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Document on product characteristics, lab results and field trials (year 4)

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Preface

This study was carried out and published as a part of the European demonstration project SYSTEMIC funded by the H2020 programme (project number 730400). The project SYSTEMIC focuses on five large-scale biogas plants where innovative nutrient recovery and re-use (NRR) processing techniques are implemented and monitored throughout the timespan of the project (2017-2021). One of the tasks within the SYSTEMIC project is to report on i) the characterisation of the recovered products, including nutrient content and presence of unwanted substances and ii) the performance of these products as fertilisers or soil improvers tested on laboratory and field scale. The overall objective is to obtain information about the effectiveness of the recovered fertilisers and soil improvers relative to conventional synthetic or organic products.

In this final report the results after four years of the project are reported. The D1.13 provides an overview of finalised experiments which include results on product testing from the five demo plants (Acqua & Sole (Italy), Am-Power (Belgium), Groot Zevert Vergisting (the Netherlands), BENAS (Germany) and Waterleau NewEnergy (Belgium), and three outreach locations: Bojana (Croatia), Waternet (the Netherlands) and RIKA Biofuels (UK).

The results of this report serve as an input for Environmental Impact Assessment (D1.15 '*Final report on environmental impact assessment of recovered products*') and Life Cycle Assessment (D2.6 '*Final report on LCA analysis and sustainability indicators of products'*). The underlying report includes all results collected during the SYSTEMIC project on product characterisation and testing, i.e. it merges results from confidential deliverables 1.10, 1.11 and 1.12.

We would like to acknowledge the plant owners and staff of Acqua & Sole, Am-Power, BENAS and GNS, Waterleau NewEnergy and Groot Zevert Vergisting whom regularly delivered product samples and information about their nutrient composition. Furthermore, we would like to acknowledge outreach location Bojana, Waternet and RIKA Biofuels, for providing their products for testing. Finally, we would like to acknowledge Faculty of Agriculture from Zagreb University (Croatia), University of Milano (Italy), Wageningen Environmental Research (the Netherlands) and Ghent University (Belgium) for testing obtained products and providing the respective information on their fertiliser and/or soil enhancer performance.

The authors

Summary

The information compiled in this report is divided over 5 chapters. Introduction is given in **Chapter 1**.

Chapter 2 focuses on characterisation of products collected and analysed from five SYSTEMIC demo plants. The characterisation complies information on macronutrients, micronutrients and heavy metals. In 2020, the monitoring was extended with analysis of organic pollutants (residues from pesticides, herbicides and pharmaceuticals). The content of nutrients in digestate varied between different biogas installations, according to the type and composition of processed feedstock, and biogas process conditions (e.g. organic loading rate, hydraulic retention time, temperature). Moreover, product characterisation has confirmed that application of NRR technology changes the initial composition of the treated digestate and up-concentrates nutrients in the recovered products. The number of compounds (herbicides, pesticides, and pharmaceuticals) detected varied between the plants and can be related to the feedstock of the plant. Residues of pharmaceutically active compounds were detected in digestate of AD plants where animal manure is part of the ration. However, no residues of pharmaceuticals were detected in digestate obtained by thermophilic digestion of sewage sludge. Finally, no residues were detected in purified water or ammonium sulphate solution.

Chapter 3 covers nine experiments which are reported in the form of extended abstracts: as certain experiments have been published in peer-review scientific journals or are currently under review or under preparation for submission to peer-review scientific journal. The first two experiments deal with assessment of nitrogen (N) and carbon (C) mineralisation rates of raw digestate (section 3.1) and solid fraction (SF) of digestates (section 3.2) from SYSTEMIC demo plants. For raw digestates, results showed that N release and mineralisation were significantly positively correlated with the initial NH_4^+-N:total N ratio of the products and negatively correlated with total C:total N. The N mineralisation was observed for all products and humifiable fraction of C ranged from 50 to 81% for raw digestates, suggesting that these materials could be suitable candidates to increase C storage in agricultural soils. For SFs, the various SFs showed similar patterns of C mineralisation and it was concluded that the nature of the organic matter (OM) was the main factor controlling C mineralisation in the different treatments. In terms of N, some SFs may cause temporary N immobilisation.

The third experiment (section 3.3) aimed to investigate the short-term emissions of CO_2, N_2O and CH_4 from end-products obtained from SYSTEMIC demo plants and from certain outreach locations. Despite their high N contents, the N_2O emissions from the end-products were lower than their mineral counterparts (urea and calcium ammonium nitrate (CAN)). N_2O emissions were lowest in the SFs, ammonium sulphate (AS) solutions and permeate water from reverse osmosis (RO). CO_2 emissions in some of the biobased fertilisers were high owing to their high OM contents. However, CO_2 emissions from biobased fertilisers may be considered as biogenic and, therefore, do not contribute to a net increase in atmospheric CO_2. Meanwhile, CH_4 emissions from all fertilisers were negligible i.e. not significantly different from the unfertilised soil control.

The fourth experiment (section 3.4) assessed the suitability of organic fibres recovered from digestate to replace peat in regular potting soil mixtures. Results have shown that organic fibres from GZV and BENAS are suitable to replace respectively 13 and 30% of peat in regular potting soil mixtures without inducing negative effects on plant growth. For the organic fibres of GZV, the limiting parameter is the electrical conductivity (EC) value (salt content) which is high due to the use of sulphuric acid in the treatment process. Organic fibres of BENAS have a lower EC value and hence are more suitable to replace peat in potting soil mixtures.

In the fifth experiment (section 3.5), the suitability of struvite as fertiliser was tested and compared to diammonium phosphate (DAP) in a pot experiment. Results showed that struvite cannot fully replace DAP as a starter fertiliser for maize but could be effective as a slow release fertiliser.

The sixth experiment (section 3.6) aimed to assess phosphorus (P) speciation and easily extractable P in SF's of digestate produced by the SYSTEMIC demo plants. P speciation and availability strongly differs among SFs of digestate. Besides total P content, suppliers of SFs should also inform farmers about the available fraction of P and its suitability as a P fertiliser or as a soil improver. Finally, the use of iron-salts or iron-rich feedstocks in the digester has a negative effect on the availability of P and should therefore be discouraged.

The last three experiments concern field trials where end-products of WNE, Bojana and A&S were tested in maize cultivation for respectively one, two and three-year growing season.

The results from **one-year field trial in Belgium** on WNE end-products (section 3.7) have shown no significant difference in biomass yields between treatments using synthetic mineral N and biobased fertilisers, nor on the soil post-harvest nitrate residue. When it comes to apparent N recovery (ANR) and N fertiliser replacement value (NFRV), it seems that to the relatively high soil N contribution masked the contribution of N fertiliser and, along with unfavourable weather conditions, resulted in high standard deviations of the calculated ANR and NFRV values: statistically significant difference only observed between CAN and digestate.

In the **Croatian two-year field trial** on end-products from Bojana AD plant (section 3.8), the highest dry grain yield was reported for synthetic N fertiliser treatment (NPK) followed by treatment where liquid fraction (LF) of digestate was applied in combination with NPK (at ratio 50:50). Over the period of 2-years, the calculated ANR was highest in NPK treatment, followed by treatments where LF of digestate and SF of digestate were applied in combination with NPK. For all biobased treatments NFRV was below 100%, with LF of digestate + NPK treatment resulting in highest NFRV of 78±10% in 2018 and 83±9% in 2019 – all by assuming that mineral fertilisation is 100% efficient, i.e. NFRV = 100%. Similar as in Belgian trial, on the basis of nitrate residue it was concluded that the use of LF and SF of digestate from Bojana AD plant should not additionally increase the risk of nitrate leaching compared to the use of synthetic N fertilisers.

Finally, from a **three-year field trial in Italy** (section 3.9) it was concluded that the use of the digestate produced by A&S did not cause negative effects on the soil (in terms of accumulations of heavy metals or organic pollutants), environment (in terms of odour, ammonia and GHG emissions) or on the maize produced, compared to the use of urea (in both treatments recovered AS solution was applied as topdressing). The only significant difference observed after three years of experimentation concerns the higher OC content in soils fertilised with digestate. When it comes to NFRV, the digestate treatment had a similar efficiency of that provided with urea (assumption 100%) by resulting in NFRV of 83.7%.

Chapter 4 reports on product evaluation and compliance with European Fertiliser Product Regulation (FPR) (2019/1009) and currently proposed RENURE criteria to authorise manure-derived recycled N fertilising products to be used above the application standard of 170 kg total N ha^{-1} y^{-1} for manure-derived N fixed by the Nitrates Directive. For SYSTEMIC demonstration plants, in FPR, Component Material Categories (CMC) 4 (Fresh crop digestate) and CMC 5 (Digestate other than fresh crop digestate) are relevant. In addition, criteria for CMC 10 (By-products within the meaning of Directive 2008/98/EC) which covers processed animal manure is relevant, along with CMC 11 that covers industrial by-products including e.g. AS solution obtained through stripping and scrubbing. However, criteria for CMC 10 and CMC 11 have not yet been defined. Finally, AS solution from BENAS and RO concentrate from GZV comply with the currently proposed RENURE criteria and as such have potential to be exempt from the imposed application standard of 170 kg total N ha^{-1} y^{-1}.

The document concludes with **Chapter 5** where an overall conclusion on reported results is given per SYSTEMIC demo plant and in relation to the promised impact of the project.

List of abbreviations

AD: Anaerobic digestion
AmP: Am-Power
ANR: Apparent nitrogen recovery
AOX: Adsorbable organic halides
AS: Ammonium sulphate
A&S: Acqua & Sole
CAN: Calcium ammonium nitrate
DAF: Dissolved air flotation
DAP: Di-ammonium phosphate
DM: Dry matter
EC: Electrical conductivity
EIA: Environmental impact assessment
FM: Fresh matter
GHG: Greenhouse gas emission
GWP: Global warming potential
GZV: Groot Zevert Vergisting
IX: Ionic exchange
JRC: Joint Research Centre
LF: Liquid fraction
MAC: Maximum allowable concentration
MF: Microfiltration
NFRV: Nitrogen fertiliser replacement value
NRR: Nutrient recovery and reuse
OC: Organic carbon
OM: Organic matter
PAHs: Polycyclic aromatic hydrocarbons
PCBs: Polychlorinated biphenyls
RO: Reverse osmosis
SF: Solid fraction
SFA: Segmented flow analyser
SOM: Soil organic matter
SOF: Solid organic fertiliser
TMF: Tailor-made fertiliser
TOC: Total organic carbon
UGhent: Ghent University
UMIL: University of Milan
WENR: Wageningen Environmental Research
WFPS: Water filled pore space
WNE: Waterleau NewEnergy
WWTP: Waste water treatment plant

List of definitions

Term	**Definition**
Digestate	Solid material remaining after the anaerobic digestion of a biodegradable feedstock.
Liquid fraction (LF) of digestate	LF of digestate after separation of digestate by a decanter centrifuge or screw press.
Solid fraction (SF) of digestate	SF of digestate after separation of digestate by a decanter centrifuge or screw press.
Reverse osmosis (RO) concentrate	Concentrate remaining after removal of water from a liquid stream (e.g. LF of digestate or condensed water) by RO.
Permeate water	Permeate after reverse osmosis, which needs further purification by means of ionic exchange prior to discharge to surface water.
Purified water	Water recovered from digestate by means of RO and IO (ionic exchange), purified to be used as process water or to be discharged to surface water.
Low phosphorus (P) soil improver	Solid fraction of the digestate after flushing with water and sulphuric acid to remove most of the P.
Precipitated phosphate salts	Precipitated phosphate salts, obtained by precipitation of phosphate (PO_4) with calcium, and which are recovered as a sludge.
Dried SF of digestate	SF of digestate after a thermal drying process.
Evaporator concentrate	LF of digestate, after evaporation of water and volatile components including ammonia.
Ammonium sulphate (AS) solution	Solution of AS obtained after ammonia stripping followed by recovery of gaseous ammonia in sulphuric acid (Acqua&Sole) or with gypsum (FibrePlus at BENAS).
Condensed ammonia water	Condensate after evaporation of LF of digestate with a high content of ammonium, and treated by RO to reduce the water content.
Condensed water	Condensate after evaporation of LF of digestate which contains water and volatile compounds including ammonia, bicarbonate and volatile organic acids.
Low nitrogen (N) organic fibres	SF of digestate obtained by a screw press from digestate after N stripping-scrubbing in the FibrePlus system and used for production of fibre.
Organic fibres	GZV: Organic fibres with a low N and P content, recovered from digestate by means of a screw press after two or three washing steps to remove P, salts and fine particles. BENAS: SF obtained by a screw press from digestate after N stripping-scrubbing in the FibrePlus system and used for production of fibre.
Calcium carbonate sludge	Precipitate of calcium and carbonate produced as a side product of the FibrePlus N stripping unit at BENAS by the reaction of striped gas containing ammonia and carbon dioxide with gypsum ($CaSO_4$) leading to the formation of ammonium sulphate and calcium carbonate precipitate.
Micro-filtration (MF) concentrate	Concentrate after treatment of LF of digestate by means of micro filtration (MF concentrate).

1 Introduction

The current European policy strongly focuses on the transition from a linear economy towards a circular economy (EC, 2015). Main goal is 'an economic sustainable growth by increasing the value of products, materials and raw materials as long as possible in the economy'. To facilitate the transition, the European Commission has implemented a new Fertiliser Regulation (Regulation on fertilising products 2019/1009) that will enter into force on July 16th 2022. The regulation focuses on the production of fertilisers from renewable raw materials which are classified into different categories. There is much attention on the organic fertilisers and organo-mineral fertilisers. Another main development is that the European Commission will set up criteria for nitrogen (N) fertiliser derived from manure which may be applied above the N application standard for manure as substitute for (synthetic produced) mineral N fertilisers. The first step towards this development is the Science for Policy report "*Technical proposals for the safe use of processed manure above the threshold established for Nitrate Vulnerable Zones by the Nitrates Directive (91/676/EEC)*" by the Joint Research Centre (JRC) (Huygens et al., 2020). The objective of the report is to help define those harmonised criteria that could allow N fertilisers, partially or entirely derived from manure through processing, to be used in areas subject to the ceiling of 170 kg total N ha^{-1} y^{-1} prescribed in the Nitrates Directive (91/676/EEC) following identical provisions applied to chemical N fertilisers. The concerned materials are called RENURE, from 'REcovered N from manURE'. Furthermore, there is an European initiative to increase the soil organic C stock in the soil with 4 promille (so called '4 per 1000' initiative', https://www.4p1000.org/).

Agricultural production is highly dependent on the availability of (fresh) water, macronutrients N, phosphorus (P) and potassium (K), and a healthy soil. P is a life-essential irreplaceable element and the P reserves are limited. The current worldwide P reserves are estimated at 70 000 Tg P and the world mining production in 2018 was 270 Tg P (USGS, 2019). Essentially, all chemical fertiliser and P in feed is mined from phosphate-rich rocks which are located in a few places (mainly Morocco 75%, but also in China, Finland and USA). As Europe has no significant phosphate mines, it is highly dependent on the import of phosphate ore (De Ridder et al., 2012). Large quantities of fossil fuel are used for the production of mineral N fertilisers. N fertiliser production is based on the Haber-Bosch process, which requires 22 GJ t^{-1} NH_3 fossil energy (EFMA, 2004). Besides the application of mineral fertiliser in agriculture, also organic biomass (like manure, digestate and compost and in some countries also sewage sludge) is used as source of organic matter (OM) to improve the soil quality and as sources of macro (N, P and K), secondary (Ca, Mg and S) and micro (B, Cu, Fe, Mn, Mo, Zn, etc.) nutrients. Furthermore, there is a tendency to create more value out of organic biomass 'waste' streams e.g. by producing biogas as substitute for natural gas and by recovering nutrients as substitute for the 'synthetic produced' mineral fertilisers.

Within the Horizon 2020 project SYSTEMIC (Grant Agreement no. 730400) innovative nutrient recovery and reuse (NRR) techniques are implemented at five large scale biogas plants (hereinafter referred to as demonstration plants). The overall objective of the SYSTEMIC project is to reach a break-through in reuse of nutrients recovered from biowaste (manure, sewage sludge as well as food, feed and agricultural waste) in the agricultural production cycle. The focus of the project is on demonstration of circular economy solutions for biowaste management by an effective combination of anaerobic digestion (AD) and novel NRR technologies at five full-scale demonstration plants. By implementing NRR techniques at biogas plants the digestate will change in composition and, depending on the processing techniques, different types of biobased fertilisers will be produced. Consequently, new nutrient management strategies for agricultural land will become available, since different products are recovered from digestate. From an agronomic point of view the available nutrients can be applied more in line with the requirements of the crops. However, also the environmental impact can change due to the changes in applied products (both quality and quantity).

The main aim of this report is to analyse the produced end products (from five demonstration plants) on relevant agronomical and environmental parameters and to show-case the agronomic performance of the products on laboratory and field scale as a potential fertiliser and/or soil enhancer. The full quantification of the environmental impact of changes in nutrient management strategies on agricultural land due to nutrient recovery is reported in D1.15 *'Final report on environmental impact assessment of recovered products'* (Schoumans et al., 2022).

Since the product testing is an important aspect of the SYSTEMIC project, a master plan on documenting product characteristics, lab results and field trials for all NRR end-products and digestate is given in Table 1-1. The plan specifies the work that has been done during the 4 years of the SYSTEMIC project.

Table 1-1 'Master plan' of product testing in Systemic project. Letter 'X' indicates in which type of assessment a certain product was included, and in which chapter/section results are reported.

Anaerobic digestion (AD) plant	**Type and internal classification of the products (i.e. feed, intermediate, end-product and water)**		**Nutrient content and heavy metals (Ch. 2)**	**Micro-pollutants (Sec. 2.2)**	**P speciation in solid fertilisers (Sec. 3.6)**	**N incubation (Sec. 3.1 - 3.2)**	**C incubation (Sec. 3.1 - 3.2)**	**Pot trials (Sec. 3.4 - 3.5)**	**Field trials (Sec. 3.7 - 3.9)**	**NH_3 and GHG emissions (Sec. 3.3)**
SYSTEMIC demonstration plants										
GZV										
	Feed of NRR	Digestate	X	X		X	X			X
	End product	RO concentrate	X	X						X
	End product	SF of digestate	X	X	X	X	X			X
	End product	Low P soil improver	X	X	X	X	X	X	X	X
	End product	Precipitated phosphate salts	X	X						
	Water	Purified water	X	X						
Am-Power										
	Feed of NRR	Digestate	X	X		X	X			X
	End product	RO concentrate (old process until November 2018)*	X							X
	End product	Evaporator concentrate (part of new process in 2020)	X	X						X
	End product	Dried SF of digestate	X	X	X	X	X			X
	Water	Purified water	X	X						
Acqua & Sole										
	End product	Digestate**	X	X		X	X		X	X
	End product	Ammonium sulphate solution	X	X					X	X
Waterleau NewEnergy										
	Feed of NRR	Digestate	X	X					X	X
	End product	Dried SF of digestate	X	X	X					X
	End product	Dried SF of digestate mixed with evaporator concentrate	X		X					
	End product	Evaporator concentrate	X	X					X	X
	End product	Condensed ammonia water	X						X	X
	Water	Purified water	X	X						

Anaerobic digestion (AD) plant	Type and internal classification of the products (i.e. feed, intermediate, end-product and water)		Nutrient content and heavy metals (Ch. 2) *	Micro-pollutants (Sec. 2.2)	P speciation in solid fertilisers (Sec. 3.6)	N incubation (Sec. 3.1 - 3.2)	C incubation (Sec. 3.1 - 3.2)	Pot trials (Sec. 3.4 - 3.5)	Field trials (Sec. 3.7 - 3.9) **	NH_3 and GHG emissions (Sec. 3.3)
BENAS										
	Feed of NRR	Digestate	X	X		X	X			X
	End product	LF of digestate	X	X						
	End product	Ammonium sulphate solution	X	X						X
	End product	SF of digestate	X	X	X					
	End product	Low N organic fibres	X	X	X			X		
	End product	Calcium carbonate sludge	X							
SYSTEMIC Outreach locations										
RIKA										
	Feed of NRR	Digestate (replaced by third party)				X	X			X
	End product	SF of digestate				X	X			
	End product	Ammonium sulphate solution								X
Bojana										
	Feed of NRR	Digestate							X	X
	End product	LF of digestate				X	X		X	X
	End product	SF of digestate				X	X		X	X
Waternet										
	End product	Precipitated phosphate salts						X		

NH_3: ammonia; GHG: greenhouse gas emission; RO: reverse osmosis; SF: solid fraction; LF: liquid fraction; P: phosphorus; GZV: Groot Zevert Vergisting; N: nitrogen; C: carbon; NRR: nutrient recovery and reuse.

** After November 2018, Am-Power installed an evaporator and RO concentrate is not produced in the new NRR configuration.*

*** Digestate from Acqua & Sole is defined as an end product because N stripping-scrubbing is coupled to AD, and hence N content in digestate has been reduced.*

2 Product characteristics

2.1 Physicochemical characterisation

Within the SYSTEMIC project, five biogas installations implemented different approaches for the reuse of nutrients from digestate. The five large-scale demonstration plants are located in Belgium (Am-Power and Waterleau NewEnergy), Germany (BENAS), Italy (Acqua & Sole), the Netherlands (Groot Zevert Vergisting). Table 2-1 gives an overview of the feedstock and produced products of the demonstration plants.

Table 2-1 Overview of feedstock and produced biobased fertilisers (and other end-products) at five demonstration plants.

Name	Location	Feedstock quantity (2020)	Feedstock	Biobased fertilisers and other end-products
Groot Zevert Vergisting	Beltrum (NL)	115 kt y^{-1}	Pig slurry, Biowaste from agro-industry	• RO concentrate • MF concentrate • SF of digestate • Low-P soil improver • Precipitated P salt • Purified water
Am-Power	Pittem (BE)	134 kt y^{-1}	Biowaste from agro-food industry	• Evaporator concentrate • Dried SF of digestate • Condensed water
Acqua & Sole	Vellezzo Bellini (IT)	77 kt y^{-1}	Sewage sludge, Biowaste	• AS solution • Digestate
BENAS	Ottersberg (DE)	87 kt y^{-1}	Corn silage, Poultry litter	• AS solution • Calcium carbonate sludge • LF of digestate • SF of digestate
Waterleau NewEnergy	Ypres (BE)	60 kt y^{-1}	Pig slurry Biowaste Sewage sludge	• Condensed ammonia water • Evaporator concentrate • Dried SF of digestate • Purified water

At **Groot Zevert Vergisting** (GZV), digestate is separated into an SF of digestate and LF of digestate by means of a decanter centrifuge. The LF of digestate is further processed in an advanced membrane system consisting of microfiltration and reverse osmosis (RO) producing an RO concentrate with >90% of N as N-NH_4 and that is being used as an alternative for synthetic N fertiliser (RENURE product). As a side product, a microfiltration (MF) concentrate is produced with a high N but low P content that is used as an organic N fertiliser. The SF of digestate is sold as an organic P fertiliser in Germany. Part of the SF of digestate is treated with the RePeat process where P is precipitated in the form of a calcium phosphate sludge hereafter referred to as precipitated P salts. The remaining low-P soil improver is now being used on sandy soils in the region of the plant as part of a pilot. Though farmers are interested in low-P soil improvers, the market value is low when used in agriculture. Therefore, GZV is exploring opportunities for creating of a new market for low-P soil improvers as an alternative for peat in potting soil thereby creating a higher value for their product. The reported composition of end-products from GZV was taken in the period 2019-2021. At **Am-Power** (AmP), for the first two years of the project, digestate was processed with a NRR system where the LF of digestate was transformed into RO concentrate and permeate by means of a dissolved air flotation (DAF) and RO system. The SF of digestate was dried into an organic soil improver and exported to P-deficient regions. In November 2019, AmP started with the construction of the evaporation and the new RO system with the aim of producing an evaporator concentrate and purified water. The construction and fine-tuning of the first part of the evaporator system lasted for the whole 2019. In January – February

2020 a sampling campaign was performed to assess the efficiency of the vacuum evaporator. In this intermediate phase, the condensed water (i.e. condensate from evaporator) resulted to have a pH of 9.9 ± 0.08 and an N content of about 2.5 ± 0.28 g kg^{-1}. The high pH and N of the condensed water required a substantial dosage of sulphuric acid to lower the pH in order to retain mineral N in the RO concentrate. As a consequence, AmP decided to implement an acidification step prior to the evaporation unit in order to decrease the amount of NH_3 evaporated. The final set-up includes an acidification step prior to the evaporator and the reported compositions of condensed water and evaporator concentrate were taken in the period October 2020 – April 2021. Results from this monitoring campaign indicated a strong decrease of N content in the condensed water (<1 g kg^{-1}). Nevertheless, condensed water needs post treatment via the RO in order to meet criteria for discharge onto surface water. During several tests, feeding of the condensed water to the RO resulted in quick fouling of membranes, likely due to the presence of fatty acids, preventing the correct operation of this step. As such, condensed water is considered as a final product from AmP's NRR system within the context of this report. At time of writing, AmP is still searching for solutions to circumvent this issue, enabling the correct operation of the final RO step in order to turn condensed water into dischargeable water.

At **Acqua & Sole** (A&S), N is recovered via N-stripping and scrubbing from digestate in the form of ammonium sulphate (AS) solution and the resulting N-depleted digestate is applied locally on agricultural land. Within SYSTEMIC project, A&S implemented a novel N-scrubber with a higher N removal and recovery efficiency as compared to the previous N scrubber. The composition of digestate and AS solution generated after the implementation of the novel N adsorbing system has been assessed by analysing these products from October 2020 until April 2021.

At **BENAS**, N is recovered from digestate via an N-stripping and scrubbing system which relies on flue gas desulphurisation (FGD) gypsum instead of sulphuric acid solution. As a result, AS solution and calcium carbonate ($CaCO_3$) sludge are generated. N-depleted digestate is recirculated back to the digester and digestate from the post-digester is separated into a LF and a SF that are eventually applied on the fields. The composition of digestate, SF of digestate, LF of digestate, AS solution and $CaCO_3$ has been assessed by analysing these products from August 2017 to February 2021 in dedicated sampling campaigns.

At **Waterleau NewEnergy** (WNE), the SF of digestate is dried and exported to P deficient regions. The LF of digestate is first subjected to an aeration step for partial chemical oxygen demand (COD) removal and successively processed into a vacuum evaporator. Within the evaporator, N is stripped from the condensate to generate condensed ammonia water and N poor condensate (i.e. process water). The concentrate after evaporation (referred to as evaporation concentrate) is either applied on fields as organo-mineral fertiliser or mixed with the dried SF of digestate. Process water is either recirculated within the process or treated in a RO system to generate RO permeate or purified water. The quality of digestate and final products was assessed between January 2019 and March 2021.

The characteristics of digestate and different final products produced at SYSTEMIC demo plants are reported in this chapter. The parameters included are:

- pH, electrical conductivity (EC), dry matter (DM), organic matter (OM), total organic C (TOC)
- **Primary and secondary macronutrients:** N, P, K, S, Ca, Mg
- **Micronutrients:** Zn, Mn, Fe, Cu, Na, Co, Mn
- **Heavy metals:** Al, Cd, Ni, Pb, Cr, Hg, As
- **Emerging organic pollutants:** Residues of herbicides, pesticides and pharmaceutically-active compounds
- **Pathogens:** *Salmonella* spp., faecal coliforms or *E. coli*, Enterococcaceae

2.1.1 Digestate

The content of nutrients in digestate varied between different biogas installations, according to the type and composition of processed feedstock (Table 2-2). All digestates have a slightly alkaline pH varying between 8.1 and 8.6 which is due to the digestion process. The EC ranged from 26 to 46 mS cm^{-1}. The highest DM and OM content (107 and 83 g kg^{-1} FM respectively) were found in digestate from BENAS, due to processing feedstocks with a high DM content such as silage maize, while the highest concentrations of TN and TP were found in digestate from A&S, which reflected the characteristics of the processed feedstock (mainly sewage sludge). GZV digestate contained the highest fraction of NH_4-N (5.2 g kg^{-1} FM) and displayed the highest NH_4-N/TN ratio amounting to 61%. Compared to the other demonstration plants, digestate produced at BENAS had the highest share of TK (6.8 ± 1 g kg^{-1} FM). Major concentrations of trace elements were found as expected in the digestate of A&S. A&S digestate also contains a consistent amount of Fe, probably due to the use of these chemicals for P precipitation on the wastewater treatment plants (WWTP) that deliver sewage sludge to A&S. Considerable amounts of Al and Fe were found also in digestate from AmP (respectively 5090 ± 241 and 19933 ± 1324 mg kg^{-1} DM), as a result of the use of food industry sludge as AD feedstock.

It must be specified that digestate produced at A&S and BENAS was characterised after being subjected to N-stripping, thus displaying a lower N content compared to raw digestate if there was no N stripper. The full characterisation of raw digestate (before being subjected to N-stripping) is available for BENAS but not for A&S and it is reported in deliverable D1.5.

Table 2-2 Chemical characterisation of digestate produced at Groot Zevert Vergisting (GZV), Am-Power (AmP), Acqua & Sole (A&S), BENAS and Waterleau NewEnergy (WNE). For GZV, AmP and WNE, the composition of raw digestate is given whereas for A&S and BENAS, the composition of the end product after N stripping is given.

Parameters	**Unit**	**GZV** [a] Raw digestate	**AmP** [b] Raw digestate	**A&S** [c] After N-stripping	**BENAS** [d] After N-stripping	**WNE** [e] Raw digestate
pH	-	8.2 ± 0.13	8.1 ± 0.12	8.6 ± 0.096	8.3 ± 0.20	8.5 ± 0.29
EC	mS cm^{-1}	47± 3.5	26 ± 0.9	7.3 ± 0.20	28 ± 4.2	34 ± 2.5
DM	g kg^{-1} FM	81 ± 3.8	81 ± 4.5	106 ± 3.2	107 ± 22	57 ± 8.8
OM	g kg^{-1} FM	59 ± 3.3	50 ± 3.1	63 ± 2.3	83 ± 8.6	33 ±5.0
TOC	g kg^{-1} FM	-	25 ± 6.7	34 ± 4.3	39 ± 13	14 ±4.3
TN	g kg^{-1} FM	7.3 ± 0.66	4.9 ± 0.29	8.0 ± 0.31	7.2 ±1.7	6.5 ± 0.33
NH_4-N	g kg^{-1} FM	5.0 ± 0.33	2.3 ± 0.52	3.7 ± 0.074	3.6 ± 0.92	3.7 ± 0.68
TP	g kg^{-1} FM	1.7 ± 0.1	1.4 ± 0.19	3.4 ± 0.40	1.4 ± 0.38	1.0 ± 0.45
TK	g kg^{-1} FM	4.5 ± 0.2	3.3 ± 0.31	0.59 ± 0.062	6.1 ± 1	3.9 ± 1.6
TS	g kg^{-1} FM	0.67 ± 0.039	1.0 ± 0.13	1.1 ± 0.021	1.1 ± 0.19	0.93 ± 0.48
Ca	g kg^{-1} FM	1.7 ± 0.064	1.6 ± 0.15	5.9 ± 0.68	3.1 ± 1.6	1.2 ± 0.49
Mg	g kg^{-1} FM	1.0 ± 0.054	0.38 ± 0.062	0.59 ± 0.094	0.69 ± 0.14	0.31 ± 0.16
Na	g kg^{-1} FM	1.6 ± 0.21	2.4 ± 0.27	0.21 ± 0.015	0.44 ± 0.29	2.5 ± 0.97
Cu	mg kg^{-1} DM	325 ± 219	76 ± 10	347 ± 41	54 ± 20	156 ± 61
Zn	mg kg^{-1} DM	693 ± 31	337 ± 55	1060 ± 105	328 ± 100	505 ± 94
Al	mg kg^{-1} DM	655 ± 45	5090 ± 241	33250 ± 5728	651 ± 26	2292 ± 952
Cd	mg kg^{-1} DM	<0.4	<1.3	0.87 ± 0.24	0.79 ±0.45	<7.2
Co	mg kg^{-1} DM	1.5	2.4 ± 0.59	6.2 ± 0.5	2.6 ± 1.2	3.1 ± 1.3
Ni	mg kg^{-1} DM	15 ± 4.1	15 ± 1.4	54 ± 6.0	8.7 ± 2.6	16 ± 6.8
Pb	mg kg^{-1} DM	<5	<13	70 ± 12	6.0 ± 5.2	4.3 ± 2.8
Cr	mg kg^{-1} DM	16 ± 8.6	16 ± 1.5	74 ± 5.7	6.9 ± 2.9	15 ± 5.3
Cr VI	mg kg^{-1} DM			<0.5	<1	<1.0
Hg	mg kg^{-1} DM	<0.05		<1.3	0.02	<0.014
As	mg kg^{-1} DM	<1		7.7 ± 1.7	0.99	0.97
Fe	mg kg^{-1} DM	2450 ± 354	19933 ± 1324	18700 ± 5657	13433 ± 6067	5138 ± 859
Mn	mg kg^{-1} DM		246 ± 21	464 ± 85	566 ± 774	326 ± 53
Salmonella spp.	MPN g^{-1} DM	present	absent	Absent		
Fecal coliforms	MPN g^{-1} DM		<10			
E. coli	CFU g^{-1}	250		28 ± 1.3		
Enterococcaceae	CFU ml^{-1} MPN g^{-1} FM	2500		Absent		
Weed seeds (viable)			absent	absent		

[a] For GZV average of samples taken in the period September 2020 – February 2021 (n=5 for all parameters, except for heavy metals were n=2).

[b] For Am-Power average of samples taken in the period October 2020 – April 2021 (n=10 for all parameters, except for micronutrients, heavy metals and pathogens were n=5).

[c] For A&S, average of samples taken in the period October 2020 - April 2021 (n=8 for all parameters, except for pH, Fecal coliforms and Salmonella where n=3; for TS, Ca, Mg, Na, Al, Co, Fe and Mn were n=2).

[d] For BENAS average of samples taken in the period February 2018 – April 2021 (n=7 for all parameters, except for DM, TN, NH_4-N n=9; for EC, OM, Al, Co n=6; for TOC n=2, for Cr IV, Hg, As where n=1).

[e] For Waterleau NewEnergy average samples taken in the period February 2020 – March 2021 (n=10 for all samples, except for DM, NH_4-N, TP, TK, TS, Ca, Mg where n=11; for EC, Al, Co n=9; fro Hg, Cr IV and As n=1).

2.1.2 Ammonium sulphate solution and condensed ammonia water

Similarly to synthetic fertilisers, ammonium sulphate (AS) solution generated at A&S and BENAS contained TN entirely in mineral form (NH_4-N) (Table 2-3). Small discrepancies between TN and NH_4-N content should be attributed to the different analytical methodologies. The nutrient content in AS is strictly dependent on process conditions and efficiency, thus translating into high variability in NH_4-N (45-70 g kg^{-1} FM) and TS (54-85 g kg^{-1} FM) concentrations between AS solution produced at different plants. The weakly acidic/alkaline pH of AS solutions should not pose a risk to soil acidification and machinery corrosion, however, the high EC values (119-221 mS cm^{-1}) may represent a threat for salt-sensitive crops. Contents of TP and all other macro- and micronutrients and trace elements were mostly below the detection limits or detected in the order of mg kg^{-1} FM. If present, most of these nutrients and trace elements are probably derived from the sulphuric acid solution or the (FGD)-gypsum added during the NH_3 washing step after the stripping phase. FGD-gypsum used at BENAS was collected from a coal power plant and traces of elements other than Ca and S are therefore expected to be present. FGD-gypsum is comparable to natural gypsum with reference to heavy metal content, thus making it a promising product in agricultural applications (Watts and Dick, 2014). Both solutions, that are rich in N and S, were characterised by very low amounts of TOC: <1.6 and 0.35 ± 0.12 g kg^{-1} FM for A&S and BENAS respectively. In both cases, pathogens were absent or below the limit of quantification. According to the available literature, Bolzonella et al. (2018) recorded higher TN content in the recovered AS solution. The authors monitored a digestate processing system where the LF of digestate obtained via screw press and settler separation entered in a stripping and scrubbing unit. The AS solution contained 26 g kg^{-1} TN. Differently from the previous case, Ledda et al. (2013) described a digestate processing cascade where digestate was first mechanically separated and the LF of digestate was subsequently processed in a membrane filtration system. The centrate was treated in a stripping and scrubbing system resulting in 22-31% AS solution with a content of 51-61 g kg^{-1} TN.

At WNE, the evaporator system includes a stripping step where NH_3 is removed from the condensate and concentrated in a solution named condensed ammonia water (Table 2-3). Mineral and total N content resulted to be similar and differences are probably due to the different analytical methodologies. Over the monitoring period the NH_4-N content had a pretty high variation (± 27 g kg^{-1}). As such condensed ammonia water might not be suitable as mineral N fertiliser. Moreover, the high pH (about 11) may result in high N volatilisation and decreased fertiliser use efficiency.

Table 2-3 Chemical characterisation of AS solution produced at Acqua & Sole (A&S) and BENAS and condensed ammonia water produced at Waterleau NewEnergy (WNE).

Parameters	**Unit**	**A&S** [a] **Ammonium sulphate**	**BENAS** [b] **Ammonium sulphate**	**WNE** [c] **Condensed ammonia water**
pH	-	5.9 ± 1	7.8 ± 0.22	11 ± 0.46
EC	mS cm^{-1}	118 ± 3.3	221 ± 12	125 ± 23
DM	g kg^{-1} FM	360 ± 12	232 ± 23	-
TOC	g kg^{-1} FM	<1	0.35 ± 0.12	0.43 ± 0.17
TN	g kg^{-1} FM	75 ± 3.8	46 ± 3.7	96 ± 23
NH_4-N	g kg^{-1} FM	71 ± 0.31	45 ± 3.9	99 ± 19
TP	g kg^{-1} FM	0.012 ± 0.00035	0.0023 ± 0.0021	0.0005 ± 0.00069
TK	g kg^{-1} FM	0.017 ± 0.012	0.0063 ± 0.004	0.00055 ± 0.00075
TS	g kg^{-1} FM	85 ± 6.2	54 ± 4.4	0.50 ± 0.46
Ca	g kg^{-1} FM	0.043 ± 0.024	1.2 ± 0.29	0.00047 ± 0.00055
Mg	g kg^{-1} FM	0.068 ± 0.0022	0.010 ± 0.0088	0.00088 ± 0.00053
Na	g kg^{-1} FM	0.019 ± 0.01	0.0045 ± 0.0022	0.0014 ± 0.0011
Cu	mg kg^{-1} FM	<5	0.023 ± 0.024	<0.054
Zn	mg kg^{-1} FM	<8.5	012 ± 0.045	<0.054
Al	mg kg^{-1} FM	<0.1	0.73 ± 0.31	<0.25
Cd	mg kg^{-1} FM	<0.2	<0.011	<0.054
Co	mg kg^{-1} FM	<0.1	0.0059 ± 0.0046	<0.054
Ni	mg kg^{-1} FM	<1.1	0.20 ± 0.21	<0.054
Pb	mg kg^{-1} FM	<1	<0.011	<0.054
Cr	mg kg^{-1} FM	<0.23	0.015 ± 0.0044	<0.054
Hg	mg kg^{-1} FM	<0.25	-	-
As	mg kg^{-1} FM	<0.98	-	-
Fe	mg kg^{-1} FM	<9.8	12 ± 8.9	0.66 ± 0.089
Mn	mg kg^{-1} FM	2.4 ± 0.74	0.27 ± 0.18	<0.054

Salmonella spp.	CFU mL^{-1}	absent	
	in 25 g		absent
E. *coli*	CFU mL^{-1}	absent	
	CFU g^{-1}		<10
Enterococcaceae	CFU mL^{-1}	absent	
	CFU g^{-1}		<1

[a] *For Acqua & Sole average of samples taken in the period October 2020 - April 2021 (for all parameters n=4).*

[b] *For BENAS average of samples taken in the period April 2017 – March 2021 (for pH n=9; for DM, TN, NH$_4$-N n=7; for EC, TS n=6; for TOC, TP, TK, Ca, Mg, Na n=5; for Salmonella spp., E.coli, Enterococcaceae n=2).*

[c] *For Waterleau NewEnergy average of samples taken in the period January 2019 – March 2021 (for all parameters n=10, except for pH, EC, TN n=11; for TOC, Cu, Co, Cr n=9; for NH$_4$-N n=6; for Fe, Mn, Ni n=8 and for Al n=7).*

2.1.3 Liquid fraction of digestate

Table 2-4 gives the composition of the LF of digestate produced at GZV, AmP, BENAS and WNE. Only for BENAS is the LF of digestate an end-product; the other demo plants process LF into RO concentrate or evaporator concentrate. The LFs had comparable pH, however the BENAS's LF had a higher DM content, which translates in a higher nutrient content. Similarly, the high OM content of BENAS translated into a higher TN content. The higher DM and OM in BENAS LF of digestate may be due the high amount of undigested fibres, since the main feedstock source is silage maize. The ratio of the DM content in LF of digestate over the DM content in digestate (DM$_{LF}$:DM$_{digestate}$) of the screw press used at BENAS resulted to be 0.84, suggesting a poor solids separation. This is in agreement with Akhiar et al. (2017), where a DM$_{LF}$:DM$_{digestate}$ in screw press above 0.8 was observed. The use of decanter centrifuges in all other demo plants (GZV, AmP and WNE) resulted in lower DM content in the LF. This was especially observed for AmP and WNE, where polymeric flocculants are used during the separation step. LF of digestate from WNE displayed high NH$_4$-N:TN ratio (0.78) compared to the others (0.64 in AmP, 0.53 in BENAS and 0.69 in GZV). With almost 80% of TN in the form of mineral N, LF of digestate from WNE represents an interesting substitute for mineral N fertilisers (Tambone and Adani, 2017). For all demo plants, LF of digestate has a lower TP content as compared to unseparated digestate due to recovery of P in the SF.

Table 2-4 Chemical characterisation of liquid fraction of digestate produced at Groot Zevert Vergisting (GZV), Am-Power (AmP), BENAS and Waterleau NewEnergy (WNE).

Parameters	**Unit**	**GZV** [a]	**AmP** [b]	**BENAS** [c]	**WNE** [d]
pH	-	8.3 ± 0.11	8.3 ± 0.12	8.3 ± 0.17	8.8 ± 0.26
EC	mS cm^{-1}	49 ± 2.8	30 ± 3.1	30 ± 2.8	32 ± 2.2
DM	g kg^{-1} FM	49 ± 1.6	26 ± 5.0	95 ± 17	25 ± 2.9
OM	g kg^{-1} FM	32 ± 1.9	14 ± 5.0	67 ± 14	12 ± 2.4
TOC	g kg^{-1} FM	-	2.9 ± 1.2	26 ± 2.3	4.4 ± 1.6
TN	g kg^{-1} FM	6.8 ± 0.63	4.0 ± 0.62	6.8 ± 1.7	5.1 ± 0.91
NH$_4$-N	g kg^{-1} FM	4.7 ± 0.32	2.4 ± 0.78	3.6 ± 0.91	3.3 ± 0.77
TP	g kg^{-1} FM	0.62 ± 0.072	0.21 ± 0.061	1.6 ± 0.36	0.29 ± 0.079
TK	g kg^{-1} FM	4.7 ± 0.18	3.1 ± 0.39	6.3 ± 1	2.9 ± 0.69
TS	g kg^{-1} FM	0.51 ± 0.029	0.21 ± 0.092	1.0 ± 0.20	0.56 ± 0.19
Ca	g kg^{-1} FM	0.94 ± 0.051	0.18 ± 0.10	3.5 ± 1.3	0.22 ± 0.15
Mg	g kg^{-1} FM	0.24 ± 0.066	0.031 ± 0.014	0.71 ± 0.22	0.039 ± 0.039
Na	g kg^{-1} FM	1.7 ± 0.23	2.1 ± 0.27	0.50 ± 0.26	1.8 ± 0.40
Cu	mg kg^{-1} DM	330 ± 85	25 ± 9.4	65 ± 17	69 ± 53
Zn	mg kg^{-1} DM	1015 ± 30	98 ± 30	386 ± 97	106 ± 47
Al	mg kg^{-1} DM	695 ± 65	327 ± 196	712 ± 135	311 ± 122
Cd	mg kg^{-1} DM	0.5 ± 0.029	<21	<1.8	0.61 ± 0.064
Co	mg kg^{-1} DM	2.2 ± 0.055	4.1 ± 0.59	3.0 ± 1.5	3.6 ± 1.7
Ni	mg kg^{-1} DM	20 ± 5.7	19 ± 2.2	10 ± 3.1	17 ± 11
Pb	mg kg^{-1} DM	<5	<21	7.3 ± 6.3	<16
Cr	mg kg^{-1} DM	6.2 ± 0.065	<9.5	6.9 ± 2.7	7.7 ± 4.4
Fe	mg kg^{-1} DM	2450 ± 354	1656 ± 993	15720 ± 6670	835 ± 434
Mn	mg kg^{-1} DM	400 ± 10	80 ± 6.8	926 ± 206	77 ± 16
Hg	mg kg^{-1} DM	<0.05	-	-	-
As	mg kg^{-1} DM	<1	-	-	-

[a] *For GZV average of samples taken in the period September 2020 – February 2021 (n=5 for all parameters, except for heavy metals were n=2).*

[b] *For Am-Power average of samples taken in the period October 2020 – April 2021 (n=10 for all parameters, except for micronutrients and heavy metals were n=5).*

[c] *For BENAS average of samples taken in the period January 2018 – March 2020 (n=10 for all parameters, except for pH, OM n=9; for EC, Cu, Fe, Zn n=7; for Na, Al, Cd, Co, Cr, Mn, Ni, Pb n=6 and for TOC n=2).*

[d] *For Waterleau NewEnergy average of samples taken in the period January 2019 – March 2021 (for all parameters n=7).*

2.1.4 Concentrates

Table 2-5 gives the composition of RO concentrate generated at GZV and of evaporator concentrates generated at AmP and WNE.

In general, RO concentrate produced at GZV has a low DM and OM content, on average 37 ± 4.8 and 14 ± 4.3 g kg^{-1}. Average TN concentration in the RO concentrate from GZV was 8.1 ± 0.8 g kg^{-1} FM. TN was almost entirely present as NH_4-N (99%), which make RO concentrate an interesting substitute for synthetic mineral fertiliser, according to RENURE criteria. In addition, TP is low due to pre-treatment of the LF by microfiltration. TK and Na represented a considerable proportion of the salts in the RO concentrates, amounting to 7.9 ± 0.38 and 3.1 ± 0.44 g kg^{-1} FM, respectively. The high pH (8.4 ± 0.17) of the RO concentrates may cause volatilisation of NH_3 during the application of the product. Therefore, distribution of the product by injecting it into the soil is advisable. In comparison to a different digestate processing technology, such as the vibratory shear enhanced processing membrane filtration described by Vaneeckhaute et al. (2012), RO concentrate from GZV achieved higher K concentrations (7.9 ± 0.38 kg^{-1} FM compared to 2.9 ± 1.0 kg^{-1} FM). Nevertheless, the TN content of recovered RO concentrates from the two processes was comparable (8.1 ± 0.8 kg^{-1} FM and 7.3 ± 1.6 kg^{-1} FM).

The evaporator concentrate from AmP is characterised by a high mineral-N:TN ratio, as a consequence of the acidification step, which prevents NH_4-N to volatilise as NH_3. A secondary effect of the acidification is the increment of TS from 0.21 ± 0.1 g kg^{-1} FM in LF of digestate (Table 2-4) to 12 ± 1.6 g kg^{-1} FM in the concentrate after evaporation. Differently form AmP, the LF fed to the evaporator implemented at WNE is not acidified, resulting in a lower NH_4-N content in the concentrate 4.5 ± 5.6 g kg^{-1} FM, despite the high variability over time.

Table 2-5 Chemical characterisation of RO concentrate produced at Groot Zevert Vergisting (GZV) and evaporator concentrate produced at Am-Power (AmP) and Waterleau NewEnergy (WNE).

Parameters	**Unit**	**GZV** [a] RO concentrate	**AmP** [b] **Evaporator concentrate**	**WNE** [c] **Evaporator concentrate**
pH	-	8.4 ± 0.17	6.2 ± 0.28	7.7 ± 1.5
EC	mS cm^{-1}	89 ± 6.3	74 ± 6.3	96 ± 15
DM	g kg^{-1} FM	37 ± 4.8	115 ± 23	187 ± 27
OM	g kg^{-1} FM	14 ± 4.3	63 ± 14	91 ± 20
TOC	g kg^{-1} FM	-	28 ± 15	44 ± 9.3
TN	g kg^{-1} FM	8.1 ± 0.8	9.9 ± 1.8	13 ± 4.3
NH_4-N	g kg^{-1} FM	8.0 ± 0.77	5.3 ± 0.62	4.5 ± 5.6
TP	g kg^{-1} FM	0.15 ± 0.13	0.92 ± 0.26	2.1 ± 0.67
TK	g kg^{-1} FM	7.9 ± 0.38	10 ± 0.82	22 ± 4.6
TS	g kg^{-1} FM	1.5 ± 0.51	12 ± 1.6	11 ± 5.5
Ca	g kg^{-1} FM	0.059 ± 0.013	0.73 ± 0.34	1.4 ± 0.76
Mg	g kg^{-1} FM	0.04 ± 0.036	0.13 ± 0.074	0.24 ± 0.095
Na	g kg^{-1} FM	3.1 ± 0.44	7.0 ± 1.2	14 ± 2.4
Cu	mg kg^{-1} DM	<100	30 ± 13	51 ± 29
Zn	mg kg^{-1} DM	<550	118 ± 50	130 ± 75
Al	mg kg^{-1} DM	15 ± 5.0	1762 ± 649	372 ± 138
Cd	mg kg^{-1} DM	<0.41	<0.51	0.37 ± 0.23
Co	mg kg^{-1} DM	<2	3.0 ± 0.45	3.2 ± 1.5
Ni	mg kg^{-1} DM	14 ± 7.4	15 ± 2.5	21 ± 8.3
Pb	mg kg^{-1} DM	<5.1	<5.1	3.1 ± 1.8
Cr	mg kg^{-1} DM	<5.1	7.3 ± 2.7	7 ± 3.1
Cr IV				<1
Fe	mg kg^{-1} DM	93 ± 25	5178 ± 2157	2104 ± 930
Mn	mg kg^{-1} DM	<110	105 ± 41	89 ± 55
Hg	mg kg^{-1} DM	<0.058	-	0.012
As	mg kg^{-1} DM	<1.1	-	0.61
Salmonella spp.	in 25 g^{-1}	Absent	-	-
E. coli	MPN g^{-1}	<3	-	-
Enterococcaceae	MPN g^{-1}	<3	-	-

[a] For GZV average of samples taken in the period September 2020 – February 2021 (n=5 for all parameters, except for heavy metals were n=2).

[b] For Am-Power average of samples taken in the period October 2020 – April 2021 (n=10 for all parameters, except for micronutrients and heavy metals were n=5).

[c] For WNE average samples taken in the period February 2020 – March 2021 (n=10 for all samples except for pH, DM, TN, TP, TK, TS, Ca, Mg n=10;for Co, Cd n=9; for Al n=7; For Hg, Cr IV and As n=1).

2.1.5 Solid organic fertilisers and soil improvers

The lower water content of SFs of digestate translates into easier storage and transport compared to digestate and LF of digestate. As such, mechanical separation is a primary treatment essential when transport of digestate is required over long distances. The P-poor SF of digestate (i.e. low P soil improver) produced at GZV had a P content of 1.1 ± 0.25 g kg^{-1} FM (Table 2-6) which is about eight times lower compared to the untreated SF of digestate. The N content of the low P soil improver decreased from 12 ± 0.35 to 5.3 ± 0.37 g kg^{-1} FM and OM decreased from 242 ± 4.6 to 212 ± 14 g kg^{-1} FM. Moreover, the TS content increased from 1.9 ± 0.11 to 5.8 ± 0.94 g kg^{-1} FM due to the use of sulphuric acid in the RePeat system for the solubilisation and extraction of P. SF of digestate from BENAS was characterised by relatively high DM and OM values, due to the greater quantity of undigested fibres. The N:P ratio confirmed preferential segregation of P in SF of digestate: the ratio decreased from 5.1 in digestate and LF of digestate to 3.3 in SF of digestate. Compared to LF of digestate, SF of digestate was richer on average in TN, TP, TS, TCa and TMg. With the exception of Fe, SF of digestate produced at BENAS had the lowest content of micronutrients and trace elements due to the use of energy crops as major feedstock rather than manure or biowaste. The low N fibres are separated from the digestate after N stripping and contain the lowest amount of TN among all soil improvers (5.8 g kg^{-1} FM). Despite K and Na (and their ionic forms K^+ and Na^+) are very soluble, thus dissolved in the liquid phase (Masse et al., 2005), their behavior in LF and SF of digestate was similar for both GZV and BENAS streams. This is probably due to the low separation efficiency of the decanter without polymer addition (GZV) and the screw press (BENAS) for these elements. The soil improvers generated at AmP and WNE were dried SFs of digestate in both cases. The former had a DM content of 823 ± 70, the latter had a DM content of 904 ± 134 g kg^{-1} FM, which explain the high concentration of each macronutrient. In regards with trace elements, the dried SF of digestate from WNE has a higher content of Cu (266 ± 69 mg kg^{-1} DM) and Zn (772 ± 169 mg kg^{-1} DM), due to the use of animal manure as feedstock and the higher DM concentration during the evaporation step.

Table 2-6 Chemical characterisation of available soil improvers: solid fraction (SF) of digestate and low P soil improver at Groot Zevert Vergisting (GZV), dried SF of digestate at Am-Power, SF of digestate and low N fibres at BENAS, and dried SF of digestate at Waterleau NewEnergy (WNE).

Parameters	**Unit**	**GZV** [a] **SF of digestate**	**GZV** [b] **low P soil improver**	**AmP** [c] **Dried SF of digestate**	**BENAS** [d] **SF of digestate**	**BENAS** [e] **Wet low N Fibres**
pH	-	8.8 ± 0.15	6.7 ± 0.48	8.1 ± 0.25	8.5 ± 0.29	7.8 ± 0.14
EC	mS cm^{-1}	-	-	6.3 ± 1.5	4.7 ± 0.5	
DM	g kg^{-1} FM	313± 2.8	237 ± 16	823 ± 74	241 ± 22	243 ± 2.9
OM	g kg^{-1} FM	242 ± 4.6	212 ± 14	529 ± 29	202 ± 17	227 ± 5.3
TOC	g kg^{-1} FM	-	-	288 ± 26	96 ± 5.7	
TN	g kg^{-1} FM	12 ± 0.35	5.3 ± 0.37	23 ± 2.9	7.1 ± 1.7	2.7 ± 1.0
NH_4-N	g kg^{-1} FM	6.6 ± 0.33	2.0 ± 0.19	1.3 ± 0.59	3.0 ± 1.3	0.73 ± 0.38
TP	g kg^{-1} FM	8.9 ± 0.8	1.1 ± 0.25	19 ± 3.2	2.2 ± 0.49	1 ± 0.68
TK	g kg^{-1} FM	4.6 ± 0.34	1.1 ± 0.25	14 ± 1.7	5.6 ± 1.4	2.2 ± 1.6
TS	g kg^{-1} FM	1.9 ± 0.11	5.8 ± 0.94	11 ± 1.8	1.3 ± 0.33	0.49 ± 0.057
Ca	g kg^{-1} FM	7.7 ± 0.61	0.99 ± 0.075	23 ± 3.6	4.0 ± 1.6	1.2 ± 0.51
Mg	g kg^{-1} FM	6.4 ± 0.49	2.0 ± 0.27	5.5 ± 1.2	1.3 ± 0.30	0.78 ± 0.26
Na	g kg^{-1} FM	1.5 ± 0.15	-	8.6 ± 1.7	0.39 ± 0.26	<0.14
Cu	mg kg^{-1} DM	91 ± 17	70	88 ± 15	20 ± 6.6	<110
Zn	mg kg^{-1} DM	376 ± 30	268	405 ± 75	158 ± 61	58 ± 11
Al	mg kg^{-1} DM	-	-	7507 ± 1150	194 ± 49	144 ± 29
Cd	mg kg^{-1} DM	<0.4	<0.4	<0.17	<0.74	
Co	mg kg^{-1} DM	<1	<1	1.4 ± 0.36	1.0 ± 0.42	
Ni	mg kg^{-1} DM	11 ± 3.5	7.0	15 ± 2.9	3.6 ± 2.6	
Pb	mg kg^{-1} DM	<5	<5	<1.7	2.8 ± 2.6	
Cr	mg kg^{-1} DM	10 ± 0.33	10 ± 0.33	25 ± 4.5	3.2 ± 2.7	
Cr VI	mg kg^{-1} DM	-	-	-	<1	
Hg	mg kg^{-1} DM	<0.05	<0.05	0.03	<0.01	
As	mg kg^{-1} DM	<1	<1	1.3	0.45	
Fe	mg kg^{-1} DM	2200 ± 265	-	28579 ± 4399	5831 ± 1907	

Mn	mg kg^{-1} DM	170	130	322 ± 19	336 ± 75	-

Parameters	**Unit**	**BENAS** [f]	**WNE** [g]
		Dried low N Fibres	**Dried SF of digestate**
pH	-	5.9	7.9 ± 0.41
EC	mS cm^{-1}		7.7 ± 1.2
DM	g kg^{-1} FM	895	904 ± 134
OM	g kg^{-1} FM	871	637 ± 22
TOC	g kg^{-1} FM		325 ± 64
TN	g kg^{-1} FM	5.8	29 ± 4.7
NH$_4$-N	g kg^{-1} FM	0.20	4.6 ± 2.8
TP	g kg^{-1} FM	1.3	24 ± 3.5
TK	g kg^{-1} FM	0.86	15 ± 1.7
TS	g kg^{-1} FM	2.5	10 ± 1.1
Ca	g kg^{-1} FM	3.6	36 ± 9.4
Mg	g kg^{-1} FM	0.66	9.3 ± 0.90
Na	g kg^{-1} FM	0.28	8.9 ± 0.77
Cu	mg kg^{-1} DM	10	266 ± 69
Zn	mg kg^{-1} DM	71	772 ± 169
Al	mg kg^{-1} DM	235	3927 ± 925
Cd	mg kg^{-1} DM		0.5 ± 0.32
Co	mg kg^{-1} DM		2.5 ± 1.2
Ni	mg kg^{-1} DM	198	14 ± 4.5
Pb	mg kg^{-1} DM		9.7 ± 5.3
Cr	mg kg^{-1} DM	350	33 ± 11
Cr VI	mg kg^{-1} DM		<1
Hg	mg kg^{-1} DM		0.02
As	mg kg^{-1} DM		1.2
Fe	mg kg^{-1} DM	5028	9430 ± 2629
Mn	mg kg^{-1} DM		506 ± 96

[a] For GZV average of samples taken in the period September 2020 – February 2021 (n=5 for all parameters, except for heavy metals were n=2).
[b] For GZV average of samples taken in the period September 2020 – February 2021 (n=3 for all parameters, except for heavy metals where n=1).
[c] For Am-Power average of samples taken in the period October 2020 – April 2021 (n=10 for all parameters, except for micronutrients and heavy metals were n=5).
[d] For BENAS average of samples taken in the period April 2017 –April 2021 (n=12 for all parameters except for OM n=10; for Cu, Zn, Fe n=8; for Na, Mn, Pb n=7; for EC, Al, Cd, Co, Cr, Ni n=6; for TOC n=2).
[e] For BENAS average of samples taken in the period July - September 2021 (n=3)
[f] For BENAS average of samples taken in September 2021 (n=1).
[g] For WNE average of samples taken in the period June 2020 – February 2021 (n=8 for all samples except for NH$_4$-N, Mn, Cd, Cr, Ni, Pb n=7; for EC, OM, Na n=6; for Hg, Cr IV and As n=1).

2.1.6 Calcium carbonate and precipitated P salts

$CaCO_3$ sludge generated at BENAS had a high DM and Ca content, amounting to 698 ± 48 g kg^{-1} FM and 227 ± 35 g kg^{-1} FM, respectively (Table 2-7). Due to its high pH, it can be used as a liming agent without causing alkalinisation because it dissolves only in acid soils.

GZV produced precipitated P salts with about 17% DM which are rich in calcium phosphate and gypsum. The OM content of the precipitated P salts amounted to 40% of DM point to a poor separation of P and OM within the RePeat system thus hampering the use of the P salts in mineral fertiliser industry.

Table 2-7 Chemical characterisation of calcium carbonate sludge produced at BENAS and precipitated P salts produced at GZV.

Parameters	**Unit**	**BENAS** [a]	**GZV** [b]
		(Calcium carbonate)	**(Precipitated P salts)**
pH	-	7.9 ± 0.25	7.3 ± 0.22
EC	mS cm^{-1}	17 ± 2.6	0.0046 ± 0.0046
DM	g kg^{-1} FM	698 ± 48	171 ± 16
OM	g kg^{-1} FM	32 ± 7.3	70 ± 2.8
TOC	g kg^{-1} FM	0.68 ± 0.16	-
TN	g kg^{-1} FM	13 ± 3.1	46 ± 4.6
NH$_4$-N	g kg^{-1} FM	10 ± 6.2	5.2 ± 0.54
TP	g kg^{-1} FM	0.18 ± 0.041	9.3 ± 1.4
TK	g kg^{-1} FM	0.39 ± 0.21	2.6 ± 0.039
TS	g kg^{-1} FM	29 ± 13	15 ± 3.1

Ca	g kg^{-1} FM	227 ± 35	17 ± 4.3
Mg	g kg^{-1} FM	0.26 ± 0.16	5.5 ± 1.0
Na	g kg^{-1} FM	0.14 ± 0.10	1.2
Cu	mg kg^{-1} DM	4.0 ± 1.7	114
Zn	mg kg^{-1} DM	16 ± 2.7	414
Al	mg kg^{-1} DM	983 ± 189	1000
Cd	mg kg^{-1} DM	0.1 ± 0.044	<0.4
Co	mg kg^{-1} DM	0.47 ± 0.077	<1
Ni	mg kg^{-1} DM	3.8 ± 1.4	9.0
Pb	mg kg^{-1} DM	1.6 ± 0.08	<5
Cr	mg kg^{-1} DM	4.3 ± 0.73	21
Fe	mg kg^{-1} DM	909 ± 105	2567 ± 249
Mn	mg kg^{-1} DM	71 ± 34	1100
Salmonella spp.	in 25 g	absent	present
E*. coli*	CFU g^{-1}	<10	
	MPN g^{-1}		450
Enterococcaceae	CFU g^{-1}	<1	
	MPN g^{-1}		45000

[a] For BENAS average of samples taken in the period April 2017 – March 2021 (for pH n=9; for DM, TN, NH_4-N n=7; for EC, TS n=6; for TOC, TP, TK, Ca, Mg, Na n=5; for Salmonella spp., E.coli, Enterococcaceae n=2).

[b] For GZV average of samples taken in the period September 2020 – February 2021 (n=3 for all parameters, except for heavy metals where n=1).

2.1.7 Condensed and Purified water

GZV produces purified water by treating the RO permeate by an IX. This purified water is discharged to the surface water. As shown in Table 2-8, GZV's water leaves the plant with an NH_4-N concentration of 0.2 ± 0.095 mg L^{-1}. In the Netherlands, discharge limits are determined case by case by waterboards.

Before the implementation of the evaporator, AmP reused all generated permeate water on site for cleaning or for the production of polymer solution. With the new process, the biogas plant is planning to discharge the permeate water to the surface water. The production of water that meets criteria for discharge means a massive reduction in costs for transportation of digestate. At the time of writing, the RO unit to treat the condensed water generated at AmP was not operational. As a result, this stream is now the final product of this demonstration plant. The RO step is an essential step of the NRR system as it would allow to decrease the TN content (currently around 950 mg L^{-1} FM) and eventually meet Flemish discharge limits (15 mg TN L^{-1}).

WNE treats the process water from the evaporation system into a double pass RO system. Permeate from the first RO reaches a quality sufficient for the preparation of polymer solution. Permeate from the second RO (purified water) can be disposed into surface water, as it meets Flemish discharge limits for COD (125 mg L^{-1}), TSS (35 mg L^{-1}), TN (15 mg L^{-1}) and TP (2 mg L^{-1}).

Table 2-8 Chemical characterisation of permeate water produced at Groot Zevert Vergisting (GZV) and Am-Power.

Parameters	**Unit**	**GZV** [a]	**AmP** [b]	**WNE** [c]
		Purified water	**Condensed water**	**Purified water**
pH	-	5.3 ± 1.1	9.6 ± 0.2	6.6 ± 1.6
EC	mS cm^{-1}	0.045 ± 0.090	1678 ± 814	
DM	mg L^{-1} FM		-	<2
BOD	mg O_2 L^{-1}		-	<3
COD	mg O_2 L^{-1}		-	<7
TOC	mg L^{-1} FM			0.7
TN	mg L^{-1} FM	0.28 ± 0.078	950 ± 460	9.4 ± 1.9
NH_4-N	mg L^{-1} FM	0.20 ± 0.095	680 ± 570	
TP	mg L^{-1} FM	<0.10	<0.27	<0.2
TK	mg L^{-1} FM	<0.4	<0.27	
TS	mg L^{-1} FM	2.9 ± 3.3	29 ±5.3	2.4
Ca	mg L^{-1} FM	<1.2	<0.27	
Mg	mg L^{-1} FM	<0.15	<0.27	
Na	mg L^{-1} FM	<0.30	<0.27	
Cu	mg L^{-1} FM	<0.01	<0.027	
Zn	mg L^{-1} FM	0.025 ± 0.016	<0.027	
Al	mg L^{-1} FM	<0.03	<0.027	
Cd	mg L^{-1} FM	<0.000005	<0.027	
Co	mg L^{-1} FM	0.0000090 ± 0.0000057	<0.027	

Ni	mg L^{-1} FM	0.00038 ± 0.00039	<0.027	
Pb	mg L^{-1} FM	<0.000040	<0.027	
Cr	mg L^{-1} FM	0.000065 ± 0.000030	<0.027	
As	mg L^{-1} FM	<0.00002	-	
Fe	mg L^{-1} FM	<0.09	<0.027	
Mn	mg L^{-1} FM	<0.01	<0.027	

[a] For GZV average of samples taken in the period September 2020 – February 2021 (n=5 for all parameters, except for heavy metals were n=2).

[b] For Am-Power average of samples taken in the period October 2020 – April 2021 (n=10 for all parameters, except for micronutrients and heavy metals were n=5).

[c] For WNE average of samples taken in the period December 2020 – May 2021 (n=3 for all parameters, except for TOC and TS where n=1).

2.2 Organic micro pollutants

2.2.1 Sampling and analyses

For each demonstration plant, samples were collected from digestate and end products two or three times except for WNE which was only once because they had not entered the project during the sampling campaign in 2020. Samples were taken by the plant owners. All samples were sent to the laboratory of Lufa Nord West, Hameln (Germany) for a screening on residues of pesticides and herbicides (218 active substances examined) and pharmaceuticals residues (58 active substances examined). The detection limit depended on the sample matrix and hence, varied among the samples and is given in Table 2-9.

Appendix A1 contains a full lists of analyses substances and detection limits for 2020 and 2021. The herbicides glyphosate, AMPA, Glufosinat, and MPPA were only analysed in 2021 since they were not part of the standard package but were only analysed upon request. The screening on residues of pharmaceuticals consists mostly of residues of veterinary pharmaceuticals though most of the antibiotics included in the screening are also commonly used as human medicine. This focus on veterinary medicines was suitable for screening of products of GZV, Benas and WNE whom all have manure as part of their ration.

For A&S, using sewage sludge as their main feedstock, digestate was additionally send to LabAnalysis in Casanova Lonati (Italy) for a screening on residues of nine widely used medicines and hormones as listed below with detection limit on fresh matter (FM):

- Ciprofloxacin (0.01 mg kg^{-1})
- Sulfamethoxazole (0.01 mg kg^{-1})
- Fenofibrate (0.01 mg kg^{-1})
- Gemfibrozil (0.01 mg kg^{-1})
- Carbamazepine (0.01 mg kg^{-1})
- Metroprolol (0.10 mg kg^{-1})
- Diclofenac (0.01 mg kg^{-1})
- Ethynyl estradiol (0.01 mg kg^{-1})
- Estradiol (0.01 mg kg^{-1})

2.2.2 Results

Results of the screening on residues of herbicides, pesticides and pharmacologically active substances on samples of end products are shown in Table 2-9. The detection limit varied among the samples ranging from 0.001 mg kg^{-1} FM in purified water to 0.01 – 0.05 mg kg^{-1} in fertilising products.

Table 2-9 Results of screening on residues of herbicides, pesticides and pharmacologically active substances. Sample-specific detection limit and concentrations are in mg kg^{-1} fresh product. 1) and 2) refer to the first sampling round (2020) and second sampling round (2021), respectively.

	Sample-specific detection limit (mg kg^{-1})	**Herbicides, Pesticides (mg kg^{-1})**	**Pharmacologically active substances (mg kg^{-1})**
Groot Zevert Vergisting			
Digestate	0.01	1): Chloropropham (0.048) Fluazifop free acid (0.039) 2): Glyphosate (0.120)	1): Flubendazol (0.036) Sulfadiazin, sulfapyrimidin (0.014) 2): Not detected
SF of digestate	0.01	1): Chloropropham (0.161) Fluazifop free acid (0.066) 2): Not detected	1): Flubendazol (0.154) Sulfadiazin, sulfapyrimidin (0.011) 2): Not detected
RO concentrate	0.01	1): Not detected 2): Glyphosate (0.228) AMPA (0.081)	1): Sulfadiazin, sulfapyrimidin (0.041) 2): Not detected
Low-P soil improver	0.01	1): Not analysed 2): Not detected	1): Not analysed 2): Not detected
Purified water	0.0005	1): Not detected 2): Not detected	1): Not detected 2): Not detected
BENAS			
Digestate	0.05	1): Piperonylbutoxide (0.043) 2): Piperonylbutoxide (0.058)	1): Not detected 2): Not detected
AS solution	0.01	1): Not detected 2): Not analysed	1): Not detected 2): Not analysed
SF of digestate	0.05	1): Piperonylbutoxide (0.048) 2): Piperonylbutoxide (0.097)	1): Not detected 2): Not detected
Am-Power			
Dried SF of digestate	0.05	1): Chlorpropham (0.230), Cypermethrin (0.029), Diflufenican (0.011), Diurion (0.078), Permethrin (0.035), Piperonylbutoxide (0.067), Prosulfocarb (0.031), Spinosad (0.014) 2): Chlorpropham (0.110), Fluopyram (0.071)	1): Sulfaguanidin (0.024), Thiabendazole (0.101) 2): not detected
Evaporator concentrate	0.05	1): Chlorpropham (0.037), Cotinin (0.044), Permethrin (0.011) 2): Glyphosphate (0.235) AMPA (0.202)	1): Sulfadiazin, Sulfapyrimidin (0.022), Sulfaguanidin (0.018) Thiabendazole (0.015) 2): Not detected
Permeate water	0.001	1): Not detected 2): Chlorpropham (0.0011)	1): Not detected 2): Not detected

Acqua & Sole	**Sample-specific detection limit (mg kg^{-1})**	**Herbicides, Pesticides (mg kg^{-1})**	**Pharmacologically active substances (mg kg^{-1})**
Digestate	0.05	1): Permethrin (0.041) Terbutryn (0.016) 2): Not detected 3): Not detected	1): Not detected 2): Not detected 3): Not detected
AS solution	0.05	1): Not detected 2): Not detected	1): Not detected 2): Not detected
Waterleau NewEnergy			
Digestate	0.05	Glyphosate (0.166) AMPA (0.074) Cypermethrin (0.094) Difenoconazole (0.677) Fludioxonil (0.936) Silthiofam (0.288)	Not detected
Dried SF of digestate	0.05	Glyphosate (0.101) Chlorpropham (0.075) Cotinin (0.269) Difenoconazole (0.596) Fludioxonil (1.02) Silthiofam (0.453)	Not detected
Evaporator concentrate	0.05	Glyphosphate (0.224) Difenoconazole (0.288) Fludioxonil (0.586)	Not detected
Purified water	0.001		Not detected

Sampling dates: GZV: 06-02-2020, 27-01-2021. BENAS: 09-03-2020, 04-02-2020 (no Ammonium sulphate). Am-Power: 31-03-2020, 01-02-2021. Acqua&Sole: 01-04-2020 (digestate only), 04-12-2020, 01-04-2021. WNE: 01-02-2021

Groot Zevert Vergisting

Digestate and end-products of GZV were analysed twice. In the first monitoring round, residues of two herbicides/pesticides and two pharmaceuticals were detected in digestate and fertilising products of GZV, whereas no residues were detected in the purified water. The residues detected in digestate and the SF of digestate were Chlorpropham (herbicide), Fluazifop (pesticide), Flubendazol (wormer) and Sulfadiazin/Sulfapyrimidin (antibiotic). In de second monitoring round, above mentioned residues were not detected. Only glyphosate (herbicide) and AMPA (degradation product of glyphosate) were detected in digestate and RO concentrate. It must be noted however, that the first screening did not include glyphosate and AMPA.

The distribution of the residues among end-products differs among the type of residues detected. Chloroprophame, Fluazifop and Flubendazol are present in elevated concentrations in the SF as compared to the digestate, whereas Sulfadiazin and Glyphosate end up in the RO concentrate. No residues were detected in purified water after RO.

Chlorpropham is used as herbicide and sprout supress on potatoes. The use of Chlorpropham is prohibited in the EU since 8 January 2020 (European pesticides database, 2020) meaning that potato residues treated with chlorpropham before the product was banned could still end up at GZV in the course of 2020. In 2021, no residues of Chlorpropham were detected. Chlorpropham is included in the Dutch decree on water quality objectives in the context of the Water Framework Directive. The maximum allowable concentration (MAC) of Chlorpropham in water – which is defined as the annual average level at which no harmful effects are expected - amounts to 4 microgram/l as derived by the Dutch National Institute for Public health and the environment (Vonk and Smit, 2011). Glyphosate is a commonly used herbicide with an MAC of 77 µg/l according to the RIVM[1].

Fluazifop is an herbicide which is most effective on grasses. The MAC value for Fluazifop in surface water is 3.8 µg/l as reported by the Dutch RIVM[2]. Sulfadiazine and Sulfapyrimidin are veterinary antibiotics which are not prescribed to humans anymore. Flubendazol is used as an active compound in products against worms, larvae and eggs, and is applied to chicken and pigs.

BENAS

Digestate and SF of digestate were analysed twice and at both times, Piperonylbutoxide was detected. No other residues were detected. Also, no residues were detected in the AS solution.

Piperonylbutoxide is used as a synergist in insecticide products based on Pyrethrinen and Pyrethroïden. The compound is itself not active as an insecticide, but it increases the effectiveness of the insecticides. The Dutch RIVM[2] reported a MAC value for Piperonylbutoxide in surface water (0.000083 µg/l) and sediment (0.39 µg/kg).

Am-Power

In the dried SF of digestate, residues of nine herbicides/pesticides and two pharmaceutically active compounds were detected. Far little compounds were detected in the second sampling round (2021) as compared to the first sampling round in 2020, but there is no known explanation for this difference.

The residues included Chlorpropham (used as herbicide and sprout supress on potatoes, sales prohibited since January 2020), Cypermethrin (insecticide, used to control parasites in cattle, sheep, poultry and pets), Diflufenican (herbicide), Diuron (herbicide), Permethrin (insecticide, o.a. used to kill lice and bedbugs), Piperonylbutoxide (synergists in insectides), Prosulfocarb (herbicide), Spinosad, (insecticide of biological origin, allowed in organic agriculture) and Fluopyram (fungicide). Despite the fact that Am-Power does not take in animal manure, residues of pharmaceutically active compounds were detected, including the antibiotics Sulfadiazin, Sulfapyrimidin, Sulfaguanidin and an anti-parasitic agent (Tiabendazole), and these residues may have entered the AD plant via excreta of pets included in source-separated biowaste from households.

The dried SF of digestate contained a larger number of residues as compared to the evaporator concentrate, indicating that most residues tend to accumulate in the SF probably due to being associated with organic matter. A different trend was however observed for the antibiotics and Tiabendazole which are detected in similar concentrations in the evaporator concentrate. Glyphosphate and its degradation product AMPA were only detected in the evaporator concentrate but not in the SF of digestate. A similar trend was observed in samples of GZV and this points to Glyphosphate being highly water-soluble.

Permeate water – which is used on-site for cleaning purposes but not discharged to surface water – was free of residues during the first monitoring campaign but contained 0.001 mg/kg Chlorpropham in the second monitoring round. The fact that the concentration of Chloropropham in permeate water was 100 times lower as compared to the dried SF of digestate, and that other residues as detected in the evaporator concentrate were not detected in permeate water, indicates that the treatment process effectively retains organic micro-pollutants within the SF and concentrate, producing a clean permeate water. If Am-Power is to discharge the permeate water onto surface water, an additional treatment step with RO or IO would be needed to meet criteria for N-NH_4 and these additional treatment steps would also act as a security filter removing any residues or organic micro-pollutants that ended up in the permeate water.

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[1] https://rvszoeksysteem.rivm.nl/ accessed: July 2021

[2] https://rvszoeksysteem.rivm.nl/ accessed: July 2021

Acqua & Sole

Digestate of A&S, which is produced from sewage sludge and biowaste, was analysed three times. In the sampling round, residues of one herbicide (Terbutryn) and one pesticide (Permetrhin) were detected. Residues of pharmaceutically active compounds were not detected in the digestate in any of the three samples. Additional analyses by LabAnalysis in Casanova Lonati on residues of nine pharmaceuticals that are widely by humans also showed that there were no residues present *above the detection limit* of 0.01 mg/kg. AS solution of Acqua&Sole was analysed twice and no residues were detected.

Waterleau NewEnergy

Digestate and SF of digestate contained residues of some herbicides (Chlorpropham, Glyphosate, AMPA), insecticides (Cypermetrhin, Fludioxonil) and fungicides (Difenoconazole, Fludioxonil).
No residues of pharmaceutically active compounds were detected. All residues detected in the ingoing digestate were also detected in the dried SF of digestate whereas only three residues were detected in the evaporator concentrate. This trend is similar to the trend observed in samples of GZV and Am-Power. No residues were detected in purified water.

2.2.3 Discussion

Overall, these analyses show that fertilisers from biowaste and manure can contain residues of herbicides, pesticides and pharmaceuticals. The number of compounds detected varied among the demo plants and is likely related to differences in feedstock. All compounds detected are permitted in the EU except for Chloropropham that has been phased out in 2020 but might still be present in residues of potatoes. SF generally contain a larger number of residues as compared to liquid concentrates though some residues show the opposite trend. Sewage sludge is generally considered as a hazard due to the presence of emerging contaminants. This study showed that digestate from sewage sludge did not contain any residues of commonly used pharmaceuticals whereas other studies showed that sewage sludge contains a residues of a variety of pharmaceutically active compounds (Tavazzi et al., 2012). Acqua&Sole runs the digester under thermophilic conditions which may lead to a higher degradation rate for pharmaceuticals as compared to digestion under mesophilic conditions, as is done on the other four demo plants. To confirm this, more research is needed including screening on residues of the ingoing sewage sludge. It is known that pharmaceuticals are partly degraded during AD but little is known about the effect of temperature and results in literature are inconsistent (Carballa et al., 2007).

Also, purified water, which is RO permeate after polishing in an IO, was free of residues indicating that these compounds cannot pass the membranes in the RO system and hence there is no risks in discharging purified water to surface water. This is in line with results from a recently completed monitoring study to assess quality of permeate water from manure treatment installation were no residues of veterinary antibiotics were detected in permeate water (Hoeksema et al., 2021). An exception here was Chloropropham which was detected in one sample from permeate water after RO at Am-Power, but this is no point of concern since water is re-used on-site.

AS solution was free of residues indicating that these compounds do not volatilise upon stripping of ammonia.

2.3 Polymers

From the five demonstration plants, GZV, WNE and Am-Power use polymers/flocculants (Table 2-10). These demonstration plants need polymers and other additives in solid-liquid separation apparatus such as the decanter and DAF in order to improve the solid-liquid separation. Acqua & Sole does not need any polymers because they do not have a solid-liquid separation step. For BENAS, solid-liquid separation with a screw press is sufficient and this does not require use of polymers. GZV is currently using a different polymer and anti-foaming agent than they used for the majority of 2019 because the ones they use now perform better.

There is still an ongoing debate about the criteria for the use of synthetic polymers, such as fertiliser coatings, as additive for fertilising products to be traded under the new EU Fertiliser Regulation. It is expected that only polymers which are fully degradable to water and carbon dioxide will be allowed as an additive to EC Fertilisers. The same criteria will likely apply to polymers used in separation of digestate and this may mean that the polymers based on poly-acrylamide hamper trade of biobased fertilisers, most notably SF from digestate under the new EU Fertiliser Regulation. However, so far, a standardized analytical protocol to determine the degradation rate and end products has not yet been published meaning that it is yet impossible to assess whether the polymers used by the demonstrations plants comply or not.

At Am-Power, polymer consumption has been substantially reduced after implementation of the new NRR process avoiding the use of polymer onto the DAF. Am-Power and WNE use Ecoflok polymers produced by Zeta b.v., Belgium[3]. The active ingredient is a polyacrylamide. According to the accompanying safety factsheets, Ecoflok is easily biodegradable. The suppliers mention a hydrolysis rate of 70% within 28 days, however, the hydrolysis products are not mentioned. It is therefore uncertain whether the product could meet requirements of the new EC Fertiliser regulation which states that products must be degradable to H_2O and CO_2. For the polymers used by GZV, information of degradability was not available.

Another substance of concern are phosphonates which are organophosphorus compounds known as active ingredient of fungicides and in anti-scaling products used in RO installations. Phosphonate cannot be taken up by plants and hence cannot be considered as a nutrient in contrast to phosphate. Phosphonates shall not be intentionally added to any EU fertilising product. Unintentional presence of phosphonates shall not exceed 0,5 % by mass (EU 2019/1009).

Table 2-10 Polymer use on the demonstration plants after implementation of NRR

	Brand name	**Active ingredients**	**Producer**	**Description**	**Dosage**
GZV	Zetag 8147	Polyacrylamide	BASF	Cationic polymer of medium charge added on decanter for improved removal of fines from the liquid fraction	225 g per m^3 of decanter influent
Am-Power (New process)	Ecoflok 6440	Adipic acid, sulfamic acid	ZETA	Flocculant used on decanter centrifuge. DAF is not part of the new process.	0.3% solution, Dosage to decanter centrifuge: 0-50 L/m^3 of digestate. Optimal dosage is still under investigation
Waterleau NewEnergy	Ecoflok 5440	Cationic polyacrylamide	ZETA	Powder flocculant used on decanter centrifuge	0.16% solution About 110 L/m^3 of digestate

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[3] www.zeta-water.be

3 Product testing in laboratory, pot and field setting

3.1 Nitrogen and carbon mineralisation potential from digestate

This section has been redrafted after:

Reuland et al., 2022. *'Assessment of the nitrogen fertilising value and carbon sequestration potential of digestates from different feedstocks'*. Under preparation.

i) Introduction

The study set out to assess the nitrogen (N) and carbon (C) mineralisation rates of (Table 3-1):

- Four raw digestates from Systemic demo plants: Am-Power, Acqua & Sole, BENAS and Groot Zevert Vergisting;
- A fifth raw digestate, from the mono-digestion of chicken manure was provided by a third party (D_CM). It was included as a substitute to the RIKA Biofuels demo plant (later replaced by Waterleau NewEnergy);
- Additionally, the SF of digestate from RIKA Biofuels (SFD_RIK) was also examined.

Table 3-1 Overview of tested products and acronyms used for the C & N mineralisation experiments

Product	Feedstock	Origin
D_AMP	Raw digestate from: 69% food waste, 11% food industry sludge, 7% raw animal manure, 6% glycerine & fatty substrates, 3% solid fraction manure, 4% other substrates	Am-Power, BE
D_A&S	Raw digestate from: 85% sewage sludge, 9% agro food waste, 7% digestate from anaerobic treatment of source-separated food waste	Acqua & Sole, IT
D_BNS	Raw digestate from: 44% maize, 31% rye silage, 14% chicken manure, 5% grass, 4% corn grain, 1% other solids, <1% millet	BENAS, DE
D_GZV	Raw digestate from: 67% pig manure, 16% co-substrate from the dairy and feed industry, 9% slaughterhouse manure, 4% dairy cattle manure, 4% glycerine	Groot Zevert Vergisting, NL
D_CM	Raw digestate from: 100% chicken manure	RIKA substitute, third-party plant, IE
SFD_RIK	Solid fraction of digestate from: 100% chicken manure	RIKA Oaklands demonstration plant, UK
COM_1	Commercial compost from source-separated waste from households and gardens (used for N incubations)	/
COM_2	Commercial compost from source-separated waste from gardens and parks (used for C incubations)	/
U_PS	Undigested pig slurry	St. Amandshof, BE

ii) Methodology

The mineralisation rates of these products were compared to those of undigested pig slurry (U_PS) and commercial compost (COM), as reference materials. Commercial compost 1 (COM_1) was used for N incubation, whereas compost 2 (COM_2) was used for C incubations (due to insufficient quantities of COM_1 for C incubations). The mineralisation rates were assessed via aerobic incubation experiments, in which predetermined amounts of each product were applied to 260 g (N incubations) and 275 g (C incubations) of preincubated soil. The respective mixtures of soil and products were placed in PVC tubes, packed to a bulk density of 1.4 g cm^{-3}, and water content was increased to 50% WFPS. For N, destructive sampling was carried out every 20 days, during which NH_4^+-N and NO_3^--N were measured. For C, each tube was placed inside an airtight glass jar with a NaOH 0.5 M vial to trap CO_2. The treatments were then incubated over 149 days, during which time the NaOH traps were periodically removed and back titrated (702 SM Titrino) with 0.5 M HCl to measure the evolved CO_2 after precipitating the carbonates with 2 mL of 0.5 M $BaCl_2$. The detailed description of the protocols can be found in Egene et al. (2020), based upon the Public Waste Agency of Flanders (OVAM, 2002, Oriënterend onderzoek naar de invulling van de begrippen mineralenrijk - mineralenarm, humusrijk D/2002/5024/06) and the Flemish institute for technological research (VITO, 2010, Bodem-Bepaling van snel vrijkomende organische stikstof).

Regarding N incubations, N release (N_{rel}) was calculated as the difference between the mineral N measured in the amended soil and the mineral N measured in the control (i.e. unfertilised soil), expressed as a percentage of total N added (De Neve et al., 1996). Net N mineralisation (N_{min}) was calculated by subtracting the mineral N content of the treatment on day 0 from the mineral N at all subsequent measurements and expressed as percentage of organic N (Norg). Regarding C incubations, a second-order kinetic model was fitted to the C_{min} pattern of each of the tested products (based on 17 sampling points taken over 149 days) to extrapolate the humification coefficient (HC) of each treatment after 365 days. The HC was used to calculate the effective organic matter (EOM), defined as the OM that remains in the soil after one year.

iii) Results and Discussion

A positive N_{min} (% Norg added) was observed for all products on the final day of the experiment (day 127) (Table 3-2). From highest to lowest: 52% (U_PS); 39% (D_GZV); 35% (D_CM); 33% (D_BNS); 21% (D_AMP); 20% (D_A&S); 16% (SFD_RIK); 4% (COM_1) (Table 3-2). N release results were as follows on the final day: 87% (U_PS); 81% (D_CM); 78% (D_GZV); 72% (D_AMP); 69% (D_BNS); 63% (D_A&S); 48% (SFD_RIK); 9% (COM_1).

Table 3-2 Overview the main parameters and results from C & N incubations.

Parameter	D_AMP	D_A&S	D_BNS	D_GZV	D_CM	SFD_RIK	COM_1	COM_2	U_PS
DM (g kg^{-1} FM)	85	110	107	78	80	812	531	349	29
OM (g kg^{-1} FM)	51	71	80	57	60	323	158	293	16
TN (g kg^{-1} FM)	7.2	7.9	8.9	7.7	7.7	28.1	7.6	9.0	4.0
NH_4-N (g kg^{-1} FM)	4.7	4.2	4.7	4.9	5.4	10.6	0.0	/	2.9
HC (%)	61	81	74	50	57	86	/	99	57
EOM (g kg^{-1} FM)	31	58	59	29	34	278	/	290	9
P (g kg^{-1} FM)	2.1	3.9	1.9	1.8	0.8	4.4	1.7	0.4	0.4
OM:P	24	18	43	32	72	74	92	765	37
EOM:P	15	15	31	16	43	63	/	725	23
TC (g kg^{-1} FM)	21.9	33.9	39.5	20.0	21.8	157.2	81.0	163	7.9
TC:TN	3.0	4.3	4.5	2.6	2.8	5.6	10.7	27.0	2.0
NH_4-N:TN	0.65	0.53	0.53	0.64	0.70	0.38	0.00	0.00	0.72
N_{min} (%Norg)	21	20	33	39	35	16	4	/	52
N_{rel} (%TN)	72	63	69	78	81	48	9	/	87

DM = dry matter; OM = organic matter; TN = total nitrogen; NH_4-N = ammonium nitrogen; TC = total carbon; N_{min} = mineralisation of organic nitrogen on day 127; N_{rel} = nitrogen release on day 127; HC = extrapolated humification; EOM = effective organic matter; P = phosphorus.

With between 60% and 80% of the total-N applied having been released from the digestates on day 127, results agree with previously reported trends (Sigurnjak et al., 2017; Tambone & Adani, 2017). Moreover, on the final day of incubations, N_{rel} ($p < 0.01$) and N_{min} ($p < 0.05$) were significantly positively correlated with the initial NH_4^+-N:Total N ratio of the products and negatively correlated with total C:total N ($p < 0.01$ and $p < 0.05$ respectively), in alignment with findings from previous studies (Barduca et al., 2021; Tambone & Adani, 2017). Thus, NH_4^+-N:Total N and TC:TN of products constituted reliable predictors of N availability in the soil. Also of note, N immobilisation probably occurred between days 0 and 20 for some products (D_GZV; D_CM; D_AMP; COM_1) and between days 0 and 40 for others (SFD_RIK; D_BNS; D_A&S), followed by subsequent remineralisation.

For the C incubations (Figure 3-1), the total C:total N ratio was significantly negatively correlated ($p < 0.01$) to the amount of mineralised C (C_{min}), highlighting the importance of this parameter as a reliable predictor of C mineralisation. This observation agrees with other studies that reported C:N stoichiometry as a crucial parameter affecting C and N priming (Chen et al., 2014; Hicks et al., 2019). C_{min} from the treatments was calculated by subtracting the amount of cumulative CO_2 evolved from the untreated soil and was expressed as a percentage of added organic C (Corg) from each product. Results showed that the humifiable fraction of C from the products ranged from 50 to 81% for raw digestates and 86% for SFD_RIK (Table 3-2), suggesting that these materials would be suitable candidates to increase carbon storage in agricultural soils.

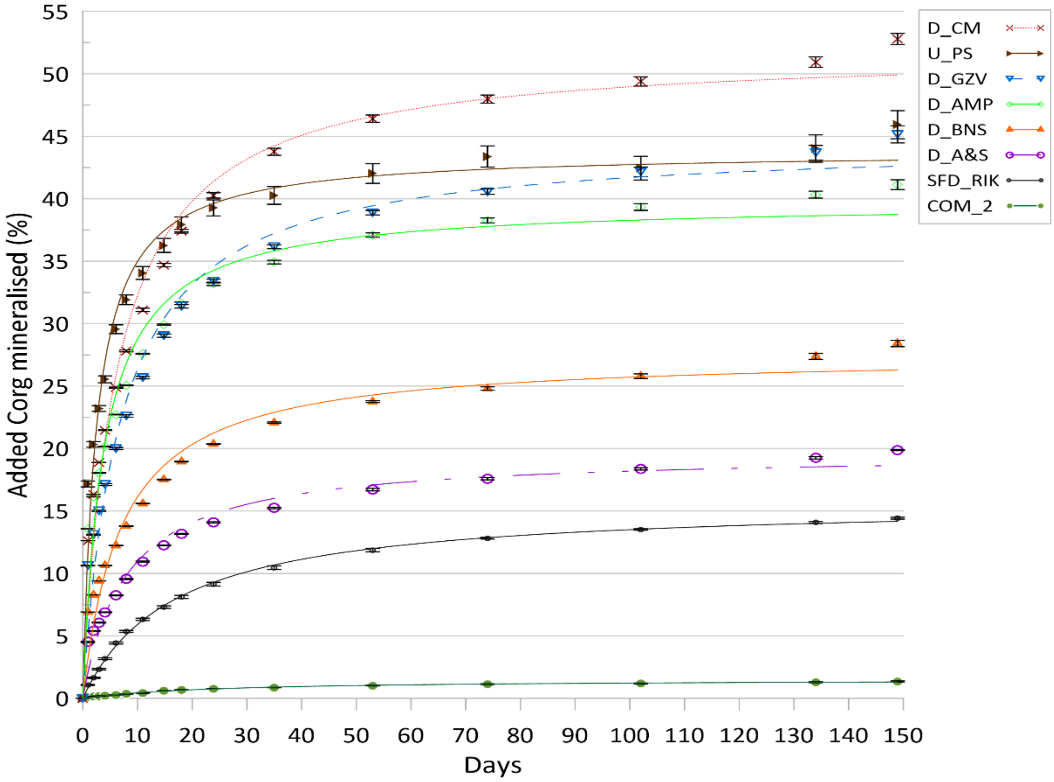

Figure 3-1 Cumulative mineralisation of added Corg from products (%) from tested products over 149-day incubation experiment (mean value ± standard deviation, n=3; where absent, error bars fall within symbols) with raw digestate from AM-Power (D_AMP); Acqua y Sole (D_A&S); BENAS (D_BNS); Groot Zevert Vergisting (D_GZV); raw digestate from the mono-digestion of chicken manure (D_CM); the SF from RIKKA (SFD_RIK); commercial compost used for C incubations (COM_2); undigested pig slurry (U_PS). Lines represent the fitted curve results; symbols are experimental data.

Compared with COM_2 and the control soil, the addition of digestates and U_PS resulted in a marked increase in microbial activity as suggested by the higher respiration and C_{min} activity. Thus, the digestates might have contained more easily degradable organic compounds than aerobically stabilised composts as previously pointed out by Kirchmann & Bernal (1997). A spike of evolved CO_2-C was observed during the first 20 hours of the incubations after which CO_2-C dropped rapidly for all treatments. In terms of C_{min} rates, the greatest gap was observed between COM_2 (1% C_{min}) and the other treatments, showcasing the higher stability and C recalcitrance of COM_2, probably due to a higher presence of lignocellulosic compounds, thus confirming its primary function as soil improver. In general, products with a lower C:N ratio led to a higher C_{min} activity as reported previously (Riffaldi et al., 1996). The C_{min} values in this study were generally similar to those reported in other studies on digestate (Alburquerque et al., 2012; de la Fuente et al., 2013).

iv) Conclusion

A significant correlation was observed between Total C:total N with respectively N release and C mineralisation rates in this study. Thus reaffirming that total C:total N is a useful predictor of C & N kinetics in soil. The N_{min} rates (% Norg) of the studied digestates on the last day of the experiment ranged from 20 to 39% (16% for the SFD_RIK). In other words, this suggests that anywhere between 60 to 80% of Norg was still present in the fertilised soil treatments after 127 days. Therefore, more attention might be given to the remaining organic N fraction still contained in raw digestates once applied. In this respect, field trials would be warranted to assess the long-term effect of digestates in a plant-soil system to ascertain the synchronisation between plant N uptake and subsequent mineralisation of the remaining Norg pools contained in these products thereby also assessing the N leaching potential of these products. Also, an initial N immobilisation phase was observed for all treatments, in light of which appropriate timing of N fertilisation to avoid any counterproductive effects on crop growth might also be taken into consideration.

The amounts of humifiable C (HC) after one year from the C incubation experiment ranged from 50 to 81% for the raw digestates (and 86% for SFD_RIK), which suggests that, without reaching the levels of the compost (99% for COM_2), these products have an equally interesting C sequestration potential in soil aside from their fertilising value.

3.2 Nitrogen and carbon mineralisation potential from solid fraction of digestate

This section has been redrafted after:

Egene, C.E., Sigurnjak, I., Regelink, I., Schoumans, O.F., Adani, F., Michels, E., Sleutel, S., Tack, F., Meers, E., 2021. *Solid fraction of separated digestate as soil improver: implications for soil fertility and carbon sequestration.* Journal of Soils and Sediments, 21(2). p.678-688. Doi: 10.1007/s11368-020-02792-z

i) Introduction

This study aimed to compare the stabilities of organic carbon and nitrogen present in solid fractions of digestate derived from different techniques (i.e. screw press, decanter, drying, P-stripping via RePeat) by estimating their mineralisation potential in soil.

ii) Methodology

Five solid fractions (SF) of digestates were collected from two Systemic demonstration plants, Am-Power and Groot Zevert Vergisting (Table 3-3). Each SF was added to a sandy loam soil, mixed thoroughly and studied in soil incubation tests. Set-ups with compost and biochar were also prepared and used as reference products. As an estimate for the conversion of organic C to mineral C in the solid fractions, the CO_2 evolution in the mixtures was measured via the alkali trap method at predetermined intervals over 81 days. The mineral N (NH_4^+ and NO_3^-) released in the mixtures was measured bi-weekly for 112 days using a flow analyser after KCl extraction.

Table 3-3 Overview of the products used in the incubation test

Code	Description	Origin
DEC-SF	Solid fraction of co-digested pig slurry after separation with a decanter	GZV, NL
SCP-SF	Solid fraction of co-digested pig slurry after separation with a screw press	GZV, NL
DRY-SF	Dried solid fraction from digestate of organic residues from the agro- food industry	Am-Power, BE
LOW-P SF	Low P soil improver obtained after treatment of DEC-SF with sulphuric acid to extract P	GZV, NL
BCHR	Biochar produced from the low P soil improver	Produced at the lab (UGhent)
COMP	Reference material: commercial compost from source-separated household- and garden waste	Attero, NL

iii) Results and Discussion

At the end of the incubation period, the highest C mineralisation was observed in the SF of digestate obtained after screw press (SCP-SF) with 23%. Meanwhile, the C mineralisation of the SF of digestates after decanter (DEC-SF), after decanter and drying (DRY-SF), and after decanter and P- stripping (P-POOR SF) did not significantly differ from each other. The least C mineralisation was in compost (COMP) and biochar (BCHR) with 6 and 4%, respectively. In general, the curves indicate that the mineralisation of the SFs followed similar patterns that differed from the compost and biochar treatments (Figure 3-2).

The humification coefficients (HC) of the SFs of digestate ranged between 51 and 75%, meaning that at least 51% of the initial organic carbon applied will remain in the soil after one year. Although these values were lower than in biochar (91%) and compost (93%), the SFs of digestate were more stable than undigested pig manure slurry, dairy manure slurry, and chicken manure with HCs of 33%, 45% and 33% respectively. The effective organic matter (EOM) which is the organic matter that is left in the soil after 365 days, was also determined. DRY-SF had an EOM/P ratio of 27 kg kg^{-1} while that of the LOW P-SF was 204 kg kg^{-1}, meaning that 7.5 times more EOM per kg of added P can be applied to soil as compared with DRY-SF.

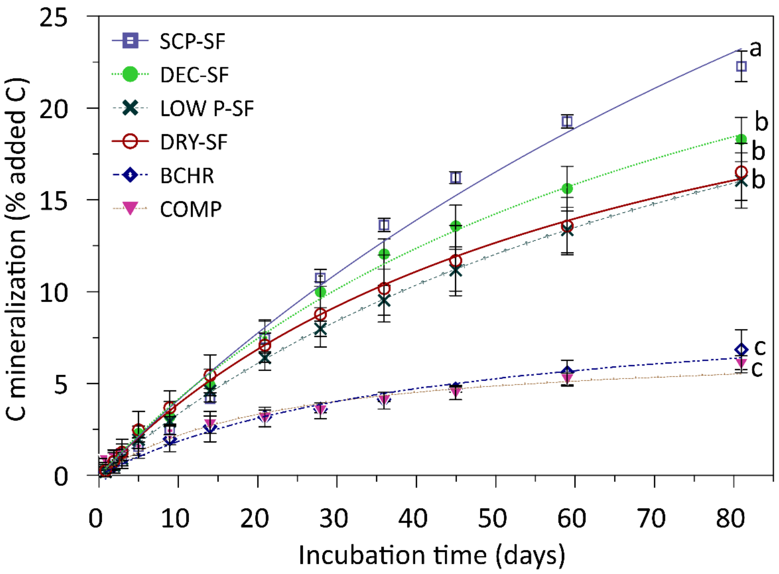

Figure 3-2 Cumulative amount of C mineralised after addition of 9000 kg OC ha^{-1} to soil during the 81-day incubation period at 10 °C (mean value ± standard deviation, n = 3). Lines represent the curve-fitting result; symbols are experimental data. Different lower case letters indicate significant differences between C mineralisation means at day 81.

Temporary N immobilisation was observed in LOW P-SF, SCP-SF, and BCHR, and this was attributed to their high C/N ratios. DEC-SF, DRY-SF and COMP, showed a positive N mineralisation, with the highest N mineralisation of 28% (in DEC-SF) (Figure 3-3). Correlation analysis showed that % N mineralisation and C/N ratio were negatively related ($r = -0.88$, $P < 0.05$) which confirmed that N is more likely to be net mineralised from substrates with a low C/N ratio. The high immobilisation of N from SCP-SF was likely due to its high carbon to organic N ratio (76) in combination with its high NH_4^+-N content that was readily available for fast initial microbial immobilisation (Calderón et al. 2005). LOW P-SF incorporation also induced temporary net N immobilisation in the first 56 days of incubation, and this was also attributed to the high carbon to organic N ratio (32) of this biosolid. The mineralisation patterns of SCP-SF and LOW P-SF, however, do suggest a re-mineralisation of the immobilised N.

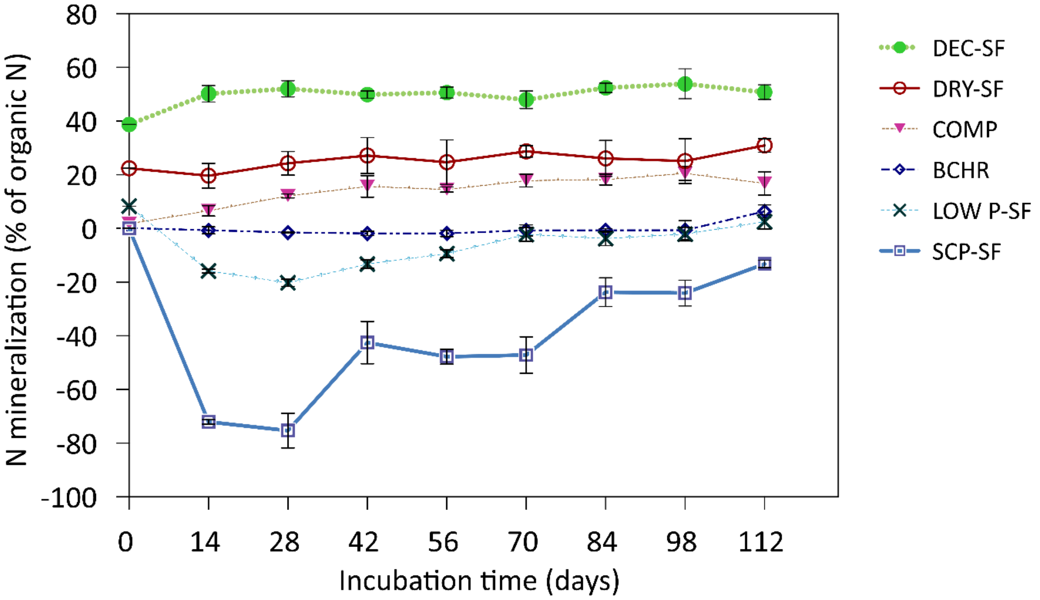

Figure 3-3 N mineralisation (% of organic N applied) during 112 days of incubation

iv) Conclusion

The C and N mineralisation potential of LOW P-SF was compared with conventional SFs of digestate to gain an understanding of the stability of the present organic matter. The various SFs showed similar patterns of C mineralisation and it was concluded that the nature of the organic matter was the main factor controlling C mineralisation in the different treatments. In terms of nitrogen, some SFs may cause temporary nitrogen immobilisation. When considering the amount of effective organic matter (EOM) that can be applied to soils within the phosphate limits, i.e. the EOM/P ratio, LOW P-SF had the highest potential to be used as an organic soil improver compared to the other SFs. As such, reducing the P concentration in SFs of digestates facilitates their application in many European soils.

3.3 GHG emission from tested end-products of five demo plants

This section has been redrafted after:

Egene et al., 2022. *Short-term greenhouse gas emissions from soil amended with novel digestate-derived nitrogen fertilisers.* Under preparation.

i) Introduction

The aim of this study was to investigate the short-term emissions of CO_2, N_2O, CH_4 from digestate-derived fertilisers obtained from different NRR technologies (hereinafter referred to as biobased fertilisers) that are present at five Systemic demo plants. Eighteen biobased fertilisers were collected from five Systemic demonstration plants (Table 3-4). Each fertiliser was characterised for dry matter (DM) content, organic matter (OM) content, total carbon (TC), total nitrogen (TN), and mineral nitrogen (N_{min}).

Table 3-4 Description, main characteristics, and the equivalent application rate of carbon at 170 kg N/ha for the biobased and mineral fertilisers

Source	**Fertiliser**	**Code**	**DM**	**OM**	**TN**	**TC**	**N_{min}**	**C/N**	**C added**
			%	**%DM**	**g kg^{-1} FW**			**-**	**kg ha^{-1}**
GZV	Digestate	GZV-DIG	7.9	72	8.1	30	5.4	3.7	621
	Solid fraction (SF) of digestate	GZV-SF	33	76	12	136	7.2	12	1959
	Low P soil improver	GZV-LOWP	27	89	4.5	120	1.2	27	4533
	Reverse osmosis concentrate	GZV-ROC	3.6	29	9.1	5.7	8.7	0.6	106
Am-Power	Digestate	AmP-DIG	8.1	62	3.4	29	2.2	8.4	1433
	SF of digestate	AmP -SF	26	65	9.1	100	2.1	11	1868
	Dried SF of digestate	AmP-DRYSF	47	63	24	310	1.1	13	2196
	Evaporator concentrate	AmP-EVA	6.3	74	7.8	22	6.3	2.8	483
	Permeate water	AmP-PW	1.5	-	3.1	5.7	2.6	1.8	313
Waterleau NewEnergy	Digestate	WNE-DIG	5.2	62	6.5	17	3.4	4.9	840
	Liquid fraction of digestate	WNE-LF	2.4	50	4.9	5.9	2.8	1.12	203
	Evaporator concentrate	WNE-EVA	17	57	10	51	0.3	3.4	864
	Dried SF of digestate	WNE-DRYSF	94	71	31	343	7.1	11	1857
	Ammonia water	WNE-NH3	-		53	<0.1	53	<0.1	1.6
Acqua&Sole	Digestate	A&S-DIG	9.3	59	7.0	29	4.4	4.1	697
	Ammonium sulphate solution	A&S-AS	38	-	76	0.1	76	<0.1	0.2
BENAS	Digestate	BNS-DIG	8.1	80	5.1	33	2.4	6.45	1097
	Ammonium sulphate solution	BNS-AS	21	-	41	0.26	41	<0.1	1.1
Mineral	Calcium ammonium nitrate	CAN	99	-	300	2	300	<0.1	1.1
	Urea	UREA	99	-	460	200	460	0.43	87

ii) Methodology

Incubation experiments were conducted in soil microcosms which consisted of a 1L Duran bottle adapted with a GL45-thread Smart Cap (model: SW45-2A). The incubation experiments were carried out in four batches from February to June 2021. Each batch included four or five products, one blank (unfertilised control soil), and two mineral fertilisers (urea and calcium ammonium nitrate (CAN) as positive controls). All fertilisers were applied at an equivalent rate of 170 kg total N/ha to 568 g of pre-incubated soil and thoroughly mixed. The moisture content in each microcosm was brought to 80% water filled pore space and maintained throughout the experiment.

Over an incubation period of 18 days, emissions of CO_2, N_2O, CH_4 were measured using the Gasera One Multi-gas analyser (Turku, Finland) equipped with a photo-acoustic infrared analyser. Fluxes of CO_2, N_2O, CH_4 were then calculated from the change in concentration over time taking into account the volume of the headspace, tubing, and area of the soil surface.

iii) Results and Discussion

N_2O emission

Among the biobased fertilisers studied, N_2O emissions were highest in the raw digestates from AmP (0.11%) and WNE (0.09%) with values comparable to emissions from CAN (~0.10%). Raw digestates from GZV, BNS, and A&S produced lower N_2O with 0.06, 0.07, 0.08 % of applied N, respectively. Although the emissions from raw digestates varied among the four batches of incubations, the raw digestate within each batch (or demo plant) remained the highest N_2O emitter relative to the digestate-derived fertilisers. This suggests that fertilisers derived from the primary and secondary processing of digestates are likely to emit less N_2O.

The mineral fertilisers emitted the highest percentage of N_2O in all four incubation batches with values ranging from 0.11 to 0.14 % of UREA N applied and 0.09 – 0.12 % of CAN N applied. This was attributed to their rapid hydrolysis in soil which occurs within hours after application, leading to increased NH_4 availability followed by nitrification (van der Weerden et al., 2016) and N_2O production. On the other hand, N_2O emissions from the biobased fertilisers were related to the availability of organic carbon which is an energy source for denitrifying bacteria. Their increased activity decreases oxygen content in the soil which promotes denitrification of the nitrified initial NH_4 of the fertilisers, thereby releasing more N_2O (Velthof and Rietra, 2019).

CO_2 emission

At the end of the incubation, the raw digestates mineralised between 8 and 28 % of applied carbon. Such high variability was attributed to differences in the origin of the digestates and amounts of labile organic C present. The SFs of digestates showed slow and near linear mineralisation patterns with values between 3 and 7 % of applied carbon. This suggests that SFs of digestates are more resistant to microbial decomposition compared to the other organic fertilisers studied. The evaporator concentrates from AmP and WNE showed different mineralisation patters. Carbon mineralisation was faster in AmP-EVA during the first few days of incubation before decreasing strongly. On the other hand, C mineralisation started slowly in WNE-EVA before increasing quickly after the fourth day. The slow C mineralisation phase in WNE-EVA coincided with the low N_2O emissions observed and was attributed to the low availability of mineral N at the start of the incubation.

CH_4 emission

Methane emissions from the studied fertilisers were generally low. For most of the fertilisers studied, CH_4 uptake was greater than CH_4 emissions for the duration of the incubations. The exceptions were evaporator concentrates from AmP and WNE with net positive emission of 4 and 1 mg m^{-2}, respectively. These observed differences were, however, not significantly different from the soil control.

Global warming potential and environmental impact

The global warming potential (GWP) estimated over 100 years (IPCC, 2007) was determined for each of the studied fertilisers. The results are presented as the sum of grams of CO_2, N_2O, and CH_4 emitted expressed as grams of CO_2 equivalents per 100 grams of N added to the soil (Figure 3-4).

The raw digestates from GZV, AmP, and WNE had the highest GWP expressed in g eq CO_2 per 100 g N applied, followed by the evaporator concentrates from AmP, WNE. For most of the fertilisers, CH_4 was taken up rather than emitted, however, the benefit gained by CH_4 consumption was offset by the increase in CO_2 and N_2O emissions. The results showed that CO_2 emissions contributed significantly more to GWP than N_2O and CH_4 in the biobased fertilisers. This was attributed to their high organic matter contents which undergo mineralisation to release inorganic carbon. It is important to note that from the life cycle assessment perspective, the decomposition of biobased substrates releases biogenic carbon i.e. CO_2 produced through biological processes involving living organisms (USEPA, 2010; WRI, 2014). Therefore, the mineralization of biobased fertilisers in soils is considered to be carbon-neutral because it does not contribute to the net increase of CO_2.

N_2O was the highest contributor to GWP in urea while the N_2O contribution from CAN was comparable to those from the raw digestates and evaporator concentrates. Ammonium sulphates and reverse osmosis waters reduced the GWP compared to the raw digestates which confirms that primary and secondary phase separation helps to decrease the environmental impacts related to GHG emission from the application of digestates in soils.

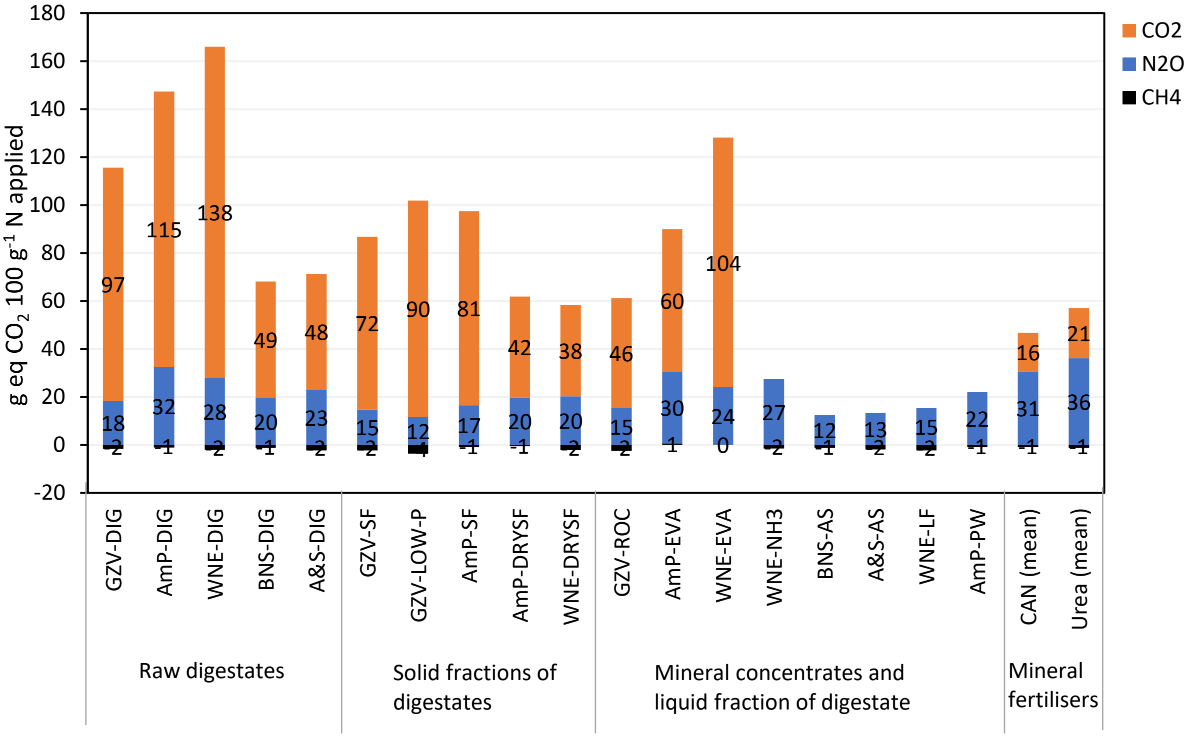

Figure 3-4 Global warming potential for the studied fertilisers calculated as the sum of grams of CO_2, N_2O, and CH_4 (expressed as grams of CO_2 equivalents) per 100 grams of total N applied to the soil. All CO_2 emission data for WNE-NH_3 water, BNS-AS, A&S-AS, WNE-liquid fraction, AmP-Permeate water, and CAN fertilisers were not shown because they contain little or no organic carbon, therefore, any CO_2 produced was attributed to the positive priming effect of native soil organic carbon which stimulates mineralisation.

iv) Conclusion

The objective of this study was to assess the GHG emissions from soil after the application of biobased fertilisers obtained from five SYSTEMIC demonstration plants. N_2O emissions were lowest in the ammonium sulphate solutions, water from reverse osmosis, and LF of digestate. In general, none of the biobased fertilisers emitted more N_2O as compared to urea or CAN. CO_2 emissions in some of the biobased fertilisers were high owing to their high OM contents. However, CO_2 emissions from biobased fertilisers may be considered as biogenic and, therefore, do not contribute to a net increase in atmospheric CO_2. Meanwhile, CH_4 emissions from all fertilisers were negligible i.e. not significantly different from the unfertilised soil control. Within each demo plant, primary and secondary phase separation of digestates seemed to decrease the GWP relative to the raw digestates.

3.4 Organic fibres as alternative for peat in potting soil and substrate

This study has been redrafted after:

Eveleens, B., Blok, C., Regelink, I. 2020. Solid fraction of digestate as growing medium in potting soil.

i) Introduction

Every year large quantities of peat are being excavated to be used as growing media and soil improver. The quantities used annually in agriculture are 40 Mm^3 worldwide, 27 Mm^3 in Europe, and 3.9 Mm^3 in the Netherlands. The main use is as constituent of potting soils for production of container plants. The excavation of peat not only affects the landscape from which the peat is harvested but also results in the emission of the carbon dioxide consolidated in the peat mass. It is therefore in the public interest that more durable alternatives for peat in potting soils are found. The potting soil industry realises that the excavation of peat is a non-sustainable practice and are therefore in search for alternatives.

Organic fibres recovered from digestate may serve as a sustainable and circular alternative for peat in potting soil. SYSTEMICs demonstration plants GZV and BENAS produce organic fibres with low nutrient values which could be suitable as an alternative for peat in potting soil, substrate or growing media. The market value for peat and peat-alternatives is about 15 Euro per m^3 (which is 30-40 euro per tonne) which is much higher than market values of soil improvers such as compost (0-5 euro per tonne) and organic manure (negative value, -10 to -25 euro per tonne). Hence, the potting soil market offers good opportunities for plant owners to increase the revenues from their products and thereby increase the profitability of their business.

Potting soil constituents must meet numerous and strict requirements. Potting soil mixtures are usually prepared from various constituents with compensating properties until overloading occurs. Compensation is possible if, for example, a too wet and too dry material are mixed to create a resulting potting soil of exactly the desired moisture properties. Overloading occurs, for example, when a constituent brings much more of a nutrient than is needed by the crop. This can only result in the restriction of this constituent to a very low contribution to the mix.

Within the SYSTEMIC project, organic fibres from Groot Zevert Vergisting and BENAS have been thoroughly analysed and tested in a pot experiment in order to assess the perspectives for usage as ingredient in potting soil.

ii) Methodology

Low-P soil improver (GZV-fibres) and organic fibres (BENAS-fibres) were sampled and analysed on physico-chemical parameters relevant for potting soil (Table 3-5). A potting soil experiment was performed using mixtures of biobased fibres with regular potting soil. The mixing ratio was determined based on the physico-chemical characteristics of the biobased fibres and the criteria for potting soil.

For GZV, the mixture consisted of 13% of GZV-fibres and 83% of regular potting soil because the high EC-value (salts) limited the use of GZV-fibres in the potting soil mixture. For BENAS, the mixture consisted of 30% of BENAS-fibres and 70% of regular potting soil. Other nutrients were added to obtain equal levels of macro- and micro-nutrients in the potting soil mixtures and the reference potting soil. A pot experiment was performed with chrysanthemum and begonia's. Plant growth and nutrient status in the potting soil was determined at the end of the experiment.

iii) Results and Discussion

Physico-chemical properties of the biobased organic fibres and regular peat-based potting soil are given in Table 3-5. GZV-fibres are characterised by a low pH value of 5.4, due to the use of sulphuric acid in

RePeat system to remove P, which meets criteria for potting soil. However, the EC value of the GZV-fibres amounts to 5.4 whereas an EC value of 1.0 is regarded the maximum because high EC levels negatively effect germination and growth of salt-sensitive plants. The high EC value of GZV-fibres is due to the addition of sulphuric acid in the RePeat process to extract P. BENAS-fibres have a low EC value and are therefore more suitable for use in potting soil. Both biobased fibres have high OUR (oxygen uptake rates) as compared to potting soil, pointing to a risk for anoxic conditions in the growing media but this can be controlled by mixing the organic fibres with regular potting soil. Physical properties of the biobased fibres, including water holding capacity and bulk density, are similar to peat.

The pot experiment showed that replacing peat by GZV-fibres or BENAS-fibres had no effect on plant growth (Figure 3-5). Hence, biobased fibres could safely replace up till 30% of peat in potting soil mixtures while maintaining yields similar to those with regular potting soil.

Table 3-5 Characteristics of organic fibres from demonstration plants GZV and BENAS as compared to characteristics of potting soil based on peat[a].

		GZV	***BNS***	***Peat-based potting soil***
		Low-P soil improver	***Low-N organic fibres***	
pH	(-)	5.4	8.1	5.7
EC	(mS/cm)	5.4	1.1	0.6
Na	(mmol/l)	5.1	0.9	0.4
Cl	(mmol/l)	3.6	1.8	0.2
SO_4	(mmol/l)	23	2.5	0.7
NH_4	(mmol/l)	21	0.3	0.1
NO_3	(mmol/l)	0.2	0.2	2.3
Bulk density	(g/l)	99	91	95
Total pore space	(% v/v)	94	94	95
Water holding capacity at pF 3 (saturation)	(% v/v)	71	65	68
Oxygen uptake rate	(mmol O_2/kg/h)	9.6	8.1	2.3

[a] Nutrients extracted with demineralised water using a 1:1,5 v/v mixing ratio

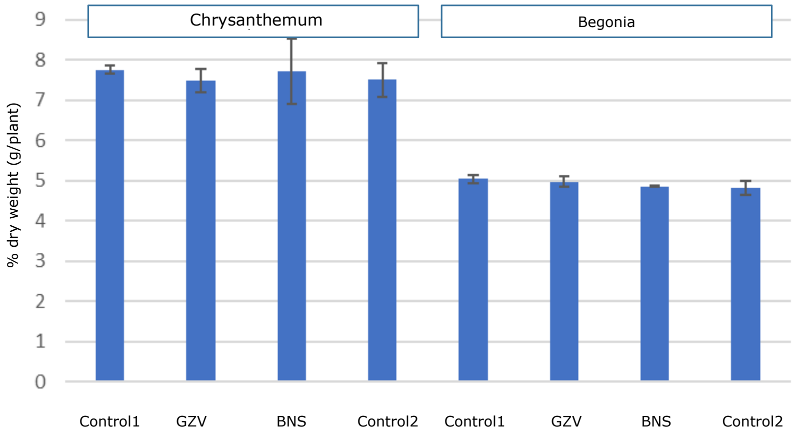

Figure 3-5 Plant dry weight of Chrysanthemum and Begonia grown on standard potting soil mixtures (control1, control2) and potting soil mixtures with 13% (v/v) GZV-fibres and 30% (v/v) organic fibres from BENAS

iv) Conclusion

Organic fibres recovered from digestate of GZV and BENAS were found to be suitable to replace respectively 13 and 30% of peat in regular potting soil mixtures without inducing negative effects on plant growth. For the organic fibres of GZV, the limiting parameter is the EC value (salt content) which is high due to the use of sulphuric acid in the treatment process. An additional leaching step to reduce the salt content could improve suitability of the GZV-fibres. For GZV, it is advised to focus on replacement of peat in substrates for cultivations were the salt levels is not limiting f.e. substrates for mushrooms. Organic fibres of BENAS have a lower EC value and hence more suitable to replace peat in potting soil mixtures.

3.5 Effectiveness of precipitated P salt as P fertiliser

This section has been redrafted after:

Ferron, et al., 2022. *Effectiveness of struvite as a phosphorus fertiliser for maize: a pot experiment.* Under preparation.

i) Introduction

Demo plant GZV has invested in a full-scale installation for separation of SF of digestate into a precipitated P salt and a low-P soil improver. Within this process, P is extracted from the SF of digestate by leaching SF with water and sulphuric acid at a pH-level of 5.5. Secondly, P is recovered by increasing the pH towards 7.0 through the addition of calcium-hydroxide or magnesium-hydroxide. Depending on the choice of the base salt added, phosphate precipitates either as calcium phosphate ($CaHPO_4.2H_2O$) or struvite ($MgNH_4PO_4.6H_2O$). GZV aims to sell the precipitated P salts to the fertiliser industry as a raw material for granular organic fertilisers. However, little is known about the effectiveness of precipitated P salts as a P fertiliser as compared to synthetic P fertilisers. The objective of this pot experiment was to assess the effectiveness of struvite as compared to a synthetic P fertiliser (di-ammonium phosphate, DAP) as a P fertiliser supporting growth of maize on soils with a low P status. At the start of the experiment, GZV had not yet finalised the construction of the new installation. Therefore, struvite was chosen as the precipitated P salt and collected from a waste water treatment plant.

ii) Methodology

Maize was chosen as a test crop because there is a demand for P starter fertilisers for maize in the region of GZV. DAP is a commonly used synthetic fertiliser. Row fertilisation, which is the common practice in the Netherlands, was mimicked by placing the P granules at a depth of 5 cm and near the maize seed. In addition, a negative control (control-) receiving no P fertiliser, and a positive control (control+) receiving an excess amount of phosphate in the form of phosphate salts were added (Table 3-6). Struvite consisted of 1-2 mm granules and was obtained from outreach location Waternet (WWTP, Amsterdam) were it is recovered from digestate of sewage sludge. GZV was not yet operational at the time of the pot experiment. The experiment was performed with a low-P sandy soil (OM: 4.2%, clay: 2%, P-AL: 26 mg P_2O_5/100 g, P-$CaCl_2$: 0.3 mg P/kg) and a low-P loamy soil (OM: 6.0, Clay: 16%, P-AL: 11 mg P_2O_5/100 g, P-$CaCl_2$: <0.3 mg P/kg). Low-P soils (sand and loam) were selected to ensure that P was the growth-limiting factor despite the fact that soils in the Netherlands generally have a neutral or high P status. All other nutrients were given in surplus as compared to crop demand. Maize was harvested at a length of nearly 100 cm which was reached six weeks after germination.

iii) Results

Results (Table 3-6, Figure 3-6) show that yield increased in the order: control–, struvite, control+, DAP. Row fertilisation was effective since it gave a higher yield as compared to the control+ treatment were a larger amount of P was added but uniformly mixed with the soil. In the latter case, P is bound to the iron-hydroxides in the soil and consequently only partly available for P uptake. Row fertilisation creates hotspots of available P and maize plants can actively take up P from these hotspots.

Row fertilisation with struvite increased P uptake of maize as compared to control- treatments in which no P fertiliser was added. Hence, maize plants were able to take up P applied as struvite. However, a far greater yield was observed when applying a same amount of DAP fertiliser. Further soil analysis show that struvite is soluble in 0.01 M $CaCl_2$ and hence, is expected to become available for plant uptake over time. However, dissolution of struvite during extraction of soil with 0.01 M $CaCl_2$ also let to an overestimation of plant-available P in the soil.

Table 3-6 Treatments of the pot trial with maize and corresponding plant uptake of P and N. Struvite and DAP were given as row fertilisation (5 cm depth, near maize plant) whereas fertiliser in the control+ treatment was uniformly mixed throughout the soil (n:3)[A]

Soil	*Treatment*	**Fertiliser treatment**	**Equivalent dosage kg P_2O_5/ha**	**P-uptake (mg/plant)**	**N-uptake (mg/plant)**
Sand	***Control -***	No P fertiliser	0	0.7^{a} ± 0.04	35.6^{a} ± 2.2
	Control+	Na_2HPO_4/NaH_2PO_4	98 (uniform fertilisation)	$5.5^{a,b}$ ± 0.23	140^{b} ± 7.4
	Struvite	$MgNH_4PO_4\cdot(6H_2O)$	40 (row fertilisation)	$3.3^{a,b}$ ± 0.66	$81.9^{a,b}$ ± 8.5
	DAP	$(NH_4)_2HPO_4$	40 (row fertilisation)	10.6^{b} ± 2.9	160^{b} ± 26
Loam	***Control -***	No P fertiliser	0	1.7^{a} ± 0.04	51.6^{a} ± 0.3
	Control +	Na_2HPO_4/NaH_2PO_4	148 (uniform fertilisation)	27.5^{b} ± 0.86	315^{b} ± 11
	Struvite	$MgNH_4PO_4\cdot(6H_2O)$	40 (row fertilisation)	5.7^{c} ± 0.67	123^{c} ± 14
	DAP	$(NH_4)_2HPO_4$	40 (row fertilisation)	20.9^{d} ± 0.86	313^{b} ± 12

[a] Different letters indicate that values that are different according to a Tukey HSD test (P<0.001).

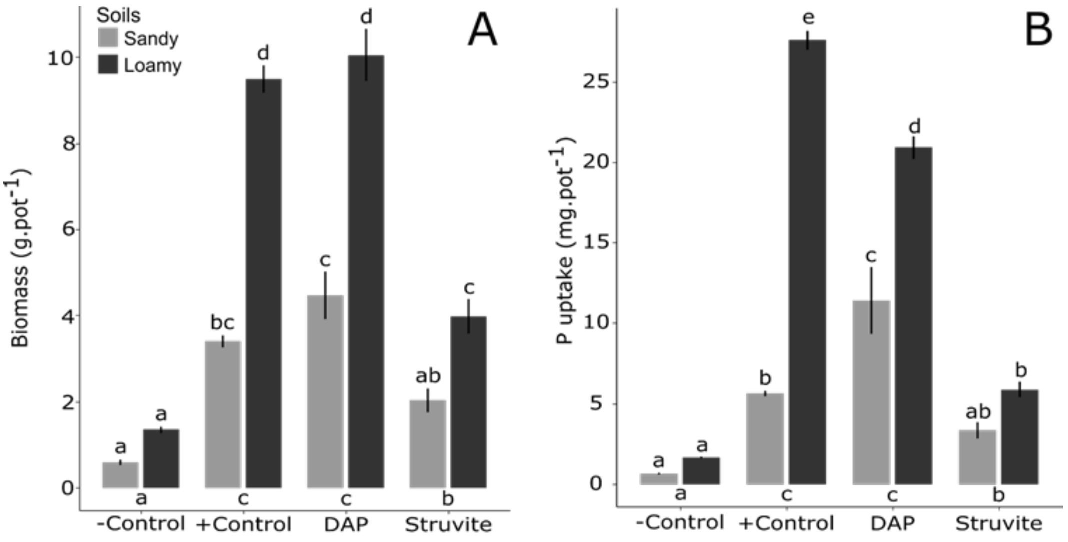

Figure 3-6 Maize performances in treatments receiving diammonium-phosphate (DAP) or struvite and as compared to a negative control (no P fertiliser) and a positive control (large amount of mineral P fertiliser). A: Biomass production (g/pot). B: Phosphorus uptake (mg/pot). Tukey HSD result are indicated next to the x axis for fertilisation effect and on top of bars for soil:fertilisation effect. Error bars are standard errors.

iv) Conclusion

To conclude, struvite is not as effective as DAP in terms of supporting young maize plants, but struvite is expected to be effective as a slow release fertiliser. It is advised to further investigate if grinding of struvite, thereby reducing its particle size and increasing the dissolution rate, is an effective post-treatment leading to a higher P availability of struvite. Care should be taken with interpretation of conventional P availability assessments, including P-CaCl2, due to struvite dissolution during extractions.

3.6 Phosphorus availability in solid fractions of digestate

This section has been redrafted after:

Regelink et al., 2022. *Efficiency of organic fertilisers from digestate and biowaste as P fertilisers.* Submitted to Agronomy

i) Introduction

Most of the phosphorus (P) used in agriculture today is derived from the mining of non-renewable phosphate rock. However, global P reserves are rapidly being depleted thus raising important questions about the long-term supply security of P. Processing biowaste and manure into safe and agronomically efficient organic fertilisers that can replace current use of P fertilisers from fossil resources is therefore essential. While farmers select solid fractions (SFs) of digestate based on their total P content, this may not be indicative for the plant available P content of the SFs which depends on the speciation of P within the organic fertiliser. P in SFs is dominantly present in the form of inorganic P which precipitates with multi-valent cations forming e.g. brushite ($CaHPO_4.2H_2O$) or struvite ($MgNH_4PO4.6H2O$). These precipitates typically dissolve under slightly acidic conditions thereby releasing ortho-phosphate ($P\text{-}PO_4$). Of particular importance are levels of Fe and Al in the SFs since P is preferentially bound to these metals forming poorly insoluble complexes. The objective of this study was to assess P-speciation and easily extractable P in SF's of digestate produced by the SYSTEMIC demo plants.

ii) Methodology

Within the SYSTEMIC project, four demo plants produce SF of digestate which are characterised by moderate to high P contents (4.7- 27.5 g P/kg DW) and should therefore primarily be seen as P fertilisers except for the low-P soil improver produced by GZV (3.6 g P/kg DW) which is a soil improver (Table 3-7).

Tabel 3-7 Description of the origin of the solid fractions (SF) of digestate from SYSTEMICs demonstration plants in which phosphorus speciation was assessed including dry weight (DW), organic matter (OM), P and Fe.

Code	Feedstock and processing	DW	OM	P	Fe	P/Fe
		(%)	(% DW)	(g/kg DW)	(g/kg DW)	(mol/mol)
AMP-SF	Dried SF of digestate of biowaste including iron-rich sludge from wastewater treatment plants	80.1	46	27.5	35.2	1.4
WNE-SF	Dried SF of digestate of manure, biowaste and sewage sludge. Iron sludge and iron-chloride is added to improve dewatering and to bind sulfide in the digester.	95.0	65	25.5	13.2	3.5
WNE-SF2	Blend of WNE-SF1 with evaporator concentrate	55.5	62	23.0	11.3	3.7
GZV-SF1	SF of digestate of pig manure and biowaste	31.3	76	28.0	2.8	18.0
GZV-SF2	Low-P organic soil improver obtained after leaching P with water and sulphuric acid in the RePeat process.	22.6	93	3.8	2.3	3.1
BNS-SF2	SF of digestate of energy crops	66.2	92	4.7	5.5	1.5
BNS-SF1	Low N fibres obtained by removing N through stripping	24.5	91	6.2	5.5	2.0

Besides P, the SF's show a wide variation in Fe-contents (2.3-35 g Fe/kg DW) and differences in Fe-content can be related to the source of the feedstock and the use of iron salts during processing. For example, WNE add iron water (a side product from the production of drinking water from ground water) and iron chloride ($FeCl_3$) in order to bind sulphide in the digester and to improve digestate dewatering. This explains higher Fe-contents as compared to SF's produced by GZV and BENAS were no iron is added. The highest Fe-content (35 g Fe/kg DW) was found in the SF produced by Am-Power though they do not add iron-containing additives. The high Fe-content is likely due to the intake of iron-rich sludges from flotation units of waste-water treatment plants in agro-food industry as well to the intake of garden waste containing soil.

The SF's were analysed for (i) easily available P by extracting SF with 0.01 M $CaCl_2$ after adjusting the pH of the mixture to 5.5. Inorganic was determined after extraction with 0.5 M H_2SO_4. P-total was determined after dry combustion followed by extraction with 0.5 M H_2SO_4. Organic P is calculated as the difference between total-P (measured after oxidation at 550°C followed by extraction with 0.5 M H_2SO_4) and inorganic-P (measured after extraction with 0.5 M H_2SO_4). Reactive Fe and Al, which have a high affinity for adsorption of P, were measured after extraction by 0.2 M ammonium oxalate at pH 3.0 for 4 hours in the dark. The % of P bound to Fe and Al was calculated assuming a maximum binding capacity of 0.5 M P per mol Fe+Al. Residual P is defined as the difference between total P and the sum of the other defined P pools. Based on the analytically-derived P-pools, P species were distinguished as schematically depicted in Figure 3-7.

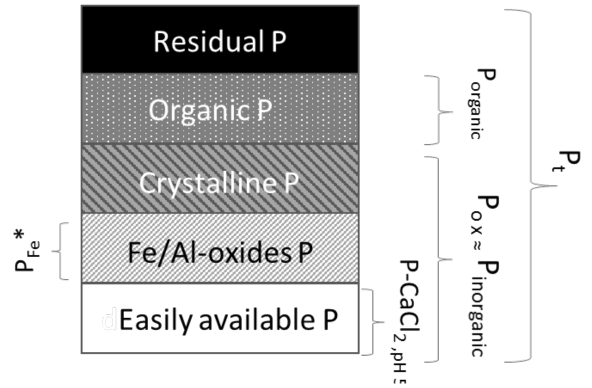

*Figure 3-7 Phosphorus (P) species a distinguished in the OFs and their relation with the analytical P pools. *Fe-bound P is derived using the P binding capacity (see Regelink* et al., *2021)*

iii) Results and Discussion

Results of the assessment of P-speciation in the SOF's (Figure 3-8) show a wide range in the fraction of easily available P (20-85% of total-P). Highest fraction of available P was found in the SF of digestate of GZV (GZV-SF1). The fraction of easily available P is lower in the low-P soil improver of GZV (GZV-SF2) which is due to removal of the easily available fraction during treatment with sulphuric acid in the RePeat process. Lowest fraction of available P was found in the SOF of Am-Power. A relatively large fraction of P was classified as crystalline P which did not dissolve in 0.01 M $CaCl_2$ though it may become plant-available over time. The fraction of easily available P correlated negatively with the molar P/Fe ratio pointing to fixation of P by metal salts added during processing or by soil in case garden waste or iron-rich sludge was used as a feedstock.

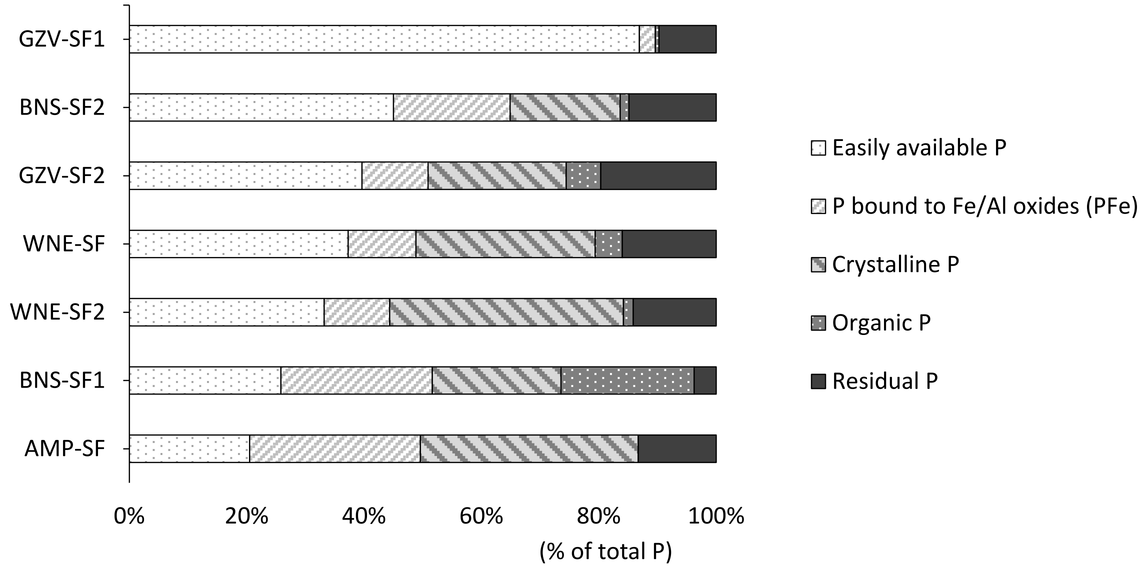

Figure 3-8 Speciation of phosphorus (P) in solid organic fertilisers produced by demo plants GZV, BENAS (BNS), Waterleau NewEnergy (WNE) and Am-Power (AMP) plotting in order of decreasing fraction of 'easily available P'. As a reference, compost of vegetable, fruit and garden waste (VFG-COMP) is included.

iv) Conclusion

Phosphorus speciation and availability strongly differs among solid fractions of digestate produced by SYSTEMICs demonstration plant. Besides total P content, suppliers of solid fractions should also inform farmers about the available fraction of P and its suitability as a P fertiliser or as a soil improver. Solid fractions with a low fraction of available P possess a lower risks on increasing P leaching from soil and are preferred under conditions were soil P status is sufficient. However, for fertilisation of soils with a low P status, a solid fraction with a high fraction of available P is preferred. Moreover, the use of iron-salts or iron-rich feedstocks in the digester has a negative effect on the availability of P and should therefore be discouraged.

3.7 One-year field trial experiment in Belgium

This section has been redrafted after:

Luo et al., 2022. *Assessing the potential value of biobased products from evaporation process in the substitution for synthetic mineral nitrogen fertiliser: soil incubation and field experiment.* Under preparation.

i) Introduction

Substitution of synthetic mineral fertilisers by biobased products derived from agro-waste is considered to be a key strategy towards more sustainable agriculture. At the site of Waterleau NewEnergy (Ypres, Belgium), a nutrient recovery cascade consisting of anaerobic digestion (AD), centrifugation and evaporation technology is developed at a scale of 120 000 tonnes feedstock (45% manure) per year. To evaluate the nitrogen fertiliser replacement value (NFRV) of the biobased products derived from this nutrient recovery cascade as compared to synthetic mineral N fertilisers, a field trial on maize was designed with the following seven treatments in quadruplicate: 1) N unfertilised control (CON); 2) calcium ammonium nitrate (CAN) as synthetic mineral N fertiliser; 3) pig manure (PM) used as a feedstock for the AD; 4) raw digestate (DIG); 5) liquid fraction of digestate (LFD) after centrifugation; 6) evaporator concentrate (EVA); and 7) ammonia water (AW).

ii) Methodology

The field trial was conducted at the experimental farm of Ghent University in Bottelare (Belgium). Maize (*Zea mays* LG31220, France) was sowed on 12th May 2020 and was harvested on 22nd September 2020 to determine the biomass yield and N uptake. Before fertilisation and after harvest, soil samples were taken at 0-90 cm depth to determine the level of mineral N and the post-harvest leaching risk. The N application rate was set at 105 kg total N ha^{-1}, accounting for 70% of the recommended rate based on the maize demand and the soil N status. The NFRV of each biobased treatment was calculated by comparing the apparent N recovery (ANR) to that of synthetic mineral N fertiliser. The ANR and NFRV are calculated according to Eq. 3-1 and Eq. 3-2:

$$ANR = \frac{N\ Uptake_{biobased\ treatment} - N\ uptake_{control}}{total\ N\ applied} \qquad \text{Eq. 3-1}$$

$$NFRV\ (\%) = \frac{ANR_{biobased\ treatment}}{ANR_{reference}} \times 100 \qquad \text{Eq. 3-2}$$

The average temperature during the growing season was 18.7±3.6 °C and the precipitation was 1.5±3.8 mm day^{-1}. However, it was relatively dry during the early stage of the growing season (3rd May till 3rd June with an average temperature at 23.3±4.5 °C and the precipitation at 0.05±0.16 mm day^{-1}) that the germination of some maize plant was delayed and thus resulted in heterogeneous growth in the following stages and high deviations by harvest.

iii) Results and Discussion

The LFD treatment resulted in the highest average values in FW and DW biomass yield and crop N uptake, with significant ($p<0.05$) difference compared to CON, DIG, EVA and AW treatments but no significant difference compared to CAN and PM treatments (Table 3-8). Consequently, the calculated ANR and NFRV were significantly ($p<0.05$) higher in LFD treatment compared to DIG, EVA and AW treatments. The DIG, EVA and AW treatments did not differ significantly from CON (i.e. unfertilised) treatment in terms of biomass yield or N uptake. When comparing biobased treatments to the synthetic reference, i.e. CAN treatment, significant difference was only observed in N uptake and the calculated ANR of DIG treatment which had lower values than CAN treatment.

Table 3-8 The biomass yield (t ha^{-1}) in fresh (FW) and dry weight (DW) and the content (on dry weight basis) of macronutrients (g kg^{-1}) in maize shoot at harvest (n = 4). The small letters refer to one-way ANOVA and post-hoc pair-wise comparisons (Fisher's Least Significant Difference (LSD) test) between treatments on each sampling date at each depth, with a significant difference at the 5% level. CON = no-N control, CAN = calcium ammonium nitrate, PM = pig manure, DIG = digestate, LFD = liquid fraction of digestate, EVA =evaporator concentrate, AW = ammonia water.

	Unit	CON	CAN	PM	DIG	LFD	EVA	AW
FW	t ha^{-1}	36±2 a	42±3 cbd	43±2 cd	39±2 ac	44±5 d	38±3 ac	37±6 ab
DW	t ha^{-1}	14±0 a	15±1 ab	16±1 b	14±1 a	17±2 b	14±1 a	13±2 a
N uptake	kg ha^{-1}	135±5 a	172±8 bc	182±10 bc	140±19 a	200±37 c	155±14 ab	159±31 ab
ANR	-	/	0.35±.08 bc	0.45±0.09 bc	0.04±0.18 a	0.62±0.35 c	0.20±0.13 ab	0.23±0.30 ab
NFRV	%	/	/	126±27 bc	12±52 a	175±99 c	55±37 ab	65±84 ab

The low values of the calculated ANR (Table 3-8) could be attributed to the relatively high soil N contribution which was notable in crop N uptake of 135±5 kg ha^{-1} in CON (i.e. unfertilised) treatment. In the study of Joris et al. (2020) by applying ^{15}N labelled mineral N fertiliser on sugarcane, they also reported 80-85% of the N uptake derived from soil, while 15–20% was derived from fertiliser. It seems that the high soil N supply could have met most of the crop N demand during the growing season which masked the contribution of N fertiliser. Furthermore, a two-year field experiment with winter and spring wheat by de França et al. (2021) reported that in the first experimental year, the soil influence on crop parameters was more pronounced compared to the second year. Therefore, the low precipitation at the beginning of growing season in our study might have resulted in a low soil moisture and subsequent water stress to the young maize plants which impacted the development of seminal roots and reduced the uptake of extra nutrients. This might have also led to the heterogeneous growth of maize plants starting from a heterogeneous germination, resulting in high standard deviations of the calculated ANR and NFRV values. All these aspects seem to have further blocked the effect of fertiliser treatments and ended up with no significant difference in biomass and nutrient yields in DIG, EVA and AW treatments compared to unfertilised CON treatment.

Before fertilisation, the soil mineral N (SMN) at a depth of 0-90 cm ranged from 76 to 94 kg N ha^{-1} (Table 3-9), with no significant difference observed among treatments. After harvest, the average values of the SMN till 90 cm were in the range of 100-150 kg N ha^{-1}, with significant difference ($p<0.05$) only observed in EVA and AW treatments as compared to unfertilised CON treatment (Table 3-9). However, when compared with the synthetic reference, i.e. CAN treatment, there was no significant difference observed in biobased treatments regarding the SMN after harvest.

Table 3-9 Concentration of mineral N (kg ha^{-1}) in soil from surface to 90 cm deep before fertilisation and after harvest (n = 4). The small letters refer to one-way ANOVA and post-hoc pair-wise comparisons (Fisher's Least Significant Difference (LSD)) between treatments on each sampling date at each depth, with a significant difference at the 5% level. CON = no-N control, CAN = calcium ammonium nitrate, PM = pig manure, DIG = digestate, LFD = liquid fraction of digestate, EVA = Evaporator concentrate, AW = ammonia water.

	CON	CAN	PM	DIG	LFD	EVA	AW
Before fertilisation	89±40	87±44	94±68	87±35	79±30	76±23	79±26
After harvest	104±18 a	111±19 ab	139±34 ab	131±30 ab	122±17 ab	145±24 b	144±24 b

According to the latest guideline for nitrate residue in Flanders (VLM, 2020), the SMN residues after harvest were higher than the first threshold value (75 kg ha^{-1}) for maize cultivation on non-sandy field (area type 2 based on the located area of the tested site), some plots even higher than the second threshold value (150 kg ha^{-1}). This indicated a high risk of post-harvest nitrate leaching for winter period, thus caution should be taken to improve the fertiliser management in the following years.

iv) Conclusion

Based on the preliminary first-year results, the tested biobased products showed no significant difference with the synthetic reference using calcium ammonium nitrate (CAN) regarding fresh and dry biomass yields. Significantly lower N uptake was only observed in the biobased treatment using digestate as compared to the CAN treatment. These results suggested high potential of the tested pig manure, liquid fraction of digestate, evaporator concentrate and ammonia water as substitutions for synthetic mineral N fertilisers. However, though no significant difference was observed in the post-harvest soil mineral N (SMN) residues of biobased treatments as compared to the CAN treatment, the high SMN could indicate an increased N leaching risk which suggests a need for an improved fertiliser management in the following seasons.

3.8 Two-year field trial experiment in Croatia

This section has been redrafted after:

Šatvar et al., 2022. *Digestate derivatives as potential synthetic nitrogen fertiliser replacements in a two-year maize field experiment in Croatia*. Under preparation.

i) Introduction

The Eastern member states (e.g. Croatia) are still facing a lack of knowledge on chemical composition of digestate, absence of certification schemes on the national level and unclear legislative framework, resulting in slower implementation of biogas technology. The NRR technology is currently not taken up by Croatian biogas plant operators. To stimulate the development of biogas sector and hence nutrient recovery it is important to assess the fertiliser value of digestate against conventional mineral nitrogen (N) fertiliser and demonstrate to farmers that, next to the biogas production, digestate could also have a market value. Therefore, the aim of this experiment was to determine, in a two-year field experiment, fertiliser value of SF and LF of digestate by assessing: a) their effect on crop yield and NFRV as compared to the conventional use of mineral N fertilisers and animal manure; b) soil properties as the experiment was conducted on very acidic soil (pH_{KCl} 4.21±0.09) and c) NO_3^- -N residue in post-harvest period.

ii) Methodology

The used SF and LF of digestate were obtained from outreach location Bojana (Čazma, Croatia) that treats maize silage and liquid cattle manure (LCM). The following seven experimental treatments were established in four replicates: i) synthetic mineral fertiliser (NPK, i.e. NPK 15-15-15 + CAN 27% N), ii) liquid cattle manure (LCM), iii) solid fraction of digestate (SFD), iv) liquid fraction of digestate (LFD), v) a mix of SFD + NPK, vi) a mix of LFD + NPK and vii) unfertilised control (C). Fertilisation of 140 kg total N ha^{-1} for each treatment and year (2018 and 219) was performed as sub-equilibrium fertilisation. This dosage was chosen in order to prevent subsequent nitrate leaching after harvest as there were no catch crops each year after maize harvest. NPK, SFD and LFD were added to soil manually in order to assure accurate dosage. After application, the fertilisers were immediately incorporated into soil by rotary harrow (depth 10 cm) to reduce the ammonia volatilisation and in within 2-3 days, sowing took place. As a test crop, fodder maize (Zea mays L.) hybrid R 0725 (Corteva agriscience, FAO vegetation group 570) was sown at a seed density of 80 000 ha^{-1}.

iii) Results and Discussion

DW grain yield followed same trend in all treatments in both years, however the DW grain yield in 2019 was lower in all treatments as compared to 2018. In 2018 the highest yield was achieved in NPK treatment 13.1 t ha^{-1}, while in 2019 LFD+NPK treatment resulted in the highest yield of 11.5 t ha^{-1} (Figure 3-9). Control treatment gave lowest DW grain yield in both years, amounting to 8.6 t ha^{-1} in 2018 and 7.1 t ha^{-1} in 2019. As shown, production of maize dry grain yield can vary from year to year, also depending on the weather conditions, soil characteristics and many other factors. In 2018, when maize was in vegetative stage V6, hail made damage on the plant. During 2019, total dry grain yield was lower in all treatments because corn rootworm (*Diabrotica virgifera virgifera*) that appeared during the reproductive stage R1 of maize silking was feeding on silk. Cvjetićanin et al. (2017) reported that between year 2013 to 2017 maize dry grain yield production in Croatia ranged 6.5 – 8.5 t ha^{-1}, while average dry grain yield in this two-year experiment was 7.9 t ha^{-1} in unfertilised (control) treatment, 12.1 t ha^{-1} for NPK, 9.9 t ha^{-1} for LCM, 9.4 t ha^{-1} for SFD and 9.6 t ha^{-1} for LFD.

In 2018 the significantly ($p < 0.05$) higher N uptake was observed in NPK (279±16.1 kg ha^{-1}) and LFD+NPK (256±10.2 kg ha^{-1}) treatments, while in 2019 the highest N uptake was recorded in NPK (265±1.1 kg ha^{-1}) and combinations of NPK with SFD (236±13.3 kg ha^{-1}) and LFD (243±12.8 kg ha^{-1}) (Table 3-10). Lowest N uptake recorded was observed in control treatment for both 2018 and 2019 season. N uptake could be affected by soil pH reaction (immobilisation of elements) of the experimental field. While field had acidic reaction, 4.2_{KCl}±0.1 in 2018 and 3.9_{KCl}±0.1 in 2019, the critical soil pH range for corn is 5 to 5.5 (Rhoads and Manning, 1989) and experimental field had even lower pH.

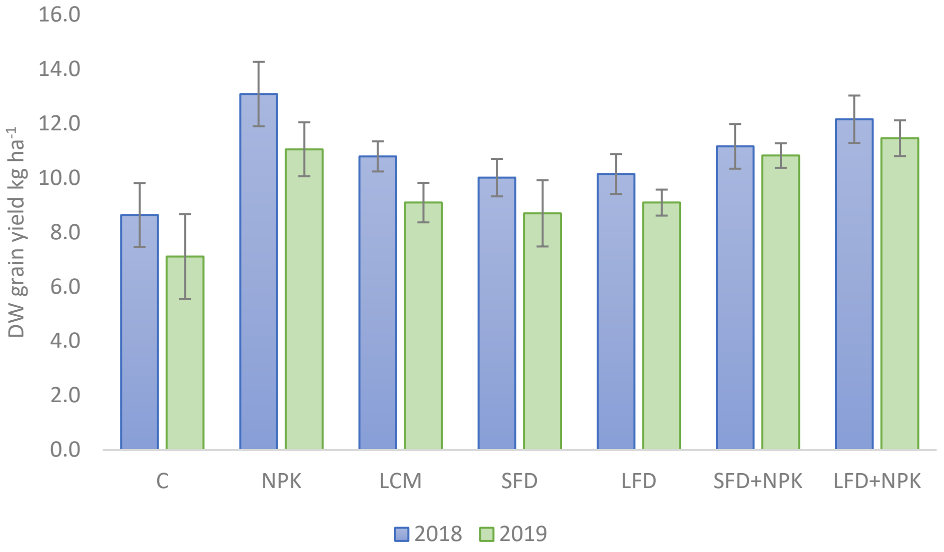

Figure 3-9 Dry grain yield kg ha^{-1} in 2018 and 2019; C: control; NPK: synthetic mineral fertiliser; LCM: liquid cattle manure; SFD: solid fraction of digestate; LFD: liquid fraction of digestate; SFD+NPK: a mix of solid fraction of digestate with synthetic mineral fertiliser; LFD+NPK: a mix of liquid fraction of digestate with synthetic mineral fertiliser.

Calculated ANR in 2018 was statistically highest in NPK (0.77±0.1), followed by LFD+NPK (0.60±0.1) treatment as compared to other treatments (Table 3-10), while in 2019 the highest ANR was recorded in NPK (0.93±0.1) and combinations of SFD (0.72±0.1) and LFD (0.77±0.1) with NPK. Consequently, the highest NFRV in 2018 was obtained in LFD+NPK 78±10% treatment, whereas LFD+NPK treatment with 83±9% resulted in highest NFRV for 2019 – all by assuming that mineral fertilisation is 100% efficient, i.e. NFRV = 100%.

Table 3-10 Mean ± standard deviation of maize nutrient uptake, Apparent N recovery (ANR) and N fertiliser replacement value (NFRV) for the seven different fertilisation treatments (n = 4) at harvest in year 2018 and 2019. NPK fertilisation is assumed to be 100% efficient; NFRV = 100%.

Parameters	**C**	**NPK**	**LCM**	**SFD**	**LFD**	**SFD+NPK**	**LFD+NPK**
				Year 2018			
Total N kg ha^{-1}	171±19.5a	279±16.1c	215±5.3b	213±6.3b	221±1.7b	228±9.0b	256±10.2c
ANR	-	0.77±0.1c	0.32±0.0a	0.30±0.0a	0.36±0.0a	0.41±0.1a	0.60±0.1b
NFRV (%)	-	100d	41±10ab	39±7a	47±7ab	53±10b	78±10c
				Year 2019			
Total N kg ha^{-1}	135±31.4a	265±11.1c	188±12.6b	176±8.5b	198±8.6b	236±13.3c	243±12.8c
ANR	-	0.93±0.1d	0.38±0.1ab	0.29±0.1a	0.45±0.1ab	0.72±0.1c	0.77±0.1cd
NFRV (%)	-	100c	41±5a	31±6a	48±2a	77±8b	83±9bc

C: control; NPK: synthetic mineral fertiliser; LCM: liquid cattle manure; SFD: solid fraction of digestate; LFD: liquid fraction of digestate; SFD+NPK: a mix of solid fraction of digestate with synthetic mineral fertiliser; LFD+NPK: a mix of liquid fraction of digestate with synthetic mineral fertiliser.

The lower ANR and NFRVs in all biobased treatments in compare to reference NPK are probably a result of lower initial NH_4-N/total N in tested products (Reijs et al., 2007), specially in 2018 (0.13 for LCM, 0.08 for SFD and 0.12 for LFD). As NH_4-N/total N was higher in 2019 (0.33 for LCM, 0.19 for SFD and 0.43 for LFD), due to feedstock input changed in biogas plant, NFRV slightly but not significantly increased in all treatments. As mentioned, the NH_4-N/total N ratio of the LFD and SFD depends on the feedstock input of AD (mechanical separation unit remained the same), and in the end can reflect NFRV.

As for the NO_3^--N residue in the soil, for both years NPK and LFD+NPK treatments resulted in highest NO_3^--N residue after harvest (Figure 3-10). On the other hand the lowest nitrate residue was measured in treatments without NPK application, indicating that in these experimental conditions utilisation of LFD and SFD should not additionally increase the risk of nitrate leaching compared to the applied synthetic fertilisers.

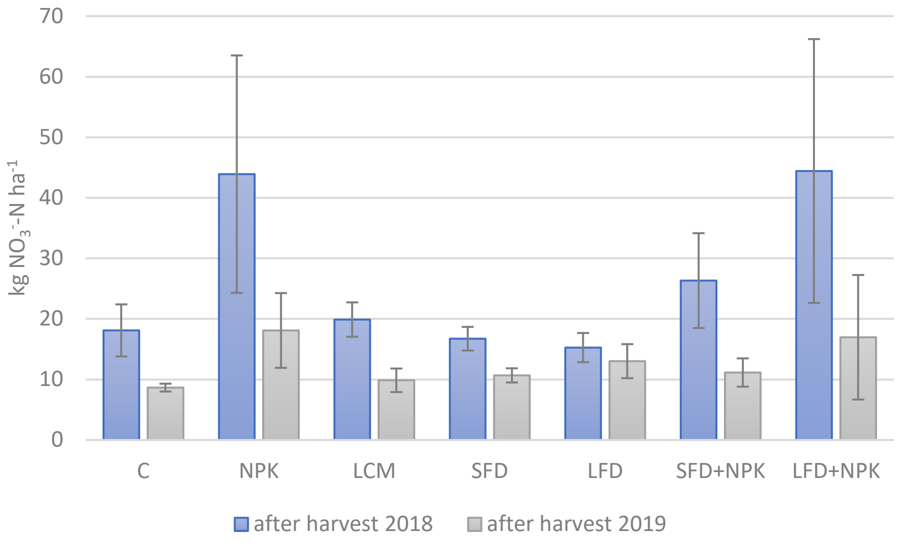

Figure 3-10 NO_3^--N residue kg ha^{-1} in soil (0-90 cm) after harvest time for the seven different fertilisation treatments; C: control; NPK: synthetic mineral fertiliser; LCM: liquid cattle manure; SFD: solid fraction of digestate; LFD: liquid fraction of digestate; SFD+NPK: a mix of solid fraction of digestate with synthetic mineral fertiliser; LFD+NPK: a mix of liquid fraction of digestate with synthetic mineral fertiliser.

iv) Conclusion

In this two-year field trial, NPK and LFD+NPK gave highest dry grain yield of all treatments in both 2018 and 2019, which was in the range of Croatian average yields. There was no statistical difference in ANR and NFRV for NPK treatment in comparison to LFD+NPK (50:50) treatment in 2019. It is apparent that combination of both organic and synthetic fertiliser can result in higher percentage of NFRV. From the results of NO_3^-N residues in the soil it is obvious that SFD and LFD should not additionally increase the risk of nitrate residue or leaching compared to synthetic fertilisers.

3.9 Three-year field trial experiment in Italy

This section has been redrafted after:

Pigoli, A., Zilio, M., Tambone, F., Mazzini, S., Schepis, M., Meers, E., Schoumans, O., Giordano, A., Adani, F. 2021. *Thermophilic anaerobic digestion as suitable bioprocess producing organic and chemical renewable fertilisers: a full scale approach.* Waste Management 124, 356-367. Doi: https://doi.org/10.1016/j.wasman.2021.02.028

Zilio, M., Pigoli, A., Rizzi, B., Geromel, G., Meers, E., Schoumans, O., Giordano, A., Adani, F. 2021. *Measuring ammonia and odours emissions during full field digestate use in agriculture.* Science of The Total Environment 782, 146882. Doi: https://doi.org/10.1016/j.scitotenv.2021.146882

Zilio et al., 2022. *Soil assessment, pollutants, nitrate leaching and GHG emissions during the full field digestate use in agriculture.* Under preparation

Rizzi et al., 2022. *Effects of digestate on the nitrogen cycle in soil under full scale assessment.* Under preparation

i) Introduction

The aim of the work was to verify the feasibility of using digestate and ammonium sulphate (AS) solution produced from treated sewage sludge in agriculture to replace mineral fertilisers, with particular focus on agronomic performance and environmental impacts. The experimentation took place on a full-scale maize crop, comparing the use of the digestate produced by the Acqua & Sole plant with the use of a mineral fertiliser (urea) for a duration of three consecutive agronomic seasons. Description of AD plant and production and characterisation of used fertilisers have been reported by Pigoli et al. (2021).

ii) Methodology

Every year in pre-sowing, the plots fertilised with digestate received a dose of total nitrogen of 370 kg N ha^{-1} as digestate injected into soil (TAN/TKN: 0.54 - 0.61), while the plots fertilised with urea received a dose of 185 kg N ha^{-1}, spread on surface. All plots then received an additional 100 kg N ha^{-1} in form of AS solution in topdressing fertilisation (through fertigation). The experiments concerned in particular: i) change in the chemical and agronomic characteristics of the soil; ii) accumulation of inorganic and organic pollutants in the soil; iii) risk of nitrate leaching; iv) odour and ammonia emissions during and after spreading; v) emission of greenhouse gases (GHG) during an agronomic season after the spreading; vi) effect on soil N-related microbial communities; vii) annual production of maize grains and content of micro-elements and inorganic pollutants; vii) efficiency of the digestate as a fertiliser vs. urea.

iii) Results and Discussion

To determine the effect on the soil characteristics, soil samples were taken from all experimental plots before the start of the three years of experimentation, and after the third. The only significant difference observed in the soils at the end of the project concerns the organic carbon content. In fact, after three years in soils fertilised with digestate, an increase in the amount of total organic carbon (TOC) was observed compared to both unfertilised and urea fertilised soils (12.3 ± 0.4, 11.9 ± 0.2 and 11.3 ± 0.4 g C kg^{-1} dw for digestate, urea and unfertilised respectively). In particular, after the third year of experimentation, in the soil fertilised with digestate the more stable OC fraction increased significantly compared to the soil fertilised with urea (13.8 ± 1.3 and 9.58 ± 0.1% of TOC respectively). This stable C accumulation was probably due to the high level of biological stability achieved by the digestate after the anaerobic digestion process (BMP: 57 ± 23 L_{biogas} kg^{-1} dw).

Particular attention was given to environmental impacts related to the use of digestate. After three years of experimentation, the concentrations of inorganic pollutants (i.e. heavy metals) and organic pollutants (including emerging pollutants) were measured in all the experimental plots, and the values obtained in the plots fertilised with digestate did not differ from those of the plots fertilised with urea and unfertilised plots.

To examine the risk of nitrate (NO_3^-) leaching in the experimental soils, soil cores were taken out at a depth of one meter, during the 2019-2020 agronomic seasons (Figure 3-11).

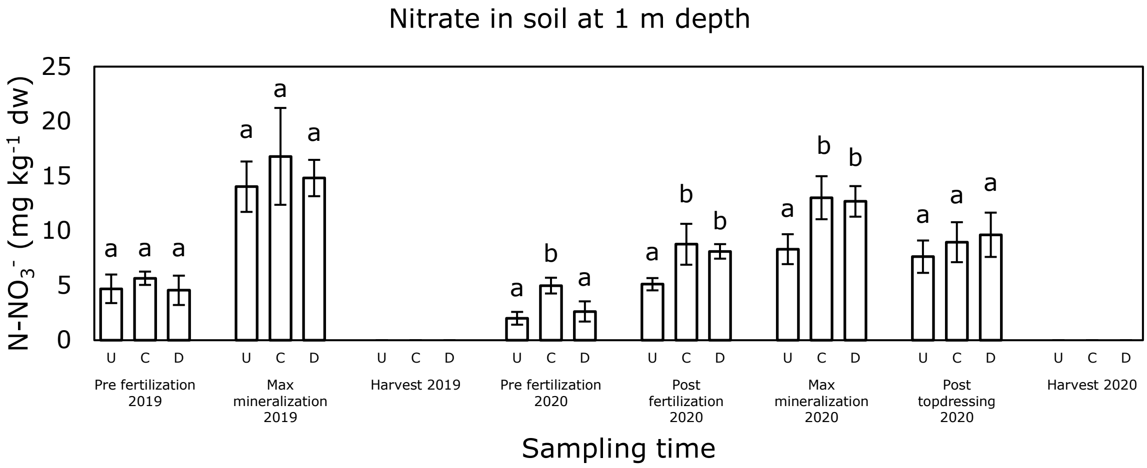

Figure 3-11 Concentrations of nitric nitrogen (N-NO_3^-) in experimental soils at 1 meter depth during the crop seasons 2019 and 2020 (mean ± SD; n=3). U: Unfertilised, C: Chemical (urea), D: digestate. Letters are referred to One-way ANOVA (p<0.05; Tukey post-test) comparing the three treatments within each sampling time.

The concentrations of nitrates measured in soils fertilised with digestate were always equivalent to those in soils fertilised with urea, and in some cases also to those unfertilised. Even the N related microbial populations of the soil have not been affected using digestate. Their abundance was measured by gene quantification, monitoring it up to a depth of one meter during the 2019 agronomic season.

Immediately after fertilisation in pre-sowing, ammonia (NH_3) and odour emissions were also measured. Ammonia was measured during the 90 hours after pre-sowing fertilisations for three consecutive years. The cumulative mean N-NH_3 emission was 25.6 ± 9.4 kg N ha^{-1} for fertilisation with digestate (corresponding to 11.6 ± 4% of the TAN), and 24.8 ± 8.3 kg N ha^{-1} for fertilisation with urea (corresponding to 13.4 ± 4.5 % of the TAN). The amounts of ammonia emitted using the two fertilisers were statistically equivalent. Even the emissions of odours after fertilisation were found to be equivalent between the plots fertilised with digestate and those fertilised with urea, demonstrating that the injection of the digestate into the soil, together with its stabilisation, is able to strongly reduce both ammonia and odours emitted. These data (regarding ammonia and odours) have been published by Zilio et al. (2021).

Furthermore, greenhouse gas emissions (N_2O, CO_2 and CH_4) from the experimental soils were measured starting from pre-sowing fertilisation and for the following 10 months (Table 3-11).

Table 3-11 Cumulated emissions (mean ± SD, n = 6) of N_2O, CO_2 and CH_4 measured from the experimental plots during the crop season 2020 and the following months (36 samplings). Letters are referred to One-way ANOVA (p<0.05, Tukey post-test).

Period	**Fertiliser**	**Total nitrogen dosed (kg N ha^{-1})**	**Total N_2O emitted (kg N ha^{-1})**	**Total CO_2 emitted (kg C ha^{-1})**	**Total CH_4 emitted (kg C ha^{-1})**
From 28/05/2020 to 17/03/2021	Unfertilised	0	1.71 ± 1.1 (a)[a]	5698 ± 935 (a)	0.066 ± 0.06 (a)
	Chemical	285	10.3 ± 6.8 (b)	6144 ± 1491 (a)	0.053 ± 0.04 (a)
	Digestate	461	7.59 ± 3.2 (ab)	6216 ± 1160 (a)	0.036 ± 0.03 (a)

[a] Letters are referred to One-way ANOVA comparing in each row the three experimental soils in January 2021 (p<0.05; Tukey post-test).

The data showed, also in this case, that the emission from soils fertilised with digestate was equivalent to that from urea fertilised soils. This data confirms the high stability of the organic substance contained in the digestate and brought to the soil. As for the performance of the digestate as a fertiliser, the results obtained show that it is comparable to that achieved with urea. In fact, ammonia nitrogen concentrations in soils fertilised with digestate and urea during the agronomic season have always been equivalent, never causing deficiencies or excesses of available nitrogen. The plants were able to use the ammonia nitrogen provided with the digestate with similar efficiency of that provided with urea (NFRV of digestate= 83.7%), producing equivalent amounts of grain (18.1 ± 2.9 and 17.4 ± 1.2 Mg ha^{-1} respectively for digestate and urea), and without accumulating heavy metals or other dangerous elements.

iv) Conclusion

The reported data show that the use of the digestate produced by Acqua&Sole for three consecutive years in a full-scale trial did not cause detectable negative effects on the soil, environment or on the maize produced, compared to the use of urea. As for the effect on the soil characteristics, the only significant difference observed after three years of experimentation concerns the higher organic carbon content in soils fertilised with digestate. No accumulations of heavy metals or organic pollutants were observed in the soils. The nitrate concentrations in the soil below the plots fertilised with digestate have always been equivalent to those of soils fertilised with urea. Equivalent were also ammonia and odours emissions detected immediately after fertilisation. Finally, the greenhouse gases emission (N_2O, CO_2 and CH_4) from soils fertilised with digestate was equivalent to that from urea fertilised soils. As for the performance of the digestate as a fertiliser, the results obtained show that it is comparable to that obtained with urea. The plants were able to use the ammonia nitrogen provided with the digestate with the same efficiency of that provided with urea, producing equivalent quantities of grain and without accumulating heavy metals or other dangerous elements. Overall, all these data show that the digestate produced by Acqua&Sole plant can completely replace other synthetic fertilisers without affecting the environment or loss of agronomic performance.

4 Product evaluation and compliance with regulation

4.1 Compliance with EU Fertiliser Product Regulation

The new EU Fertiliser Product Regulation (FPR) (2019/1009[4]), laying down rules for free trade of fertilisers within the EU, will come into force in 2022. The FPR covers mineral-, organic- and organic-mineral fertilisers as well as soil improvers, growing media and biostimulants. The implementation of the new EU FPR may offer improved possibilities for SYSTEMICs plant owners to sell their recovered fertilisers as a CE fertiliser to other EU countries. The EU FPR is a facultative regulation and does not replace national legislation. Fertilisers with a CE label can be *traded* throughout the EU. The application of EC fertilisers, including application rate limits, is regulated by national or local legislation.

The EU FPR defines CMC's (Component Material Categories) and PFC's (Product Function Categories) (Figure 4-1). The CMC's describe which input materials are allowed to produce an EC fertilising product whereas criteria for the composition of fertilising products are set in the PFC's description. Blending is allowed though the individual input material shall not contain any of the substances for which maximum values are indicated in the PFC description in such quantities as to jeopardise the EU fertilising product's compliance with the applicable requirements for that PFC.

CMC, annex II
1 Virgin material
2 Plants, plant part, plant extract (untreated)
3 Compost
4 Fresh crop digestate
5 Other digestate
6 Food industry by-products
7 Micro organisms
8 Nutrient polymers
9 Polymers other than nutrient polymers
10 Derived animal by-products (1069/2009/EC))
11 Industrial byproducts (2008/98/EC
12 Precipitated P salts (STRUBIAS)

PFC, annex I
1 fertiliser **- organic** **- organo-mineral** **- inorganic**
2 Liming material
3 Soil improver **- organic** - inorganic
4 Growing media
5 Inhibitors
6 Biostimulants - microbial - non-microbial
7 Blend PCMs

Figure 4-1 Overview of the Component Material Categories (CMC) and Product Function Categories (PFC) in the EU Fertiliser Product Regulation. Criteria for CMC 10-12 are not yet defined.

The EU Fertiliser Product Regulation may be beneficial for SYSTEMICs demonstration plants if:

- The end product is exported to another EU country (if applied locally, national legislation applies (Table 4-1)
- The input material from which the fertiliser is produced, complies with criteria of one of the CMCs
- The end product, which may be a blend of several CMCs, complies with criteria of one of the PFCs

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[4] Regulation (EU) 2019/1009 of the European Parliament and of the Council of 5 June 2019 laying down rules on the making available on the market of EU fertilising products and amending Regulations (EC) No 1069/2009 and (EC) No 1107/2009 and repealing Regulation (EC) No 2003/2003 (Text with EEA relevance)

For SYSTEMIC demonstration plants, CMC 4 (Fresh crop digestate) and CMC 5 (Digestate other than fresh crop digestate) are relevant. Animal manure is allowed as feedstock for the digester under CMC 5 under the conditions that 'an endpoint has been reached in the manufacturing chain in accordance with regulation (EC) No 1069/2009, implying hygiënisation of manure. CMC 11 covers industrial by-products including e.g. ammonium sulphate solution obtained through stripping and scrubbing. However, also for CMC 11 criteria are not yet definite.

The EC No 2019/1009 does not includes allowed post-processing technologies for digestate. In 2022, the EC published a draft amendment to include post-processing technologies for digestate under CMC4 and CMC5. This draft[5] includes solid/liquid separation as well as 'physical processing to remove water that do not chemically modify the digestate or fraction'.

GZVs dominant feedstocks are residues from food and feed processing and animal manure; Both seem to be expected under CMC5 though residues from feed processing are not explicitly mentioned. The SF of digestate does however not meet criteria for a PFC-1 fertilising product since the criteria for a minimum nutrient content of 30 g kg^{-1} FM (sum of N, P_2O_5, K_2O) is not met. An additional drying step to increase DM and nutrient contents would be necessary in order to comply with the criteria. For the precipitated P salts, a designation as an EC fertiliser would be valuable since it would enable GZV to sell the P salts to producers of organic fertilisers which are traded over Europe. Criteria for CMC 12 are however not yet definite but based on the STRUBIAS report, a maximum TOC content of 3% of the dry matter content is expected which is unachievable for GZV that yet produces P salts with 20% TOC. Also for the low-P soil improver, a designation as an EC fertiliser would open up opportunities to sell the product to potting soil producers however, it is yet unclear whether the the production process which includes the use of sulphuric acid and multiple leaching steps, is accepted under the FPR. RO concentrate of GZV does not comply with the minimum nutrient content required for a liquid fertiliser under the FPR (Table 4-2) which is of no concern since this product is traded locally.

BENAS' feedstock of the digester are mostly energy crops and its digestate, and therefore they could meet criteria of CMC 4. In 2017 and 2018, also poultry litter was digested but the intake of poultry litter has been reduced over time. At the BENAS plant, the SF after N-stripping is used as raw material for the production of cardboard products. The remaining digestate does not meet criteria for liquid organic fertilisers under the PFR because the criteria for the sum of the nutrients (> 30 g/kg FM) is not met (Table 4-2). BENAS produces an AS solution. Ammonium salts, including AS solution, will be covered under CMC 11 (Industrial by-products) but criteria and definitions are not yet known. If criteria for CMC 11 are met, AS solution could be traded as a liquid macronutrient fertiliser under the PFR (Table 4-3).

Am-Power processes source separated biowaste which is allowed as a feedstock for digestate under CMC 5 and hence its digestate may serve as input material if other criteria are met as well. Dried SF of digestate of Am-Power meets all criteria solid organic fertilisers PFC1(A) (Table 4-4) including criteria for heavy metals. The other product of Am-Power is an evaporator concentrate which, however, does not fit within any of the PFCs due to its TOC and total nutrient content being too low. Hence, Am-Power still relies on the national legislation for exporting evaporator concentrate towards France.

Acqua & Sole's main feedstock is sewage sludge. Digestate of sewage sludge is exempted from the list of input materials for fertilisers with a CE label. A&S can however still continue selling and applying digestate regionally following the regional legislation on the use of sewage sludge (Table 4-1). A&S has a REACH registration for the AS solution that is being produced by means of stripping and scrubbing ammonia from digestate. Products with a REACH registration are allowed as input material under CMC 1 (Virgin Materials). The composition of AS solution complies with criteria for PFC 1 and may be traded as a Compound Liquid Inorganic Macronutrient fertiliser (Table 4-3) and also blended with other input materials as long as the end product fits within one of the PFCs.

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[5] Ref. Ares(2022)932249 - 09/02/2022

Waterleau NewEnergy processes multiple feedstock including animal manure and sewage sludge. The addition of sewage sludge exempts the end products from being traded as a CE fertilising products. WNE will therefore continue exporting the dried SF of digestate and evaporator concentrate under the Flemish waste legislation (Vlarema) and French fertiliser regulation (NF U42). If WNE excludes both sewage sludge and raw animal manure from its digester, its digestate could comply with criteria of CMC 5 and its dried SF could be labelled as solid organic fertiliser (PFC1(A)). Evaporator concentrate however, does not meet criteria for the minimum amounts of nutrients (>30 g/kg FM) and TOC (similar as in case of AmP) and export of evaporator concentrate is therefore solely possible via national regulations or after blending with the dried SF towards a tailor made fertiliser that fits within one of the PFCs.

Table 4-1 Overview of national legislation to which end-products of SYSTEMIC's demo plants have to comply.

	Country were to be applied	Relevant regulations	EU FPR[1]
Am-Power Biowaste from agro-food industry			
Evaporator concentrate	Belgium (or France)	Flemish waste legislation (Vlarema)	Does not comply with PFCs
Dried SF of digestate	France	Flemish waste legislation (Vlarema) and French criteria (NF U42-001)	CMC5, PFC 1
GZV[2] Pig slurry, biowaste from agro-industry			
RO concentrate	the Netherlands	RENURE	Not relevant (used locally) and would not meet criteria for minimum nutrient contents in EU FPR (PFC1)
SF of digestate	Germany		CMC 5, PFC1
Precipitated P salts	Export	-	CMC 12, but does not comply with proposed limit for max. TOC.
Low-P organic soil improver	the Netherlands	-	Processing method not included in FPR
Acqua & Sole Sewage sludge, biowaste			
AS solution	Italy or sold to fertiliser industry	REACH-registered	CMC1 (REACH-registered) or CMC 11, PFC 1,7
Digestate	Italy, Lombardy	Regional regulation: DGR X/2031/2014, DGR X/5269/2016 and DGR X/7076/2017	Excluded as EC fertiliser because of the intake of sewage sludge
BENAS Corn silage, poultry litter			
AS solution	Germany	-	CMC 11 PFC1 (inorganic fertiliser)
Digestate, LF and SF of digestate	Germany	Ordinance on biowaste	CMC4&5 (CMC)
Low-N organic fibres	Germany	Not relevant (no fertilising product but used a raw material for fibre board production)	Processing technique not covered by FPR
Waterleau NewEnergy Pig slurry, biowaste, sludge from industrial waste water			
Dried SF of digestate	France	Flemish waste legislation (Vlarema) and French fertiliser regulation (NF U42-001)	Could comply with CMC 5 if they cease intake of sludge from industrial waste water treatment installations. End products could comply with product specifications under PFC1
Evaporator concentrate	Belgium or the Netherlands	Flemish waste legislation (Vlarema) RENURE	
Condensed ammonia water	Belgium	Not relevant because product is not used as a fertiliser but sold to industry	

Table 4-2 Criteria for liquid organic fertilisers in the Product Fertiliser Regulation and composition of evaporator concentrate and RO concentrate produced by Am-Power, Waterleau NewEnergy (WNE), Groot Zevert Vergisting (GZV) and BENAS.

Parameters	**Unit**	**PFC 1(A)(II)**	**Evaporator concentrate (Am-Power)**	**Evaporator concentrate (WNE)**	**RO concentrate (GZV)**	**LF of digestate (BENAS)**
Nutrient content*	g kg^{-1} FM	≥ 30*	**24**	**45**	**18**	**18**
TN	g kg^{-1} FM	≥ 10 or	9.9	13	8.1	6.8
P_2O_5	g kg^{-1} FM	≥ 10 or	2.1	4.8	0.34	3.7
K_2O	g kg^{-1} FM	≥ 10	12	27	10	7.6
TOC	g kg^{-1} FM	≥ 50	**28**	**44**	**7****	**26**
Cu	mg kg^{-1} DM	≤ 600	34	51	<100	65
Zn	mg kg^{-1} DM	≤ 1500	118	130	<550	386
Cd	mg kg^{-1} DM	< 1.5	<0.51	0.37	<0.41	<1.8
Cr (VI)	mg kg^{-1} DM	< 2	-	<1	-	-
Hg	mg kg^{-1} DM	< 1	-	0.012	<0.058	-
Ni	mg kg^{-1} DM	< 50	15	21	14	10
Pb	mg kg^{-1} DM	<120	<5.1	3.1	<5.1	7.3
As	mg kg^{-1} DM	<40	-	0.61	<1.1	-

* The sum of nutrients N, P_2O_5 and K_2O shall be at least 30 g kg^{-1} FM

** TOC calculated as 0.45 * Loss on ignition.

Table 4-3 Criteria for Compound Liquid Inorganic Macronutrient fertiliser in the Product Fertiliser Regulation and composition of Ammonium Sulphate produced by BENAS and Acqua & Sole (A&S).

Parameters	**Unit**	**PFC 1(C)(I)(b)(ii) (FPR)**	**Ammonium sulphate (BENAS)**	**Ammonium sulphate (A&S)**
Nutrient content*	g kg^{-1} FM	≥ 70*	185	287
TN	g kg^{-1} FM	≥ 15	46	75
SO_3	g kg^{-1} FM	≥ 7.5	137	212
TOC	g kg^{-1} FM	≤ 10	0.35	<1
Cu	mg kg^{-1} DM	≤ 600	0.18	<14
Zn	mg kg^{-1} DM	≤ 1500	0.52	<24

* The sum of nutrient contents (TN, P_2O_5, K_2O, MgO, CaO, SO_3, Na_2O) shall be at least 70 g kg^{-1} FM.

Table 4-4 Criteria for solid organic fertilisers in the Product Fertiliser Regulation and composition of solid fraction Am-Power, Waterleau NewEnergy (WNE), Groot Zevert Vergisting (GZV) and BENAS.

Parameters	**Unit**	**PFC 1(A)(I)**	**Dried SF of digestate (Am-Power)**	**Dried SF of digestate (WNE)**	**SF of digestate (GZV)**	**SF of digestate (BENAS)**
Nutrient content*	g kg^{-1} FM	≥ 40*	84	104	38	21
TN	g kg^{-1} FM	≥ 10 or	23	29	12	7.1
P_2O_5	g kg^{-1} FM	≥ 10 or	44	57	20	5
K_2O	g kg^{-1} FM	≥ 10	17	18	5.5	6.7
TOC	g kg^{-1} FM	≥ 150	288	325	-	96
Cu	mg kg^{-1} DM	≤ 600	88	266	91	20
Zn	mg kg^{-1} DM	≤ 1500	405	772	376	158
Cd	mg kg^{-1} DM	< 1.5	<0.17	0.5	<0.4	<0.74
Cr (VI)	mg kg^{-1} DM	< 2	-	<1	-	<1
Hg	mg kg^{-1} DM	< 1	0.03	0.02	<0.05	<0.01
Ni	mg kg^{-1} DM	< 50	15	14	11	3.6
Pb	mg kg^{-1} DM	<120	<1.7	9.7	<5	2.8
As	mg kg^{-1} DM	<40	1.3	1.2	<1	0.45

* The sum of nutrients N, P_2O_5 and K_2O shall be at least 40 g kg^{-1} FM, and N or P_2O_5 or K_2O shall be at least >10 g kg^{-1} FM.

** TOC calculated as 0.45 * Loss on ignition.

4.2 Compliance with criteria for RENURE products

Both the use of manure, and products derived from manure, are regulated by the Nitrates Directive (91/676/EEC). This Directive aims to protect waters against pollution caused by nitrates from agricultural sources. Some regions are therefore designated as 'Nitrate Vulnerable Zones' (NVZ) where an application limit of 170 kg total N of livestock manure/ha/year applies. As the N requirement of the crop might be higher than this application standard, the crops' additional N needs must be met with synthetic mineral fertilisers.

In the Nitrates Directive 'livestock manure' is defined as 'waste products excreted by livestock, even in processed form'. As manure is abundantly available in many livestock producing regions of the EU, farmers are unlikely to want to pay for recovered nutrients from manure when the recovered products are also restricted by the same application limit. This means that creating added value for recovered nutrients can be extremely challenging. If farmers could use nutrients recovered from manure under the same conditions as synthetic fertilisers in NVZ, thereby removing them from the application standard of 170 kg total N/ha/year, this could be a major boost for creating a market for the recovered nutrients and therefore the feasibility of business cases of nutrient recovery and reuse (NRR) in Europe, and allow the manure to be used locally rather than transported over long distances.

The European Commission has recognised this barrier and has recently published the RENURE report (Recovered Nitrogen from manURE) which proposes criteria (Table 4-5) to authorise manure-derived recycled nitrogen fertilising products to be used above the application standard of 170 kg total N/ha for manure-derived N fixed by the Nitrates Directive. Authorisation to use RO concentrate as a substitute for mineral N fertiliser is of particular importance for GZV since it would enable them to sell RO concentrate to farmers in the nearby region rather than to transport the concentrate to regions with a demand for animal manure. RO concentrate complies with the criteria as set by the JRC, similarly as AS solution.

Table 4-5 Criteria for RENURE products and composition of ammonium sulphate (BENAS) and RO concentrate (GZV)

Parameters	**Unit**	**RENURE product (JRC)**	**Ammonium sulphate (BENAS)**	**RO concentrate (GZV)**
TOC:TN**		≤ 3	0.0076	-
Mineral N:TN**		≥ 0.9	1.0	0.99
Cu	mg kg^{-1} DM	≤ 300	0.13	<100
Zn	mg kg^{-1} DM	≤ 800	0.12	<550

** For RENURE products either the threshold for TOC:TN ratio or NH_4-N:TN ratio should be met.

5 General conclusion and discussion

5.1 Overall conclusion

The following main conclusions can be drawn from this report:

- The preformed **product characterisation** has confirmed that application of NRR technology changes the initial composition of the raw digestate and results in different recovered products that contain either higher concentration of nutrients than the raw (untreated) digestate or other ratio's between macro-nutrients as compared to digestate (section 2.1).

- **Organic micro pollutants** analyses show that fertilisers from biowaste and manure can contain residues of herbicides, pesticides and pharmaceuticals. The number of compounds detected varied among the demo plants and is likely related to differences in feedstock. All compounds detected in the analysed products are permitted in the EU except for Chloropropham that has been phased out in 2020 but might still be present in residues of potatoes (section 2.2). No residues were detected in purified water that is being discharged onto surface water.

- For SYSTEMIC demonstration plants, in **Fertiliser Product Regulation**, Component Material Categories (CMC) 4 (Fresh crop digestate) and CMC 5 (Digestate other than fresh crop digestate) are relevant. In addition, criteria for CMC 11 that covers industrial by-products including e.g. AS solution obtained through stripping and scrubbing are relevant however have not yet been defined (section 4.1).

- AS solution from BENAS and RO concentrate from GZV comply with the currently proposed **RENURE criteria** and as such have potential to be exempt from the imposed application standard of 170 kg total N/ha from the Nitrates Directive (section 4.2).

- **Nitrogen and carbon incubation tests** have shown that raw digestate and SF of digestate have a potential to be used as a source of C since, according to performed tests, at least 50% of applied C will remain available after the first year of application. This contributes to '4 per 1000' initiative that aims to demonstrate that agriculture, and in particular agricultural soils can play a crucial role where food security and climate change are concerned (section 3.1 and 3.2).

- Despite the high N content in recovered end products, the N_2O **emissions on laboratory scale** were lower than their mineral counterparts (urea and calcium ammonium nitrate). CO_2 emissions in some of the biobased fertilisers were high owing to their high OM contents. However, CO_2 emissions from biobased fertilisers may be considered as biogenic and, therefore, do not contribute to a net increase in atmospheric CO_2. Meanwhile, CH_4 emissions from all fertilisers were negligible i.e. not significantly different from the unfertilised soil control. Primary and secondary phase separation seemed to decrease the GWP relative to the raw digestates which had the highest GWP (section 3.3).

- **Struvite** is not as effective as DAP in terms of supporting young maize plants, but struvite is expected to be effective as a slow release fertiliser. It is advised to further investigate if grinding of struvite, thereby reducing its particle size and increasing the dissolution rate, is an effective post-treatment leading to a higher P availability of struvite. Care should be taken with interpretation of conventional P availability assessments, including P-$CaCl_2$, due to struvite dissolution during extractions (section 3.5).

- **P-speciation** in the solid fractions has shown a wide range in the fraction of easily available P (20-85% of total-P). Highest fraction of available P was found in the SF of digestate of GZV, whereas the lowest fraction of available P was found in the SF of digestate of Am-Power. The fraction of easily available P correlated negatively with the molar P/Fe ratio pointing to fixation of P by metal salts added during processing or by soil in case garden waste or iron-rich sludge was used as a feedstock (section 3.6).

- **Organic fibres** recovered from digestate of GZV and BENAS were found to be suitable to replace respectively 13 and 30% of peat in regular potting soil mixtures without inducing negative effects on plant growth. Due to high salt content in fibres of GZV it is advised to focus on replacement of peat in substrates for cultivations were the salt (EC) levels is not limiting e.g. substrates for mushrooms. Organic fibres of BENAS have a lower EC value and hence are more suitable to replace peat in potting soil mixtures (section 3.4).

- Results from **field trials** show that the use of tested biobased fertilisers results in a similar crop yield as the use of conventional mineral N fertilisers (i.e. CAN and urea). Also, there were no significant differences observed in regard to the nitrate residue in the soil layer of 0-90 cm. However, in some instances lower crop N uptake was observed, which led to lower ANR and NFRV values for certain biobased products. In **Belgian 1-year trial** LF of digestate had NFRV (175±99) similar to the one of CAN (126±27). Low precipitation at the beginning of growing season and heterogeneous growth of maize plants starting from a heterogeneous germination, might have led to high standard deviations of the calculated ANR and NFRV values. In Croatian **2-year field trial**, LF of digestate in combination with NPK resulted in highest NFRV value of 83±9 % as compared to the sole use of conventional NPK. In **Italian 3-year field trial** maize plants were able to use the N provided with the digestate with similar efficiency of that provided with urea (NFRV of digestate= 83.7%). In general, studies on NFRVs tend to show a notable variation across different field experiments. This variation stems from the effects of variable weather and soil conditions on the performance of both biobased products and the used references (section 3.7 – 3.9).

5.2 Conclusion per demonstration plant

5.2.1 Groot Zevert Vergisting (the Netherlands)

As a demonstration plant located in the Netherlands, a country that is faced with nutrient surplus and strict N and P application limits, GZV aims to increase mineral N content and reduce P content in raw digestate by producing N and P-rich biobased fertilisers. Moreover, the high water content in raw digestate (> 90% water; DM = 8.1%) makes it challenging and not economically feasible to store and transport raw digestate. As a first step in tackling these issues decanter mechanical separation of raw digestate is used to produce NK-rich LF of digestate and PC-rich SF of digestate. By implementing GENIUS technology (an advanced membrane system consisting of microfiltration and RO) on LF of digestate (NH_4-N/total N = 0.69 N/P = 11), GZV managed to increase NH_4-N/total N and N/P ratio from 0.68 and 4.3 in raw digestate to 0.99 and 54 in **RO concentrate**, respectively. In the Netherlands, field trials (not part of SYSTEMIC, Ehlert, 2020) have been performed to test a blend of RO concentrate (produced by GZV) with ammonium sulphate and urea. The agricultural effectiveness of the new fertilising product was comparable to that of the synthetic mineral N fertiliser blend in the terms of grass yield. The mineral N in soil was comparable to the blend of mineral N fertilisers, however, it was high as a result of the drought which deteriorated the quality of the grass sod. The lower the quality of the grass sod, the higher the quantity of mineral N was observed in the soil 0-90 cm layer after the last cut. The practical observation is that the high pH (8.4 ± 0.17) of the RO concentrates may cause volatilisation of NH_3 during the application of the product. Therefore, distribution of the product by injecting it into the soil is advisable and mandatory in the Netherlands. Along with RO concentrate, permeate water is produced and treated with an IX to produce **purified water**. The purified water is discharged to the surface water at NH_4-N concentration of 0.2 ± 0.095 mg L^{-1} (Table 2-8; section 2.1.7).

As most of P is concentrated in SF of digestate (NH_4-N/total N = 0.55 N/P = 1.34), GZV implemented RePeat system where P from SF of digestate is precipitated in the form of a calcium phosphate sludge (hereafter referred to as precipitated P salts). This process also results in a residue called low-P soil improver. The **SF of digestate and low-P soil improver** have shown to be a good source of C, by exhibiting HC value of 70% and 73% (section 3.2), similar to the one of raw digestate (78%; section 3.1) but somewhat lower than commercial compost (93%; section 3.2). Nevertheless, when considering the amount of effective organic matter (EOM) that can be applied to soils within the phosphate limits, i.e. the EOM/P ratio, low P-soil improver (EOM/P ratio = 204) had the highest potential to be used as an organic soil improver compared to the other SFs (EOM/P ratio ranges 23-49), raw digestate (EOM/P ratio = 16; section 3.1) and even commercial compost (EOM/P ratio = 86; section 3.2). As such, reducing the P concentration in SFs of digestates facilitates their application in many European soils. Moreover, tests on P availability have shown that the highest fraction of available P was found in the SF of digestate of GZV, whereas the fraction of easily available P was lower in the low-P soil improver of GZV due to removal of the easily available fraction during treatment with sulphuric acid in the RePeat process (section 3.6). Furthermore, initial tests have shown that low-P soil improver also has a potential to be used as sustainable alternative for peat in potting soils (section 3.4). Currently the limiting parameter is the EC value (salt content) which is high due to the use of sulphuric acid in the treatment process. An additional leaching step to reduce the salt content could improve suitability of the GZV-fibres. For GZV, it is advised to focus on replacement of peat in substrates for cultivations were the salt levels are not limiting e.g. substrates for mushrooms. The tests **on precipitated P salts** in the form of struvite (sourced from Waternet outreach location as struvite was not yet produced at GZV) have shown that struvite is not as effective as DAP in terms of supporting young maize plants, but is expected to be effective as a slow release fertiliser (section 3.5).

5.2.2 Am-Power (Belgium)

Being located in the similar regional concept as GZV, Am-Power aims primarily to reduce water content from raw digestate in order to reduce its transport and storage costs. The solution to achieve this goal was seen in implementation of an evaporator system which would allow water evaporation in the form of condensed water, and also production of residue called evaporator concentrate. The condensed water would then be treated in RO system, resulting in RO concentrate and permeate water suitable for discharge to the surface water. The condensed water would be mixed with dried SF of digestate and exported to France. Since in intermediate testing phase of evaporator it was observed that the condensed water had a high pH and total N content, decision was made to implement an acidification step prior to the evaporation unit, in order to decrease the amount of NH_3 evaporated.

As a first step, mechanical separation is performed on raw digestate (NH_4-N/total N = 0.42 N/P = 4) and resulting LF of digestate (NH_4-N/total N = 0.64 N/P = 17) is acidified with sulphuric acid and fed to the evaporator. By doing so, Am-Power managed to reduce water content by increasing DM content from 8.1% in raw digestate to 11.5% in **evaporator concentrate**. Furthermore, an increase in NH_4-N/total N ratio was observed from 0.42 in raw digestate to 0.77 in evaporator concentrate. The NH_4-N/total N ratio of 0.77 was seen as a marketable benefit of adding N when being mixed with **SF of digestate** that has a NH_4-N/total N ratio of 0.04. In general, raw digestate and dried SF of digestate of Am-Power have a high HC (%) amounting respectively to 61% (section 3.1) and 75% (section 3.2), suggesting that at least 61 - 75% of initial OC applied will remain in the soil after one year. Moreover, SF of digestate is a good source of P (N/P ratio = 1.21), however, the lowest fraction of available P was found in Am-Power's SF. This is probably related to the high Fe-content (35 g Fe/kg DW) in SF which is likely due to the intake of iron-rich sludges from flotation units of waste-water treatment plants in agro-food industry as well to the intake of garden waste containing soil. Finally, addition of sulphuric acid did reduce an N content in the **condensed water** (from 2.5 ± 0.28 g kg^{-1} to <1 g kg^{-1}). Nevertheless, during several tests, feeding of the condensed water to the RO resulted in quick fouling of membranes, preventing the correct operation of this step. As such, condensed water is considered as a final product from AmP's NRR system. At time of writing, AmP is still searching for solutions to circumvent this issue, enabling the correct operation of the final RO step.

5.2.3 Waterleau NewEnergy (Belgium)

Since being faced with similar regional problems as Am-Power, similar NRR solution was implemented at WNE where vacuum evaporation is used on LF of digestate, followed by RO treatment. This NRR technology cascade was implemented before the SYSTEMIC project, and differs from the one of Am-Power in terms of evaporator within which N is stripped from the condensate to generate N poor condensate (process water) and condensed ammonia water. The resulting concentrate from the evaporation (referred to as evaporation concentrate) step is either applied on fields as soil improver or mixed with the dried SF of digestate. Half of condensed ammonia water is mixed with the evaporator concentrate and the other is used as reductant in the DeNOx system which treats the exhaust gases of a Belgian waste incineration plant. Process water is either recirculated within the process or treated in a RO system to generate RO permeate or purified water.

By implementing this NRR technology cascade, WNE managed to reduce water content by increasing DM content from 5.7% in raw digestate to 19% in **evaporator concentrate**. Furthermore, a reduction in NH_4-N/total N ratio was observed from 0.61 in raw digestate to 0.46 in evaporator concentrate, as WNE recovers N in the form of 100% mineral **condensed ammonia water** with total N content of 9.3%. WNE treats the process water from the evaporation system into a double pass RO system. Permeate from the first RO reaches a quality sufficient for the preparation of polymer solution. **Permeate** from the second RO (purified water) can be disposed into surface water, as it meets Flemish discharge limits for COD (125 mg L^{-1}), TSS (35 mg L^{-1}), TN (15 mg L^{-1}) and TP (2 mg L^{-1}).

In general, condensed ammonia water is not perceived as a good N fertiliser since it has high pH (11) and hence N can volatilise easily. This was also confirmed in 1-year field trial with maize where products of WNE were tested as compared to the use of CAN. In this field trial, despite being 100% in mineral N form, condensed ammonia water performed relatively poor in terms of FW and DW maize yield, resulting on average to the lowest maize yield which was similar to the one of unfertilised control. Similar results were obtained with raw digestate and evaporator concentrate, whereas the treatment with LF of digestate resulted in the highest average values in FW and DW biomass yield and crop N uptake, with significant ($p<0.05$) difference compared to unfertilised control, digestate, evaporator concentrate and condensed ammonia water treatment, but no significant difference compared to CAN and pig manure (Table 3-8; section 3.7). Consequently, the calculated ANR and NFRV were significantly ($p<0.05$) higher in LF of digestate treatment compared to digestate, evaporator concentrate and condensed ammonia water treatment. When comparing biobased treatments to the synthetic reference, i.e. CAN treatment, significant difference was only observed in N uptake and the calculated ANR of the treatment with digestate which had lower values than CAN treatment. When assessing soil mineral N in 0-90 cm soil depth, there was no significant difference observed in biobased treatments as compared to CAN treatment at harvest time.

5.2.4 Acqua & Sole (Italy)

A&S produces an organic liquid fertiliser (digestate) from sewage sludge and agro-food biowaste to be used as fertiliser and carbon source on fields in the region of the plant. These N-rich substrates may lead to an excess of NH_3 levels in bioreactors, thus causing the failure of the digestion process. By coupling N-stripping to their AD, A&S found a solution to maintain NH_3 levels below inhibiting concentrations in the bioreactor, while at the same time producing C-rich digestate low in mineral N (NH_4-N/total N ratio = 0.46) and recovering ammonia in the form of 100% mineral AS solution (7.5% total N). As low C content in soils is an issue in Italy, the utilisation of digestate as soil improver is seen as a valuable tool to tackle this. However, restrictions on N application on agricultural land limit their use, making it necessary to find solutions to lower their N content. In this context, N-stripping represents an interesting solution.

A three-year field trial in maize cultivation was conducted in Italy where performance of the **N-stripped digestate** from A&S was tested against urea. Both treatments received the recovered **AS solution** in topdressing fertilisation (through fertigation). The reported data show that the use of the digestate produced by A&S for three consecutive years in a full-scale trial did not cause detectable negative effects on the soil, environment or on the maize produced, compared to the use of urea. As for the effect on the soil characteristics, the only significant difference observed after three years of experimentation concerns the higher OC content in soils fertilised with digestate. The laboratory C incubation also showed that digestate from A&S had the highest HC (among the tested digestates) amounting to 81% (section 3.1). No accumulations of heavy metals or organic pollutants were observed in the soils. The nitrate concentrations in the soil below the plots fertilised with digestate have always been equivalent to those of soils fertilised with urea. Equivalent were also ammonia and odours emissions detected immediately after fertilisation. Finally, the greenhouse gases emission (N_2O, CO_2 and CH_4) from soils fertilised with digestate was equivalent to that from urea fertilised soils (similar was confirmed on lab scale; section 3.3). As for the performance of the digestate as a fertiliser, the results obtained show that it is comparable to that obtained with urea. The plants were able to use the ammonia N provided with the digestate with the same efficiency of that provided with urea, producing equivalent quantities of grain and without accumulating heavy metals or other dangerous elements. Overall, all these data show that the digestate produced by A&S plant can completely replace other synthetic fertilisers without affecting the environment or loss of agronomic performance. For the practical use of AS solution it should be stated that the slightly acidic pH (5.9) of AS solution should not pose a risk to soil acidification and machinery corrosion, however, the high EC value (118 mS cm^{-1}) may represent a threat for salt-sensitive crops.

5.2.5 BENAS (Germany)

Similar as to A&S AD plant, BENAS treats chicken manure that is N-rich feedstock. Due to NH_3 inhibition of the anaerobic bacteria, chicken manure remains a difficult stream to digest and restriction on N application rate makes hard to get rid of it after processing. This leads to high transportation cost over large distances. BENAS, producing up to 400 t d^{-1} of digestate, has been hereby forced to search for a digestate treatment technology that lowers the NH_3 content of the digestate. The solution was found in N-stripping which allows them to generate **digestate** that contains 50% of total N in mineral form and recover the rest of NH_3 in the form of **AS solution** (4.6% of total N). As compare to A&S where sulphuric acid is used as adsorbing agent, BENAS uses flue gas desulphurisation (FGD) gypsum which also allows production of **$CaCO_3$ sludge**. The CaCO3 sludge has a high DM and Ca content, amounting to 698 ± 48 g kg^{-1} FM and 227 ± 35 g kg^{-1} FM, respectively (Table 2-7; section 2.1.6). Due to its high pH, it can be used as a liming agent without causing alkalinisation because it dissolves only in acid soils.

The N-stripped digestate showed to be a good source of stabile OC, suggesting that at least 74% of initial OC applied will remain in the soil after one year. Since 2020, the production of N-free fibers started again, yet not continuously. The N-stripped digestate outgoing from the N-stripper is separated into a liquid and a solid fraction. The former is recycled in the AD, the latter is processed in a fibre molding or paper making machine to products with residual moisture. Finally, the residual moisture, is dried with excess heat generated from the AD plant. The final dried product is collected as NH_3-free fibers and it is suitable for different applications in the fiber and timber industries (i.e. fiberboard) or as alternative for peat used in potting soil (section 3.4). BENAS-fibres have a low EC value and are therefore more suitable for use in potting soil than the ones of GZV. Both biobased fibres have high OUR (oxygen uptake rates) as compared to potting soil, pointing to a risk for anoxic conditions in the growing media but this can be controlled by mixing the organic fibres with regular potting soil. Physical properties of the biobased fibres, including water holding capacity and bulk density, are similar to peat.

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Annex

The Annex provides an overview of pesticides and pharmacologically active substances that were analysed by LUFA on end-products from five demo plants.

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Deutsche
Akkreditierungsstelle
D-PL-14165-01-00

LUFA
NORD-WEST

Attachement to analytical report

Version: 1

Hameln, 06/04/2021

Order-no.: 2494429
Sample receipt: 25/02/2021
Sample-no.: **21UO000598**
Sample type: Gärrest
Description: Benas digestate, Probe B_1
(as declared by sender)

Pesticides - analyzed substances

Fungizide Paket

Acibenzolar-S-methyl (<0.050 mg/kg), Anilazine (<0.050 mg/kg), Azoxystrobin (<0.050 mg/kg), Benalaxyl, sum of isomers (<0.050 mg/kg), Benomyl (<0.050 mg/kg), Benthiavalicarb, sum of stereoisomers (<0.050 mg/kg), Benzovindiflupyr (<0.050 mg/kg), Bitertanol, sum of isomers (<0.050 mg/kg), Bixafen (<0.050 mg/kg), Boscalid (<0.050 mg/kg), Bromuconazole, sum of isomers (<0.050 mg/kg), Bupirimate (<0.050 mg/kg), Carbendazim (<0.050 mg/kg), Carbendazim, sum (<0.050 mg/kg) - (Summe aus Carbendazim, Benomyl), Carboxin (<0.050 mg/kg), Chinomethionat (<0.050 mg/kg), Cyazofamid (<0.050 mg/kg), Cyflufenamid, sum of isomers (<0.050 mg/kg), Cymoxanil (<0.050 mg/kg), Cyproconazole (<0.050 mg/kg), Cyprodinil (<0.050 mg/kg), Dichlofluanid (<0.050 mg/kg), Diclobutrazol (<0.050 mg/kg), Difenoconazole (<0.050 mg/kg), Dimethomorph, sum of isomers (<0.050 mg/kg), Dimoxystrobin (<0.050 mg/kg), Diniconazole (<0.050 mg/kg), Diphenylamine (<0.050 mg/kg), Dodemorph (<0.050 mg/kg), Dodine (<0.050 mg/kg), Epoxiconazole (<0.050 mg/kg), Ethirimol (<0.050 mg/kg), Ethoxyquin (<0.050 mg/kg), Etridiazole (<0.050 mg/kg), Famoxadone (<0.050 mg/kg), Fenamidone (<0.050 mg/kg), Fenarimol (<0.050 mg/kg), Fenbuconazole, sum of isomers (<0.050 mg/kg), Fenfuram (<0.050 mg/kg), Fenhexamid (<0.050 mg/kg), Fenpiclonil (<0.050 mg/kg), Fenpropidin, sum (<0.050 mg/kg), Fenpropimorph, sum of isomers (<0.050 mg/kg), Fluazinam (<0.050 mg/kg), Fludioxonil (<0.050 mg/kg), Fluopicolide (<0.050 mg/kg), Fluopyram (<0.050 mg/kg), Fluoxastrobin, sum of isomers (<0.050 mg/kg), Fluquinconazole (<0.050 mg/kg), Flusilazole (<0.050 mg/kg), Flutolanil (<0.050 mg/kg), Flutriafol (<0.050 mg/kg), Fluxapyroxad (<0.050 mg/kg), Fuberidazole (<0.050 mg/kg), Furmecyclox (<0.050 mg/kg), Hexaconazole (<0.050 mg/kg), Hymexazol (<0.050 mg/kg), Imazalil, sum of isomers (<0.050 mg/kg), Iprodione (<0.050 mg/kg), Iprovalicarb (<0.050 mg/kg), Isopyrazam (<0.050 mg/kg), Kresoxim-methyl (<0.050 mg/kg), Mandipropamid, sum of isomers (<0.050 mg/kg), Mepanipyrim (<0.050 mg/kg), Mepronil (<0.050 mg/kg), Metalaxyl (<0.050 mg/kg), Metconazole (<0.050 mg/kg), Methfuroxam (<0.050 mg/kg), Myclobutanil, sum of isomers (<0.050 mg/kg), Nuarimol (<0.050 mg/kg), Ofurace (<0.050 mg/kg), Oxadixyl (<0.050 mg/kg), Oxathiapiprolin (<0.050 mg/kg), Oxycarboxin (<0.050 mg/kg), Penconazole, sum of isomers (<0.050 mg/kg), Pencycuron (<0.050 mg/kg), Picoxystrobin (<0.050 mg/kg), Prochloraz (<0.050 mg/kg), Procymidone (<0.050 mg/kg), Propiconazole, sum of isomers (<0.050 mg/kg), Proquinazid (<0.050 mg/kg), Prothioconazole, sum of isomers (<0.050 mg/kg), Pyraclostrobin (<0.050 mg/kg), Pyrazophos (<0.050 mg/kg), Pyrifenox (<0.050 mg/kg), Pyrimethanil (<0.050 mg/kg), Quinoxyfen (<0.050 mg/kg), Silthiofam (<0.050 mg/kg), Spiroxamine, sum of isomers (<0.050 mg/kg), Tebuconazole (<0.050 mg/kg), Tetraconazole (<0.050 mg/kg), Thiabendazol, 5-Hydroxy- (<0.050 mg/kg), Thiabendazole (<0.050 mg/kg), Thiophanat (<0.050 mg/kg), Thiophanate-methyl (<0.050 mg/kg), Tolclofos-methyl (<0.050 mg/kg), Tolylfluanid (<0.050 mg/kg), Triadimefon (<0.050 mg/kg), Triadimenol, sum of isomers (<0.050 mg/kg), Triazoxide (<0.050 mg/kg), Tricyclazole (<0.050 mg/kg), Tridemorph (<0.050 mg/kg), Trifloxystrobin (<0.050 mg/kg), Triflumizol (<0.050 mg/kg), Triforine (<0.050 mg/kg), Triticonazole (<0.050 mg/kg)

Herbizide Paket

3,4-Dichloranilin (<0.050 mg/kg), Aclonifen (<0.050 mg/kg), Alachlor (<0.050 mg/kg), Atrazin, Desethyl- (<0.050 mg/kg), Atrazin, Desisopropyl- (<0.050 mg/kg), Atrazine (<0.050 mg/kg), Beflubutamid (<0.050 mg/kg), Bifenox (<0.050 mg/kg), Bromacil (<0.050 mg/kg), Buturon (<0.050 mg/kg), Carbetamide, sum of isomers (<0.050 mg/kg), Carfentrazone-ethyl (determined as carfentrazone and expressed as carfentrazone-ethyl) (<0.050 mg/kg), Chlorbromuron (<0.050 mg/kg), Chloridazon (<0.050 mg/kg), Chlorotoluron (<0.050 mg/kg), Chloroxuron (<0.050 mg/kg), Chlorpropham (<0.050 mg/kg), Clomazone (<0.050 mg/kg), Cyanazin (<0.050 mg/kg), Cycloat (<0.050 mg/kg), Cycloxydim (<0.050 mg/kg), Desmedipham (<0.050 mg/kg), Desmetryn (<0.050 mg/kg), Di-allate (<0.050 mg/kg), Dichlorbenzamid, 2,6- (<0.050 mg/kg), Difenoxuron (<0.050 mg/kg), Diflufenican (<0.050 mg/kg), Dimefuron (<0.050 mg/kg), Dimethachlor (<0.050 mg/kg), Dimethenamid (<0.050 mg/kg), Diuron (<0.050 mg/kg), Ethidimuron (<0.050 mg/kg), Ethofumesat (<0.050 mg/kg), Fenoxaprop-Ethyl (<0.050 mg/kg), Fenoxaprop-free acid (<0.050 mg/kg), Fenuron (<0.050 mg/kg), Florasulam (<0.050 mg/kg), Fluazifop, free acid (<0.050 mg/kg), Fluazifop-butyl (<0.050 mg/kg), Flufenacet (<0.050 mg/kg), Flumioxazine (<0.050 mg/kg), Fluometuron (<0.050 mg/kg), Fluridone (<0.050 mg/kg), Flurochloridone, sum of isomers (<0.050 mg/kg), Fluroxypyr (<0.050 mg/kg), Fluroxypyr-1-methylheptylester (<0.050 mg/kg), Flurprimidole (<0.050 mg/kg), Flurtamone (<0.050 mg/kg), Hexazinon (<0.050 mg/kg), Imazamox (<0.050 mg/kg), Isoproturon (<0.050 mg/kg), Isoxaben (<0.050 mg/kg), Isoxadifen (<0.050 mg/kg), Isoxadifen-ethyl (<0.050 mg/kg), Isoxaflutol (<0.050 mg/kg), Lenacil (<0.050 mg/kg), Linuron (<0.050 mg/kg), Mesotrion (<0.050 mg/kg), Metamitron (<0.050 mg/kg), Metamitron-desamino (<0.050 mg/kg), Metazachlor (<0.050 mg/kg), Methabenzthiazuron (<0.050 mg/kg), Metobromuron (<0.050 mg/kg), Metolachlor, sum of isomers (<0.050 mg/kg), Metosulam (<0.050 mg/kg), Metoxuron (<0.050 mg/kg), Metribuzin (<0.050 mg/kg), Molinate (<0.050 mg/kg), Monolinuron (<0.050 mg/kg), Monuron (<0.050 mg/kg), Napropamide, sum of isomers (<0.050 mg/kg), Neburon (<0.050 mg/kg), Paclobutrazol, sum of isomers (<0.050 mg/kg), Pendimethalin (<0.050 mg/kg), Pethoxamid (<0.050 mg/kg), Phenmedipham (<0.050 mg/kg), Picolinafen (<0.050 mg/kg), Pinoxaden (<0.050 mg/kg), Profluralin (<0.050 mg/kg), Prohexadion (<0.050 mg/kg), Prometryn (<0.050 mg/kg), Propazin (<0.050 mg/kg), Propyzamide (<0.050 mg/kg), Prosulfocarb (<0.050 mg/kg), Pyraflufen-ethyl (<0.050 mg/kg), Pyridat (<0.050 mg/kg), Pyridat-Metabolit (<0.050 mg/kg), Quinmerac (<0.050 mg/kg), Quinoclamin (<0.050 mg/kg), Sebuthylazin (<0.050 mg/kg), Sethoxydim (<0.050 mg/kg), Simazine (<0.050 mg/kg), Simetryn (<0.050 mg/kg), Sulcotrione (<0.050 mg/kg), Tebutam (<0.050 mg/kg), Tebuthiuron (<0.050 mg/kg), Tembotrione (<0.050 mg/kg), Tepraloxydim (<0.050 mg/kg), Terbumeton (<0.050 mg/kg), Terbuthylazin-desethyl (<0.050 mg/kg), Terbuthylazine (<0.050 mg/kg), Terbutryn (<0.050 mg/kg), Topramezone (<0.050 mg/kg), Tri-allate (<0.050 mg/kg), Triapenthenol (<0.050 mg/kg), Trietazin (<0.050 mg/kg), Trifluralin (<0.050 mg/kg), Trinexapac (<0.050 mg/kg), Trinexapac-ethyl (<0.050 mg/kg)

Insektizide Paket

3,4,5-Trimethacarb (<0.050 mg/kg), Abamectin (<0.050 mg/kg), Acetamiprid (<0.050 mg/kg), Acrinathrin (<0.050 mg/kg), Aldicarb (<0.050 mg/kg), Aldicarb-Sulfon (<0.050 mg/kg), Aldicarb-Sulfoxid (<0.050 mg/kg), Aminocarb (<0.050 mg/kg), Amitraz (<0.050 mg/kg), Azamethiphos (<0.050 mg/kg), Bendiocarb (<0.050 mg/kg), Benfuracarb (<0.050 mg/kg), Benzoximat (<0.050 mg/kg), Bifenthrin, sum of isomers (<0.050 mg/kg), Binapacryl (<0.050 mg/kg), Buprofezin (<0.050 mg/kg), Butocarboxim (<0.050 mg/kg), Butocarboxim-sulfoxid

#1 = IfF, Oldenburg; #2 = IfT, Oldenburg; #3 = IfL, Oldenburg; #5 = Analysis was don in another laboratory; #6 = Method is not accredited
„<…“ = LOQ

The results refer to the present material of the sample. It is not allowed to copy parts of this analytical report without authorization of the LUFA Nord-West. The accreditation is valid for the in D-PL-14165-01-00 listed scope.

Attachement to analytical report Version: 1 Hameln, 06/04/2021

Order-no.:	2494429	Sample receipt:	25/02/2021
Sample-no.:	**21UO000598**		
Sample type:	Gärrest		
Description: (as declared by sender)	Benas digestate, Probe B_1		

(<0.050 mg/kg), Butoxycarboxim (<0.050 mg/kg), Carbaryl (<0.050 mg/kg), Carbofuran (<0.050 mg/kg), Carbosulfan (<0.050 mg/kg), Chlorantraniliprole (DPX E-2Y45) (<0.050 mg/kg), Chlordimeform (<0.050 mg/kg), Chlorfluazuron (<0.050 mg/kg), Chlorobenzilate (<0.050 mg/kg), Chlorthiophos (<0.050 mg/kg), Cinerin I (<0.050 mg/kg), Cinerin II (<0.050 mg/kg), Clofentezine (<0.050 mg/kg), Clothianidin (<0.050 mg/kg), Cotinin (<0.050 mg/kg), Cyfluthrin, sum of isomers (<0.050 mg/kg), Cyhalothrin, sum of isomers (<0.050 mg/kg), Cypermethrin, sum of isomers (<0.050 mg/kg), Deltamethrin (<0.050 mg/kg), Desmethyl-formamido-pirimicarb (<0.050 mg/kg), Desmethyl-pirimicarb (<0.050 mg/kg), Dichlorvos (<0.050 mg/kg), Dicrotophos (<0.050 mg/kg), Diethofencarb (<0.050 mg/kg), Diflubenzuron (<0.050 mg/kg), EPN (<0.050 mg/kg), Esfenvalerate (<0.050 mg/kg), Ethiofencarb (<0.050 mg/kg), Ethiofencarb-sulfon (<0.050 mg/kg), Ethiofencarb-sulfoxid (<0.050 mg/kg), Ethoprophos (<0.050 mg/kg), Etofenprox (<0.050 mg/kg), Fenazaquin (<0.050 mg/kg), Fenbutatin oxide (<0.050 mg/kg), Fenoxycarb (<0.050 mg/kg), Fenpropathrin (<0.050 mg/kg), Fenpyroximate (<0.050 mg/kg), Fenvalerat (<0.050 mg/kg), Fenvalerat, sum (<0.050 mg/kg) - (Summe aus Fenvalerat, Esfenvalerate), Flonicamid (<0.050 mg/kg), Flucythrinate (<0.050 mg/kg), Flufenoxuron (<0.050 mg/kg), Fluvalinate, sum of isomers (<0.050 mg/kg), Fosthiazate (<0.050 mg/kg), Furathiocarb (<0.050 mg/kg), Hexythiazox (<0.050 mg/kg), Imidacloprid (<0.050 mg/kg), Indoxacarb, sum of isomers (<0.050 mg/kg), Isazofos (<0.050 mg/kg), Isoxathion (<0.050 mg/kg), Jasmolin I (<0.050 mg/kg), Jasmolin II (<0.050 mg/kg), Lufenuron, sum of isomers (<0.050 mg/kg), Methiocarb (<0.050 mg/kg), Methiocarb-sulfon (<0.050 mg/kg), Methiocarb-sulfoxid (<0.050 mg/kg), Methomyl (<0.050 mg/kg), Milbemycin A4 (<0.050 mg/kg), Naled (<0.050 mg/kg), Nicotine (<0.050 mg/kg), Oxamyl (<0.050 mg/kg), Permethrin, sum of isomers (<0.050 mg/kg), Phenothrin (<0.050 mg/kg), Piperonylbutoxide (<0.050 mg/kg), Pirimicarb (<0.050 mg/kg), Pirimiphos-methyl (<0.050 mg/kg), Prallethrin (<0.050 mg/kg), Promecarb (<0.050 mg/kg), Propamocarb (<0.050 mg/kg), Propargite (<0.050 mg/kg), Propetamphos (<0.050 mg/kg), Propoxur (<0.050 mg/kg), Pymetrozine (<0.050 mg/kg), Pyrethrin I (<0.050 mg/kg), Pyrethrin II (<0.050 mg/kg), Pyrethrins (<0.050 mg/kg) - (Summe aus Pyrethrin II, Pyrethrin I, Cinerin II, Cinerin I, Jasmolin II, Jasmolin I), Pyridaben (<0.050 mg/kg), Pyriproxyfen (<0.050 mg/kg), Resmethrin, sum of isomers (<0.050 mg/kg), Rotenone (<0.050 mg/kg), Spinosad, sum (<0.050 mg/kg), Spirodiclofen (<0.050 mg/kg), Spirotetramat (<0.050 mg/kg), Tebufenozide (<0.050 mg/kg), Tebufenpyrad (<0.050 mg/kg), Teflubenzuron (<0.050 mg/kg), Tetramethrin (<0.050 mg/kg), Thiacloprid (<0.050 mg/kg), Thiamethoxam (<0.050 mg/kg), Thiodicarb (<0.050 mg/kg), Thiofanox (<0.050 mg/kg), Thiofanox-sulfon (<0.050 mg/kg), Thiofanox-sulfoxid (<0.050 mg/kg), Tribufos (<0.050 mg/kg)

Pharmacologically active substances - analyzed substances

Chloramphenicol (<0.0500 mg/kg), Chlorotetracycline (<0.0500 mg/kg), Ciprofloxacin (<0.0500 mg/kg), Clofibrat (<0.0500 mg/kg), Clofibrinsäure (<0.0500 mg/kg), Cotinin (<0.0500 mg/kg), Dapson (<0.0500 mg/kg), Demeclocyclin (<0.0500 mg/kg), Dimetridazol (<0.0500 mg/kg), Doxycycline (<0.0500 mg/kg), Enrofloxacin (<0.0500 mg/kg), Ethopabat (<0.0500 mg/kg), Fenbendazol (<0.0500 mg/kg), Florfenicol (<0.0500 mg/kg), Florfenicol-amin (<0.0500 mg/kg), Flubendazol (<0.0500 mg/kg), Halofuginon (<0.0500 mg/kg), Lasalocid-Sodium (<0.0500 mg/kg), Maduramicin (<0.0500 mg/kg), Mebendazol (<0.0500 mg/kg), Metronidazol (<0.0500 mg/kg), Metronidazol-OH (<0.0500 mg/kg), Minocyclin (<0.0500 mg/kg), Monensin (<0.0500 mg/kg), Narasin (<0.0500 mg/kg), Nicotine (<0.0500 mg/kg), Norfloxacin (<0.0500 mg/kg), Oxolinsäure (<0.0500 mg/kg), Oxytetracycline (<0.0500 mg/kg), Robenidin (<0.0500 mg/kg), Rolitetracyclin (<0.0500 mg/kg), Ronidazol (<0.0500 mg/kg), Salinomycin (<0.0500 mg/kg), Semduramicin (<0.0500 mg/kg), Sulfabenzamid (<0.0500 mg/kg), Sulfacetamid (<0.0500 mg/kg), Sulfachloropyridazine (<0.0500 mg/kg), Sulfachlorpyrazin (<0.0500 mg/kg), Sulfadiazin, Sulfapyrimidin (<0.0500 mg/kg), Sulfadimethoxin (<0.0500 mg/kg), Sulfadimidin Sulfamethazin (<0.0500 mg/kg), Sulfadoxin (<0.0500 mg/kg), Sulfaethoxypyridazin (<0.0500 mg/kg), Sulfaguanidin (<0.0500 mg/kg), Sulfamerazin (<0.0500 mg/kg), Sulfamethazin (<0.0500 mg/kg), Sulfamethizol (<0.0500 mg/kg), Sulfamethoxazol (<0.0500 mg/kg), Sulfamethoxypyridazin (<0.0500 mg/kg), Sulfanilamid (<0.0500 mg/kg), Sulfanitran (<0.0500 mg/kg), Sulfapyridin (<0.0500 mg/kg), Sulfaquinoxalin (<0.0500 mg/kg), Sulfathiazol (<0.0500 mg/kg), Sulfisoxazol (<0.0500 mg/kg), Tetracycline (<0.0500 mg/kg), Thiabendazole (<0.0500 mg/kg), Trimethoprim (<0.0500 mg/kg)

#1 = IfF, Oldenburg; #2 = IfT, Oldenburg; #3 = IfL, Oldenburg; #5 = Analysis was don in another laboratory; #6 = Method is not accredited
„<…“ = LOQ

SYSTEMIC
Circular solutions for biowaste

The SYSTEMIC project has received funding from the European Union’s Horizon 2020 Framework Programme for Research and Innovation under Grant Agreement no. 730400

Horizon 2020

Systemic large-scale eco-innovation to advance circular economy and mineral recovery from organic waste in Europe

Consortium

Wageningen University and Research (NL)
Am-Power (BE)
Groot Zevert Vergisting B.V. (NL)
Acqua & Sole S.r.l. (IT)
RIKA Biofuels Development Ltd. (UK)
GNS Gesellschaft für Nachhaltige Stoffnutzung mbH (DE)
A-Farmers Ltd (FI)
ICL Europe (NL)
Nijhuis Water Technology (NL)
Proman Management GmbH (AU)
Ghent University (BE)
Milano University (IT)
Vlaams Coördinatiecentrum Mestverwerking (BE)
European Biogas Association (BE)
Rural Investment Support for Europe (BE)

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