Green fertilizer upcycling from manure: Technological, economic and environmental sustainability demonstration

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Final Report Summary - MANUREECOMINE (Green
Executive Summary:

Intensive agriculture has a vital role in sustaining the population growth. Synthetic nitrogen (N) and phosphorus (P) fertilisers are massively produced to sustain intensive agriculture, with an European market reaching about €15.5 billion per year. Their production consumes fossil fuels (N fertilisers) and rock ores (P fertilisers), creating a negative environmental footprint, also caused by massive application of fertilisers on soil and their leaching in water bodies. Manure production by European cows and pigs estimated around 1.2 billion tonnes per year. The content of nutrients in manure makes it an exquisite mining resource for bio-based fertilisation, however its application as such is strictly regulated in several European areas, denominated nutrient vulnerable zones (NVZ), due to its direct link to eutrophication. The ManureEcoMine project aimed at linking these two major agricultural fluxes in Europe: synthetic fertilisers and manure.

The project ManureEcoMine proposed a synergic approach with integrated manure processing technologies targeting the upcycling of nutrients manure as “green”, bio-based fertilisers. The technology core of the ManureEcoMine process featured a flexible, modular system for manure anaerobic co-digestion, ammonia stripping and scrubbing, solid/liquid separation and ultrafiltration, struvite precipitation and residual biological nitrogen removal. These technologies were proved at technically relevant scale in a pilot installation designed and built within the ManureEcoMine project. The pilot operation demonstrated the robustness of the ManureEcoMine technology core and its flexibility in term of manure mix quality and feasibility of the process chain. Laboratory support was provided along the entire project for the characterisation of influents, evaluation of the optimal operating parameters and lab validation of pilot processes. Moreover, laboratory tests demonstrated technologies different than those implemented in the ManureEcoMine system. An important aspect was the demonstration of the added value of combining thermophilic anaerobic digestion with ammonia side stripping, which enhanced the biogas production and the release of N and P, with potential benefits for nutrient recovery. The nutrients recovered from the lab tests and pilot operation were analysed for their fertilising potential and blended with growing media into tailor-made fertilisers, which were used for greenhouse tests on different types of plants. Struvite and ammonium sulphate recovered during the lab and pilot operations were successfully used as P and N supplies, respectively, for plant growth, with no significant modification in the rhizosphere (micro)biology. Trace contaminants migration through the entire process was assessed and a risk management plan was created, identifying the solid digestate fraction as the main contaminants (mainly, antibiotics and heavy metals) output of the system. Negligible amount of contaminants were detected in the nutrient products struvite, ammonium sulphate and related fertilising blends, and in final irrigation water. A Life Cycle Assessment showed that the synergic ManureEcoMine process combination decreased the environmental burden associated to manure, thanks to lowered emissions and environmental credits (avoided processes) associated with the nutrients recovery and energy production. Finally, the ManureEcoMine process was studied for its economic viability in terms of process costs, revenues from the generated fertilisers and reduced synthetic fertiliser application. The process resulted competitive at the cost of €18/ton manure processed, with the first combination of different ManureEcoMine technologies entering the market at 10% cost reduction compared to the current estimated cost. Considering the exhaustion of fossil fuels and depletion of P ores expected within 100 years, the ManureEcoMine market potential
results competitive in the near future, when synthetic fertilisers prices are expected to increase. ManureEcoMine demonstrated the technological feasibility of manure nutrients extraction and their potential benefit as N and P sources for plant fertilisation. The process footprint positively impacted the environment for almost all the environmental categories investigated.

Project Context and Objectives:
The world population is expected to increase in the next years up to 9 billion people by 2050, along with the average life standard and food market needs, hence stimulating intensive agriculture and animal husbandry. To sustain such growth, farmers make use of large amounts of synthetic fertilisers, which yearly consumption in the European Union is estimated around 11 million tonnes of nitrogen (N) and 1.1 million tonnes of phosphorus (P). Due to the dependency on fossil fuel (N), or the mining from exhaustible mineral deposits (P, K), fertiliser prices over the past 20 years (1990-2010) have increased at about 8.0% per year. The cost associated to such consumptions is currently estimated around €15.5 billion per year, representing a significant expense in the food production chain. Synthetic N fertilisers are produced through the Haber-Bosch process, which consumes around 3-5% of world’s natural gas, accounting for about 1-2% of the world’s annual energy consumption. P fertilisers are generated using rock resources. P rock is enlisted among the critical raw material, crucial for the economy and essential for maintaining and improving the quality of life. Biobased fertilisers are a pressing need in order to cope with the future perspective of increasing food demand and depletion of P ores and fossil fuels. Finally, long-term application of synthetic fertilisers renders exhausted soils devoid in organic carbon. As a consequence, the agricultural soil (i) sequesters less carbon, contributing to global warming, and (ii) is less productive due to lower pH buffering, water and nutrient retention, resulting in eutrophication.

Manure has been used since the past as natural fertiliser, however the variability of its composition makes it difficult to evaluate the exact supply needed for crops. European cows and pigs are estimated to produce around 1.2 billion tonnes of manure per year, corresponding to about 500,000 Olympic swimming pool. The amount of N and P in manure varies between 2 - 6 g N L^-1 and 0.2-1.6 g P L^-1, thus making manure an exquisite mining resource for nutrients reuse in agriculture. If calculated at synthetic market price, the nutrient load in manure accounts for about €10.7 billion per year. However, intensive animal husbandry and agriculture led to hotspot areas in Europe, denominated nutrient vulnerable zones (NVZ) which largely overlap with areas with intensive animal husbandry. This combination makes manure use as fertiliser very difficult, since specific legislations apply in NVZ, in order to limit nutrients leaching damages and minimise eutrophication threat. Currently, most of manure processing technologies applied on NVZ and beyond aim at the dissipation of manure in order to remove the nutrient load and thus facilitate its disposal, however no nutrients nor value is generated in these processes. A more sustainable approach in the use of nutrients in agriculture and farming has become a raw nerve, including, as first, the major environmental consequences of the disposal of animal husbandry waste.

ManureEcoMine was projected and carried out targeting the improvement of the resource efficiency in a time of greater competition for limited resources such as nutrients, increasing energy prices and carbon constraints. This was achieved by linking two major nutrient fluxes in Europe: agricultural, synthetic fertilisers and manure. With this regard, the ManureEcoMine project firstly aimed to extract nutrients from manure into concentrated sources, which are easier and more cost efficient for transportation from intensive livestock zones to nutrient shortage areas. This would greatly contribute to the reduction of both the environmental burden of manure in NVZ and the use of synthetic fertilisers. ManureEcoMine combined several technologies implemented in the wastewater treatment field. Such
technologies have been combined into a synergistic process designed for manure processing while efficiently recovering nutrients such as N, P and K. The process was translated into a pilot plant installation which was operated for 20 months in 2 different locations, with full laboratory and analytical support. The nutrient potential of the products recovered was assessed and tested in greenhouse plant growth tests. The complete process was demonstrated not only on the technological level, but also on the environmental, contaminant and economic perspectives.

The main technological objectives of the ManureEcoMine project were:

• Manure upgrading. ManureEcoMine proposed a technological combination targeting manure upgrade rather than dissipation. The organic and nutrient (N, P and K) compounds were separated into concentrated streams for an effective nutrient upcycling and transportation to nutrient depleted areas. Technologies like anaerobic digestion (thermophilic and mesophilic), ammonia stripping and scrubbing, solid/liquid separation, membrane filtration (ultrafiltration), struvite precipitation and short-cut biological nitrogen removal were combined in two process configurations for maximised nutrient recovery, maximum energy autonomy, minimal emissions, with an effluent quality suitable for irrigation.

• A process demonstrated at technologically relevant scale. The project consortium aimed at demonstrating the technology core of the ManureEcoMine with a pilot installation. The plant was designed and built based on the influent characteristics and process requirements, and operated to daily process 150 L manure and co-substrates, corresponding to the production of 50 pigs or 12 cows. The system was designed to process cow and pig manure, as well as co-substrates locally available through the entire year. Laboratory testing was in place along the entire project, in order to support the pilot plant design and operation in all its phases, and identify the optimal technical parameters to operate the installation.

• Demonstration of process robustness. The pilot installation was operated for 20 months in two different locations in Europe, being Zeeuws Vlasnderen in The Netherlands (10 months) and Catalonia in Spain (10 months). The aim was to prove that the system was able to process manure and co-substrates of very different regions, and successfully cope with fluctuations in substrates composition.

• Flexibility as main feature of the technology core. In addition, the system was designed and build with a modular approach, thus allowing the omission or reallocation of one or more units depending on the process requirements. This would enable the recovery of different products, such as struvite (N/P) or K-struvite (K/P).

• Nutrient recovery efficiency. The objective of the technological aspects of the project was to obtain an N recovery over 60% as ammonium sulphate and struvite, P recovery over 95% as struvite and solid digestate, and K recovery around 25% as K-struvite.

On the fertilising potential perspective, the objectives of the project were:

• Evaluation of fertiliser potential. Preliminary tests were expected to give indications on the nutrient composition of the recovered products and their effect on plant germination. This would indicate the optimal N/P/K composition of the novel fertilisers blended within the project.

• Tailor-made blended fertilisers. The recovered products were blended into high-performance fertilisers to stimulate nutrients uptake of different plant, including ornamental and edible plants. Several growing media were screened to optimally provide support for nutrients release and uptake by plants. The final result was expected to be tailor-made blends with specific nutritional and physicochemical composition, depending on the crop requirements.

• Struvite and ammonium sulphate proved as P and N fertilising sources. The main products expected from the project were struvite and ammonium sulphate, which were supposed to efficiently serve as bio-based nutrients (P and N, respectively) sources for plants. Greenhouse tests were in carried out for an in-
depth analysis of the effect of the new “green” fertilisers on plant growth.
• Soil microbial response to the application of recovered nutrients. The consortium aimed at assessing the fertilisers effect on a wide perspective. Not only plants growth but also soil stability evaluation following the addition of tailor-made fertilisers was included in the project.

The overall process was studied for environmental, safety and economic evaluation:
• Environmental assessment of the process. Life cycle assessment of the ManureEcoMine process was carried out for identifying the environmental profile of the entire process chain, with a special focus on the technology core. The environmental assessment included improvements on global warming potential (greenhouse gas emissions), eutrophication potential, acidification potential, ozone depletion potential, and freshwater and human toxicity. In addition, the process energy self-sustainment through energy and heat production by anaerobic co-digestion and reduced energy requirement through short cut biological removal (60% less than conventional nitrification/denitrification) was proved.
• Contaminants risk management of the entire ManureEcoMine system. Trace contaminants assessment of the entire process project was carried out, in order to identify the main input and output sources of contamination. The study proved that the main nutrients products ammonium sulphate and struvite, as well as the final irrigation water had drastically reduced load of contaminants. This enables the reduction of contaminants uptake in the soil due to manure upcycling.
• Economic sustainability and market uptake of ManureEcoMine. The economic study was included to evaluate the market potential of the ManureEcoMine system. The cost for the process to competitively enter the market was expected around €20/m3 of treated manure. The process was compared to other systems currently implemented in an intensive animal husbandry area such as Flanders.

Finally, the ManureEcoMine project aimed at providing academia, industry and society with novel means for manure upcycling and bio-based fertilisation, by:
• Improved state of the art through process optimisation and demonstration. The pilot operation over 20 months was intended to implement the state of the art demonstrating the feasibility of manure processing, and nutrients recovery and reuse potential thanks to synergistically combined process. In addition, in-depth study on fertilising potential and testing greatly enriched the know-how on recovered nutrient application and effect.

• Providing farmers and wastewater treatment companies with new means for manure processing. The technologies included in the ManureEcoMine process are known for full scale (e.g. anaerobic co-digestion) or lab/pilot level (e.g. ammonia stripping and scrubbing) application. These technologies and their combination have a significant market potential for farmers and industries operating in the wastewater treatment field. The project partner Colsen already did pilot scale test on a sewage sludge thermophilic digester and removed > 80 % of the nitrogen
• Providing fertiliser blending companies with new biobased sources for P and N fertilisers. Similarly to the technological potential uptake for wastewater treatment companies, new fertilising products would be available for fertilising blending company. The project aimed at demonstrating the suitability of such products as P, N and K sources for novel, tailor-made fertilisers.
• Giving society a new perspective for circular economy and waste upgrade. The ManureEcoMine perspective aimed at boosting the perspective of circular economy. Two open-door events at the pilot plant installation were used, among other dissemination events, for reaching the society at large and demonstrating that upcycling of manure would contribute at reducing the agricultural environmental impact.
Project Results:
WP1 - Manure characterisation - Lab support for pilot design and optimisation for pilot operation
Task 1.1 - Characterisation of manure and selection of co-substrates
The aim of this task was to obtain accurate influent data at the early stage of the project to be provided to the technology partners for the lab-scale technological optimization studies in WP1 and pilot design and construction in WP2. Samples of manures and co-substrates available in each scenario location (the Netherlands and Spain) were provided by farming partners. A complete and deep analysis of product was carried out. The characterization included nutrients (N, P, K), organic matter, dissolved ions (Mg2+, Ca2+, SO42-, among others), dry matter, solids (total, suspended, volatile solids) and physic-chemical parameters (pH, electrical conductivity, alkalinity, among others). Special attention was paid to the bio-methane production (BMP) potential and to the presence of trace contaminants (TC; mycotoxins, veterinary drugs, heavy metals). Results showed significant differences in some parameters between pig and cow manure, such as nitrogen and phosphorus content and trace contaminants, while the BMP tests revealed necessary the performance of anaerobic co-digestion for the process energy self-sufficiency. The co-substrates selected were mainly by-products (wastes) from agro-food industry, including fats, segregates or vegetable wastes mixtures. Besides increasing BMP, some co-substrates represented additional nutrient inputs. Corn was analysed, as it is currently used as co-substrate, but was discarded to avoid competence with livestock production, since it is used as fodder. In summary, this task provided the influent conditions and main constrains to deal with as starting point for the technical part of the project (for the lab-scale technological optimization works of WP1. Pilot plant design and construction in WP2, pilot operation in WP3 and WP4, as well as risk analysis in WP7). All results generated by task 1.2 were compiled in the public Deliverable 1.1

Task 1.2 - Optimisation of thermophilic and mesophilic anaerobic digestion
The main objective of this task was the optimization of thermophilic and mesophilic anaerobic digesters for maximal energy recovery and nutrient release from solid wastes. Two scenarios were evaluated: the Netherlands (NL; thermophilic, main substrate: pig manure, co-substrates: maize silage and food waste) and Spain (ES; mesophilic, main substrates: cow and pig manure, co-substrates: bioiberica fat, segregates and powdered corn). Since mesophilic digestion of animal manure is a well-established technology, the main focus of the ES scenario was the definition of the optimal mixture of substrates for co-digestion, the selection of appropriate inocula to accelerate the start-up period and the definition of the strategy to reach the operational conditions that would maximize methane production without compromising digester stability. In contrast, more attention was paid to NL scenario, as thermophilic digestion of N-rich substrates (manure, food waste, etc.) might be limited and hindered by free ammonia inhibition. Acute ammonia inhibition tests demonstrated that thermophilic biomass can tolerate short-term medium levels of total ammonium nitrogen (TAN), up to 6 g N-TAN L-1. Nonetheless, thermophilic biomass may confront high free ammonia levels when an endurance period, in which TAN concentration is slowly increased, is applied. In order to control ammonia concentration in the thermophilic reactor, ammonia side-stream stripping with air was adopted. In the stripping column, the biomass was exposed to high temperature (65°C), high pH (> 9) and oxygen at saturation levels. The effects of these conditions on the anaerobic process were studied by trying different recycling ratios between the digester and the stripping column. It was demonstrated that it is possible to define a recycling ratio that can keep ammonia concentrations in the digester below inhibitory levels without compromising methane production and digester stability. The benefit of connecting a thermophilic digester to a stripping unit when treating N-rich
substrates was also proved, as coupling side-stream stripping allowed to increase 2.5-fold the organic loading rate without affecting the digester performance.

Task 1.3 Assessment of ammonia stripping reactor configurations and operation to minimize aeration and heat

The work performed in this task focused on the optimization of ammonia side stream removal at small scale as sound basis for subsequent design, operation and optimization of the pilot process. Moreover, the development of new process modifications of the stripping process was addressed. A stripping and scrubbing unit was built out of glass elements. The plant was continuously improved and adapted according to the needs of different experimental set ups. Two different ammonia concentration ranges (start up and steady state) were supplied by project partners. To identify optimal operational conditions for those concentrations, different air flow rates were tested. The higher the air flow, the faster and more efficient is the stripping operation. For most of the conducted experiments the stripping temperature was fixed to a level efficiently obtained from the waste heat of a combined heat and power (CHP) plant.

Stripping plants can be operated in either batch or continuous mode. The pilot plant stripping unit was designed as batch set-up. However, continuous operation offers several advantages and was therefore experimented at lab scale. On a direct comparison, batch stripping was more effective. If necessary operational downtime is factored in, continuous operation had a similar or better performance. During stripping manure is strongly aerated and heated for several hours. Both, air and the temperature increase might affect the microbial community in the digestate and possibly the digesters performance as well. A series of bio-methane potential tests were set up to investigate the effects of stripping on the residual biogas production capacity of stripped digestate. As expected, stripping had a negative impact on the biogas production, but it was reversible in approximately two weeks. These results triggered experiments in which air as strip gas was replaced with biogas or CHP off gas. Due to the high CO2 content (40-50%) biogas was not suitable. However, off gas (15% CO2, 5-7% O2) seemed to be a viable alternative to air, especially as the waste heat can also be utilised. It was also demonstrated that substitution of sulphuric acid by nitric acid as scrubbing solution does not affect the recovery efficiency or process performance. The product obtained in such manner, ammonia nitrite (instead of ammonia sulphate), has a higher fertilizer value. This was proven in further experiments addressing impact of recovered products on plant growth.

In the conducted experiments, side stream ammonia stripping proved to be a valuable and effective process for ammonia reduction in an anaerobic digester. The captured ammonia, once bound in acid and crystalized/concentrated, is a valuable base for organic fertilizers production.

Task 1.4 Study of process conditions for ammonium or potassium struvite at different levels of dissolved and suspended organics, while minimizing energy input of energy and chemicals

A crystallizer was designed specifically to recover nutrients (as fertilizers) from the digested manure after solid/liquid (S/L) separation, able to work indistinctly in batch or continuous mode. Optimal conditions (pH, Mg:P ratio, temperature) for struvite and K-struvite recovery were first determined with simulated manure in batch tests, and then validated in the crystallizer with real manure influent. Struvite was recovered at pH 8.5 and room temperature, while K-struvite was recovered at pH 9.5-10.5 and 37 °C, in both cases the Mg:P ratio was set at 1.5-2. Good P recovery efficiencies (65-98%) were obtained either before or after the biological nitrogen removal step. The crystallizer was able to generate a recovered product with good fertilizing properties treating digested manure. In presence of high ammonium concentration (before BNR),
mainly pure struvite crystals were obtained (N-P fertilizer), while at low ammonium concentration (10-100 mg N-NH4+) up to 5% of K was recovered (as K-struvite) resulting in a N-P-K fertilizer. The ammonium threshold for K-struvite recovery was determined, allowing the possibility for recovering either pure struvite, K-struvite or a mixture. Different influents were tested to investigate the effect of suspended solids (SS) on precipitation and recovery performance. SS solids after S/L separation did not affect the P recovery efficiency (neither crystallization nor settling of the precipitate), since they mainly consisted in colloidal SS that just passed through the system. At low SS (treating digested manure permeate from membrane separation) very pure struvite crystals were obtained. At higher SS (up to 2 g TSS L-1 were tested; digested manure centrate), suspended solids acted as nuclei during crystal nucleation, but then only crystal growth occurred in time. This added some volatile fraction into the final product, which decreased in time as crystals grew. This limited organic content in the fertilizer could be interesting as soil amendment. Also, the up-flow velocity in the riser (controlled by aeration) was used to control the final particle size of the product recovered.

Figure 1. Overview of the struvite recovery study at laboratory level.

Task 1.5 Optimization of biological nitrogen removal process configuration and operation/control aiming at minimal energy use and minimal greenhouse gases emission

The processes targeted in the laboratory approach for shortcut biological nitrogen removal were partial nitritation anammox (PNA) and nitritation/denitritation (Nit/DNit). Manure showed an inhibitory effect on anammox cultures. Different PNA biomasses and manure digestate streams and concentrations were used, aiming at identifying the optimal condition for biological nitrogen removal through PNA. Concentrations higher than 25% of manure digestate did not enable satisfactory microbial growth and strongly inhibited PNA cultures. However, both salinity and heavy metal (excluding Fe3+) at manure digestate stream concentration did not show inhibition on PNA. A lab-scale PNA reactor was set-up using the liquid fraction of pig manure digestate from the MEM pilot installation, after ultrafiltration. Results were encouraging, with a total nitrogen removal over 75%. However, it needs to be stressed that anammox cultures have slow growth rates and thus long period for biomass adjustment, first with synthetic medium and then with real manure digestate, are needed. At the same time, the Nit/DNit process was tested in lab for residual nitrogen removal from the manure digestate of the ManureEcoMine pilot. Complete nitrogen removal was achieved with this process, which resulted less sensitive to manure digestate than PNA. Nit/DNit is known to produce nitrous oxide (N2O), which is a greenhouse gas 300-fold more potent than carbon dioxide (CO2). Strategies implemented at lab level and tested over a period of 3 months proved the feasibility of reducing N2O emission up to 99%, thus suppressing the environmental threat represented by the Nit/DNit process.

Task 1.6 Trace contaminant support for individual technological optimization at lab-scale

In order to assess the contaminants migration throughout the ManureEcoMine technology scheme, lab tests were carried out adding a mix of trace contaminants to the feeding. Antibiotics concentration was sharply reduced during thermophilic anaerobic digestion and ammonia stripping, hence suggesting that temperature affects the degradation of antibiotics. The effluent of the struvite precipitation process showed extremely low antibiotics concentration (around 3-4% of the initial load), while the final water produced by biological nitrogen removal as partial nitritation anammox had less than 25% antibiotics present in the influent water. Regarding heavy metal contamination, anaerobic digestion and ammonia stripping had
almost no effect on metals concentration. On the contrary, solid/liquid separation represented the main process for heavy metals reduction, with over 80% of contaminants precipitated in the solid fraction. The effluents of both struvite precipitation and biological nitrogen removal had less than 4% of the initial heavy metals. The laboratory tests showed that it is possible to concentrate almost all contaminants in the solid fraction and leave the liquid phase almost uncontaminated, by fine tuning the solid/liquid separation process. The results obtained at lab level were used as base information for the contamination assessment of the ManureEcoMine pilot installation.

WP2 - Pilot plant design/engineering/construction - Pilot plant installation/ disinfection/ moving
Tasks 2.2 - 2.6 Development of a technological design; detail engineering of the technological design; pilot construction; commissioning with technical start-up
A pilot plant study represents an intermediate step in the evaluation of the feasibility of successful lab experiment as a preliminary step prior to building a full-scale installation. It is therefore a critical tool in the process development scheme. Colsen was responsible for the design, engineering and construction of the multifunctional pilot (Figure 2). The pilot itself was considered the core of the project and allowed flexible and efficient manure nutrient recovery.
A modular set-up was created, which facilitated a high degree of flexibility and transportability. The following modules were constructed:
• Thermophilic digester: This module converts the organic fraction (i.e. 85% manure) to biogas.
• Container 1:
  o Feeding system for the thermophilic digester to supply the digester with manure and vegetable waste.
  o Solid-liquid separation:
    • Centrifuge/filter press
    • Ultrafiltration ceramic membranes
  • Container 2:
    o AMFER: ammonia stripping and scrubbing. Efficient recovery of ammonia from the digested manure and recovery as nitrogen-fertilizer.
  • Container 3:
    o ANPHOS: struvite crystallization reactor. Allowing to effectively remove and recover the remaining phosphate after solid-liquid separation.
    o Biological nitrogen removal: aiming at the conversion of all remaining nitrogen.
The ManureEcoMine pilot installation was built in sea containers so to facilitate its transport to Spain and back to the Netherlands.

Figure 2. State of the art pilot for nutrient recovery from manure

WP3 - Technological optimization of the pilot plant
Tasks 3.1 - 3.5 Optimization of thermophilic digestion combined with ammonia stripping - NL; optimization of solid-liquid separation after digestion- NL; optimization of nitrogen removal through biological processes - NL; optimization of nitrogen and phosphorus removal through struvite precipitation - NL
The pilot setup was in operation in the Netherlands for 10 months. During that period it was demonstrated that the concept of fertiliser production from manure is feasible. Up to 80% of the nitrogen input could be efficiently recovered in pure form (as a salt) in the AMFER® (ammonia stripper) installation, and an average removal of 45% was obtained. This is a patented Colsen technology, which is especially suitable
for recovering nitrogen from waste streams high in nitrogen concentration, such as manure, with minimal input of heat. Both ammonium sulphate and ammonium nitrate can be produced in this way. Also high volatile fatty acids were indicated to have a significant impact on the nitrogen removal efficiency of the AMFER®.

Apart from the recovery of valuable nitrogen fertilizer, there is an important secondary effect. The thermophilic digestion process benefited greatly from the attached AMFER® unit, as a detoxification step for the digester. The use of the AMFER® resulted in an increase of the process stability and allowed a larger throughput of feed to the digester and consequently in the production of larger amounts of biogas in comparison to mesophilic digestion. Pathogens and antibiotics were degraded effectively and at higher rates (even up to 100%) than in comparisons to conventional techniques and mesophilic digestion. For the solid-liquid separation centrifuge, filter press and ceramic ultrafiltration membranes were tested. High ratios of phosphorus and organic matter removal were observed, without the need to add polymer in the centrifuge. The use of ceramic membranes itself can be considered as a breakthrough during processing of digested manure. It was demonstrated that all remaining suspended solids were effectively removed. The cleaning of the membranes was quite intensive however and the flow could not be fully recovered with conventional cleaning agents. The retention of high amounts of phosphorus in the solid fraction during the separations processes led to a reduce recovery of this nutrient in the ANPHOS® unit, which is specially suitable to recover struvite, a high grade phosphorus-fertiliser. The ultrafiltration unit was by-passed, recovery of struvite was not possible, since the high solids content of the influent seemed to prevent recovery. The struvite recovery unit requires the use of a magnesium source for struvite formation. Three different magnesium sources were successfully tested, hence demonstrating the robustness of the process.

During the subsequent Spanish testing scenario, the total phosphorus recovery was further tested using other manure sources and with a different process set-up.

Figure 3 gives an overview of all process units and their correlations.

Figure 3. Process flow of the pilot operations in the Netherlands and demonstration of nutrient recovery potential

Task 3.2 Optimization of mesophilic digestion - ES

The mesophilic anaerobic digester was inoculated in Vic (Spain) in December 2015, with three different inoculums (treating WWTP sludge, cow and pig manure respectively) to increase biodiversity. The digester, operated at 37ºC, treating a mixture of cow manure, pig manure and segregates, a residue from the sugar industry over a period of 10 months.

The optimisation phase consisted in identifying the optimal substrate and co-substrates proportions, leading to an optimised balance between biogas production and nutrients recovery. The optimised feed mixture was finally identified as 52% cow manure, 43% pig manure and 5% segregates. Further optimisation was done along the operation, by increasing the organic loading rate (OLR) throughout time. The operation strategy consisted on a minimum hydraulic residence time (HRT) of 20 days and a target OLR of 3-4 g COD L-1 d-1. During the start-up, the feeding composition was maintained constant and the OLR was progressively increased by lowering the HRT. The digester was monitored in terms of biogas production, volatile fatty acids (VFAs), temperature, pH, amongst others, and subsequent weekly increments of the organic load were done according to the digester stability, leading to increased biogas production rates. The target OLR (3 g COD L-1 d-1) was achieved after 76 days of operation. The slow
but stepwise start-up of the mesophilic anaerobic digester ensured a long term stable reactor without volatile fatty acids increase or free ammonia inhibition.

Task 3.3 Optimization of solid-liquid separation after digestion - ES
The optimization consisted on recovering a higher fraction of P in the digestate liquid fraction, since high P losses during separation were observed in the NL (80% retained in the solid fraction). The main objective of the task was to test whether a higher quantity of P could be recovered in the centrate by previous acidification of the digestate. Results indicated that without acidification, the P in the centrate was only 25% of the influent concentration. However, when the digestate pH was decrease to 6, almost all P (98% of the influent concentration) passed in the centrate. This was a huge improvement in P recovery and the potential struvite production. Additionally, optimisation of the organic fraction removal during centrifugation treating not acidified digestate was done, by changing the centrifuge operating parameters. The ultrafiltration unit was also optimised. Commercial cleaning agents were tested leading to an increase by 68% in permeability compared to regular acid-base cleaning agents. Additionally, the acid cleaning times and concentration of acid agents were optimised until complete cleaning was achieved, reducing membrane clogging issues. Ultrafiltration of partially acidified and not acidified centrate took place, in both cases most of the P (about 80%) was retained in the retentate, demonstrating that complete acidification (pH 4.5) of the digestate was needed to maximise P release in the final liquid effluent, therefore a second acidification step of the centrate was tested, showing that 94% P the UF incoming concentration can be recovered.

Task 3.4 Optimization of nitrogen removal through biological processes - ES
The BNR reactor was inoculated in March 2016 with an anammox biomass to be operated as a partial nitritation-anammox (PNA) system. After testing the unit during more than 80 days, biomass was unable to process the synthetic medium. Therefore, the system was finally switched to nitritation-denitritation (Nit/DNit) reactor. The BNR was re-inoculated in July 2016 with nitrifying-denitrifying biomass from a real scale reactor. Initially, only pig manure was fed and then it was progressively changed into a mixture of 50% pig manure and 50% pilot struvite effluent from centrate. Temperature was controlled and pH was adjusted by addition of sulphuric acid. The BNR operated below a nitrogen loading rate of 0.13 g N L-1 d-1, however stability was not achieved since ammonium accumulated in the reactor due to the poor oxygen mass transfer. To prevent free ammonia inhibition, the feeding was intermittently supplied to the BNR unit. Significant changes were done pursuing a better oxygen transfer, including mixing improvement by the installation of a higher frequency variator. In addition, the diffusion in the reactor was improved by the installation of an aquarium diffuser and finally the aeration capacity was increased to suitable levels. In spite of all efforts, it was not possible to maintain nitrogen loading rate stability. However, it was possible to operate the BNR via nitrite leading to lower oxygen and therefore lower energy demand than a conventional nitrification-denitrification system. Overall removal efficiencies were about 65% removal of nitrogen as ammonium, however lab scale tests proved that much higher removal efficiencies can be achieved under more controlled conditions.

Task 3.5 Optimization of nitrogen and phosphorus removal through struvite precipitation - ES
The struvite unit was deeply optimised. Different wastewaters were tested (centrate vs. permeate) under different pH conditions (acidic vs. alkaline) and different hydraulic residence time (2 h, 3.5 h and 4.5 h), using magnesium hydroxide as Mg source.
During the operation in Spain, the mesophilic digester received around 500-700 mg L⁻¹ of P but only 4-7% of the P was able to enter the struvite reactor because of the high P retention in the solid fraction during the solid/liquid separation steps. For this reason, optimisation of the P recovery was crucial for the P recovery in the struvite unit. Higher P concentrations were achieved with complete acidification of the digestate prior to the centrifuge, with almost all P able to reach the struvite unit. pH control to 8.5 was essential for struvite precipitation from acidified permeate to prevent from P accumulation in the reactor. With pH control, removal efficiencies higher than 90% are obtained through struvite production from acidified permeate. Struvite precipitation from digestate centrate was tested. The idea was to investigate more flexible ways of recovering P without using the membranes, which may not be affordable in some conditions. Struvite production from centrate was obtained, however the maximum P removal efficiency was 60%, thus lower than using manure digestate after membrane ultrafiltration.

**WP4 - Demonstrative operation of the pilot plant**

**Task 4.1 Steady-state operation of the pilot at Alphen, the Netherlands**

The pilot plant in the Netherlands reached the steady-state condition during the last months of operation. The ammonia stripping and scrubbing unit was successfully connected to the thermophilic anaerobic digester as side process, with digestate being recirculated over the stripping unit. No negative effects on biogas production nor digester performance were noticed during the coupling of anaerobic digester and ammonia stripping units. On the contrary, positive results were recorded in the digester performance as increased biogas production, since digestion inhibition of free ammonia was greatly reduced by the activity of the stripping unit. The organic loading rate of the thermophilic digester was around 4 kg COD m⁻³ d⁻¹ and a digestate recirculation ratio of 130% digestate/total feed was obtained.

During solid/liquid separation through decanter centrifuge, P retention in the solid fraction was over 80%. The overall separation (centrifuge and ultrafiltration membranes) resulted in a P concentration in the final permeate liquid stream of about 7% of the P present in the initial feed of the ManureEcoMine pilot installation.

Struvite crystallisation using the liquid fraction of manure digestate was not significant, while struvite was obtained using the ultrafiltrated (after membrane filtration) fraction as influent of the crystalliser unit.

Struvite allowed the recovery of over 90% of the P load of the unit influent, however the great solid retention during the solid/liquid separation led to an overall recovery of about 6% of the P present in the feed substrates (figure 4).

Due to time restrictions, biological nitrogen removal was not operated during the operation in the Netherlands.

**Figure 4. Destiny of organic dry matter (ODM), total nitrogen (TN) and total phosphorus (TP) during the ManureEcoMine process implemented in the Netherlands.**

**Task 4.2 Steady-state operation of the pilot in Raurell farm from Spain**

Two steady state periods were reached during the operation in Spain: (1) transforming the digestate into permeate with an acidification step to maximize P recovery; (2) transforming digestate into centrate without the acidification step. Both schemes included struvite precipitation and biological N removal. During these periods, optimal settings for each unit, identified along the optimization phase, were applied. The overall operation scheme implemented during the activity in Spain is shown in figure 5.

The anaerobic digestion was operated in steady state for 181 days, with an optimum feed mixture of 52%
cow manure, 43% pig manure and 5% segregates. The estimated average OLR was 3.3 g COD L-1 d-1 and HRT of 23 days, with an average biogas production of 0.9 m3 m-3 d-1. It was demonstrated that no free ammonia inhibition occurred, demonstrating that stripping of ammonium was not needed in this scenario.

At long term, complete acidification (pH 4.5) of the digestate was not easily achieved at pilot scale since acidification was not part of the initial pilot design. Therefore, a two-step acidification process was done, acidifying not only the digestate but also the centrate. With partial acidified digestate, the P recovery improved from 20 to 70% in the digestate liquid fraction but high retentions occurred in the membranes. With complete acidified centrate, the P recovery in the membranes improved from 18 to 86%.

The struvite unit was operated under two different optimized conditions: (1) generating struvite from centrate without pH control and (2) generating struvite from acidified permeate with pH control. Struvite was produced from both centrate and acidified permeate, however lower productions were achieved with centrate due to the higher suspended solids content, normally retained by the membranes. The obtained struvite products were analysed through X-ray diffraction demonstrating that struvite was the main crystal in both scenarios, however with centrate other amorphous products co-precipitated, while with permeate only crystalline products were found, indicating that the higher presence of ions caused interferences in the precipitation of struvite.

Finally, the BNR was operated as a nitritation - denitritation system, however due to design limitations, it was not possible to achieve the steady state operation of this unit. Considering lab results, it is expected that BNR operated as Nitritation - Denitritation system would be efficient at real scale in degrading the N levels present in the ManureEcoMine effluent, with complete ammonium removal efficiencies and improved control system for minimising NOx gases emissions.

In overall, the pilot operation in Spain demonstrated that it is possible to transform manure into “green energy” maximising the nutrients recovery into valuable fertilisers with positive effects on plant growth and generating irrigation water.

Figure 5. Pilot operation strategy implemented in Spain.

Task 4.3 Demonstration of controllable trace contaminant levels

Residues and trace contaminants analyses were conducted to evaluate the migration of contaminants from the raw manure and co-substrates to final products such as digestate, struvite, ammonium sulphate and irrigation water to the soil and green plants produced within the ManureEcoMine project. The streams generated at the pilot plant in the Netherlands and Spain allowed to monitor the complete ManureEcoMine operation at pilot level and provided relevant information for the selection of the operation schemes. Comparing the two ManureEcoMine scenarios (NL and ES), mycotoxins resulted of minor relevance for contamination as their concentration was very low and they were fully degraded along the process. Pesticides and disinfectants were also in low concentration, although not completely degraded during the process, and no priority substances listed in the Stockholm Convention were found in influent streams and products. Heavy metals were in high concentration and little degraded along the ManureEcoMine process, however they accumulated in the solid fraction of the solid/liquid separation process, thus ensuring highly reduced load in the products and final water. Similarly, antibiotics in the influent streams were in relatively high concentration, however most of them were degraded during the thermophilic anaerobic digestion. The struvite produced as well as the final irrigation water had a heavy metals content of about 4% of the initial input. Finally, the solid fraction was the main output carrier of trace contaminants, with the
solid/liquid separation efficiency having a relevant impact on contaminants content in the final irrigation water.

**Task 4.5 Individual and overall performance evaluation of the processes and optimal technological scheme establishment**

In order to establish a final optimal technological scheme, local/customer requirements, maximum energy production, low energy consumption, maximum nutrient recovery needs and achievable N/P recovery efficiencies were considered. During the pilot plant optimization, technologies like acidification were implemented in order to provide an optimal scheme of maximized nutrients recovery. In the nitritation-denitrification process, reduced GHG emissions can be achieved by modulating aeration and feeding cycles during the operation. In addition, the ES configuration of the pilot plant included an additional operative configuration, in which the UF step was eliminated and struvite production from the centrate was obtained. This solution was tested to provide farmers and manure operators with the possibility of reducing the process costs and complexity.

Additionally, a study based on 21 different technological schemes was carried out and the schemes compared on technological and economic points of view, in order to provide a tool for the farmers and manure processing companies for evaluating possible options for manure treatment. Different optimal configurations can be selected based on the local requirements for manure disposal and soil nutrients burden. It is considered that a combined scenario (thermophilic anaerobic co-digestion coupled with ammonia stripping, acidification, decanter centrifuge and membrane UF, struvite production and biological nitrogen removal by Nitritation/Denitritation) would provide an optimal process from a technological perspective. This scheme would allow working at high organic loading rates therefore maximising energy and heat production (thermophilic anaerobic co-digestion) and nutrients (N, P) recovery. However, specific assessments should be carried out in order to identify whether an optimal technological process corresponds to an economically affordable scenario for farmers and manure processing companies. With this respect, different economic levels were identified, analyzing in which conditions the single and combined ManureEcoMine technologies become economically viable in specific areas, within their case study.

**WP5 Green fertiliser blending**

**Task 5.1 Determination of the nutritional quality of various fertilizer formulations coming from the first lab tests and safety of the blended products**

Proper nutrition is essential for satisfactory crop growth and production. The determination of the nutritional quality of various blends and the efficient application of the correct types and amounts of fertilizers for the supply of the nutrients are important parts for achieving profitable yields. The struvite sample received from the first lab tests was chemically analysed and used in first preliminary plant trials. The chemical composition of the growing medium blended with struvite was comparable to a growing medium with organic fertilizer, however significant increases in phosphorous (7-fold higher) and magnesium (1.4-fold higher) contents were detected compared to the treatment with an organic fertilizer with the same total nitrogen level as a reference. The company Greenyard Horticulture Belgium received several samples from the cooperating partners and plant tests with Lepidium and Ocimum basilicum were performed. However, these first preliminary tests with struvite from a real waste water treatment plant showed decreased seed emergence and fresh weight with Ocimum basilicum due to possible toxicity effects. In addition, phytotoxicity tests were performed with Lepidium. In this test, the germination
percentage of the seeds and the fresh weight of Lepidium of the different mixes were determined. For the standard, a germination percentage of 80% is obligatory and for growing media in combination with an organic fertilizer a germination percentage of 70% is needed. This test was performed with ammonium struvite and ammonium sulphate provided by Colsen and collected from the potato production company Lamb Weston, which was visited in the framework of the ManureEcoMine project. The germination was not negatively affected by the addition of ammonium sulphate and ammonium struvite except for the highest addition of ammonium sulphate (50 mL L⁻¹ growing medium), where no seeds emerged. The seed emergence was always higher than the requested 80%. However, the fresh weight was negatively influenced by the addition of ammonium sulphate and struvite.

Task 5.2 Fertiliser and growing media blending in order to meet the final demand of a cash crop and an ornamental plant on physical and nutritional level

For the experiment with the ornamental plant Viola cornuta, 15 fertilizing blends were prepared using the different recovered nutrients from the ManureEcoMine processing activities. Two controls, a combination without fertilizer and a combination with a slow release fertilizer (Osmocote/Everiss) were used. Triple super phosphate (TSP) and potassium sulphate (K₂SO₄) were used in the fertilizer blend in order to compensate potential chemical deficiencies. The blends had a N-P-K ratio of 1–0.26-0.61 and the nutrient concentration was 900 g N m⁻³, 235 g P m⁻³ and 548 g K m⁻³. From the results obtained, it can be concluded that the different recovered nutrients can be used as potential fertilizers. The pH, electrical conductivity, nitrogen, phosphorous and potassium content were influenced by the fertilizer blend used. Indeed very low or very high values of the chemical characteristics can endanger the growth of the plant. The use of ammonium sulphate impacted the electrical conductivity, which increased up to 2,000 µS cm⁻¹. This caused osmotic stress, resulting in a decreased water uptake and possible chemical burning of the roots. In the worst case, plants can even die. It seems that the growing media in combination with ammonium struvite, ammonium nitrate and potassium sulphate as fertilizers and in combination with potassium struvite, ammonium struvite, ammonium sulphate and potassium sulphate had the best chemical characteristics for the growth of Viola cornuta.

Task 5.3 Fertiliser and soil enhancer blending in order to meet the final demand of outdoor or field crops on physical and nutritional level to evaluate the recovered fertilizers in WP6

More than 23 different recovered nutrients were tested for their chemical composition and purity with X-ray diffraction. From the results obtained it can be concluded that the different recovered nutrients as potential fertilizers can be blended into growing media. Germination was negatively impacted, as observed also at the test performed at FZJ, however, fresh weight was increased when the recovered nutrients from the pilot plant were used. This was also in line with the tests made by FZJ. From the results of the X-ray diffraction analysis, we could conclude that the struvite was not pure, hence these results need to be interpreted with caution. However, struvite with 80-90% purity can be produced from wastewater treatment plants. Tests performed with single recovered nutrients as carried out by FZJ and in pre-tests at Greenyard Horticulture Belgium showed quite good plant performance, however aiming to meet the demand of the plant, blending the different recovered nutrients with other single fertilizers, such as triple super phosphate, potassium sulphate, potassium nitrate were required in order to have the right amount and correct ratio of nutrients in the mix. Even though single product tests showed good plant growth performance, industrial application and use of these recovered nutrients to produce blends is limited until now. This is due to the fact that the chemical composition of the different recovered nutrients showed high
variability, taken into account that nutrients were recovered from a pilot plant and still had a high variability, with struvite crystals not 100% pure. However, it has to be stressed that the struvite samples analysed were produced under different operation parameters at the pilot plant (in Spain and in the Netherlands), thus fluctuations were also due to varied operating settings. Industrial application demands high reproducibility and, as seen from the chemical analyses, this is not guaranteed or given, yet. Hence, from an industrial point of view, there is an increased demand for quality control in comparison to the standard used fertilizer, resulting in an increased work load and consequently higher costs.

WP6 Effect of green fertiliser blend on plant and soil
Task 6.1 Evaluating the effects of recovered nutrients of various fertiliser formulation on plant performance and biomass yield
Several experiments were done to evaluate the effect of recovered nutrients on plant performance. These studies included not only the observation of the effects in aboveground biomass, but also the effect in root architecture and nutrient cycling. The effect of recovered products were tested in different plant species like lupine, maize, tomato and viola. In one of the experiments, it was compared the effects of the struvite and a common rock phosphate-based P fertilizer (triple super phosphate TSP), on crop yield, P dynamics (bioavailability and recovery) and changes in root architecture, as well as differences between two plant species (maize and narrow-leafed lupine) with different nutrient acquisition strategies and with different N sources (ammonium or nitrate). Results suggested that struvite can be used as a sustainable and effective alternative for TSP on acidic substrates. Both treatments increased significantly the shoot dry weight of both crops compared to no-P control, being the struvite-ammonium treatment significantly higher than the TSP-ammonium one in maize plants.

Figure 5. Lupine and maize dry weight (DW) of leaves and stems as affected by phosphorus (P) sources (TSP triple super phosphate and struvite) and nitrogen sources applied (NH4+ ammonium, NO3 nitrate).

No significant differences were observed in the root total length and root diameters between struvite and TSP, however both treatments developed more and smaller roots (related to higher P uptake) than the no-P application.

Task 6.2 Evaluation of plant nutrient availability at different soil pH
The overall objective of this study was to observe the differences in the plant performance in varying growing media pH values and to identify the mechanisms that account for variation in the efficiency of P acquisition and use from different P sources.
There were significant differences in the lupine growth performance at three different pH. The highest growth was observed at neutral conditions. Variations of pH showed a decrease in the plant growth, more pronounced if the pH value increased over 7. Figure 6 shows the variation in the plant performance between pH, with the struvite addition, and comparison between the struvite treatment, commercial available P (K2PO4 = KP) and no-P addition at neutral pH. It can be noticed that struvite treatment had a significant positive effect in plant growth, in comparison to no-P addition, only at acidic and neutral pH.

Figure 6. Differences between pH with struvite treatment (left); at pH neutral, differences between P treatments (right)
Task 6.3 Determination of nutrient leaching and effects on soil health and rhizosphere (micro)biology

The main objective of this study was to determine the effect of recovered nutrients like struvite, on the functionality and composition of the microbial community associated with the bulk zone and the rhizosphere over time, and determine which of both factors (plant species or fertiliser) had a bigger effect on the bacterial community. The microbial nutrient turnover is an important factor that affects availability for soil applied fertilisers, and the comparison between recovered and commercial fertilisers in the bacterial community was a necessary step to increase the value and stability of those recovered products. The results showed that the overall microbial communities of lupine and tomato were independent and not impacted by the use of the fertiliser. Major differences were observed in the relative abundance of bacteria in the rhizosphere in comparison to the bulk zone. Plant effect is determinant on the differences in the relative abundances of the communities in the different growing media microbiomes.

Low nutrient use efficiency by plants and consequently higher risk of nutrient leaching results in eutrophication of water bodies. For optimal plant growth, with low losses to the environment, the timing, condition and quantity of fertilisers applied are fundamental, because the nutrient demand of the plant may not be concomitant with the nutrient release from the fertiliser. Nutrient leaching is an important dissipation factor for soil applied fertiliser formulations. To evaluate the stability and leaching character of the different fertiliser blends in relation to plant nutrient uptake, growth and performance, nutrient leaching was evaluated employing column tests. Results confirmed the hypothesis that the P from the struvite was more soluble and more prone to leach in acidic conditions than in alkaline growing medium, however there were no differences with mineral fertilizers. In the presence of organic acids, the acidification increased and thus the solubility of P from struvite, therefore the use of plants like lupine that can release organic acids and solubilize the struvite only when the P is needed is recommended.

Figure 7. Set up for the leaching experiment. (A): falcon tubes to which sand and struvite were added and subsequently flushed with water or citrate solution (B) shows a close up of the collecting system.

WP7 Economic/environmental sustainability – Safety management for trace contaminants

Task 7.1 - Economic sustainability optimization and demonstration

The evaluation of the total benefit of extracting nutrients from manure products and the identification of those units contributing the most to the overall process feasibility were the main goals of this task. Besides the identification of the costs of producing and selling the N and P recovered products, the nutrient extraction from a waste and its disposal costs were evaluated. The latter depends on regional balance between the amount of nutrients available from manure and the amount of nutrients disposable on agricultural land. Therefore, a spatial-equilibrium model allowed to analyse the market introduction of the ManureEcoMine technologies (also in different combinations) following different scenarios. In such scenarios, decreasing cost levels of configurations using one or more ManureEcoMine units were compared to fixed cost levels for the traditional pig manure processing techniques (separation and separation + nitrification/denitrification of liquid fraction) in a region with intensive animal husbandry such as Flanders.

The first technology to potentially enter the market was a combination of acidification, struvite precipitation and biologic treatment, at a 10% reduction of costs (6.86 €/ton compared to 7.63 €/ton in the baseline scenario). Various technologies based on digestion and N stripping started being competitive from a 25% reduction of cost (15.02 €/ton compared to 18.87 €/ton for AD + stripping), and became the dominant manure processing technologies in all further cost reduction. This market introduction was driven by an
average share of added value generated by nutrient extraction and nutrient denitrification. In the same way, anaerobic digestion and subsequent stripping and scrubbing of digestate started to be competitive at 25% reduction of cost. This market introduction was fuelled by 3 € per ton for nutrient extraction and denitrification.

Task 7.2 - Environmental sustainability optimization and demonstration
This task was mainly focused on the evaluation and demonstration of the sustainable profile of Dutch and Spanish scenarios from an environmental perspective. The Life Cycle Assessment (LCA) methodology was applied for this purpose and the entire life cycle of both systems was analysed. Moreover, additional scenarios were also defined with the aim of evaluating the performance of ManureEcoMine prototypes compared to alternative technologies. The most relevant environmental indicators were considered: climate change, terrestrial acidification, freshwater eutrophication and marine eutrophication as well as toxicity impact categories for the Dutch scenario. The characterisation factors provided by ReCiPe Midpoint (H) and IPCC methods were applied. According to the results, the ManureEcoMine prototype based on anaerobic co-digestion with biogas valorisation in a combined heat and power (CHP) unit and application of recovered nutrients as organic fertilisers was found as the most environmental-friendly option in both Dutch and Spanish scenarios, closely followed by the analogous configuration of anaerobic digestion coupled to a gas burner (instead of CHP unit). In contrast, conventional practices – where digestate is directly applied on agricultural soil – presented the worst profile with the largest impacts on most environmental indicators. Diffuse emissions mainly from both storage (with a critical role in conventional practices) and fertilisation activities showed decisive contributions in all environmental indicators together with energy requirements. However, avoided impacts derived from the valorisation of biogas and recovered nutrients used as organic fertilisers partially offset the environmental burdens of the different scenarios as they were considered environmental credits.

Task 7.3 - Risk analysis and safety management for trace contaminants
Residue and trace contaminant analysis were conducted to evaluate the migration of contaminants from the raw materials (manure and co-substrates) through the intermediate products of the manure up cycling (solid digestate, struvite, ammonium sulphate, irrigation water) to the growing media and green plants. The results on contamination and migration were used as basis for risk management and transferred to a risk management plan. The monitoring of the operation of the pilot plant in the Netherlands and Spain, as well as the additional experiments carried out at lab scale showed that the products ‘struvite’ and ‘irrigation water’ were at a low level as output streams for trace contaminants. Mycotoxines were of minor importance since their concentrations were very low and they were degraded in the ManureEcoMine processes. Similarly, pesticides and disinfectants were also of minor importance although they were not fully degraded in the process but their concentrations were low enough compared to other trace contaminants. No priority substances listed in the Stockholm Convention was found in the streams and in the products. Heavy metals were found in the highest concentrations. Antibiotics, especially tetracyclines, were found in relatively high concentrations, also in the struvite. The trace contaminants were clearly allocated to the input source and the contamination level depended on the type and quality of input materials. The solid fraction generated in the solid/liquid separation process was generally the main output carrier of trace contaminants from the system. Therefore, the solid/liquid separation efficiency impacts the partition and level of remaining contamination in the liquid process streams. The risk management plan considered all these findings and suggested a monitoring scheme on a regular basis including mitigation
WP8 Dissemination
Task 8.1 Dissemination
ManureEcoMine has been advertised through different channels by the entire consortium, often as a result of joint efforts of more partners. The ManureEcoMine project was presented to several international workshops and conferences with poster and oral communication across Europe and beyond. The industrial partners attended national and international fairs on water and wastewater treatment and reuse with booths, and promotional material on the ManureEcoMine project widely distributed. In addition, the project results were published in several scientific journals, 3 of which as open access. Dissemination to industry and wider society was ensured through the publication of the project YouTube video and the organisation of 2 open doors event at the pilot plant installation in the Netherlands and Spain. Articles on local radio, blogs and newspapers, as well as agro-technical articles in specialised press ensured to communicate the project objectives and achievements. Finally, the project goals, results, partners and dissemination events were published in 6 Newsletter issues during the course of the project.

Task 8.2 IP management
In light of the intellectual property aspect of the ManureEcoMine project, the activities related to obtaining a patent for the technology developed under the project scope were focused on defining the newness of (parts of) the ManureEcoMine technology and subsequently by preparing a patent submission request. A preliminary patent search highlighted few patents in the area of sustainable nutrient treatment technologies for manure. In light of the intellectual property aspect relevant patent searches have been conducted to further mine the patentability potential. Since part of the technology is considered common knowledge, selected niches of the technology qualified for patentability. Based on that, an extended patent search was carried out on selected elements of the technology with the purpose of defining the newness of the technology. The industrial project partner Colsen has formulated a draft patent request related to the ManureEcoMine manure processing and nutrients extraction technology.

WP9 Management

The final goal of the management activity was to ensure a smooth progress of the project towards the milestones and final objectives. Daily project management was carried out by the coordinator via email, Skype meetings, personal meetings and phone communications. During the 3-year project course, 32 deliverables were prepared based on the activities performed in the ManureEcoMine, among which 14 are publicly available on the project website www.manureecomine.eu. Every deliverable underwent an internal review by 3 persons of the project consortium. Seven project meetings took place every 6 months, allowing for personal exchange of results and taking decisions on the project operation for the following period. Three external advisers participated to most of the project meetings. The external advisers attended the meetings giving their valuable input and technological expertise, and represented a very important resource to externally assess the work progress within the project.

In order to closely follow the pilot plant activity, teleconferences were carried out every 3 weeks, involving all the partners related to the pilot plant operation and lab optimisation of the project technologies. During the meetings, the results of the pilot installation were presented and the partners discussed the activity to be carried out at the pilot during the subsequent operational period.

Potential Impact:
Manure has always had a crucial role in agriculture thanks to its content in organic matter and nutrients such as nitrogen and phosphorus. Intensive farming was able to support the increase in world’s population, however a massive application of fertilisers (mainly synthetically produced) was needed. The resulting burden of nutrients disposed on soils and crops created the need for alternative solutions for manure processing and disposal in nutrients vulnerable areas and beyond. ManureEcoMine tackled several objectives related to intensive animal husbandry and manure nutrients upcycling as fertilisers, by proposing a strategy which perfectly matches the circular economy approach of the European Union. The overall impact brought by ManureEcoMine would affect intensive animal husbandry areas and beyond, thanks to its flexibility. Moving towards a resource-efficient Europe, the project left significant marks for:

- Reduced pressure on mineral resources, by upcycling nutrients from biological, waste streams
- Less reliance on fossil fuels, directly by implementing the anaerobic digestion technology platform, and indirectly by reducing the use of the Haber-Bosch process for nitrogen-based fertiliser production
- Safety of the nutrients supplied, through a thorough assessment of trace contaminants migrations throughout the entire ManureEcoMine flux
- Reducing costs, by minimising energy requirements with comparison with current manure processing and disposal technologies, and producing biobased fertilisers blends
- Process stability and robustness, by buffering influents variations with little effect on the product generated
- Protecting environmental assets, by reducing the consumption of synthetic fertilisers and the eutrophication threat in nutrient sensitive areas.

Demonstration of new technological possibilities for efficient manure processing. The ManureEcoMine process combined several technologies for treating cow and pig manure. Together with the demonstration of the technological feasibility of the entire process and, thus, its single technology step, the pilot installation designed, built and operated within the ManureEcoMine project enabled the demonstration at pilot level of thermophilic anaerobic digestion combined with ammonia stripping.

New ready-to-go process. Anaerobic digestion is a well-known process. Its yield as energy production may be maximised by operating the system at thermophilic conditions. The instability and potential inhibition of the system generated by a thermophilic conditions can be minimised by coupling the system with an ammonia stripping unit. This combination has been already studied in the laboratory but very few trials have been demonstrated at pilot level or operated at large scale. The positive results obtained in the ManureEcoMine pilot plant demonstrated that coupling thermophilic anaerobic digestion with ammonia stripping ensures stable operation of the digester, with yields that can almost double those of the mesophilic digestion. This represents a great potential for bioenergy production particularly in those areas with green certificates and policy incentives. In addition, a subsequent step of scrubbing of the ammonia stripped air generated ammonium sulphate, which can be introduced in the market as fertiliser. Both aspects would ensure the right technological potential for an effective economic competitiveness. The industrial partner Colsen has developed full scale project proposals and is working in the market for dairy and poultry farmers and investors in green energy and nutrient recovery. Colsen is pushing local governments to invest in those innovative projects with subsidies or guarantees for financing.

Nutrient separation and recovery. The ManureEcoMine project proved the extra value of thermophilic digestion in combination with ammonia stripping. The higher conversion of organic matter resulted in a higher biogas production, but also in a better release of nitrogen and phosphorus, which provides a potential in nutrient recovery. After separation from the liquid and solid fraction of the digestate 80 % of the phosphate is concentrated in the solid fraction. This aspect of the ManureEcoMine process can be useful
for dairy farmers to, e.g., process the own manure and keep all needed nitrogen on the own land, exporting only the P-rich solid fraction. Depending on the subsidy on green energy, the cost price for dairy manure can be reduced to zero euro per ton.

Development of innovative technologies. An important technological aspect of the ManureEcoMine process was the development of the shortcut biological nitrogen removal from the liquid fraction from digestate. Although the tested partial nitrification/anammox process met several technological difficulties, the nitrification/denitrification process was successfully operated at lab scale, where it was demonstrated that lower energy than conventional nitrification/denitrification process was required. Nitritation/denitrification process makes it possible to combine manure digestion with discharge of the liquid fraction with field application/irrigation or even with discharging on municipal sewers. In this way even intensive farmers without own land can treat the own manure in a sustainable and economic way.

European application. Intensive animal husbandry and related restrictions for manure disposal are a concern pooled by several European areas. Although the ManureEcoMine was designed as technology solution for nutrient vulnerable zones in Europe which often coincide with intensive animal husbandry zones, the technology can impact a larger area of application, as big farms and agricultural facility can benefit as well of the ManureEcoMine technological solution and results. Thus, the market areas are wider than initially projected and could reach beyond intensive animal husbandry regions. Moreover, the process was demonstrated in the North (the Netherlands) and South (Spain) of Europe, thus showing that, although each nutrient vulnerable zone has geographically bound pulls for manure processing and disposal, a common solution can be adapted to different requirements.

Environmental benefit assessed through life cycle assessment. Manure processing is an important step for reducing greenhouse gas emissions to the environment. Manure management is estimated to account for 30-50% and 12-41% for total N2O and CH4 agricultural emissions, respectively. Life cycle assessment demonstrated that the ManureEcoMine process balances the emissions by producing bioenergy (electricity), reducing the overall C footprint. An additional positive impact is given by the reduced greenhouse gas (i.e. N2O) emission into the environment during the biological nitrogen removal process. Finally, the reduction of synthetic fertilisers application also greatly contributes to the positive environmental influence of the ManureEcoMine process, especially when compared to other manure processing strategies currently applied in Europe.

Stimulating market uptake. ManureEcoMine solutions were based on the study of farmers’ willingness to pay for certain by products rather than others. From this perspective, ammonium sulphate was produced instead of ammonium nitrate, as the market uptake of ammonium sulphate would be more likely promoted. On the other hands, more innovative solutions such as struvite as P fertiliser were proposed, thus exploring new bio-based alternatives for agronomical applications. Although farmers’ willingness to pay for struvite is hardly assessable, the need of separated P and C in waste streams prior to disposal in some nutrient vulnerable zones of Europe would lead to an opening of new marketable perspectives.

Contaminants safety. Trace contaminants released into the environment through waste, e.g. manure, disposal generate an additional environmental burden for agriculture. The thorough screening of all influent and effluent streams of the process carried out during the project demonstrated that most of contaminants such as pesticides, antibiotics and heavy metals are concentrated in the solid stream. The nutrient products generated in the process were devoid (ammonium sulphate) or had minimal (struvite and final irrigation water) contaminant pollution. This indicates that the great potentiality of nutrients recovery and reuse from manure does not contribute to upcycling contaminants.

Fertilising potential of the recovered products. Struvite and ammonium sulphate are known for their
fertilising properties. The ManureEcoMine project allowed to carry out an in-depth study of the N and P sources for several plants and in different growing media. Struvite represents a valuable P source for fertiliser blending companies, for the production of blends tailor-made on the plant needs. Besides the technological newness of demonstrating manure processing in an efficient and environmental friendly way, one of the main goals of the ManureEcoMine project was to prove a novel solution for easy uptake of P for plants fertilisers. The European Commission has listed P among the critical raw materials, thus generating a pull for its recovery and upcycling, since the losses of P along its cycle are enormous: around 100% of the P eaten food is excreted and only 10% is currently recirculated back to agriculture or aquaculture. Alternative solutions for P recovery may not meet the current market demand, however they will be for sure a need and fall-back plan in the near future, when P rock depletion will become more pressing and costs of its extraction will level the costs for struvite production from manure. Currently, struvite is generated as by-product in municipal wastewater treatment plant. The potential of struvite production from manure is magnified by the larger amount of P present in manure compared to municipal wastewater. The current struvite production from the latter facilitates the market introduction of the product generated from manure and the crystallising technology. Moreover, it was demonstrated that the use of the recovered fertilisers do not consistently modify the microbial community in the soil. Together with the minimal contaminants load in the products and the positive environmental impact of the ManureEcoMine technology, this demonstrated that safety in all phases was prioritised and respected.

Final products with high purity level. As expected, the recovered ammonium sulphate had minimal contaminants inclusion. On the other hand, struvite resulted of purity higher than 90%, when recovered from permeate of manure digestate, thus following the optimal technology process for such purpose. However, organic inclusions in the struvite are positively accepted, since they can increase the products fertilising potential. As currently done for P rock, a refining step should be considered for struvite to effectively enter the market and meet the stable and standardised composition required by the fertiliser blending companies.

Plug and play configuration. The ManureEcoMine technology was designed, built and operated in a modular approach, thus flexibility was ensured to meet the requirements of the farm. The technology core was designed and operated in order to maximise nutrients (nitrogen and phosphorus) recovery. In some conditions nutrients recovery may not be required nor needed, thus the ManureEcoMine process can be easily modified in order to allow maximised energy production though anaerobic digestion, or to reduce nitrogen via biological processes with minimal greenhouse gas emission and reduced operation costs. Similarly, high maintenance steps such as the ultrafiltration unit may be omitted. The module composition of the ManureEcoMine process ensures the possibility to address it towards the needs of the farm, since modules can be arranged in different configurations and some units may be omitted.

Robustness of the process. The ManureEcoMine technology proved to be robust enough to handle manure and co-substrate fluctuations with relatively low impact on the final products and their composition. Thus, substrates variations are easily absorbed by the system and do not result in modifications of, e.g. struvite purity, which may affect the fertiliser market potential. The use of fresh input streams is anyway always preferred, especially for avoiding greenhouse gasses emissions to the atmosphere and reduced digestibility potential during the storage phase.

Potential for additional nutrients streams generation. Products such as K-struvite and digestate solid cake were not investigated at pilot plant. K-struvite production was demonstrated at laboratory level as feasible on streams deprived of ammonium. This matches the modular and plug-and-play approach on which ManureEcoMine is based, since process modification would enable the production of novel products.
Flexibility in the application. The ManureEcoMine technology based on the use of manure as main substrate. However, the process can be adapted to several types of waste streams, ideally with high ammonium. This would result in reaching a wider areas of application and consequently in higher market possibility, with a large impact on different sector of Europe’s waste sectors.

Generating a pull for change through the society at large. Circular economy is the main goal of the current European society and policies, thus a need for a rise in social awareness for the fact that animal-derived products should be more expensive, if their price were to reflect their environmental impact. Potential scenarios include quality labels for sustainably produced meat and/or higher taxes on animal products with a higher environmental footprint. Secondly, and relevant for the circular economy at large: sustainability campaigning should render a consumer mind-set realizing that there are hardly real waste streams, but that side streams can be considered as safe alternatives to mine secondary resources.

Improvements in the state of the art. The ManureEcoMine contributed to advance the state of the art of manure processing, nutrient recovery and assessment of fertilising potential and effectiveness. In particular, the projected impacted the industrial uptake potential of such perspective, demonstrating technological feasibility and flexibility, environmental benefit and alternative P source effectiveness. Effective communication transfer of knowledge. The improvement generated in the start of the art was promptly transferred to target audience. Participation to the conferences and symposia enabled transferring the knowledge to academia and industry, although the latter was mainly reached through articles in agro-technical magazines, participation to wastewater treatment and reuse fairs and the pilot installation open days. The open doors event also enabled to communicate with citizen and the society at large, together with the ManureEcoMine project video (published on YouTube) and interviews published on blogs, radios and local newspapers.

Stimulate market uptake for biobased nutrients. Similarly to the technological market, the nutrients reuse and trade potential is considerable. Considering that European farmers and citizens spend around €15.5 billion per year for synthetic fertiliser, the share of fertilisers replacement with biobased ones is potentially endless. Although nutrients markets are still not completely established and directives often limit the use of organic and biobased fertilisers, new perspectives are expected to be soon implemented in this sector. The support of the European Commission for introducing more biobased fertilisers, often generated by processing by- and waste products is symbolised by the draft of the new fertilisers regulation which acknowledges the possibility to use organic and recycled nutrients such digestate and compost, and opens to different materials such as substitute P resources. This is an important barrier taken, but does not overcome pricing differences.

Main dissemination activities

Project logo and website. The consortium selected out of 6 logo proposals. The final one represents the concept of environment embracing earth, as how natural and environmental sustainable processes can support the world’s growth. Based on the logo format, other dissemination material such as project flyer, factsheet, poster and general presentation were designed and distributed in the first months of activity. In addition, on March 2014 the ManureEcoMine website www.manureecomine.eu was launched. The website worked as main platform to present the project content as well as distributing the main dissemination material produced during the activity, including publicly available deliverables. The website was also used as platform for the advertisement, organisation and reporting of the pilot plant open days, ManureEcoMine video https://www.youtube.com/watch?v=8WXdPCxFpY&t=1s. At the end of the second year of activity, the consortium produced a 8-minute video summarising the project objectives and technologies. The video introduces the audience to the ManureEcoMine world and its players, with
partners describing the own activity within the project. In addition, images of the ManureEcoMine pilot installation and open day are shown, providing the audience with a complete insight of the project technology core. The video is in English, however Spanish and Dutch subtitles are provided.

ManureEcoMine project video (left, displaying a moment of the project meeting in Gent, Belgium) and open day in Calldetenes, Spain (right, a moment of the guided tour).

Pilot installation open day. The ManureEcoMine consortium opened the doors of its pilot installation to the public during 2 events which took place in Axel, the Netherlands (April 2015) and Calldetenes, Barcelona, Spain (May 2016). In both cases, the day was opened with an overview presentation on the project structure, objective and technologies. Several guided tours to the installation followed the introductory part, where the technology partners explained in detail the steps composing the pilot installation and the challenges overcome. Both events welcomed more than 250 visitors from wastewater treatment companies, academia, water and environmental boards, technology providing companies, consultants and several other fields related to waste processing and nutrient reuse.

Open day at the ManureEcoMine pilot plant facilities: Axel, the Netherlands (top) and Calldetenes, Spain (bottom).

Newsletter. Six issues of the project newsletter were published during the project course. Most of them were produced in the second part of the project, as direct result of the outcomes generated by the consortium’s activities. Each issue featured 2 or 3 topics, including the publication of the project video and the invitation and outcome of the two pilot installation open events. Moreover, each project partner was presented along the newsletter issues, with a brief description of the main expertise beyond those enrolled in ManureEcoMine.

Conferences, symposia and fairs. The research partners attended conferences and symposia in Europe and America (Chile and USA). Overall. The project was presented at 27 events (conferences, workshops, symposia), with 27 oral communications and 17 posters. Among the oral communications, 3 were as invited speaker and 2 as key note presentations. The consortium was also awarded with a prize of best poster and best presentation awards. The industrial partners attended 3 fairs on waste and wastewater management, treatment and reuse, 2 local (Vakbeurs Aqua Nederlands and Smagua) and an international one (IFAT 2016), which was attended by 3 industrial partners, with a both dedicated specifically to the ManureEcoMine project.

Publications and articles. The presence of 5 research institutions in the consortium promoted the publication of several scientific research article. Over the entire course of the ManureEcoMine project, 11 manuscript were published on peer-review journals, 33 of them were granted open access aiming at widening the readers. Several other articles are in preparation, mainly as results of joint efforts of different project partners. Besides scientific communication, the technology partners which operated the ManureEcoMine pilot installation published 2 articles in national (Spain and the Netherlands) agro-technical magazines, based on the results and activity carried out during the pilot plant operation.

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