting from several optimizations of pilots which were made to improve efficiency and quality of resulting BBFs. In paragraphs below we will show mass and nutrients results from 2023 production campaign and compare to previous results which were measured since the beginning of the project. The technical specificity of the brought changes to pilots are detailed in deliverable D2.2.

Pyrolysis

Pyrolysis was tested on dry poultry manure, and on dried solid fraction of digestate. Table 29 and 30 show the impact of temperature reduction on mass balance and principal elements. Figure 28 represents element concentration evolution during the pyrolysis process for both materials.

The main effect of the pyrolysis process on both matters is a reduction of the global mass. This reduction depends on the matter used, pyrolysis temperature and the residence time in the oven. Trials made in Fertimanure context shows a reduction of 78% of the global mass of the poultry manure after being submitted to a temperature of 700°C during 20 min. Same trials made at 550°C during 20 min on solid digestate shows a reduction of 62% of the global mass.

This mass reduction is essentially linked to carbon volatilization. Nitrogen is also impacted by the process. On the contrary, both phosphorus and potassium remain stable during the process, hence the increasing concentration of these two elements.

Table 28. Mass balance of poultry manure pyrolysis

	Mass (kg)	**C orga (kg)**	**N (kg)**	**P (kg)**	**K (kg)**	**S (kg)**
INPUT						
Poultry manure	120 (n = 1)	38.90 ± 7.13 (n = 5)	3.29 ± 0.73 (n = 5)	1.1 ± 0.26 (n = 5)	2.72 ± 0.3 (n = 5)	0.31 ± 0.34 (n = 4)
PYROLYSIS OUTPUT						
Biochar	26.6 (n = 1)	9.31 ± 2.9 (n = 5)	0.67 ± 0.11 (n = 5)	0.64 ± 0.03 (n = 5)	2.04 ± 0.27 (n = 5)	0.19 ± 0.02 (n = 5)
Gas emission	93.4 (n = 1)	29.59 ± 2.9 (n = 5)	2.62 ± 0.11 (n = 5)	0.46 ± 0.03 (n = 5)	0.68 ± 0.27 (n = 5)	0.12 ± 0.02 (n = 5)

		C org. (%)	**N (%)**	**P (%)**	**K (%)**	**S (%)**
INPUT						
Raw material	Mean	32,42	2,74	0,91	2,26	0,26
	Standard deviation	5,94	0,61	0,22	0,25	0,28
PYROLYSIS OUTPUT						
Raw material	Mean	35,00	2,53	2,41	7,67	0,71
	Standard deviation	10,90	0,42	0,13	1,01	0,08

Table 29. Mass balance of solid digestate pyrolysis

	Mass (kg)	**C orga (kg)**	**N (kg)**	**P (kg)**	**K (kg)**	**S (kg)**
INPUT						
Solid digestate	5.61 ± 3.78 (n = 25)	2.18 ± 0.1 (n = 23)	0.10 ± 0.01 (n = 23)	0.04 ± 0.01 (n = 23)	0.09 ± 0.02 (n = 23)	0.02 (n = 1)
PYROLYSIS OUTPUT						
Biochar	2.14 ± 1.5 (n = 25)	0.97 ± 0.19 (n = 8)	0.04 ± 0.01 (n = 8)	0.04 ± 0 (n = 8)	0.09 ± 0.02 (n = 8)	0.03 ± 0.01 (n = 7)
Gas emission	3.47 ± 2.41 (n = 25)	1.21 ± 0.19 (n = 8)	0.06 ± 0.01 (n = 8)	0,00 ± 0.01 (n = 8)	0.00 ± 0.02 (n = 8)	0.00 ± 0.01 (n = 7)

	C orga (%)	N (%)	P (%)	K (%)	S (%)
INPUT					
Raw material Mean	38,83	1,72	0,78	1,58	0,40
Standard deviation	1,72	0,11	0,13	0,37	
PYROLYSIS OUTPUT					
Biochar Mean	45,32	1,70	1,97	4,03	1,17
Standard deviation	8,76	0,44	0,18	0,99	0,63

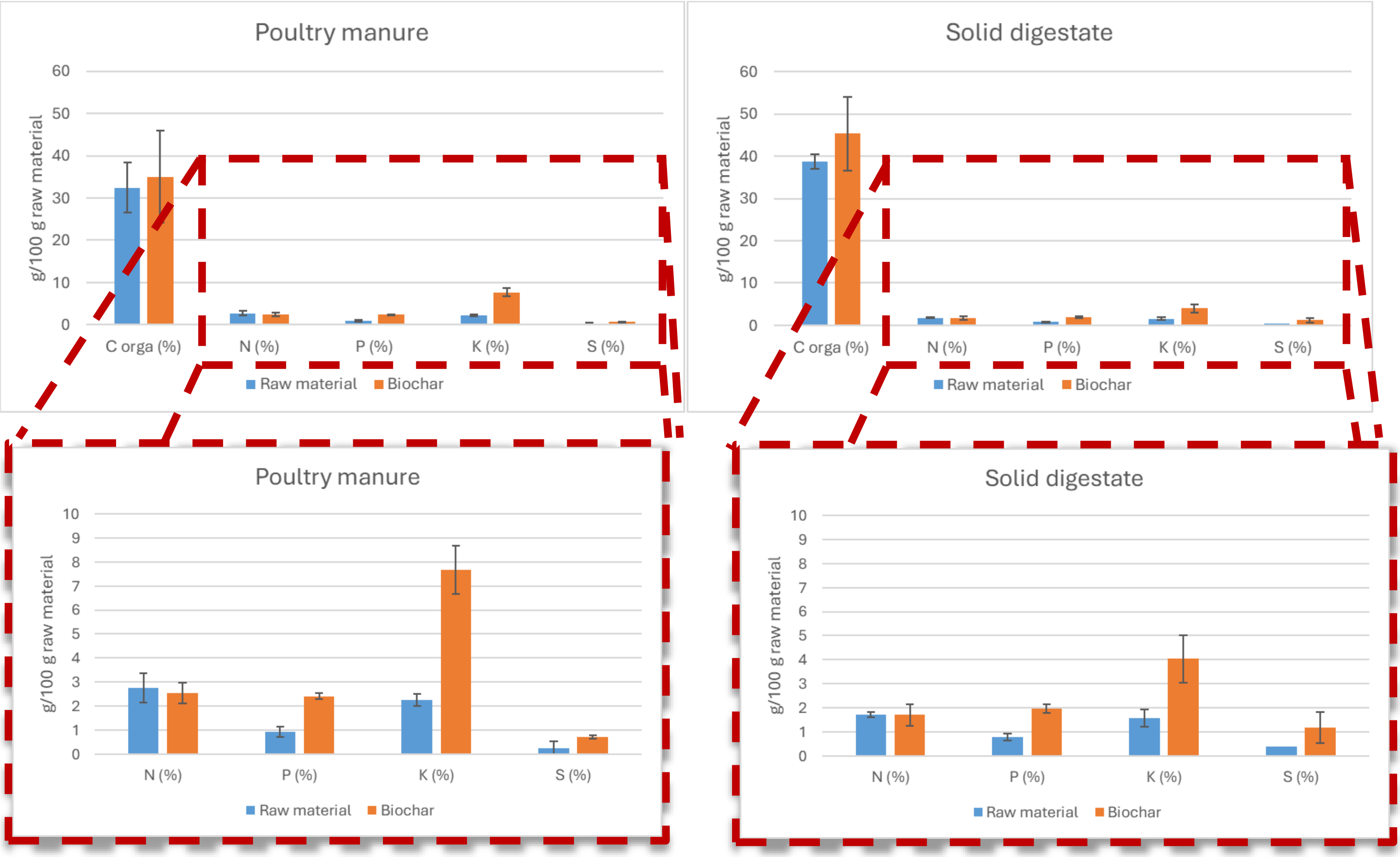


Figure 28. Evolution of element concentration before and after poultry manure pyrolysis (left) and solid digestate pyrolysis (right).

Ammonia stripping

The first conception of the stripping tower is described in Figure 29.

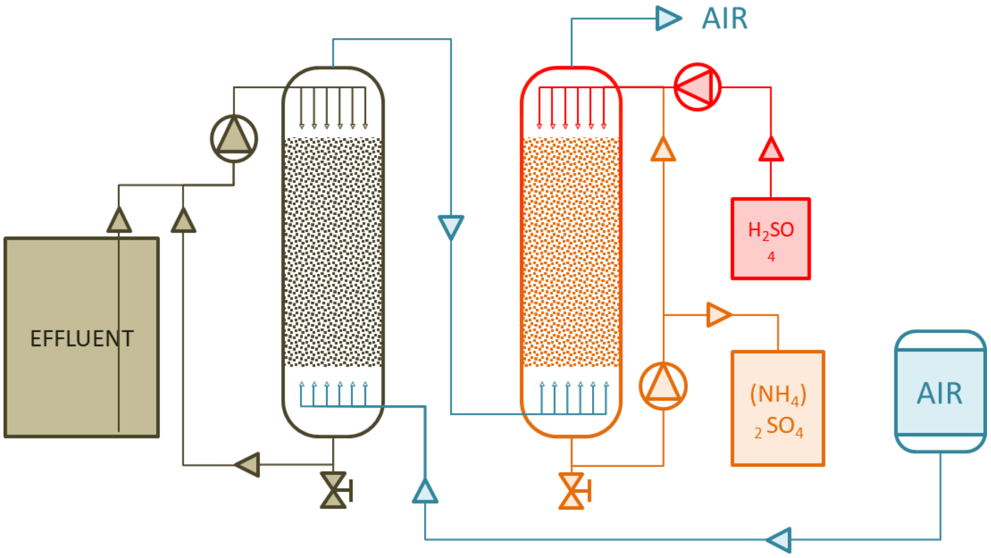


Figure 29. First conception of ammonia stripping tower.

The two columns were functioning simultaneously, one for liquid effluent circulation, and the second for sulfuric acid circulation.

With this configuration several problems appear: without optimization of physicochemical properties of effluent, ammonia volatilization is extremely poor, and on the other hand acid circulation highly reduce its trapping capacity, which is the volume capacity of the circulation is very low compared to the volume of the column. Furthermore, circulation of sulfuric acid in the system severely damaged all the system (stainless steel tower, fittings, pumps, etc.). That is why we made some modifications to the system and tested step by step every change in order to get closer to industrial yields. The Figure 30 shown the final configuration which gave us the bests yields.

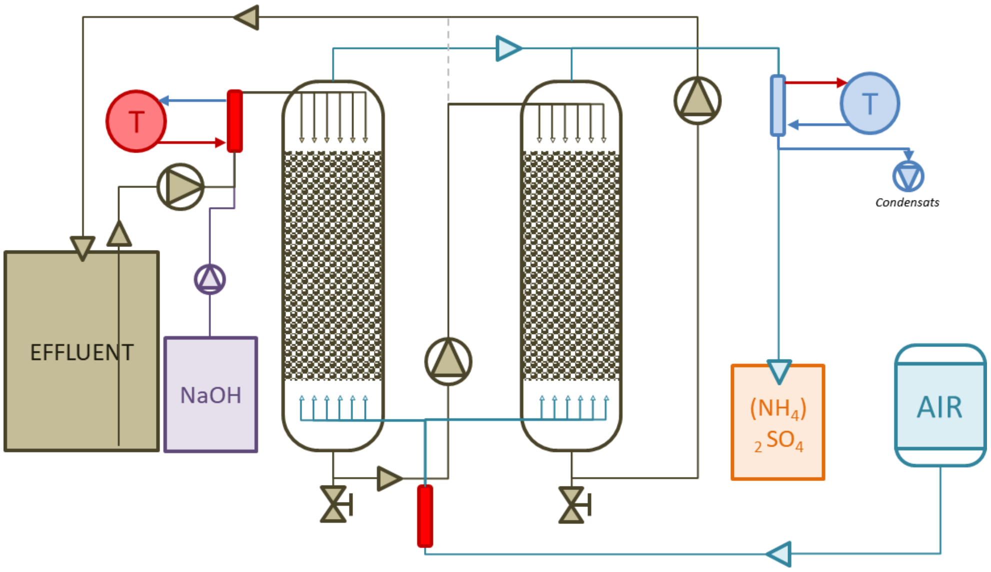


Figure 30. Final conception of ammonia stripping tower

Details of the whole modifications brought to the stripping tower are presented in deliverable D2.1. Shortly, in the final system, the ammonia volatilization is optimized thanks to:

- the addition of caustic soda (NaOH) into the effluent in order to reach a pH above 10.
- The heating of effluent and airflow at 60°C thanks to 2 heating exchangers
- The usage of the second tower as an extension of the first one for effluent circulation, in order to double the exchanging time between effluent and airflow.

The ammonia trapping (Table 30 and 31) is also optimized with bubbling charged airflow into an acid trap instead of making an acid circulation through the stripping tower (this also helps protect the process from acid corrosion)

Given that production was very variable and unstable before reaching this configuration, previous productions carried out with previous stripping systems were not considered in the final mass and element balance presented below. Ammonia stripping was carried on pig slurry and liquid digestate. Unfortunately, the second effluent was more difficult to use with our configuration. The liquid digestate is more concentrated with suspended matter, the pumps and columns saturate more quickly, requiring recurring cleaning of the whole system.

Table 30. Mass balance of pig slurry ammonia stripping

	Volume (L)	**C orga (kg)**	**N (kg)**	**N-NH_4 (kg)**	**P (kg)**	**K (kg)**	**S (kg)**
INPUT							
Pig Slurry	422.59 (n = 15)	6.75 ± 9.17 (n = 3)	6.75 ± 3.63 (n = 17)	6.29 ± 3.34 (n = 17)	0.33 ± 0.43 (n = 3)	1.05 ± 0.13 (n = 3)	0.14 ± 0.13 (n = 3)
Sulfuric acid	2	0.00	0.00	0.00	0.00	0.00	0.33
NaOH	5.25	0.00	0.00	0.00	0.00	0.00	0.00
STRIPPING OUTPUT							
FR-LK	418.46 (n = 15)	1.28 ± 1.08 (n = 3)	2.03 ± 1.08 (n = 18)	1.98 ± 1.14 (n = 18)	0.06 ± 0.03 (n = 4)	0.91 ± 0.32 (n = 4)	0.29 ± 0.37 (n = 3)
FR-AS	6.53 (n = 15)	0.00	1.00 (n = 17)	0.99 (n = 17)	0.00	0.00	0.32 (n = 1)
Loss	0.00	5.47	3.72	3.32	0.27	0.14	0.00

		C org. (%)	**N (%)**	**N-NH_4 (%)**	**P (%)**	**K (%)**	**S (%)**
INPUT							
Pig Slurry	Mean	1.60	1.60	1.49	0.08	0.25	0.03
	SD	2.17	0.86	0.79	0.10	0.03	0.03
Sulfuric acid	Mean	0.00	0.00	0.00	0.00	0.00	16.35
	SD	0.00	0.00	0.00	0.00	0.00	0.00
OUTPUT							
FR-LK	Mean	0.31	0.49	0.47	0.01	0.22	0.07
	SD	0.26	0.26	0.27	0.01	0.08	0.09
FR-AS	Mean	0.00	15.27	15.21	0.00	0.00	4.87
	SD	0.00	6.14	6.17	0.00	0.00	0.00

Table 31. Mass balance of liquid digestate ammonia stripping in French pilot

	Volume (L)	C orga (kg)	N (kg)	N-NH_4 (kg)	P (kg)	K (kg)	S (kg)
INPUT							
Liquid digestate	30 (n = 1)	0.48 (n = 1)	0.23 (n = 1)	0.16 (n = 1)	0.02 (n = 1)	0.1 (n = 1)	0.02 (n = 1)
Sulfuric acid	2	0.00	0.00	0.00	0.00	0.00	0.33
NaOH	0.00	0.00	0.00	0.00	0.00	0.00	0.00
OUTPUT stripping							
FR-LK	29 (n = 1)	0.23 (n = 1)	0.09 (n = 1)	0.04 (n = 1)	0.02 (n = 1)	0.07 (n = 1)	0.01 (n = 1)
FR-AS	3 (n = 1)	0	0.15 (n = 1)	0.15 (n = 1)	0	0	0.34 (n = 1)
LOSS	0	0.25	0	0	0	0	0

		C orga (%)	N (%)	N-NH_4 (%)	P (%)	K (%)	S (%)
INPUT							
Liquid digestate	Mean	1.60	0.78	0.54	0.07	0.33	0.05
	SD						
Sulfuric acid	Mean	0.00	0.00	0.00	0.00	0.00	16.35
	SD	0.00	0.00	0.00	0.00	0.00	0.00
OUTPUT							
FR-LK	Mean	0.78	0.30	0.15	0.07	0.24	0.03
	SD						
FR-AS	Mean	0.00	5.12	5.12	0.00	0.00	11.40
	SD	n.a					

The stripping principle consists of gasifying ammonia from an effluent and transferring it into an acid trap. Under these conditions the overall mass of the treated effluent is preserved. A variable part of ammonia is volatilized (depending on stripping duration, temperature and pH of the effluent, ratio between airflow and liquid flow). We were able to reach more than 90% of ammonia volatilization. If we refer to the commercial information provided by the manufacturers of industrial units, industrial stripping plant possibly reach more than 95% of ammonia volatilization (Figure 31).

Our stripping process also influences organic carbon content. With liquid digestate, half of the initial organic carbon is lost during the process. The mineralization of organic carbon is attributable to the massive supply of air and oxygen into the system. This effect is exacerbated with pig slurry because of soda addition. Other elements (P, K, S) remain stable during the process.

With pig slurry, the addition of caustic soda increases drastically the loss of ammonia: given the recirculation of the effluent, the overall pH in the storage tank gradually increases, and a large part of the ammonia is then lost by simple volatilization in the tank (mean of 55% of nitrogen loss during our trials). This loss is not visible with digestate because we did not add soda with this effluent.

There is also a significant loss of phosphorus, but that cannot be by volatilization. Since we do not see this loss with the liquid digestate, this loss is probably due to the phosphorus precipitation in relation with pH augmentation.

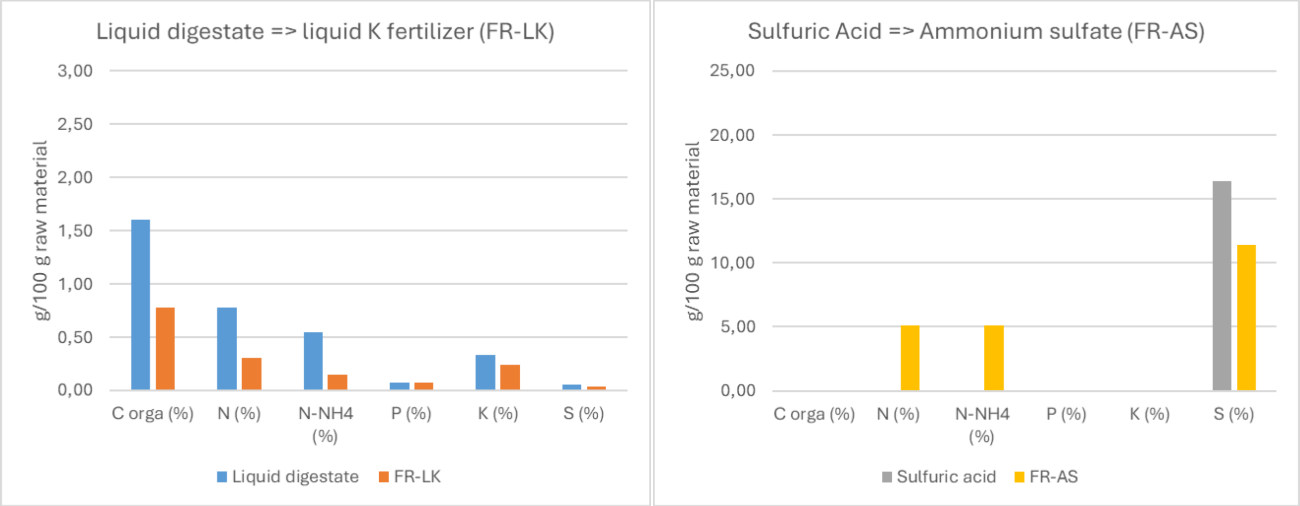


Figure 31. Evolution of element concentration before and after stripping of pig slurry ammonia and production of ammonium sulphate.

We can also notice a “reduction” of sulfur concentration during the concentration of sulfuric acid into ammonium sulfate. This effect is due to a dilution of ammonium sulfate during the process. Because of the heating of air and the effluent, outflow air is saturated with water. Despite the addition of a cooler system just before the air bubbling, all the evaporated water cannot be condensate before the acid trap, then condensate in the acid and increase the global volume of ammonium sulfate (here by 3), so that is why the global concentration of sulfur decrease by 3 (but the total amount does not change).

Concentration of final ammonium sulfate depends on the duration of bubbling into the acid trap. In order to produce usable ammonium sulfate for other trials planned in Fertimanure project, we need to keep it in liquid form. The ammonium sulfate solubility in water at 20°C is 754 g/L. If we maintained the ammonia concentration in the acid, we succeeded in going over the threshold of solubility of ammonium sulfate (181. g N-NH4/L, 18%) and produce solid ammonium sulfate (Figure 32). For production of liquid ammonium sulfate, we usually stopped the ammonia concentration before reaching this point.

Figure 32. Ammonium sulphate precipitation due to overconcentration of ammonia in the sulfuric acid trap.

Mass balance

The N, P and K mass balance for each raw material (pig slurry, poultry manure and digestate) is represented in the form of a Sankey diagram (Figure 32).

The digestate treatment is the most complete: start from the raw digestate, the first step is a phase separation thanks to a screw press, then liquid phase is used into the stripping column and the solid phase is firstly dried (without heating) then pyrolysed. The effect of the screw press is only a distribution of elements according to their affinity to water or organic matter. In that way, organic matter and phosphorus concentrate in the solid phase when nitrogen concentrate in the liquid phase. Solid digestate is dried before pyrolysis because the system cannot handle with mushy materials.

If we consider dried solid digestate and liquid digestate as two different materials, we can compare them to the poultry manure and pig slurry respectively. Poultry manure pyrolysis does not need any pretreatment because the matter is already dry, and pig slurry is also used without any pretreatment (only a quick sieving in order to eliminate all the impurities which risk clogging the system).

Concerning stripping, the most valuable difference between pig slurry and liquid digestate is the need to add soda with pig slurry in order to increase the pH of this effluent. The liquid digestate is already at a pH > 8 so the addition of soda is useless. The consequences are a significant loss of ammonia due to the equipment we used. In industrial condition, without recirculation, this ammonia would have been captured by the acid trap. There is also an impact on phosphorus precipitation. This could be a research line for phosphorus isolation into struvite, but our equipment does not allow this separation for now, phosphorus was lost during columns washing.

Regarding pyrolysis, the behavior of the two materials is very similar. The overall mass loss is slightly lower with solid digestate (62% of mass loss with solid digestate and 78% for poultry manure) due to greater loss of carbon with poultry manure. There was no nitrogen conservation during the pyrolysis process and on the contrary, we measured a significant loss of this element. For poultry manures, this constitutes a significant loss of fertilizing element, which may reconsider the interest of pyrolyzing nitrogen rich materials such as poultry manures, except to treat contaminated batches (for example during avian flu episodes, …) or to manage surplus.

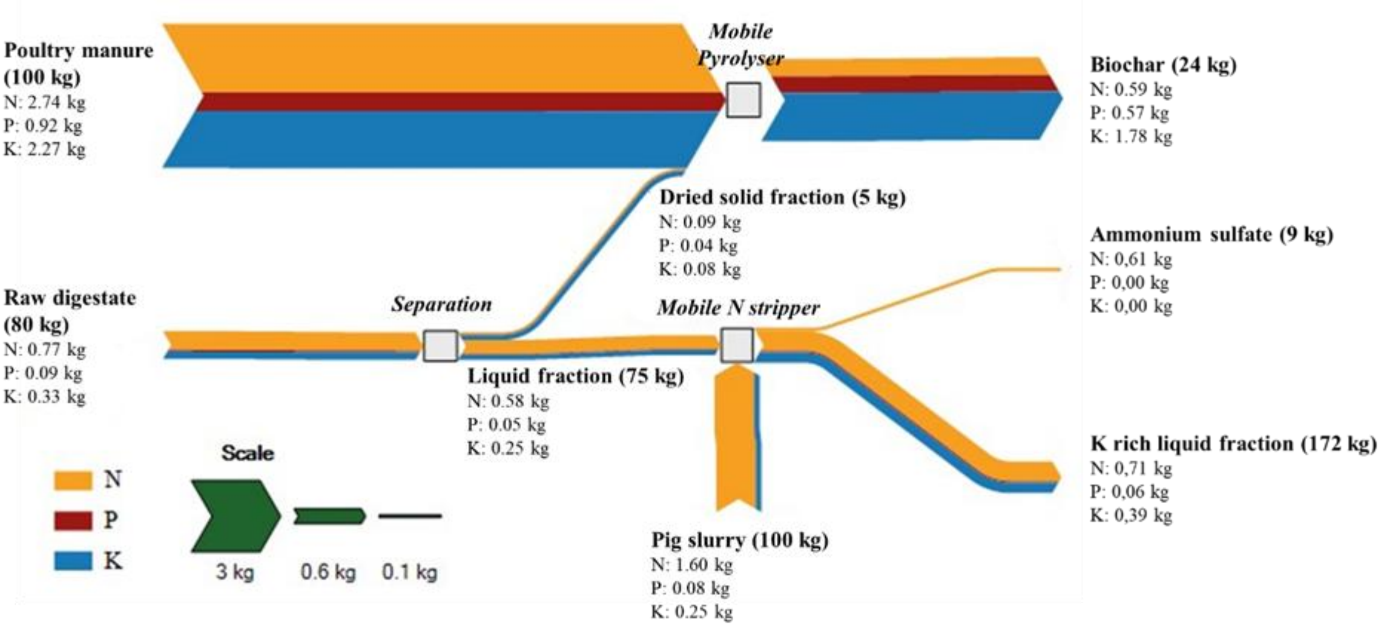


Figure 33. Mass and nutrient balance for French pilots.

3.5.2. French pilot: energy balance

French pilot uses electricity only as energy consumption. So, results are calculated using manufacturers information for energy consumption by equipment and related to operating time to produce BBFs. Results are reported in Table *32* and Table 33.

With regard to results of pyrolysis process energy consumption, the pilot is an experimental pilot dedicated for the project and with electricity as the only source of energy. Industrial pyrolysis process used fossil gas to heat the pyrolysis oven, and a part of pyrolysis gases are used to induced anaerobic condition into the oven (not N_2 as our pilot) and to preheated raw biomass.

Considering other energy consumption/production, estimations are ongoing to evaluate energy needs to dry various biomass. For the moment, manures are dried with solar energy under greenhouses, so without energy consumption. Moreover, estimations are needed to calculate recovered energy with gases boiler. Observations and measures indicated a combustion temperature reached of 700°C minimal.

Table *32* Energy balance of French pyrolysis pilot (kWh kg^{-1} of fresh treated material).

	Energy (kWh kg^{-1} treated material)
Consumption 1 (Inlet agitation)	0.0039
Consumption 2 (Feeding screw)	0.0066
Consumption 3 (Pyrolysis screw)	0.0045
Consumption 4 (Heating boiler, temperature increase)	0.015
Consumption 5 (Heating boiler, during treatment)	0.150
Consumption 6 (Outlet heating mattress)	0.069
Consumption 7 (Output screw)	0.055
Total consumption	**0.304**
Production 1 (energy recovery pyrolysis gases boiler)	not applicable with this pilot
Total production	**Not applicable with this pilot**
Net energy	**not applicable with this pilot**

Table 33. Energy balance of French stripping pilot (kWh kg^{-1} of fresh treated material).

	Energy (kWh kg^{-1} treated material)
Consumption 1 (Agitation)	0.025
Consumption 2 (Heating)	0.065
Consumption 3 (Air compressor)	0.055
Consumption 4 (Inlet pumping)	0.018
Consumption 5 (Recirculation)	0.018
Consumption 6 (Acid feeding)	0.002
Total consumption	**0.183**
Total production	**no production**
Net energy	**0.183**

3.5.3. French pilot: comparison with literature

Pyrolysis

Biochar yield and nutrient conservation

The mass yield of pyrolysis products depends on the applied temperature and heat flux (Deglise and Donnot 2020) (Figure 34). Being on fairly "low" temperatures and a slow rise in temperature due to the movement of the endless screws, the French process is similar to a slow pyrolysis process, it promotes the production of biochars and limits the production of oils or gases.

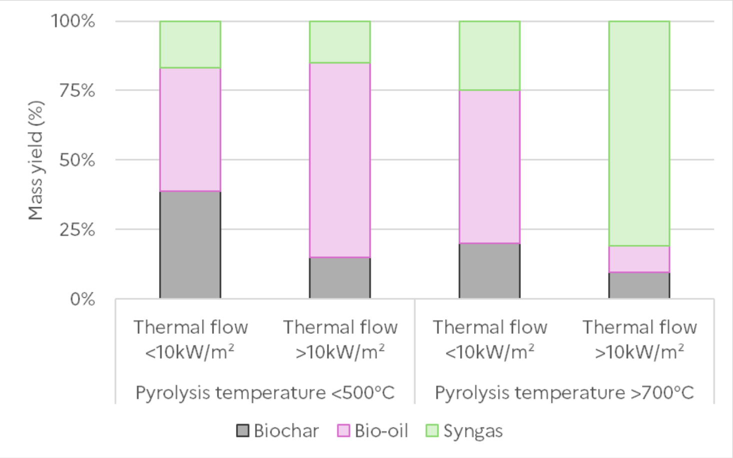


Figure 34. Influence of temperature and heat flow on pyrolysis products mass yield (data estimated from Deglise and Donnot, 2020)

The droppings biochars produced at 700°C represent approximately 20% of the initial mass, while the digestate biochars produced at 550°C represent approximately 40% of the initial mass. The biomass yields are consistent with the average values observed in the literature (Deglise and Donnot 2020).

The effect of temperature is quite important: Weiping & Mingxin (2012) which also tried to pyrolyse poultry manure, but at 600°C, obtained a yield of biochar production after pyrolysis of 45.71 % w/w, a yield significantly higher than the yield obtained at 700°C. Indeed, they also demonstrated that this yield decreases with increase of temperature with a yield of 60.13 % w/w at 300 °C to 45.71 %w/w at 600°C.

Regarding nutrient content, we have seen above that biochar production is a nutrient conservative process, with the exception of nitrogen. During the tests carried out here, the nitrogen losses represented 60% of the initial nitrogen at 550°C and 80% of the initial nitrogen at 700°C. Hadroug et al. (2019) also observed a significant N concentration decrease after pyrolysis of poultry manure at 400°c and 600°C with N losses of raw poultry manure of 57 % and 71% respectively. This was previously demonstrated by Cimo et al. (2014) who measured a N loss of raw manure of 54 % with pyrolysis at 600°C during 30 minutes. The results are quite similar to what was obtained during the FERTIMANURE project. it is indeed a shame to lose the nitrogen from these effluents. Nitrogen recovery upstream of pyrolysis could be interesting, but unfortunately this could not be explored in depth within the framework of this project. On the other hand, although they do not constitute a direct supply of nitrogen, influence of biochar on the dynamics of nitrogen in soils (N retention, protection against leaching or volatilization) is well documented (Dawar et al., 2021a and 2021b; Lee et al., 2022) and gives interesting results. These aspects are detailed in WP4 of this project.

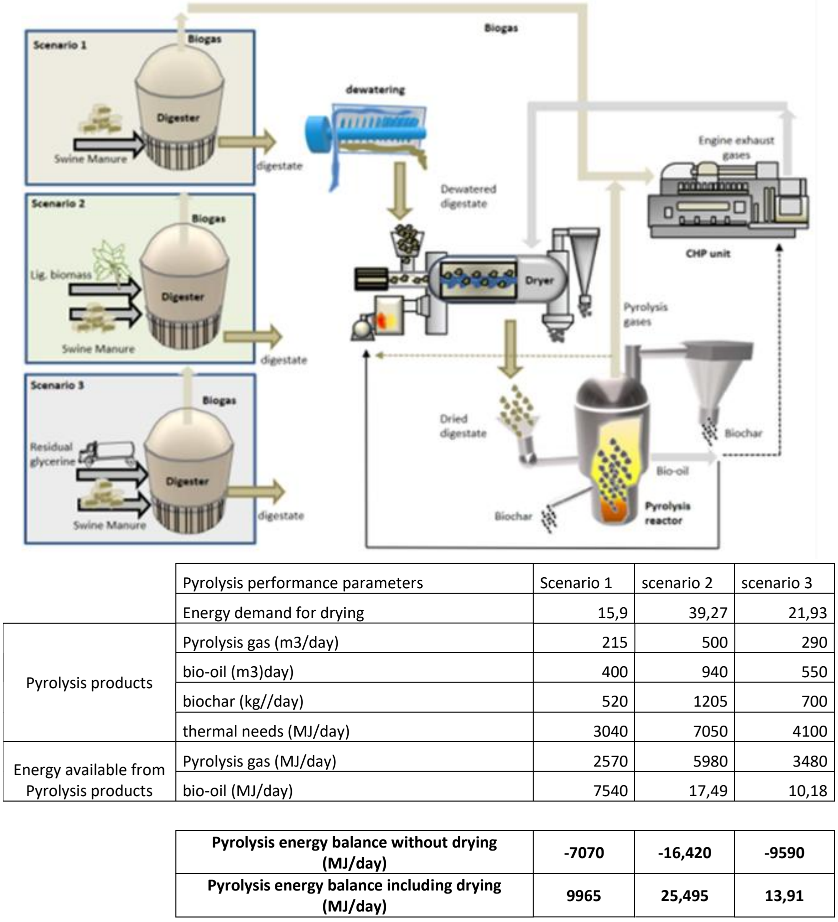
For other elements as P and K, Hadroug et al. (2019) also measured, as in our tests, an increase in their concentration in the biochars. This increase is due to the volatilization of organic matter and the conservation of mineral elements. Note that it is possible at very high temperatures to also lose potassium (Keiluweit et al., 2010).

Energy balance

For energy balance, it is not possible with the french pilot to correctly propose a calculation of energy demand and energy production only based on the temperature of the syngas combustion fumes. Indeed, we reached a smokes temperature between 750°C to 810 °C during our tests. On literature we can found some works which studied this topic of energy balance of manure pyrolysis:

Thus, Crombie et al. (2014) evaluated that pyrolysis at 450°C produced a gas product with sufficient energy content to meet the lower energy limit for maintaining the pyrolysis process while the upper energy limit was only reached for pyrolysis at 650°C. This was confirmed by other studies (Brassard et al. 2018, Gonzales et al. 2020) that pyrolysis done below 650°C shows a negative net energy balance if you do not include biomass drying in the calculation. Indeed, Gonzalez et al (2020) showed that using wet biomass as cow manure or digestate greatly penalizes energy balance (with up to 80 % of total energy inputs on studied scenarios) because of drying prior to conversion into a pyrolysis unit (Figure 35).

This impact of the drying stage concerns effluents such as manure or digestates more than poultry litter which already has higher dry matter contents (up to 65 % DM in our raw poultry manure test) and requires significantly less energy for drying or can even be introduced into pyrolysis without this step.



	Pyrolysis performance parameters	Scenario 1	scenario 2	scenario 3
	Energy demand for drying	15,9	39,27	21,93
Pyrolysis products	Pyrolysis gas (m3/day)	215	500	290
	bio-oil (m3)day)	400	940	550
	biochar (kg//day)	520	1205	700
	thermal needs (MJ/day)	3040	7050	4100
Energy available from Pyrolysis products	Pyrolysis gas (MJ/day)	2570	5980	3480
	bio-oil (MJ/day)	7540	17,49	10,18

Pyrolysis energy balance without drying (MJ/day)	**-7070**	**-16,420**	**-9590**
Pyrolysis energy balance including drying (MJ/day)	**9965**	**25,495**	**13,91**

Figure 35. Performances parameters associated with pyrolysis of digestates according to Gonzalez et al. 2020.

Ammonia stripping

Ammonium sulphate yield and nutrient conservation

The stripping process does not transform the initial biomass, it simply extracts the ammoniacal nitrogen and blocks it in an acid trap. Thus, the effluent is only slightly disturbed, the volume and other parameters remain unchanged and only the ammoniacal nitrogen content is reduced.

The concentration of ammonium sulfate produced depends solely on the production objective (the more the acid trap is kept in the system, the more it will charge, until potentially exceeding the ammonium sulfate solubility as we produced during the project (Figure 32)). On the other hand, the technologies can be compared on ammonia removal efficiency.

According to the bibliography, the efficiency of the process will be linked to several parameters: the temperature of the effluent, the pH. For example Provolo et al. 2017 tested the purification of liquid digestates by stripping, and obtained an ammonia removal efficiency of 50% at 30°C/pH 8, 69% at 40°C/pH 8, 87% at 40°C/pH 9, and 85% at 50°C/pH 8.

Vazifehkhoran et al. 2022 obtained similar results with up to 92% of ammoniacal nitrogen recovering from pig slurry, 83% from dairy cattle slurry and 67% from liquid digestate (40°C, no pH adjustment).

Abba et al. 2023 obtained an ammonia recovering up to 81% with pH at 10 and temperature at 68°C.

Concerning optimized temperature, several publications demonstrated that to optimize nitrogen volatilization, effluents must be maintained between 30°C and 40°C (Vazifehkhoran et al. 2022 , Pandey B. 2021, Georgiou D. 2019). In the case of FERTIMANURE, because of the absence of insulation, we heated it at 60°C in order to compensate for thermal losses.

Concerning preliminary slurry alkalinization, many publications demonstrate that the optimal pH range for volatilization is between 8.5 and 10. Beyond that the additional effect is negligible (Gustin et al. 2011, Vazeifehkhraon et al. 2022). That is why the pH of the slurry was corrected to 10 in FERTIMANURE. Thus we maximized volatilization, resulting in average yields of 70% which could even reach 90%. The pilot could be improved on maintaining the temperature of the effluent. Currently this is not the case and it is unfortunately the major parameter which influences the volatilization of ammoniacal nitrogen.

Energy balance

According to bibliographic data, the average energy consumed by the process is between 5–20.4 kWh/kg–NH4-N (Chen et al. 2022). As part of FERTIMANURE project, using the data from the mass balance, we used 175 kg of liquid digestate and slurry (or 2.18 kg N) to produce 9 kg of FR-AS (or 0.61 kg N). With an electricity consumption of 0.183 kWh/kg, global electric consumption is about 32.03 kWh. Compared to the mass of nitrogen recovered, we obtain 52.5 kWh/kg N-NH4 (or 3.56 kWh/kg of FR-AS).

The French pilot energy consumption is higher than what we find in the literature. This can be explained by the fact that the equipment was primarily designed to be a mobile technical demonstrator before being an economically viable pilot. An industrial installation could have higher and larger columns, with better insulation, that drastically increases efficiency while reducing energy and thermal requirements.

In addition, at the industrial level, in the context of digestate stripping, it would be possible to combine, for example, the recovery of thermal energy from cogeneration with the stripping process, and thus save on energy consumption. For example, Abba et al. 2023 manage to reduce energy consumption at 0.010 kWh/kg slurry thanks to autoconsumption of heat from anaerobic digestion cogeneration.

4. Conclusions

Deliverable 2.6 (Mass and energy balance of the on-farm pilots to WP5) aimed to describe all the mass and energy flow balances of the different on-farm pilots. The present deliverable reports final mass and energy balances defined for the five FERTIMANURE pilots.

Some interesting considerations arise from the reported results:

Spanish pilot

The Spanish pilot is composed by several treatment technologies including solid-liquid separation, biodrying, combustion, membrane-based technologies, freeze concentration, microalgae cultivation, and hydrolysis. The pilot plant complexity resulted in several viable BBFs (detailed in D 2.5) although it makes difficult the performance of proper mass and energy balances.

The main challenge was to monitor the phosphorus flow as most of the phosphorus is retained in the MF retentate in a precipitated form, but as the phosphorus salt is very sensitive to pH conditions, the mixing of this stream with RO retentate for further processing in FC caused major deviations. In addition, nitrogen balances are also complex due to some losses through ammonia volatilization.

Spanish pilot plant reported negative energy balance as some of its elements are energetically intensive (MF, FC). In the case of MF, an optimized dimensioning of the equipment could decrease the specific energy consumption.

Finally, it is important to point out the high efficiency of membrane-assisted stripping for AS production, as the specific energy consumption is tenfold lower than conventional stripping-scrubbing processes.

Dutch pilot

The Dutch pilot is a combination of several treatment technologies including manure collection robot in the stables for quick removal of dairy slurry, anaerobic digester for production of biogas (electricity and heat), solid-liquid separation by a screw press, phosphate sludge precipitation and a nitrogen (NH3) stripper-scrubber installation.

The pilot plant has common proven technologies put in practise resulting in several valuable and viable BBFs: ammonium sulphate solution high in nitrogen as ammonia, organic soil conditioner high in organic matter (carbon) and phosphate, liquid potassium fertiliser relatively high in potassium and other minerals, and wet (or future dried version) of organic rich phosphorus sludge. The products are used at own farm by the farmer but are also of interest for farmers in the region. The organic soil conditioner can also be sold to consumers or used as resource for biobased materials and products including .

Generally, the nutrient balances and nutrient recovery efficiencies are in line what would be expected for the screw press and the nitrogen scrubber-stripper. During the project optimization has taken place, finding a balance between optimal end products and minimizing costs, energy and material input, labour and time. The energy balance of the whole farm is positive if the production and use of biogas for electricity and heat is taken into account. There could be improvements in the energy efficiencies, mainly in the nitrogen stripper (which has highest energy consumption), and the position of and pipes between different processes.

German pilot

The German pilot combines treatment of the feedstock with citric acid, drying, pelletizing and an unique pyrolysis technique with scrubbing of the produced pyrolysis gas to recover ammonia. Within this pilot a unique form of pyrolysis called Thermo-Catalytic Reforming is used. TCR is a novel unit combining intermediate pyrolysis with post catalytic treatment. A resulting product is Biochar (DE-BC) with fertilizing properties and high quality. The oil and gas produced within the process can be used for energy production to maintain the process or excess energy production. As nitrogen is a valuable nutrient in the plant cycle the formed ammonia within the catalytic reforming of manure is recovered by scrubbing with phosphoric acid. The produced mono ammonium phosphate (DE-AP) is a valuable fertilizer and ready for agricultural use.

The most energy consuming step of the pilot is the drying of the manure. With advanced dryer technology in future plant sites this can be reduced for even more efficient production of biobased fertilizers.

Belgium pilot

The Belgian pilot, employing manure separation, NH_3 stripping-scrubbing followed by biological N removal, achieved noteworthy nutrient recovery from pig manure. The use of nitric acid and sulphuric acid as scrubber sorbents led to similar N recovery efficiencies, while generating different products. The Belgian pilot showcased a total energy consumption of 16.7 kWh per ton raw manure processed, with notable reductions facilitated by the incorporation of N recovery by a NH_3 stripper before N removal by NDN treatment. Comparative analysis with literature highlighted the pilot's competitive edge, with a nitrogen recovery efficiency of 32-36%, positioning it as a promising contributor to sustainable agricultural practices.

French pilot

Nitrogen stripping pilot treated pig slurry to produce ammonium sulphate with concentration of 5 % N using a very simple equipment. The demonstration to farmers, who accepted to test on their pig slurry, was an interesting challenge. Farmers were quite interesting, and demonstrations could help to promote the development of biobased fertilizer.

Pyrolysis French pilot produced biochars from poultry manure and solid digestates. Both biochars were interesting organic amendments which could also add some available nutrients (P and K), but with a significant N loss. Energy needed for pyrolysis, and more especially energy needed to dry inputs, could be an obstacle to the development of this technology for wet manure.

References

1. Abba A., Domini M., Baldi M., Pedrazzani R., Bertanza G. (2023). Ammonia Recovery from Livestock Manure Digestate through an Air-Bubble Stripping Reactor: Evaluation of Performance and Energy Balance. Energies 16 (4) 1643 https://doi.org/10.3390/en16041643
2. Bekker, R., Prinsloo, N. M., *Ind. Eng. Chem. Res. (*2009), *48*, 10156-10162.
3. Bennamoun, L., Arlabosse, P., and Léonard, A. (2013). Review on fundamental aspect of application of drying process to wastewater sludge. Renewable and Sustainable Energy Reviews, 28, 29-43. https://doi.org/10.1016/j.rser.2013.07.043
4. Bolzonella, D., Fatone, F., Gottardo, M., & Frison, N. (2018). Nutrients recovery from anaerobic digestate of agro-waste: Techno-economic assessment of full scale applications. J. Environ.l Manage. 216, 111–119. https://doi.org/10.1016/j.jenvman.2017.08.026
5. Bonmatí, A., & Flotats, X. 2003. Air stripping of ammonia from pig slurry: characterisation and feasibility as a pre- or post-treatment to mesophilic anaerobic digestion. Waste Manage. 23(3), 261–272. https://doi.org/10.1016/S0956-053X(02)00144-7
6. Brassard P., Godbout S., Pelletier F., Raghavan V., Palacios J.H. (2018). Pyrolysis of switchgrass in an auger reactor for biochar production: A greenhouse gas and energy impacts assessment. Biomass and Bioenergu 116, 99-105. https://doi.org/10.1016/j.biombioe.2018.06.007
7. Brienza, C., Donoso, N., Luo, H., Vingerhoets, R., de Wilde, D., van Oirschot, D., Sigurnjak, I., Biswas, J. K., Michels, E., & Meers, E. (2023). Evaluation of a new approach for swine wastewater valorisation and treatment: A combined system of ammonium recovery and aerated constructed wetland. Ecol. Eng. 189, 106919-.https://doi.org/10.1016/j.ecoleng.2023.106919
8. Brokmeier, L-P., Biochar Production from Municipal Sewage Sludge via Pyrolysis, University Gotland, **2022,** 11.
9. Cai, L., Gao, D., Chen, T. B., Liu, H. T., Zheng, G. D., & Yang, Q. W. (2012). Moisture variation associated with water input and evaporation during sewage sludge bio-drying. Bioresource Technology, 117, 13-19. https://doi.org/10.1016/j.biortech.2012.03.092
10. Cathcart, A., et al. (2023). "Optimising mechanical separation of anaerobic digestate for total solids and nutrient removal." Journal of Environmental Management 345: 118449.
11. Chen T.L., Chen L.H., Lin Y.J., Yu C.P., Ma H., Chiang P.C. (2021). Advanced ammonia nitrogen removal and recovery technology using electrokinetic and stripping process towards a sustainable nitrogen clycle: a review. Journal of Cleaner Production, doi: 10.1016/j.jclepro.2021.127369
12. Cherkasov, N.; Ibhadon, A.O.; Fitzpatrick, P. (2015). A review of the existing and alternative methods for greener nitrogen fixation. Chem. Eng. Process. 90, 24–33. https://doi.org/10.1016/j.cep.2015.02.004.
13. Chiumenti, A., da Borso, F., Teri, F., Chiumenti, R., Piaia, B. (2013). Full-scale membrane filtration system for the treatment of digestate from a co-digestion plant. Applied Engineering in Agriculture, 29(6), 985-990.
14. Choi, H. L., Richard, T. L., & Ahn, H. K. (2001). Composting high moisture materials: Biodrying poultry manure in a sequentially fed reactor. Compost science & utilization, 9(4), 303-311. https://doi.org/10.1080/1065657X.2001.10702049

15. Cong, H., Meng, H., Mašek, O., Yao, Z., Li, L., Yu, B., ... & Zhao, L. (2022). Comprehensive analysis of industrial-scale heating plants based on different biomass slow pyrolysis technologies: product property, energy balance, and ecological impact. Cleaner Engineering and Technology, 6, 100391. https://doi.org/10.1016/j.clet.2021.100391

16. Conti, R., Jäger, N., Neumann, J., Apfelbacher, A., Daschner, R., & Hornung, A. (2017). Thermocatalytic reforming of biomass waste streams. Energy Technology, 5(1), 104-110. https://doi.org/10.1002/ente.201600168

17. Corbala-Robles, L., Sastafiana, W. N. D., Van linden, V., Volcke, E. I. P., & Schaubroeck, T. (2018). Life cycle assessment of biological pig manure treatment versus direct land application − a trade-off story. Resour., Conserv. Recycl. 131, 86–98. https://doi.org/10.1016/j.resconrec.2017.12.010

18. Dadrasnia, A., de Bona Muñoz, I., Yáñez, E. H., Lamkaddam, I. U., Mora, M., Ponsá, S., ... & Oatley-Radcliffe, D. L. (2021). Sustainable nutrient recovery from animal manure: A review of current best practice technology and the potential for freeze concentration. Journal of cleaner production, 315, 128106. https://doi.org/10.1016/j.jclepro.2021.128106

19. Dawar, K., Fahad, S., Jahangir, M.M.R., Munir, I., Alam, S.S., Khan, S.A., Mian, I.A., Datta, R., Saud, S., Banout, J., Adnan, M., Ahmad, M.N., Khan, A., Dewil, R., Habib-ur-Rahman, M., Ansari, M.J., Danish, S., (2021a). Biochar and urease inhibitor mitigate NH3 and N2O emissions and improve wheat yield in a urea fertilized alkaline soil. Sci Rep 11, 17413. https://doi.org/10.1038/s41598-021-96771-0

20. Dawar, K., Saif-ur-Rahman, Fahad, S., Alam, S.S., Khan, S.A., Dawar, A., Younis, U., Danish, S., Datta, R., Dick, R.P., (2021b). Influence of variable biochar concentration on yield-scaled nitrous oxide emissions, Wheat yield and nitrogen use efficiency. Scientific Reports 11. https://doi.org/10.1038/s41598-021-96309-4

21. Deglise, X., Donnot, A., (2020). Bois énergie - Propriétés et voies de valorisation. Technique de l'ingénieur.

22. de Haas, D., & Andrews, J. (2022). Nitrous oxide emissions from wastewater treatment - Revisiting the IPCC 2019 refinement guidelines. Environmental Challenges (Amsterdam, Netherlands), 8, 100557-. https://doi.org/10.1016/j.envc.2022.100557

23. de Klerk, A. Catalysis (2011), *23*, 1-49.

24. Donatello, S., Tong, D., & Cheeseman, C. R. (2010). Production of technical grade phosphoric acid from incinerator sewage sludge ash (ISSA). Waste management, 30(8-9), 1634-1642. https://doi.org/10.1016/j.wasman.2010.04.009

25. Dong, D., Choi, O. K., Lee, K., Hong, Y., & Lee, J. W. (2018). Alternative route for the recovery of nitrogen as ammonium phosphate crystals from high strength waste streams. Journal of Material Cycles and Waste Management, 20, 578-584. https://doi.org/10.1007/s10163-017-0624-2

26. Elawwad, A. (2018). Optimized biological nitrogen removal of high-strength ammonium wastewater by activated sludge modeling. Journal of Water Reuse and Desalination, 8(3), 393–403. https://doi.org/10.2166/wrd.2017.200

27. Fang, L., Li, J. S., Guo, M. Z., Cheeseman, C. R., Tsang, D. C., Donatello, S., & Poon, C. S. (2018). Phosphorus recovery and leaching of trace elements from incinerated sewage sludge ash (ISSA). Chemosphere, 193, 278-287. https://doi.org/10.1016/j.chemosphere.2017.11.023

28. FAO. World Fertilizer Trends and Outlook to 2022. (2020). Available online: https://doi.org/10.4060/ca6746en (accessed on October 18th 2021).

29. Foged, Henning Lyngsø, Xavier Flotats, August Bonmati Blasi, Jordi Palatsi and Albert Magri. (2011). End and by-products from livestock manure processing - general types, chemical composition, fertilising quality and feasibility for marketing. Technical Report No. III concerning "Manure Processing Activities in Europe" to the European Commission, Directorate-General Environment. 78 pp.

30. Fournel, S., et al. (2019). "Production of recycled manure solids for bedding in Canadian dairy farms: I. Solid–liquid separation." Journal of Dairy Science 102(2): 1832-1846.

31. Frei, K. M., Cameron, D., & Stuart, P. R. (2004). Novel drying process using forced aeration through a porous biomass matrix. Drying Technology, 22(5), 1191-1215. https://doi.org/10.1081/DRT-120038587

32. Garcia-Moscoso, J. L., Obeid, W., Kumar, S., & Hatcher, P. G. (2013). Flash hydrolysis of microalgae (Scenedesmus sp.) for protein extraction and production of biofuels intermediates. The Journal of Supercritical Fluids, 82, 183-190. https://doi.org/10.1016/j.biortech.2016.06.087.

33. Georgiou, D.; Liliopoulos, V.; Aivasidis, A. (2019). Investigation of an integrated treatment technique for anaerobically digested animal manure: Lime reaction and settling, ammonia stripping and neutralization by biogas scrubbing. Bioresour. Technol. Rep. 5, 127–133. [Google Scholar] [CrossRef]

34. González, D., Guerra, N., Colón, J., Gabriel, D., Ponsá, S., & Sánchez, A. (2019). Filling in sewage sludge biodrying gaps: Greenhouse gases, volatile organic compounds and odour emissions. Bioresource technology, 291, 121857. https://doi.org/10.1016/j.biortech.2019.121857

35. González, R., Ellacuriaga, M., Aguilar-Pesantes, A., Carrillo-Peña, D., & Gómez, X. (2021). Feasibility of Coupling Anaerobic Digestion and Hydrothermal Carbonization: Analyzing Thermal Demand. *Applied Sciences*. Appl. Sci. 2021, 11, 11660. https://doi.org/10.3390/app112411660

36. Gowd, S. C., Kumar, D., Lin, R., & Rajendran, K. (2022). Nutrient recovery from wastewater in India: A perspective from mass and energy balance for a sustainable circular economy. Bioresource Technology Reports, 101079. https://doi.org/10.1016/j.biteb.2022.101079

37. Gripa, E., Campos, J. C., & da Fonseca, F. V. (2021). Combination of ozonation and microfiltration to condition landfill leachate for reverse osmosis treatment. Journal of Water Process Engineering, 43, 102264.

38. Guerra-Gorostegi, N., González, D., Puyuelo, B., Ovejero, J., Colón, J., Gabriel, D., ... & Ponsá, S. (2021). Biomass fuel production from cellulosic sludge through biodrying: Aeration strategies, quality of end-products, gaseous emissions and techno-economic assessment. Waste Management, 126, 487-496. https://doi.org/10.1016/j.wasman.2021.03.036

39. Günther, S.; Grunert, M.; Müller, S. (2018). Overview of recent advances in phosphorus recovery for fertilizer production. Eng. Life Sci. 18, 434–439. https://doi.org/10.1002/elsc.201700171.

40. Guštin, S.; Marinšek-Logar, R. (2011). Effect of pH, temperature and air flow rate on the continuous ammonia stripping of the anaerobic digestion effluent. Process. Saf. Environ. Prot. 89, 61–66. [Google Scholar] [CrossRef]

41. Gulyas, H., Bruhn, P., Furmanska, M., Hartrampf, K., Kot, K., Luttenberg, B., ... & Otterpohl, R. (2004). Freeze concentration for enrichment of nutrients in yellow water from no-mix toilets. Water Science and Technology, 50(6), 61-68. https://doi.org/10.2166/wst.2004.0360

42. Hadroug, S.; Jellali, S.; Leahy, J.J.; Kwapinska, M.; Jeguirim, M.; Hamdi, H.; Kwapinski, W. Pyrolysis Process as a Sustainable Management Option of Poultry Manure: Characterization of the Derived

Biochars and Assessment of their Nutrient Release Capacities. Water (2019), 11, 2271. https://doi.org/10.3390/w11112271

43. Hawkins, S., Robinson, K., Layton, A., & Sayler, G. (2010). Limited impact of free ammonia on Nitrobacter spp. inhibition assessed by chemical and molecular techniques. Bioresour. Technol. 101(12), 4513–4519. https://doi.org/10.1016/j.biortech.2010.01.090
44. Herbes, C., Roth, U., Wulf, S., & Dahlin, J. (2020). Economic assessment of different biogas digestate processing technologies: A scenario-based analysis. Journal of Cleaner Production, 255, 120282. https://doi.org/10.1016/j.jclepro.2020.120282
45. Hjorth, M., et al. (2010). "Solid-liquid separation of animal slurry in theory and practice. A review." Agronomy for Sustainable Development 30(1): 153-180.
46. Jubany, I., Carrera, J., Lafuente, J., & Baeza, J. A. (2008). Start-up of a nitrification system with automatic control to treat highly concentrated ammonium wastewater: Experimental results and modeling. Chem. Eng. J. 144(3), 407–419. https://doi.org/10.1016/j.cej.2008.02.010
47. Juárez, J. M., Hernando, A. L., Torre, R. M., Lanza, S. B., & Rodríguez, S. B. (2016). Saccharification of microalgae biomass obtained from wastewater treatment by enzymatic hydrolysis. Effect of alkaline-peroxide pretreatment. Bioresource Technology, 218, 265-271.
48. Kaikake, K., Sekito, T., & Dote, Y. (2009). Phosphate recovery from phosphorus-rich solution obtained from chicken manure incineration ash. Waste Management, 29(3), 1084-1088. https://doi.org/10.1016/j.wasman.2008.09.008
49. Kar, S. (2020). Analysis Methods for Lifecycle Assessment of Energy Systems and Nutrient Recovery, in Relevance to Environmental Policies. Drexel University.
50. Keiluweit, M., Nico, P.S., Johnson, M.G., Kleber, M., (2010). Dynamic Molecular Structure of Plant Biomass-Derived Black Carbon (Biochar). Environ. Sci. Technol. 44, 1247–1253. https://doi.org/10.1021/es9031419
51. Kehrein, P., van Loosdrecht, M., Osseweijer, P., & Posada, J. (2020). Exploring resource recovery potentials for the aerobic granular sludge process by mass and energy balances–energy, biopolymer and phosphorous recovery from municipal wastewater. Environmental Science: Water Research & Technology, 6(8), 2164-2179. 10.1039/D0EW00310G
52. Khan, M.N.; Mohammad, F. (2014). Eutrophication: Challenges and solutions. In Eutrophication: Causes, Consequences and Control; Springer: Dordrecht, Germany; pp. 1–15. https://doi.org/10.1007/978-94-007-7814-6_1.
53. Kleemann, R., Chenoweth, J., Clift, R., Morse, S., Pearce, P., & Saroj, D. (2017). Comparison of phosphorus recovery from incinerated sewage sludge ash (ISSA) and pyrolysed sewage sludge char (PSSC). Waste management, 60, 201-210. https://doi.org/10.1016/j.wasman.2016.10.055
54. Kröger, Michael & Klemm, Marco & Nelles, Michael. (2019). Extraction Behavior of Different Conditioned S. Rubescens. Energies. 12. 1336. 10.3390/en12071336.
55. Lee, J.-M., Park, D.-G., Kang, S.-S., Choi, E.-J., Gwon, H.-S., Lee, H.-S., Lee, S.-I., (2022). Short-Term Effect of Biochar on Soil Organic Carbon Improvement and Nitrous Oxide Emission Reduction According to Different Soil Characteristics in Agricultural Land: A Laboratory Experiment. Agronomy 12, 1879. https://doi.org/10.3390/agronomy12081879

56. Lee, S., Maniquiz-Redillas, M. C., Choi, J., & Kim, L.-H. (2014). Nitrogen mass balance in a constructed wetland treating piggery wastewater effluent. J. Environ. Sci. 26(6), 1260–1266. https://doi.org/10.1016/S1001-0742(13)60597-5

57. Li, X., Guo, J., Dong, R., Ahring, B. K., & Zhang, W. (2016). Properties of plant nutrient: Comparison of two nutrient recovery techniques using liquid fraction of digestate from anaerobic digester treating pig manure. *Science of the Total Environment*, *544*, 774-781. https://doi.org/10.1016/j.scitotenv.2015.11.172

58. Liu, B., Giannis, A., Zhang, J., Chang, V. W.-C., & Wang, J.-Y. 2015. Air stripping process for ammonia recovery from source-separated urine: modeling and optimization. J. Chem. Technol. *90*(12), 2208–2217. https://doi.org/10.1002/jctb.4535

59. Lyons, G. A., Cathcart, A., Frost, J. P., Wills, M., Johnston, C., Ramsey, R., & Smyth, B. (2021). Review of Two Mechanical Separation Technologies for the Sustainable Management of Agricultural Phosphorus in Nutrient-Vulnerable Zones. Agron. 11(5), 836–. https://doi.org/10.3390/agronomy11050836

60. Mehta, N., Anderson, A., Johnston, C. R., & Rooney, D. W. (2022). Evaluating the opportunity for utilising anaerobic digestion and pyrolysis of livestock manure and grass silage to decarbonise gas infrastructure: A Northern Ireland case study. 196, 343-357.

61. Meers, E., Rousseau, D. P., Lesage, E., Demeersseman, E., & Tack, F. M. (2006). Physico-Chemical P Removal from the Liquid Fraction of Pig Manure as an Intermediary Step in Manure Processing. Water Air Soil Pollut. 169(1-4), 317–330. https://doi.org/10.1007/s11270-006-3112-1

62. Meers, E., Tack, F. M. G., Tolpe, I., & Michels, E. (2008). Application of a Full-scale Constructed Wetland for Tertiary Treatment of Piggery Manure: Monitoring Results. Water Air Soil Pollut. 193(1-4), 15–24. https://doi.org/10.1007/s11270-008-9664-5

63. Navarro-López, E., del Carmen Cerón-García, M., López-Rodríguez, M., Acién-Fernández, F. G., & Molina-Grima, E. (2020). Biostimulants obtained after pilot-scale high-pressure homogenization of Scenedesmus sp. grown in pig manure. Algal Research, 52, 102123.

64. Neumann, J., Binder, S., Apfelbacher, A., Gasson, J. R., García, P. R., & Hornung, A. (2015). Production and characterization of a new quality pyrolysis oil, char and syngas from digestate–Introducing the thermo-catalytic reforming process. Journal of Analytical and Applied Pyrolysis, 113, 137-142. https://doi.org/10.1016/j.jaap.2014.11.022

65. Noe-Hays, A., Homeyer, R. J., Davis, A. P., & Love, N. G. (2021). Advancing the design and operating conditions for block freeze concentration of urine-derived fertilizer. ACS ES&T Engineering, 2(3), 446-455. https://doi.org/10.1021/acsestengg.1c00271

66. Oshita, K., Sun, X., Kawaguchi, K., Shiota, K., Takaoka, M., Matsukawa, K., & Fujiwara, T. (2016). Aqueous leaching of cattle manure incineration ash to produce a phosphate enriched fertilizer. Journal of Material Cycles and Waste Management, 18, 608-617. https://link.springer.com/article/10.1007/s10163-016-0528-6

67. Pandey, Bishnu, and Lide Chen. "Technologies to recover nitrogen from livestock manure-A review." Science of the Total Environment 784 (2021): 147098. https://doi.org/10.1016/j.scitotenv.2021.147098

68. Phanwilai, S., Noophan, P., Li, C.-W., & Choo, K.-H. 2020. Effect of COD:N ratio on biological nitrogen removal using full-scale step-feed in municipal wastewater treatment plants. Sustain. Environ. Res. 30(1), 1–9. https://doi.org/10.1186/s42834-020-00064-6

69. Pintucci, C., Carballa, M., Varga, S., Sarli, J., Peng, L., Bousek, J., Pedizzi, C., Ruscalleda, M., Tarragó, E., Prat, D., Colica, G., Picavet, M., Colsen, J., Benito, O., Balaguer, M., Puig, S., Lema, J. M., Colprim, J., Fuchs, W., & Vlaeminck, S. E. (2017). The ManureEcoMine pilot installation: advanced integration of technologies for the management of organics and nutrients in livestock waste. Water Sci. Technol. 75(5-6), 1281–1293. https://doi.org/10.2166/wst.2016.559

70. Provolo G., Perazzolo F., Mattachini G., Finzi A., Naldi E., Riva E. (2017). Nitrogen removal from digested slurries using a simplified ammonia stripping technique. Waste Management 69, 154-161.

71. PYREG, (2021b). Brochure Sludge. [pdf] PYREG Net-Zero Technology. Available at: 210423_broschuere_sludge_EN.pdf (pyreg.com) [Accessed: 16 Oktober 2023].

72. Qin, J., Qian, S., Chen, Q., Chen, L., Yan, L., & Shen, G. (2019). Cow manure-derived biochar: Its catalytic properties and influential factors. Journal of hazardous materials, 371, 381-388. https://doi.org/10.1016/j.jhazmat.2019.03.024

73. Rathanayake, N.; Patel, S.; Hakeem, I., G.; Pazferreiro, J.; Sharma, A.; Gupta, R.; Rees, C.; Bergmann, D.; Blackbeard, J.; Surpaneni, A.; Shah, K. (2023) Co-pyrolysis of biosolids with lignocellulosic biomass: Effect of feedstock on product yield and composition, Process Safety and Environmental Protection, 173, 75-87. https://doi.org/10.1016/j.psep.2023.02.087

74. Ravi, R., Beyers, M., Vingerhoets, R., Brienza, C., Luo, H., Bruun, S., & Meers, E. (2023). In the quest for sustainable management of liquid fraction of manure - Insights from a life cycle assessment. Sustain. Prod. Consum. https://doi.org/10.1016/j.spc.2023.11.006

75. Rivera, R., Chagnes, A., Cathelineau, M., & Boiron, M. C. (2022). Conditioning of poultry manure ash for subsequent phosphorous separation and assessment for a process design. Sustainable Materials and Technologies, 31, e00377. https://doi.org/10.1016/j.susmat.2021.e00377

76. Rico, C., Rico, J. L., & Lasa, C. (2012). Anaerobic digestion of the liquid fraction of dairy manure separated by screw pressing and centrifugation in a upflow anaerobic sludge blanket reactor at 25 C. Biosystems engineering, 112(4), 344-351.

77. Rodríguez, F. A., Reboleiro-Rivas, P., González-López, J., Hontoria, E., & Poyatos, J. M. (2012). Comparative study of the use of pure oxygen and air in the nitrification of a MBR system used for wastewater treatment. Bioresour. Technol. 121, 205–211. https://doi.org/10.1016/j.biortech.2012.06.053

78. Rodríguez-Alegre, R., Zapata-Jiménez, J., You, X., Pérez-Moya, M., Sanchis, S., & García-Montaño, J. (2023). Nutrient recovery and valorisation from pig slurry liquid fraction with membrane technologies. Science of The Total Environment, 874, 162548.

79. Rodríguez-Alegre, R., Zapata-Jiménez, J., Pérez Megias, L., Andecochea Saiz, C., Sanchis, S., Pérez-Moya, M., García-Montaño, J. & You, X. (2024). Pilot scale on-site demonstration and seasonality assessment of nitrogen recovery and water reclamation from pig's slurry liquid fraction. Journal of Environmental Management, 370, 122597.

80. Rojo, E. M., Piedra, I., González, A. M., Vega, M., & Bolado, S. (2021). Effect of process parameters on the valorization of components from microalgal and microalgal-bacteria biomass by enzymatic hydrolysis. Bioresource Technology, 335, 125256. https://doi.org/10.1016/j.biortech

81. Romero García, J.M.; Acién Fernández, F.G.; Fernández Sevilla, J.M. Development of a process for the production of l-amino-acids concentrates from microalgae by enzymatic hydrolysis. Bioresour. Technol. 2012, 112, 164–170.

82. Rout, P. R., Pandey, D. S., Haynes-Parry, M., & Goel, M. (2022). Sustainable Valorisation of Animal Manures via Thermochemical Conversion Technologies: An Inclusive Review on Recent Trends. Waste and Biomass Valorization. Waste Biomass Valor 14, 553–582 (2023). https://doi.org/10.1007/s12649-022-01916-5

83. Sadaka, S., & Ahn, H. (2012). Evaluation of a biodrying process for beef, swine, and poultry manures mixed separately with corn stover. Applied Engineering in Agriculture, 28(3), 457-463. doi: 10.13031/2013.41482

84. Samanta, P., Schönettin, H.M., Horn, H., Saravia, F., 2022. MF–NF treatment train for pig manure: nutrient recovery and reuse of product water. Membranes 12, 165. https://doi.org/10.3390/membranes12020165

85. Santoleri, J. J.; Hazardous Waste Incineration, Editor(s): Robert A. Meyers, Encyclopedia of Physical Science and Technology 2003, 3, 223-244.

86. Santos, J.; Ouadi, M.; Jahangiri, H.; Hornung, A. (2019) Integrated intermediate catalytic pyrolysis of wheat husk. Food and Bioproducts Processing, 114, 23-30. https://doi.org/10.1016/j.fbp.2018.11.001

87. Sapkota, T.B., Jat, M.L., Jat, R.K., Kapoor, P., Stirling, C., 2016. Yield Estimation of Food and Non-food Crops in Smallholder Production Systems. Methods Meas. Greenh. Gas Balanc. Eval. Mitig. Options Smallhold. Agric. https://doi.org/10.1007/978-3-319-29794-1_8.

88. Sharara, M. A., Sadaka, S., Costello, T. A., & VanDevender, K. (2012). Influence of aeration rate on the physio-chemical characteristics of biodried dairy manure-wheat straw mixture. Applied Engineering in Agriculture, 28(3), 407-415. doi: 10.13031/2013.41489

89. Skouteris, G., Rodriguez-Garcia, G., Reinecke, S. F., & Hampel, U. 2020. The use of pure oxygen for aeration in aerobic wastewater treatment: A review of its potential and limitations. Bioresour. Technol. 312, 123595–123595. https://doi.org/10.1016/j.biortech.2020.123595

90. Smet, E., Debruyne, J., Deckx, J., Deboosere, S. (2003). Manure treatment according to the Trevi-concept. Commun. Agricultural and Appl. Biological Sci. 2 (PART A), 125–132.

91. Song, E., Park, S., Han, S., Lee, E., & Kim, H. (2022). Characteristics of Hydrothermal Carbonization Hydrochar Derived from Cattle Manure. Energies, 15(23), 9195. https://doi.org/10.3390/en15239195.

92. Tambone, F., Scaglia, B., Scotti, S., & Adani, F. (2011). Effects of biodrying process on municipal solid waste properties. Bioresource technology, 102(16), 7443-7450. https://doi.org/10.1016/j.biortech.2011.05.010

93. Tambone, F., et al. (2017). "Solid and liquid fractionation of digestate: Mass balance, chemical characterization, and agronomic and environmental value." Bioresource Technology 243: 1251-1256.

94. Tampio, E., Marttinen, S., & Rintala, J. (2016). Liquid fertilizer products from anaerobic digestion of food waste: mass, nutrient and energy balance of four digestate liquid treatment systems. J. Clean. Prod. 125, 22–32. https://doi.org/10.1016/j.jclepro.2016.03.127

95. The United Nations. Take Action for the Sustainable Development Goals—United Nations Sustainable Development. United Nations Sustainable Development. (2020). Available online: https://www.un.org/sustainabledevelopment/sustainable-development-goals/ (accessed on May 17th 2022).

96. Uald-lamkaddam, I., Dadrasnia, A., Llenas, L., Ponsá, S., Colón, J., Vega, E., Mora, M., 2021. Application of freeze concentration technologies to valorize nutrient-rich effluents generated from the

anaerobic digestion of agro-industrial wastes. Sustainability 13, 13769. https://doi.org/10.3390/su132413769

97. Van Hulle, S. W. H., Callens, J., Mampaey, K. E., van Loosdrecht, M. C. M., & Volcke, E. I. P. (2012). N2O and NO emissions during autotrophic nitrogen removal in a granular sludge reactor – a simulation study. Environ. Technol.33(20), 2281–2290. https://doi.org/10.1080/09593330.2012.665492

98. Vazifehkhoran A.H., Finzi A., Perazzolo F., Riva E., Ferrari O., Provolo G. (2022). Nitrogen recovery from differnet livestock slurries with an innovative stripping process. Systainability 14 (13), 7709 https://doi.org/10.3390/su14137709

99. Wang, Y.; Zhu, Y.; Zhang, S.; Wang, Y. (2018). What could promote farmers to replace chemical fertilizers with organic fertilizers? J. Clean. Prod. 199, 882–890. https://doi.org/10.1016/j.jclepro.2018.07.222.

100. VCM, 2021. Nota VCM Waterrecuperatie uit mest. https://www.vcm-mestverwerking.be/nl/nieuws/153/vcm-nota-waterrecuperatie-uit-mest/ (accessed 20 May 2023).

101. Weiping Song, Mingxin Guo. (2012). Quality variations of poultry litter biochar generated at different pyrolysis temperatures. Journal of Analytical and Applied Pyrolysis 94 (2012) 138–145

102. Willeghems, G., De Clercq, L., Michels, E., Meers, E., & Buysse, J. (2016). Can spatial reallocation of livestock reduce the impact of GHG emissions? Agric. Syst. 149, 11–19. https://doi.org/10.1016/j.agsy.2016.08.006

103. Wijffels, R. H., & Barbosa, M. J. (2010). An outlook on microalgal biofuels. science, 329(5993), 796-799.

104. Winkler, M. K., Bennenbroek, M. H., Horstink, F. H., Van Loosdrecht, M. C. M., & Van de Pol, G. J. (2013). The biodrying concept: An innovative technology creating energy from sewage sludge. Bioresource Technology, 147, 124-129. https://doi.org/10.1016/j.biortech.2013.07.138

105. Yue, Y., Lin, Q., Xu, Y., Li, G., & Zhao, X. (2017). Slow pyrolysis as a measure for rapidly treating cow manure and the biochar characteristics. Journal of Analytical and Applied Pyrolysis, 124, 355-361.. https://doi.org/10.1016/j.jaap.2017.01.008

FERTIMANURE

INNOVATIVE NUTRIENT RECOVERY FROM SECONDARY SOURCES-PRODUCTION OF HIGH-ADDED VALUE FERTILISERS FROM ANIMAL MANURE

PROJECT COORDINATOR

Fundació Universitària Balmes (Spain)

CONSORTIUM

Ghent University (Belgium)
Wageningen Environmental Research (Netherlands)
University of Milan (Italy)
Leitat (Spain)
GreenWin (Belgium)
European Landowners Organisation (Belgium)
IPS Konzalting (Croatia)
Fraunhofer (Germany)
Dorset Green Machines (The Netherlands)
Prinsen Dairy Company (The Netherlands)
French Chamber of Agriculture (France)
Cooperativa Plana de Vic (Spain)
AlgaEnergy S.A. (Spain)
Fertinagro Biotech (Spain)
RITTMO Agroenvironnement (France)
Agrifutur (Italy)
Departament d'Agricultura, Ramaderia, Pesca I Alimentació (Spain)
Fertilizers Europe (Belgium)
Instituto Nacional de Tecnología Agropecuaria (Argentina)

PROJECT WEBSITE:

https://www.fertimanure.eu

Brief project summary

The mission of the FERTIMANURE project is to provide innovative solutions (technology. end-products. and business models) that solve real issues. ie the manure challenge. and help farmers with the challenges that they are currently facing. FERTIMANURE will develop. integrate. test and validate innovative nutrient management strategies so as to efficiently recover and reuse nutrients and other products with agronomic value from manure. to ultimately obtain reliable and safe fertilisers that can compete in the EU fertiliser market.

The FERTIMANURE project will cover both technological and nutrient management approaches. The technological side will be addressed with the implementation of 5 innovative & integrated on-farm experimental pilots for nutrient recovery in the most relevant European countries in terms of livestock production (Spain. France. Germany. Belgium. The Netherlands). whereas nutrient management will be addressed through 3 different strategies adapted to mixed and specialised farming systems:

Strategy #1 with on-farm production and use of bio-based fertilisers (BBF)(1) . **Strategy #2** with on-farm BBF production and centralised tailor-made fertilisers (TMF)(2) production. and **Strategy #3** with on-farm TMF production and use.

Definition of Bio-based fertilisers (BBFs): *Bio-based fertilisers (BBFs) are fertilising products or a component to be used in the production of (Tailor-Made) Fertilisers that are derived* ***from biomass-related resources.***

The BBFs of FERTIMANURE are "obtained through a ***physical. thermal/thermo-chemical. chemical. and/or biological processes for the treatment*** *of manure or digestate that result into a change in composition due to a change in concentration of nutrients and their ratios compared to the input material(s) in order to get better marketable products providing farmers with nutrients of sufficient quality".*

However. just separation of manure in a solid and liquid fraction (as first processing step) is excluded. These products are not conceived as a BBF. although they are valuable sources to supply nutrients on agricultural land.

LIST OF BBFs Produced in FERTIMANURE

Number	BBF-code	BBF product description
1	NL-AS	Ammonium sulphate solution
2	NL-LK	Liquid potassium fertiliser
3	NL-SC	Organic soil conditioner
4	NL-WP	Wet organic phosphorus rich fertiliser
5	NL-DP	*90% dried organic P rich fertiliser (calc)*
6	ES-NC	Nutrient-rich concentrate
7	ES-DSC	Bio-dried solid fraction
8	ES-PA	Phosphorous (ashes)
9	ES-AM	Ammonium salts
10	ES-AA	AA-based biostimulants
11	DE-BC	Biochar
12	DE-AP	Ammonium phosphate on perlite
13	BE-AN	Ammonium nitrate
14	BE-AS	Ammonium sulphate
15	BE-AW	Ammonium water
16	FR-BC	Biochar
17	FR-AS	Ammonium sulphate
18	FR-LK	Liquid K-fertiliser

Definition of Tailor-Made Fertilisers (TMFs): *A tailor-made fertiliser (TMF) is a customized fertiliser that meets with the nutrient requirements of a specific crop by taking into account the soil type. soil fertility status. and growing conditions and fertilisation practises.*

The TMFs obtained in FERTIMANURE are produced from BBFs (produced from manure or digestate and/or other recovered fertilising products that are available) and/or mineral fertilisers (MF) (and/or biostimulants).

Fully crop specific TMFs can be defined and centrally produced assuming e.g. a sufficient nutrient status of a soil type and no additional fertilisation practice.

However. on farm level the soil-crop requirements will be different due to another nutrient status of the soil and the fact that often manure/digestate will be applied on the fields which has to be taken into account as nutrient supplier. Consequently. the composition of the TMF (combination of BBF and MF) that will be used by the farmer can differ from the one produced in a centralised way.