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## I. Projects – Drivers of Innovation

#### Biogas Action - Removing the Non-Technical Barriers to Contribute to Creating Better Frameworks for the Widespread Production of Biogas and Biomethane

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#### INTRODUCTION

The project Biogas Action works on increasing biogas and biomethane production in the European Union. By removing non-technical barriers faced by stakeholders of the sector, the project contributes to evolving better frameworks.

These improvements will contribute to reaching EU 20-20-20 targets on climate change and energy sustainability which comprise

- greenhouse gas emissions 20% (or even 30%, depending on conditions) lower than 1990,
- 20% of energy from renewables,
- 20% increase in energy efficiency.

Biogas Action aims at promoting the production of sustainable biogas throughout the EU, especially by exchanging best practices, creating new business models and increasing investments in biogas production. The project is based on deep cooperation between policies on European, national and regional levels, and on their implementation in various EU regions.

#### FIRST PROJECT RESULTS

#### Download of Results: <a href="http://biogasaction.eu/downloads/">http://biogasaction.eu/downloads/</a>

Close collaboration between partners has led to the publication of a brochure on Best Regional-Integrated Biogas Plants. This brochure gives a comprehensive overview of ten biogas plants located in the partnering countries; each showcasing a model of success that can be adapted and applied in other European regions.

The brochure offers a detailed analysis of the current situation and the environment in which the project was drawn up. It also explains the driving forces behind the conception and the construction of the plants. It explains the faced obstacles, along with the involvement of the municipality and the residents of the area around each biogas plant. The financial conditions for each plant are described, including detailed analyses of local support tools and financial subsidies. The technical data are characterised in detail, listing types and amounts of feedstocks and products of the biogas process (energy, heat, renewable gas, digestate as organic fertiliser). This brochure describes biogas plants that are not only outstanding but also offer a model of success with a good replication potential in other European regions.

The development of a new industry based on farm waste in Wales along with the starting of stakeholder forums in Latvia are other examples for the partners' dedicated efforts. Specialised

guides were published in France and an online biogas platform has been established in Croatia. Extensive lobbying activities in the Netherlands as well as meetings with municipalities and national associations in Denmark have already led to regional developments. The project network has achieved cross-country activities such as the arrangement of internship experience exchanges, organisation of specialised workshops and seminars.

#### **FUTURE ACTIONS**

On the Biogas Action website, a biogas toolbox will be available which allows stakeholders to search for tools regarding their special needs. This toolbox compiles useful tools for developing biogas projects on regional, national and European level. It is a compilation of 22 European projects and 62 national tools for regional biogas deployment, providing a European-wide analysis and proactive guidelines for development of the European biogas sector at a regional level.

The project partners will develop specific biogas/biomethane intervention strategies with specific activities and measures, focusing on the removal of non-technical barriers. These measures will include institutional building of national and regional platforms, improvement of dialog and interaction between stakeholders, optimisation of business models and project financing, increase of investments in biogas/biomethane production in the EU and the exploitation of biogas potential through project generation and concrete assistance to farmers and entrepreneurs.

The project will establish systematic knowledge sharing to allow for replication on European level.

#### IMPACTS OF THE BIOGAS ACTION PROJECT

Biogas Action will contribute both to the increase of the share of sustainable bioenergy and to a reduction in the transaction costs for project developers and the authorities. The project could also actively contribute to the development of better policies, financial frameworks and market support at national and regional levels.

#### **PROJECT PARTNERS**

- Cornelissen Consulting Services CCS <u>http://www.cocos.nl</u>
- Rhônalpénergie-Environnement RAEE <u>www.raee.org</u>
- Danish Technology Centre for Biogas DFFB <u>www.dffb.dk/</u>
- European Biogas Association EBA <u>www.european-biogas.eu</u>
- EC Network ECNet <u>www.ecnetwork.dk/en</u>
- Energy Agency for Southeast Sweden ESS <u>www.energikontorsydost.se/biogassydost</u>
- AILE <u>www.aile.asso.fr</u>
- Severn Wye Energy Agency SWEA <u>www.severnwye.org.uk</u>
- Energy Institute Hrvoje Pozar EIHP <u>www.eihp.hr</u>
- Czech Biogas Association <u>www.czba.cz/</u>
- Ekodoma <u>www.ekodoma.lv</u>
- Fedarene <u>www.fedarene.org/</u>

 The International Biogas and Bioenergy Centre of Competence – IBBK www.the.international.biogas.center/

**PROJECT COUNTRY REGIONS**: Rhône-Alpes and Western part of France, Wales and UK, Croatia, Czech Republic, the Netherlands, Denmark, Latvia and South-East Sweden.

**PROJECT DETAILS**: Biogas Action has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 691755. The project started in January 2016 and will run until December 2018.

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#### TransBio: Transition to a More Cost-Effective Biogas Sector in Flanders

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#### SUMMARY

The biogas sector grew steadily the past ten years in Flanders. Today, the sector provides green electricity to approximately 120.000 families a year. Furthermore the 40 large-scale installations in Flanders process ca. 2 million tons of organic waste per year. Nevertheless, the sector is characterised by a high investment and operating cost. The TransBio project aims to identify realistic implementations with a high potential to evolve to a more cost-effective biogas sector in Flanders. Field experiments were conducted to examine the effectiveness of digestate as a fertilizer to facilitate the application of digestate as an alternative for fossil based fertilizers. A model was generated to optimize profits from variable electricity production and the potential added value from the implementation of biomethane production in Flanders.

#### INTRODUCTION

The biogas sector in Flanders represents an annual revenue of approximately 50 million euro, plus yearly investments to a value of 10 million euro over the past 10 years. The sector, including its suppliers, employs somewhere between 1.000 and 2.000 people. The biogas is typically valorised in a combined heat and power installation, providing green electricity to approximately 120.000 families, not including a surplus of green heat for an equivalent of 20.000 families. Every year, the anaerobic digestion plants in Flanders account for about 625.000 t CO<sub>2 eq.</sub> avoided emissions. Furthermore, the 40 large-scale installations in Flanders process about 2 million tons of organic waste and a surplus of 1,9 million kg N and 1,8 million kg P from manure. The processed end product is a valuable organic soil fertilizer.

The appreciation for this key technology is increasing across Europe, because of the unique combination of waste management and green energy production (Stambasky, et al., 2016). Furthermore, biogas serves as a better energy basis compared to weather dependent renewable energy resources due to a more constant and predictable energy production (Henning, Krautkremer, Hartmann, & Wachendorf, 2014).

Despite its high added value, the biogas sector is confronted with a high investment and operating cost. The sector is therefore dependent on financial support for renewable energy technologies (Stambasky, et al., 2016). Clearly all parties involved, such as governments and energy partners, want to reduce the level of support to a minimum, while biogas producers themselves strive for more robust business models and decreased support dependence. The TransBio project aims to identify realistic implementations with a high potential in order to evolve to a more cost-effective biogas sector in Flanders.

#### MATERIALS AND METHODS

A number of scenarios were identified that could induce a significant cost reduction or revenue increase. A scenario analysis was used to determine the most meaningful transitions where potential administrative and legal issues were identified and tackled. Then, the selected scenarios were further investigated for effective market introduction through training, demonstrations, symposia and general platform activities (including dedicated working groups).

The categories for scenario analysis, could be divided as follows:

- reduced costs for raw materials through the development of supply chains of current unexploited biomass (grass verge, roadside and nature management cuttings, organic waste, crop residues, alternative crops);
- increased revenues from electricity produced by a more intelligent approach to intra-day and day-ahead variation in energy prices and the use of biogas plants as "net balancing units" that could operate as a buffer for more weather dependent and inconsistent forms of energy (such as wind and solar energy);
- market diversification by upgrading biogas to biomethane and the introduction to the trading market as a green fuel;
- recovery and reprocessing of mineral constituents to high quality mineral fertilizers (N/P/K) which could act as fossil based fertilizer substitutes.

#### RESULTS

The project started in 2015 over the course of four years, consequently only preliminary results were obtained at this moment. In a first phase, we executed a technical assessment of the relevant subjects and technologies. These assessments included a market overview, with a description of current practices, followed by the identification of possible bottlenecks (both technical and legislative), and an estimation of the overall potential. In a second phase, we performed an economic assessment based on the technical descriptions.

To assess the potential economic added value of flexible energy production, Ghent University developed a model which simulates the added revenues from flexible electricity production. For modelling reasons, preliminary results are based on historic variations in the day-ahead market (DAM). The revenues were calculated based on (1) the current system of subsidies in Flanders, were there is a fixed feed-in tariff over a period of 10 years, and (2) a subsidizing scheme that allows operators to spread the same sum of subsidies over a longer period of time (for example 20 years). In the latter case, higher profits could be obtained since there is more compliant timeframe during which operators receive government support.

The use of digestate as a substitute for fossil based fertilizers is currently compromised in Flanders by legal restraints. A different business model for nutrient deposition was investigated. Currently all the digestate produced in co-digestion plants with manure intake is considered as 100% manure. In the proposed 'pro rato' system, only a percentage of the digestate, equal to the percentage of manure in the input, would be considered as manure. To overcome current legal restraints, that prevent the implementation of this 'pro rato' system, we conducted field experiments in 2015 and 2016 to demonstrate the effectiveness of digestate as a fertilizer. The results were unsatisfactory because of the large variation in the composition of the digestate and

the instability during storage. Especially the low N/P ratio appeared to be problematic to apply large amounts of digestate on the field as a substitute for mineral fertilizers.

The potential biomethane production in Flanders was evaluated on a technical and legislative level, where several legislative issues were identified. To overcome these issues, the recognition of the ecological value of biomethane and the traceability are a priority. A first meeting with the department of energy in Flanders, along with other relevant partners took place to discuss the necessity of a guarantee of origin for biomethane trade and an affiliated support scheme for biomethane production in Flanders. To establish a good working trading market and consequently creating an interesting financial climate for the production of biomethane, an alignment with a European trading system is desired. Consequently, we strived for a close collaboration with ERGaR, an association that seeks to establish and independent, transparent and trustworthy documentation scheme for mass balancing of biomethane.

#### DISCUSSION

Last year the project consortium focussed mainly on the technological assessment of alternative business models for nutrient depositions, the transition to biomethane production and the potential added revenue from flexible energy production. This resulted in the development of a baseline model for flexible electricity production. Efforts are still ongoing based on new insights in order to keep the model as up to date as possible. The model is able to optimize profits from variable electricity production and potential added value from the implementation of biomethane production in Flanders.

A new approach was suggested for the implementation of an alternative business model for nutrient depositions, under the form of a 'pro rato' system. Based on the results of passed research, only the liquid fraction of the digestate will be considered as a suitable fertilizer, because of the higher N/P ratio. Therefore, more digestate can be applied on the field before the amount of phosphorus becomes restricting. Furthermore, the composition of the liquid fraction appeared to be more stable, which facilitates the storage over time. Hence, the research in 2017 will focus on stability of the liquid fraction during long time storage, the development of on-site measurements and the possible elevation of the N/P ratio. Based on these results a decisive plan of action will be drafted for a new field campaign in 2018.

We experience a growing incentive to develop a guarantee of origin for biomethane in Flanders, since the draft version of the new renewable energy directive demands that EU member states implement a system for guarantees of origin for all renewable energy (European Commission, 2016). This project is actively participating in setting up scientifically sound scenarios for biomethane production that is coupled to a EU-wide market regulated through guarantees of origin. Accordingly, the consortium tries to estimate the impact of EU-developments on local business cases to make the Flemish biogas sector more independent of governmental support schemes. Biogas-E as well as Ghent University strive to generate data and results that are useful for the sector as a whole and are compatible with EU-wide trends in biogas.

#### ACKNOWLEDGEMENT

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#### Pocket Power! - Extending Small-Scale Anaerobic Digestion in Flanders

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#### SUMMARY

Flanders counts to date about 100 active small-scale anaerobic digesters. Those 'pocket' digesters use agricultural residues to provide energy for a company or farm. Most pocket digesters today find their application on dairy farms. The Pocket Power project will investigate the possible extension of pocket digestion to other agricultural streams such as pig manure and crop residues. In addition, Pocket Power will quantify the contribution of small-scale anaerobic digestion to the reduction of greenhouse gas emissions (compared to manure storage). The overall process optimization will be addressed as well.



Figure 1: Small-scale Anaerobic Digester

#### INTRODUCTION

Pocket digestion is a technology, where the anaerobic digestion process is applied to mainly onfarm agricultural residues for the on-site production of biogas. This biogas is combusted in a combined heat and power (CHP) unit and provides energy in the form of electricity and heat to be used on-site by the agricultural company.

The popularity of pocket digestion has increased greatly in the last few years in the Flemish region of Belgium and a number of neighbouring countries. In Flanders, there are about 100 active pocket installations to this date and it is expected that this number will increase significantly over the next years (De Dobbelaere et al., 2015).

Pocket digestion has many advantages. Most importantly, it is a tool for agricultural companies to increase their self-sufficiency in terms of energy demand and processing of residual waste streams. Second, pocket digestion contributes to the reduction of greenhouse gas emissions from manure storage (Miranda et al., 2015; Maranon et al., 2011). Due to its local character and limited scale, pocket digesters have limited transport issues and cause only a minimum disturbance of the landscape. This ensures a better level of acceptance from local inhabitants (De Dobbelaere et al., 2015).

Today, pocket digestion mainly finds its application on dairy farms. Within this niche there is still some potential for growth, but in the coming years a stabilization of the number of installations in Flanders is expected (De Geest et al., 2016). Therefore, it is important to investigate how pocket digestion can be expanded within and to other sectors. This will be the **first objective** of this project. The starting point is biomass that is still underused and shows a lot of potential for digestion on a small scale.

The **second objective** includes the quantification of the reduction of greenhouse gas emissions attributed to a pocket digester. Also, the relation of this reduction to operational variables will be investigated based on practical measurements on the one hand and model simulations on the other hand.

#### MATERIAL AND METHODS

Pocket Power comprises seven main work packages (WP), as represented in Figure 2. WP1 and WP7 focus on the overall project coordination and the distribution and communication of the results. The main research lines are described in WP2 to WP6.

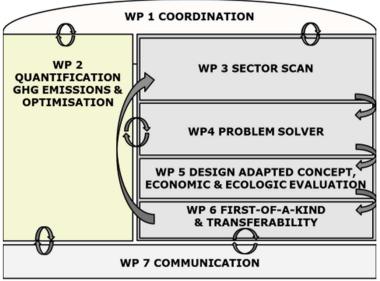


Figure 2: Work Plan of the Pocket-Power Project

WP3 to WP6 comprise the **first objective**, namely expanding pocket digestion towards other sectors. A sector scan will be performed in WP3. Based on a multi-criteria analysis the potential and impact of an extension to other sectors will be defined. Criteria of interest are biomass availability, technical needs, legal restrictions, economic feasibility and ecological impact. Eventually, the two subsectors with the highest potential will be selected.

WP4 provides technical and practical solutions for pocket digestion relating to the entire process, from input streams, storage, process stability, energy interface to utilization of waste streams. Depending on the chosen biomass, specific problems will be solved. This work package aims to find hands-on solutions for the challenges that arise from valorization of the partial streams of the two subsectors selected within WP3. WP5 makes a detailed calculation of the feasibility of the two case studies that were selected in the previous work packages. The case studies will be evaluated economically (cost-benefit analysis) and ecologically (LCA-study). The next step (WP6) is the transition to implementation and demonstration of one viable case study. WP2 focuses on the **second objective**. The second objective includes the quantification of the reduction of greenhouse gas emissions attributed to a pocket digester. Existing small-scale digesters will be used for conducting a full-scale measuring campaign on the on one hand and modelling on the other hand. In this manner, the relation between the reduction of greenhouse gas emissions and operational variables will be investigated. This knowledge will be used for process optimization in terms of maximum biogas production and valorization.

#### **RESULTS AND DISCUSSION**

The overall aim of the Pocket Power project is to respond to the demand of other sectors than the dairy sector for a profitable valorization of their agricultural residues. The first outcome will

be a priority list in which the different subsectors are ranked based on their potential for a smallscale anaerobic digester. For the two types of companies (subsectors) that are on top of the list, an adapted digestion concept from delivery of biomass to production of biogas will be developed. For at least 10 companies within one of these subsectors, assistance will be given for further innovations. For at least 100 companies a feasibility study regarding pocket digestion will be performed. Finally, the possible transferability of the adapted concept towards other unexamined agricultural subsectors will be determined.

The model and full-scale measuring campaign will provide a framework for the development of information strategies to enforce greenhouse gas emission reduction. The mechanistic model will describe the dynamic behaviour of the pocket digester. Next to the reduction of greenhouse gas emissions, the model aims at process optimization in terms of design and control for maximum biomass processing and biogas production.

The outcome of the economic and ecological studies will offer constructers a real help in expanding their business. A scientifically proven study like LCA is the ideal tool to convince opponents or governmental institutions of the benefits of this technology.

#### ACKNOWLEDGEMENT

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FLANDERS INNOVATION & ENTREPRENEURSHIP



#### **DIGESMART – Digestate from Manure Recycling Technologies**

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The project partners set out to bring together all the stakeholders for the installation of a processing step to minimize spreading of digestate and to economically valorise the minerals (N, P, K, among others) in digestate. By using recycled nutrients instead of synthetic fertilizers, it is possible to save energy, limit consumption of fossil fuels and reduce the farm's carbon footprint. The project resulted in a full-scale operational pilot plant and opened a new market for nutrient recycling.

#### THE DIGESMART PROCESS STEP BY STEP

Digested material obtained at the end of the anaerobic digestion process is centrifuged to separate the solid from the liquid fraction. The solid fraction is composted, whereas liquid fraction is treated through a high temperature stripping process (60°C) in order to obtain a mineral fertilizer. The heating process can be done using thermal energy produced by the cogeneration unit of the biogas plant, but is also aided the exothermal chemical reaction involved. Optionally, the residue from the stripping is further dried using solar energy.

#### PROJECT ENROLMENT WITH TEST RIG

DETRICON started building a first test rig with a capacity of 0.5 tons/h in June 2014. The processing unit was isolated thoroughly and the RVS material was used in order to work in low pressure and high temperature regions. The test rig had a practical stripping volume of 1 to 3 m<sup>3</sup>, and a ventilation flow of 1000 to 1800 m<sup>3</sup>/h. The system was composed of a mixing tank to add CaO, a filter, a heat exchanger (to optimise the energy balance), a scrubber and 3 stripping tanks. All subunits were connected and air could circulate from stripper to scrubber and back to the first scrubber without any exhaust, effectively forming a closed system. The operational conditions to have an optimal stripping capacity for treating the liquid fraction of digestate were determined. The search criteria were: minimal use of electrical and thermal energy, minimal use of CaO to higher the pH and lower the P concentration, maximise the stripping capacity per m<sup>3</sup> of volume and minimise the pay-back of the installation.

#### FULL-SCALE PILOT PLANT

After the start-up trials, a number of different adaptations to the design of the system were undertaken during the DIGESMART project in order to improve the market replication possibilities in Belgium and the other targeted EU-countries. The most noteworthy design changes were:

- The incorporation of the stripping and scrubbing process in a single tank, facilitating the energy recovery and making the system more efficient. Also, this integrated recovery solution implied a reduction in investments and handling in comparison with other technologies on the market.
- Conversion to a bigger stripping and scrubbing volume and greater ventilation flow in order to maximize the recovery of  $NH_3$  gas from the liquid digestate fraction.
- The use of nitric acid in the scrubbing instead of other acids due to the higher value of ammonium nitrate in comparison to other ammonium fertilizers.
- Development of a purifying step to remove odour and colour components with membranes and adsorption materials and inline concentration analysis with an accuracy of 99,97%.

The improved pilot plant was installed at the IVACO company in Gistel-Zevekote in Belgium. During the first trials the plant was able to process 2-3  $m^3/h$  of digestate, so it will be able to process liquid fraction of a biogas plant which produces about 30 000  $m^3$  of digestate per year. Nitrogen content was reduced from 4 kg N per ton to 0.5 kg N per ton. The first trials provided positive results and at current time the operation runs smoothly.

#### FIELD TRIALS GAVE GOOD AGRONOMIC RESULTS

Due to its liquid form, the recovered ammonium nitrate needs to be applied taking into account environmental effects and economic valorisation. SATA and the other partners involved tried to find the best ways to use the recovered fertiliser in the farmland. This according to the most common agricultural practice, mechanization level of farms and on-site environmental legislation. For comparison, the use of the "green fertiliser" was mostly tested against liquid synthetic fertiliser applied to the selected crop. Different field trials, greenhouse trials and a pot experiment were conducted as can be seen in table 1.

Trial Number	Сгор	Timing	Туре	Fertiliser application	Result compared to synthetic fertiliser
1	Lettuce – spring cycle	Feb 2015 – May 2015	Greenhouse	Fertigation	similar
2	Lettuce – fall cycle	Sept 2015 – Oct 2015	Greenhouse	Fertigation	similar
3	Superior bread making soft wheat	Oct 2015 – June 2016	Open field	Foliar	similar
4	Maize grain	May 2015 – Oct 2015	Open field	Fertigation	similar
5	Maize	May 2015 – Oct 2015	Open field	Spreading	similar
6	Lettuce	Jan 2016 – Mrch 2016	Growth chamber	Mixed with soil	similar

Table 1: Field trials and experiments with the	produced ammonium nitrate
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The results observed in the trials gave a solid base for making the argument that in the European agricultural system, the DIGESMART fertiliser has the same performance as the conventional liquid fertilisers. The trials in lettuce, maize and wheat conducted in two European regions make the validation of the product more solid. Nevertheless, it was noticed during the trials that attaining a stable product concentration was a work in progress (total N from 13.2% (trial 5) to above goal content 19.2% (trial 6)). In addition, the liquid formulation sets a limit on the adaptation in agriculture of the ammonium nitrate. Most practices in farming use granulated chemical fertiliser products and farmers are used to handling such products. From the agronomist's point of view, this could represent a limit for the DIGESMART-product in terms of storage space, packaging and handling.

#### SOLAR DRYING AS AN ADD-ON TECHNOLOGY

As solar drying system was designed based on a flatbed evaporator design combined with an air solar heat panel and a high efficiency module (glycol based). This module was used to further dry the liquid fraction after stripping. Four tests have been carried out during the project. Due to weather conditions, tests were mainly performed in the months of July and August.

The test on August 27, when the ambient conditions were favourable, indicated that the solar evaporator needed to be adjusted, because the evaporation process was too slow. The air entering the evaporation chamber did not interact sufficiently with the digestate and got out being far from saturated. A metal sheet with cross bands was prepared to force the process air to a closer contact with the digestate, but this device was not tested during the four tests that have been carried out in Gistel.

A possibility to ameliorate the drying rate can be searched also in reducing the airflow, in order to give more time to the air in the evaporator to collect vapour from the digestate, and enter the condenser closer to the saturation. Even with a lower airflow, a higher condensation rate is possible if the air-vapour mix enters the condenser closer to saturation. Further tests are needed to optimise the prototype.

#### **BUSINESS AND MARKETING OF THE DIGESMART SOLUTION**

A business plan model was elaborated during the project and was used for a uniform interpretation of the business opportunities that came by during the duration of the action. The input and outputs are well summarized in a front sheet in order to make it practical and easy to use. The business model can cope with different farming/biogas scenarios such as land spreading of digestate, liquid fraction treatment, selling the product or using it on farm. The input is processed in techno-economical sheets and the business results are given. Net profit, payback scheme and a theoretic break-even production point are calculated. A balance sheet is given, as well as a cash flow. Thirty-two detailed business plans were developed during the project with country specific parameters.

Next to the business model, a marketing plan was made which will be of importance for the further market replication of the DIGEDSMART-solution after the project has ended. Several commercial strategies are defined in the marketing plan in order to answer to farmers' needs and constraints. The marketing plan contains information concerning the market and future outlooks for the different targeted countries. The marketing is also taking in to account the SWOT-analysis made on the delimitated business. Competitors within the market are identified, quantified and described.

#### ENDING STRONG WITH NEW BUSINESS OPPORTUNITIES

The project ended strongly with a lot of interaction with the interested stakeholders, mainly biogas operators and farmers. The pilot site is in full operation mode and new installations are being elaborated across the target countries. Field trials have finished, the solar drying module gave interesting results and the stripping&scrubbing unit was fine-tuned and optimised. The market replication was assessed and a plan for further sales was made. Business was developed, adding up to a final market replication of 4 installation + 1 pilot.

#### ACKNOWLEDGEMENT

The DIGESMART solution aims to reduce the environmental impact of farms and biogas plants by facilitating the market uptake of ammonium stripping and further processing of digestate. The DIGESMART project is co-founded by the Eco-Innovation Initiative of the European Union (ECO/12/332882) and ran from September 2013 to August 2016.

PROJECT WEBSITE: www.digesmart.eu

# ReciDigest - Implementation of Digestate Treatment and Recirculation - Influence on Anaerobic Digestion

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#### SUMMARY

The ReciDigest project aims at improving the overall efficiency of anaerobic digesters processing organic waste and side streams by introducing digestate treatment and recirculation to the digesters. Application of ultrasound technology or oxidation-based techniques (ozone and hydrogen peroxide) on the digestate leads to the conversion of the residual refractory organic matter to easily biodegradable (soluble) organic components. When subsequently recirculating this treated digestate back to the digester, it becomes a new, secondary substrate, hence achieving a higher degree of conversion for the organic matter present in the original feed. Furthermore, ammonia inhibition caused by feeding nitrogen-rich substrates such as manure, is tackled by introducing a second treatment, i.e., the addition of a stripping & scrubbing step in the recycle stream.

#### INTRODUCTION

There is an increasing trend towards energy recovery from waste streams such as sewage sludge, animal manure and crop residues. In this regard, the use of anaerobic digestion is considered as very positive (from an economic and environmental point of view). During digestion, an energy-rich biogas is produced (55-75% CH<sub>4</sub>), which can be fed to gas engines to produce heat and electricity. Due to the presence of rigid and recalcitrant structures, the digestion process is slow, leading to long residence times, typically 15-30 days. Even with these long residence times, the overall degree of conversion is limited: only ca. 50% of the total organic dry matter is converted to gas. This means that the digestate still contains a considerable amount of organic matter, and hence represents potential for further conversion to biogas. Treating this digestate provides the ability to respond to this problem, and furthermore allows to reduce the ammonia concentration in the digester, leading to an improved system stability and hence an increased process efficiency (i.e., more biogas per ton of substrate fed to the digester). The achievement of this goal is one of the primary measures to reduce costs, further develop the biogas sector and promote renewable energy through biogas generation.

The ReciDigest project responds to this need by addressing two main technical causes of limited efficiency, i.e., (i) the limited degree of conversion of organic matter to biogas due to the presence of rigid, non-biodegradable matter in the substrate, and (ii) process inhibition due to the presence of elevated (free) ammonia concentrations in the digester. The suggested approach,

i.e., digestate treatment and recirculation, is furthermore suited to be implemented in existing installations, with limited alternations to the equipment.

#### PROJECT AIM

The ReciDigest project aims at improving the overall efficiency of anaerobic digesters processing organic waste and side streams by introducing digestate treatment and recirculation to the digesters. Application of ultrasound technology or oxidation-based techniques (ozone and hydrogen peroxide) on the digestate leads to the conversion of the residual refractory organic matter to easily biodegradable (soluble) organic components. When subsequently recirculating this treated digestate back to the digester, it becomes a new, secondary substrate, hence achieving a higher degree of conversion for the organic matter present in the original feed. The applied disintegration techniques can cause an oxidation of the present ammonia (NH<sub>3</sub>) to nitrogen-containing compounds that are not or less toxic, and which can be degraded or removed during further digestate is fed back to the digester (and nitrogen is neutralised to N<sub>2</sub>), the possible recovery of ammonia-nitrogen as a valuable mineral is also investigated. For this purpose, we will use LTL (liquid to liquid) transfer systems applied on raw extracted digestate (or liquid fraction after separation). The aim is to supply N-depleted digestate to the digester, leading to a reduced ammonia-inhibition effect and increased biogas production.

The following sub-objectives are identified:

- Evaluation of three selected disintegration techniques (i.e., ultrasound, oxidation with hydrogen peroxide and ozonation) towards their potential to increase the anaerobic biodegradability of otherwise refractory organic matter in the digestate, with the aim of recirculation this treated digestate back to the digester;
- Evaluation of the effect of the aforementioned techniques on the oxidation of the ammonia present in the digester/digestate to NO<sub>2</sub><sup>-</sup> or NO<sub>3</sub><sup>-</sup> and the microbial conversion of these components to N<sub>2</sub> (primarily through classic denitrification in the digester and anammox);
- Evaluation of the possibility to use stripping (& scrubbing) as a technique to selectively remove and recover the ammonia;
- Optimization of the combination of the abovementioned techniques to achieve the maximum obtainable increase in process efficiency

#### **PROJECT APPROACH**

#### 1/ Increasing the conversion efficiency

As indicated above, the digestate leaving the digester still contains a substantial amount of digestible organic material. In this part of the project we will examine the extent to which the conversion efficiency of biogas potential of digestate is affected when it is subjected to disintegration techniques such as ultrasound, ozone and peroxidation. A parameter study will be performed to determine the optimum treatment conditions. Previous studies already have shown that disintegration leads to the formation of more biodegradable organic material, which must have a positive impact on the biogas production potential. Towards implementation on an industrial large scale, it is important to estimate the economic potential of this application. An extensive energetic analysis of the experimental runs will therefore be carried out.

#### 2/ Issues concerning nitrogen containing compounds

Some organic streams such as animal manure contain high amounts of ammonia, but this has a negative effect on the activity of the microorganisms involved in anaerobic digestion, and can hence lead to very low biogas yields. This part of the project will examine the effectiveness of ultrasound, ozone and peroxidation on the oxidation of NH<sub>3</sub>, which oxidation products will be formed (N<sub>2</sub>, NO<sub>2</sub><sup>-</sup> and NO<sub>3</sub><sup>-</sup>), and how much of the available radicals are consumed during this conversion. The influence on the digester operation will also be examined. More specifically, we will look at possible denitrification of the formed  $NO_2^-$  and  $NO_3^-$  to N<sub>2</sub>, the conversion through anammox and how the biogas composition is influenced. By treating and recycling the digestate, the fresh feed to the digester is diluted, and hence a dilution of the ammonia concentration is established.

Next to oxidation, a reduction of  $NH_3$  will also be examined by means of stripping and recovery. The extent to which ammonia-nitrogen can be recovered as a valuable mineral, will be examined, by using LTL (liquid to liquid) transfer systems on raw extracted digestate or liquid fraction after separation. The balance between water-soluble  $NH_4^+$  and volatile  $NH_3$  in function of process parameters (T and pH) during this transfer, will be investigated. Increasing either or both parameters favors the gaseous form and hence the escape from the substrate (digestate or liquid fraction) is increased. This principle has already been examined in MIP NutriCycle as end-of-pipe technology for recovery of mineral N from organic-rich digestate. In the present context, the goal will be to improve the biogas production potential by recirculation of an N-depleted digestate to the reactor.

Figure 1 depicts the overall flow of the project and the subdivision in the different work packages (WP). WP1 investigates the influence of 3 different oxidation techniques on the release of organic matter from the digestate and the influence on the subsequent anaerobic digestion, on lab-scale batch reactors. The second WP underpins the mechanisms behind ammonia oxidation by application of these afore-mentioned oxidation techniques together with anammox, and supports the findings of WP2 on a more fundamental level. WP3 comprises the experiments on both lab- and pilot-scale of ammonia stripping & scrubbing. In WP4, all the findings come together by combining oxidation, stripping & scrubbing and anaerobic digestion on pilot-scale fermenters. The data hereby generated will be used to develop a decision tool, which allows endusers to evaluate the use of a specific or a combination of techniques suggested in this project.

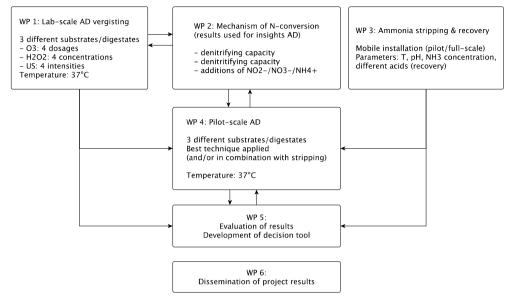


Figure 1: Overview of the different work packages of the ReciDigest project

The project is executed by 3 academic partners, from 2 universities in Flanders and includes 10 Flemish SMEs as part of the users' committee and as such contributing to the project.

#### ACKNOWLEDGEMENT

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#### **INEMAD - Reconnecting Livestock and Crop Production**

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#### CONTEXT

European agriculture is becoming more and more specialised. Farm specialisation however coevolves with an increasing reliance on external inputs and increasing environmental damage. Indeed, at this moment we experience a paradoxical situation where crop production has a need for fertiliser while livestock has an excess of nutrients. Recycling energy and materials through re-connecting crop and livestock production becomes indispensable for attaining agricultural sustainability. INEMAD will address the question of which new methods and how new arrangements should be developed to restore the recycling within the specialisation context. To realize these ambitions, the leading principle of INEMAD is a triangular enlargement (see figure) of the traditional farming systems with a "processing" system. Processing is proposed as a third system, to be linked with crop and the livestock production, in order to increase agricultural productivity while reducing external energy input and closing nutrient cycles.

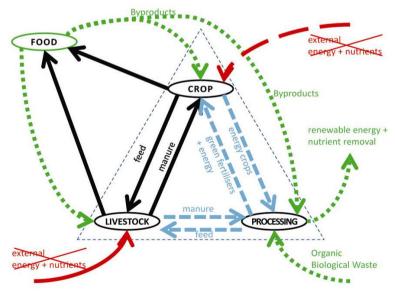


Figure 1: The nutrient flows of primary production

INEMAD is a 4-year FP7 project which started on the 1<sup>st</sup> of April 2012. Partners involved come from 8 different European countries (Belgium, The Netherlands, Germany, Denmark, Hungary, Croatia, France and Bulgaria). The leading principle of INEMAD is that processing can help to restore the nutrient cycle and decrease the energy use. Processing refers to the new upcoming sector of the bio-based economy that uses primary products to produce renewable energy and green fertilizers amongst others. Nutrient recycling can be done by biogas production and the use of digestate as fertilizer. There is a clear difference in the livestock density among regions in the participating INEMAD countries. The project looks at nutrient management strategies at the farm level scale, at the processing level, at cooperation strategies between different farms or between farms and the processing sector and how the institutional context influences these strategies.

The INEMAD project demonstrated how different strategies, technologies and policy instrument can be used to alleviate the emissions of GHG and the reliance on external inputs and helping policy makers in their choice for efficient policy instruments.

INEMAD's results were disseminated and can still be consulted on <u>www.inemad.eu</u>. Five electronic news were sent to 600 respondents. Towards the scientific community different presentations and papers were produced.

There were different stakeholder interactions during the projects lifespan. They acknowledged the need for increased dissemination of research results to practitioners. They also see the government as a central stakeholder who has an important role to play in stimulating the different types of strategies, either by offering financial support, providing a legislative framework or through creating or stimulating markets.

#### OBJECTIVES

INEMAD has a distinct focus on techniques and strategies for optimized nutrient recovery, with additional focus on opportunities for renewable energy production and carbon sequestration. The first set of objectives refers to the technological developments for nutrient recycling:

- transform waste to fertilizers
- minimize greenhouse gas effect
- reconnect livestock and crop production.

The second set of objectives refers to the socio-economic framework to reach the optimal implementation:

- analysis of legal and organisational challenges,
- prediction of economic viability
- collaboration models across Europe.

During the INEMAD project, several strategies and processes have been investigated to link the arable and livestock production, with the processing sector as an important third party. Research topics within the INEMAD project had a wide scope. There was technical lab-scale testing of technologies, there were socio-economic surveys to analyze the farmers' willingness to accept bio based fertilizers, there were policy simulations. Based on this input, the research team has created 40 working papers (WoPa's) analyzing different aspects of the strategies to improve nutrient management. The project looks at nutrient management strategies at the farm level

scale, at the processing level, at cooperation strategies between different farms or between farms and the processing sector and how the institutional context influences these strategies.

#### RESULTS

The main INEMAD results and conclusions can be clustered on the differences due to regional context, the diversified research needs but also the applicability, acceptability and the efficiency of the technologies (see figure 2). The latter one is mainly focusing on the economic-ecological trade-offs that exist when thinking and selecting about the best technology. But also, the social acceptability and aim to unravel stakeholders' awareness and perception towards these developed strategies is included.

The main results of the INEMAD project acknowledge the importance of this regional nutrient and policy context. Western European partners have a need for more fundamental and applied research. Researchers did more hypothetical simulation of policy instruments and applied research was carried on more innovative types of technology. Central European partners researched more the effect and possibilities for nutrient management techniques based on manure or organic biological waste products. There was definitely a knowledge transfer of the already established technologies in nutrient rich regions to the partner countries not facing this nutrient excess problem.

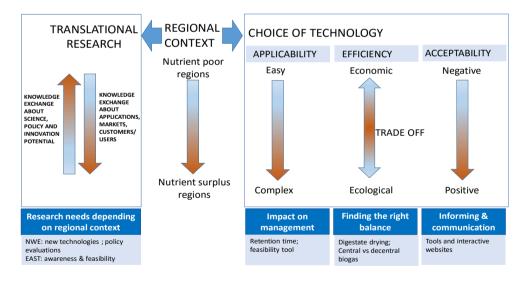


Figure 2: Overview results INEMAD project

Also, the policy context was evaluated in the different countries. Simulations were done on policy instruments to stimulate farms to respect N concentration in the leaching water, support policies for the biogas sector such as feed-in premia and investment support and simulation on relaxing some manure transport regulations. The simulations learn that policy makers should properly

overthink the policy instruments. Instruments should be selected depending on the policy objective taken into account possible conflicts with other policy objectives, the targets to be reached taken into account the (un)wanted impact these instruments can have on other ecological and economic indicators. The simulations we did can certainly help policy makers in this choice.

Different nutrient management and processing strategies were analysed in depth. Some innovative strategies were explored such as the production of algae or insects with manure products, constructed wetlands to tackle diffuse nitrate contamination, pyrolysis or phosphorous specialised catch crops. But also, existing strategies were investigated. A main conclusion is that policy instruments could enhance the right balance between economically feasible for the entrepreneur and ecologically best for society. Another conclusion was that existing strategies often face a problem of uncertainty and variability (e.g. input costs, output prices, feedstock availability, nutrient content in manure products). Yet it has been simulated that management and operating systems dealing with these technologies, can be adapted in such a way that variability can offer advantages, in a way that proactively adapting operational strategies to this variability can increase profits as opposed to simply undergoing it. Therefore, more information and dissemination plays a crucial role. Farmers were unaware of the similar performance of biobased fertilizers, which has been tested in a three-year trial within the INEMAD project. Indeed, a large survey in all participating INEMAD countries showed that this variable nutrient content in this new products was the biggest bottleneck to use them.

#### ACKNOWLEDGEMENT

The research leading to these results has received funding from the European Union's Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 289712.

#### AGROCYCLE – for a Circular Economy

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AgroCycle is a Sino-EU collaborative research venture, led by the School of Biosystems and Food Engineering at University College Dublin, Ireland. With 26 partners from across the EU, China and Hong Kong, the project is funded through the European Union's Horizon 2020 research and innovation programme, with additional funding from the governments of the Peoples' Republic of China and Hong Kong. The 36-month project (started June 1<sup>st</sup>, 2016) will deliver a protocol for the implementation of the 'circular economy' within the agri-food sector. It will develop, demonstrate and validate novel processes, practices and products for the sustainable use of agricultural wastes in applications such as fertilisers, bio-polymers and novel chemicals as well as developing technology and policy guidelines for the bioeconomy.

#### AGROCYCLE'S MAIN OBJECTIVES ARE TO:

- Achieve a 10% increase in the recycling and valorisation of agricultural waste by 2020 and;
- Further develop, demonstrate and validate novel processes, practices and products for the sustainable use of agricultural waste, co-products and by-products

The project addresses waste from several agricultural sectors: wine, olive oil, horticulture, fruit, grassland, swine, dairy and poultry. To achieve the project objectives, the work packages below are being undertaken.

#### MAPPING WASTE FLOWS

Reports available to download: www.agrocycle.eu

- Quantifying, mapping, and characterising agricultural waste flows in Europe
- Value chain logistics; and current regulatory requirements
- Sustainable extraction rates of crop residues

#### **BIOENERGY AND BIOFUELS**

- Construction of ethanol and butanol production pilot plants
- Installation of dry AD units in Ireland for poultry manure
- Development of third generation microbial fuel cells to convert biodegradable materials present in waste into electricity

#### **BIOFERTILISER PRODUCTION**

Evaluate effectiveness of biofertilisers from waste materials e.g.

- Lignosulfonates from pruning wastes
- Digestate from AD treated manures
- Natural fertilisers from rice bran

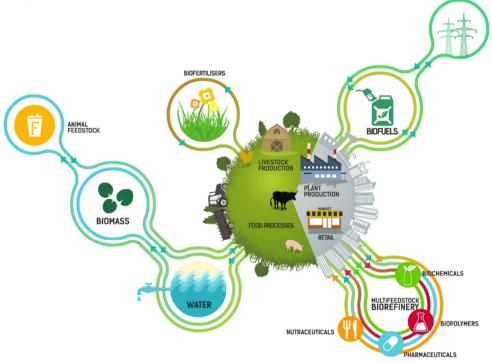
#### NOVEL CHEMICALS AND BIOPOLYMERS

- Extraction of proteins, fibres and secondary plant metabolites (SPM) from horticultural waste streams e.g. potato pulp
- Demonstrate the application of extracted biocompounds in nutraceuticals, active packaging, adhesives and coating applications

#### LIFE CYCLE ASSESSMENT

- Conduct LCA and economic and social assessments for European and Chinese waste value chains
- Consider impacts across full life cycle including upstream production of waste

#### **BUSINESS MODELS + POLICY**



- Joint stakeholder platform for knowledge/data sharing for actors in the agri-food circular economy
- Identify and develop new business models for business diversification
- Mapping out potential markets in Europe and China for agri-food wastes

WEBSITE: www.agrocycle.eu

# P-REX - Sustainable Sewage Sludge Management fostering Phosphorus Recovery and Energy Efficiency

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#### BACKGROUND AND GENERAL GOALS

Phosphorus is an essential and non-replaceable resource. Since there are no fossil deposits in Germany, Phosphorus has to be imported from other continents to sustain good harvests and proper food security for all citizen. A growing world population to be fed and increasing phosphorus demand for fertilizers triggered also by increased energy-crop production nourish concerns about repetition of price spikes and threads to our security of supply.

The project P-REX aimed at demonstrating and assessing existing and promising P recovery and recycling routes with respect to regional specifications in the European context. Besides the demonstration of technical P recovery from wastewater, also the quality assessment and market potential for the various obtained recovery materials was conducted to provide a holistic picture of what can be done already or should be done to enable still hampered options for a more sustainable P management. For sound assessments, data have been collected from literature and practice stakeholders including the demonstrators within the project. This allowed a highly reliable and sound assessment and interpretation of results. A global inventory for nutrient recovery installations (large scale) will be continued and frequently updated. (see P-REX weblink download section)

#### PROJECT ACHIEVEMENTS AND STRATEGIC RELEVANCE

The outcomes of this project have been well recognized within the EU as very practice relevant in terms of P recovery options and valorization routes.

The main results of the project are:

- The three main routes for P recovery and recycling are the traditional high quality sludge on land application, the technical struvite recovery from the liquid phase and P recovery from mono-incinerated sludge ashes. All three routes have to be seen relevant pillars for future sustainable sludge management.
- Phosphorus recovery needs to be done in an energy-efficient way to become sustainable. Focus on high P recovery rates alone is counterproductive. Synergies with better nitrogen management should be tapped. (nutrient recovery cascades)
- The legal framework in Europe, but also on national level has big impact on availability of recycling options. Still today, a level-playing-field for both, primary and secondary resources is missing. The revision of EU fertiliser regulation (EC 2003/2003) feeds the hope for improvement. There is still a concern and uncertainty based precautionary dogma to be transformed into a fact and risk assessment based pragmatism. Circular economy cannot become true without recycling!

- Some recyclates appear to be suitable to substitute/replace fossil phosphate rock as only allowed mineral P fertilizer in organic farming. Struvite and calcined phosphates (like AshDec) have been recommended by EGTOP for approval in organic farming (EC 889/2008 should be adapted accordingly)
- There is still a trade-off between recovery rate and efforts to be taken (i.e. chemicals and energy consumption)
  - Struvite recovery rates currently between 5-25% of total P in influent with operational benefits and short ROI. Provides potential of tapping synergies with higher energy efficiency and N recovery. Attempts already visible to achieve recovery rates above 40%.
  - Highest recovery rates from sludge ash (>80% of TP<sub>influent</sub>) but depending on overall energy management in worst case high energy consumption. Tendency to yield phosphoric acid instead of dubious P minerals is to be favored for the ash route.
  - Best option is integrative approach making the best and most out of existing routes and compensate routes to be restricted in future. Fertiliser manufacturers and retailers already exist and do not need to be reinvented. Recyclates provide an option to make existing production processes and therefore products cleaner.
- Technical P recovery reduces the risk of hazards linked to the application of sludge. But if sludge is safe, this route is the most sustainable, since all nutrients contained are recycled without additional efforts. Biological P removal instead of chemical P removal should be applied wherever applicable.

#### CONCLUSION

Wastewater is, besides manure and organic waste, a relevant waste stream carrying relevant quantities of P for recovery. Where sludge application on land is in question, technical alternatives will have to be implemented to compensate the losses of recycling rates. The new German sludge ordinance (draft 2015) will immediately reduce the national P recycling rate from wastewater of 20% today down to nearly Zero generating also shortage in disposal options and therefore cost explosion. If sludge complies with the requirements of the sludge and fertilizer ordinance, there is no reasonable argument for a ban. Of course, a robust sludge quality management system (i.e. like in Sweden (REVAQ)) and traceability are pre-conditions.

P recovery in form of struvite comes in line with manifold operational and environmental benefits and shall be implemented where possible. To increase the application potential, a switch from chemical P removal towards biological P removal shall be considered for bigger WWTP. Struvite recovery can always be seen as complement to the two other principle routes. Where sludge application on arable land is no option and sludge incineration infrastructure is in place, P recovery from undiluted incinerated municipal sludge is the option at hand. Here the integration of the incinerator into a reasonable energy management is crucial for energy efficiency. So far, co-incineration is more energy efficient compared to existing mono-incineration revealing clearly the trade–off between resource efficiency vs. energy efficiency. But, new mono-incinerators (like the one in Zurich) will keep up and level out the co-incineration. To meet economy of scale

requirements, P recovery from ash facilities need at least 20,000 tons of ash as minimum. Besides that, P content in ashes should not be below 8% and Fe/Al shall be kept as low as possible.

Countries without mono-incinerators should rather focus on improving the sludge quality to ensure environmental and human health risk reduction.

Struvite recovery should be combined with advanced sludge disintegration and subsequent ammonia stripping to facilitate synergies in the overall sludge treatment train of suitable WWTPs. (So-called C, N and P nutrient recovery cascades).

#### **KEY DELIVERABLES**

Reports Reports, factsheets, policy brief in EN available for download here: <u>http://p-rex.eu/index.php?id=11</u> The eMarket will be continued by the ESPP and is accessible here: <u>http://e-market.phosphorusplatform.eu/</u>

GENERAL INFORMATION DURATION: 01.09.2012 – 31.08.2015 PROJECT TYPE: EU FP7 Funding (grant agreement no. #308645) PARTNERS: 15 (from 7 countries) LOCATION OF PROJECT: Kompetenzzentrum Wasser Berlin gGmbH PROJECT MANAGER: Dr. Christian Kabbe CONTACT: Christian.Kabbe@kompetenz-wasser.de WEBLINK: www.p-rex.eu

#### COMMUNICATION

www.p-rex.eu Follow Twitter: @P\_RecoRecy

# SYSTEMIC - Systemic Large Scale Eco-Innovation to Advance Circular Economy and Mineral Recovery from Organic Waste in Europe (H2020 project)

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WEBPAGE: www.systemicproject.eu

#### SUMMARY

Manure, sewage sludge and biowaste are the most abundant nutrient-rich streams in Europe, containing large amounts of valuable phosphorus, nitrogen and organic matter. SYSTEMIC will demonstrate and evaluate innovative approaches leading to recovery and reuse of nutrients (N, P, K) from these streams. This will reduce Europe's dependency on non-renewable P fertilizers and energy-demanding N-fertilizers, and will offer sustainable solutions for the treatment of manure in regions with intensive agriculture.

At five locations in Europe, large-scale demonstration plants will be set-up to demonstrate the combination of anaerobic digestion with Nutrient Recovery and Recycling technologies (NNR). These demonstration plants will process one or more biomass sources, like manure, sewage sludge, food waste and biowaste, into valuable mineral fertilizers and soil amendments. The technical, economic and environmental performance of the demonstration plants will be evaluated and the results will be translated into business opportunities for ten outreach locations. Emerging innovation barriers will be encountered along this process and addressed in policy recommendations. Overall, the project aspires to give a boost to the implementation of innovative concepts for closing nutrient cycles at a European scale.

#### RATIONALE

Manure, sewage sludge, food waste and biowaste are the most abundant nutrient-rich streams in Europe, containing large amounts of valuable phosphorus, nitrogen and organic matter. Within a circular economy, these nutrients should be reused in an efficient way. Current treatment and disposal practices however, are inefficient in terms of nutrient utilisation for food production.

For manure, inefficient use of nutrients is typically related to regions with intensive agriculture where the agricultural sector produces more manure than can be applied on nearby land within the application standards. These application limits have been introduced in the last decades to prevent emissions of nutrients to ground- and surface waters. To date, the excess amount of manure is transported to regions with a demand for nutrients. This practice is however expensive

and associated with GHG emissions because of the large volumes and long transport distances (≈500 km). Meanwhile, arable farmers need to buy synthetic N fertilizers because manure cannot meet the NP requirements of the plant, taking into account the fertiliser standards. Additionally, transport of manure leads to a regional loss of valuable organic matter which plays a crucial role in maintaining soil quality and soil fertility. The storage of organic carbon in agricultural soils is an important pathway to mitigate GHG emissions.

As a solution, nutrients (N, P, K) should be recovered in the form of mineral fertilizers that which can be transported against low cost and can replace the purchase of industrially produced mineral fertilizers. The nutrient-poor, but C-rich residual product can then be used as soil amendment in regions with intensive livestock production. Sewage sludge and biowaste constitute a large source of P, N and organic matter, but their current re-use as fertilizers is ineffective and can pose health threats. In addition, in some EU Member States, large amounts of these valuable nutrients and organic components are still lost through incineration.

Recovery of N from these biomass resources is an effective way to reduce GHG emissions and high energy costs related to N-fertilizer production. Recovery of P will largely reduce Europe's dependency on non-renewable P-reserves. Up till 70% of Europe's demand for mineral P fertilizers can be fulfilled through recovery of P from sludge and household waste, showing the enormous potential of these waste streams as a resource for P. Though technologies to recover and reuse nutrients from biomass exist, large-scale implementation of these technologies is hampered because of poor economic perspectives, low acceptance of recovered products and/or legislative barriers.

#### OBJECTIVES

SYSTEMIC aims to develop and demonstrate viable business cases for processing of manure, sewage sludge and biowaste into valuable, customer-specific products. In order to facilitate a fast uptake of these new concepts, they should be built upon existing value-chains for waste and fertilizers and make use of proven state-of-the-art technologies for nutrient recovery and reuse. Through active dissemination of the project results and a continuous science-business-policy dialogue, SYSTEMIC will give a boost to the realisation of circular economy solutions in the EU.

#### THE SPECIFIC OBJECTIVES OF SYSTEMIC ARE:

- To demonstrate novel technologies for the recovery and reuse of nutrients from manure, sewage sludge and biowaste at five large-scale demonstration plants in Europe
- To demonstrate the technical, economic- and environmental performance of the NRR technologies
- To have a wider impact through the translating of the performance indicators into opportunities for circular economy business cases at ten outreach locations.
- To formulate policy recommendations and to derive a roadmap to support the roll out of Circular Economy Solutions for biowaste over Europe

#### SCOPE

#### Demonstration plants

In Europe, five existing AD plants will be enhanced with proven, novel nutrient recovery and reuse technologies (Table 1). The five demonstration plants are located in different regions and will present a broad pallet of NNR technologies. The demoplant in the Netherland will process digested pig slurry into mineral N, K and P-fertilizers and a P-reduced organic fertilizer. In the Netherlands, there is an urgent need for technical solutions to lower the P content of digestate because the application of manure on agricultural soil is mainly limited by its P content. In Belgium, the demoplant (AmPower) is located in a region where nutrient-rich digestate has a negative value due to excess of manure produced in the agricultural sector. They will invest in RO technology and aspire to demonstrate that the production of concentrated liquid fertilizers is economically profitable. The demoplants in the UK and Germany will demonstrate viable business cases for the conversion of poultry manure into mineral N and N-reduced digestate. The demoplant in Italy (Acqua&Sole) will invest in an advanced N-stripper in order to produce sludge-based fertilizers with a reduced N-content. They will demonstrate the agronomic value and environmental quality of sludge-derived products in order to increase the market value and acceptance of these products.

#### Business case evaluation

The demonstration plants will be thoroughly monitored and evaluated with respect to the technical performance of the NRR technologies, the viability of the overall business case and the environmental benefits compared to current practices. At all five demonstration sites, the project will build upon existing logistic chains for the supply of manure and biowaste, and the exploitation of produced products. The focus is on the technical scaling out and the actual uptake of large amounts of recovered products into existing product chains. Likewise, importance is the thorough development of the business case and defining legislative barriers to overcome.

#### Business opportunities for outreach locations

The experiences gained at the demonstration plants will be transferred to ten other AD-plants in the Europe; the so-called 'outreach locations'. For these outreach locations, current business cases will be used as a starting point and promising business cases including nutrient recovery will be formulated in relation to its regional market, legislative conditions and technological possibilities of the outreach location. This supplies the plant owners with information of new options to produce products for new markets, including information of the costs and benefits, which is needed to evaluate their own specific business cases and to decide upon an investment.

#### Innovation at a European scale

To enhance the full development of circular economy solutions for manure, sewage sludge and biowaste at the European scale, a continuous science-business-policy dialogue will be facilitated and policy recommendations will be given based on the identified regulatory barriers. The experience gained in SYSTEMIC in relation to technology, business case development, innovation processes and policy will be synthesized into a roadmap, facilitating the further roll-out of circular economy solutions for manure and biowaste streams.

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Plant	Feedstock	NNR technologies	Products (full scale)
Groot-Zevert Beltrum, The Netherlands	Pig manure, biowaste (100 000 ton)	N-stripping from liquid fraction and P- stripping from solid fraction	Biogas Ammonium sulphate Calcium phosphate P-poor soil improver
AM-Power Pittem, Belgium	Manure, biowaste (180 000 ton)	Nutrient recovery by reverse osmosis	Biogas Ammonium sulphate N+K concentrates
Acqua&Sole Pavia, Italy	Sewage sludge, biowaste (120 000 ton)	<ul> <li>N-stripping from liquid fraction</li> <li>P-recovery from liquid phase of digestate (pilot)</li> </ul>	Biogas Ammonium Sulphate Organic fertilizers Soil improvers
Oaklands Oaklands, UK	Poultry manure (50 000 ton)	Two-stage mesophilic digester (under construction) including a N stripper	Biogas Ammonium Sulphate Liquid CO <sub>2</sub> Organic P fertilizer
BENAS Ottersberg, Germany	Corn silage, poultry manure (80 000 ton)	Enhanced N stripper P recovery (pilot) Lignocellulose recovery (pilot)	Biogas Ammonium sulphate Calcium carbonate

#### **PROJECT FACTS**

The project will start on 1<sup>st</sup> June 2017 and is funded by the European Commission under call CIRC-01-2016 'Eco-innovative approaches for the circular economy: large-scale demonstration projects' of its H2020 framework (projectnr. 730400).

#### **PROJECT PARTNERS**

Wageningen Research (NL) will coordinate the four-year project in which fifteen partners from seven EU member states will participate. The project partners are: Ghent University (BE), Vlaams Coordinatiecentrum Mestverwerking (VCM, BE), Proman Management GmbH (AU), Rural Investment Support for Europe (RISE, BE), Milano University (IT), European Biogas Association (BE), AM Power (BE), Groot Zevert Vergisting (NL), Acqua & Sole (IT), RIKA Biofuels (UK), GNS (D), A-farmers (FI), ICL Europe (NL), Nijhuis Water Technology (NL).

# KASAV - Cascaded Utilisation of Food Waste for the Production of Liquid and Gaseous Biofuels

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#### INTRODUCTION

In the past, organic residues such as municipal organic waste have been utilized to produce liquid fuels only to a minor extent. The main part of municipal organic wastes is used for biogas production or disposed of by composting (Stenmarck et al., 2016). Throughout this project, a fermentative cascade is developed, which facilitates the utilization of low-cost renewable residues for the production of alternative liquid and gaseous biofuels. The process is split into three steps whereas the core element of the KASAV-process is an acetone-butanol-ethanol (ABE, solventogenesis) process unit with an upstream hydrolysis and acidification stage and a downstream biogas stage (methanogenesis). The first and third stage can be implemented as mixed microbial fermentation steps whereas the core process has to be conducted with a single microorganism under sterile conditions.

The hydrolysis gas produced in the first step is separated into  $CO_2$  and  $H_2$  via membrane technology. Using the gaseous intermediate product ( $H_2$ ) in the subsequent ABE- and biogas process stages, a fundamental improvement of the single process steps, concerning yield and productivity can be ensured (Cheng et al., 2012; Mollah and Stuckey, 1992). Additionally, higher methane concentrations and lower  $CO_2$  concentrations can be reached in the biogas stage through internal biogas upgrading and hence, external upgrading to bio-methane quality is more cost-efficient. The use of liquid intermediates from the first stage and maximization of the production rates also increases yield, productivity and stability of the ABE fermentation itself and emphasizes further research on the individual process steps.

Current drawbacks in bio-butanol production, such as low product concentrations because of product inhibition, instability of the microorganisms and long lag phases in batch-fermentation processes, will be minimized by continuous product recovery using pervaporation technology, immobilization of microorganisms on carrier materials and the development of a continuous three-stage production system.

Implementation of such a novel three stage process will have numerous positive effects on society such as:

- Contribution to the fulfilment of the energy, climate and technology policies of the European Union
- Improvement of the affordability of sustainable energy and innovative technology
- Process optimization of bioenergy production and understanding of the individual processes
- Diversification of sustainable energies

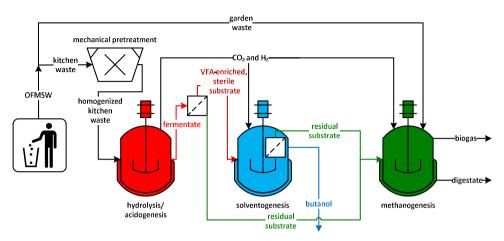


Figure 1: Process scheme of the three-stage production process

#### RESULTS

Results of this project are still under investigation but experiments on the production and optimization of the production rates and yields of the intermediates from the first stage have already been conducted. The experiments investigated the influence of the pH (5.5, 7.0 and 9.0), temperature (37, 55 and 70°C) and hydraulic retention time (HRT) of 2 and 6d on the maximization of butyric acids from the acidification of food wastes through mixed microbial fermentation. First, all process parameters can be easily implemented and therefore our experiments offer the possibility to adapt running processes towards the production of butyric acid and second, mixed microbial fermentations provide the significant technological advantage that unsterile conditions can be applied which makes the process highly attractive from the economic point of view. Unfortunately, the wide variety of different microorganisms involved makes impact of different process conditions much less predictable and process optimization is cumbersome.

Adjusting the pH values to 5.5, 7.0 and 9.0 resulted in butyric acid concentrations of 2.1 g/L, 2.8 g/L and 8.5 g/L, respectively. The most beneficial effect was achieved through temperature control. We demonstrated that raising the temperature from mesophilic to thermophilic conditions increased the butyric acid concentration by almost 300% and the percentage share on the VFA distribution nearly doubled. Additionally, the longer process times at fermentations conducted at HRT of 6d led to an increased butyric acid concentration from 10.55 g/L to 13.00 g/L, respectively. Unfortunately, the prolonged HRT resulted in lower production rates.

In the following process investigations are still ongoing and different strains have been screened for solvent production. Until now ABE concentrations of up to 14 g/L have been reached with a percentage share of butanol of almost 60%. Further research is conducted to investigate the influence of pressure on the production of ABE, yield and their change in distribution. Separation processes are investigated by the Technical University of Vienna and are still under investigation.

The residuals from the first stage which cannot be used in the ABE core process have been separated via centrifugation and have been digested in the last stage for the production of biogas. Roughly 10% of the effluent from the first stage accounted for dry mass which has both not been hydrolyzed and acidified or is biomass produced during the process. This dry mass consisted of about 87% volatile solids (VS) which yields about 600 ml  $CH_4/g$  VS. The residuals of the second stage will be processed in the last stage as well.

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### **GR3** - GRass as a GReen Gas Resource

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#### **PROJECT GOALS**

The GR3 project promotes the use of grass and other herbaceous residues from landscape management as a sustainable feedstock in biogas plants in the partner regions Belgium, Italy, Germany, Denmark and Portugal. The energy potential of these residues remains underutilized across Europe. The barriers encountered are:





Co-funded by the Intelligent Energy Europe Programme of the European Union

- Insufficient awareness and acceptance of suitable technologies for the mowing, storage and anaerobic digestion of grass residues;
- Absence or lack of cooperation between stakeholders along the value chain;
- Legal Barriers

The project aimed for an overall increase of the renewable energy production by the digestion of grass or herbaceous waste. The benefit is that these grass residues are not competing with food production as the focus was only on these residues that were harvested due to maintenance, etc. and that could not be valorised in a « higher » application (e.g. Reusing materials, recycling, etc.). Besides the increase of biogas production, also increasing the ecological landscape management as well as protect permanent grassland from land use changes was a positive outcome of the project.

In order to reach these goals and to set up some new value chains and business plans for the digestion of grass residues, different types of information had to be collected and disseminated to the stakeholders involved. In the figure below an overview is given of the information collected in the project:

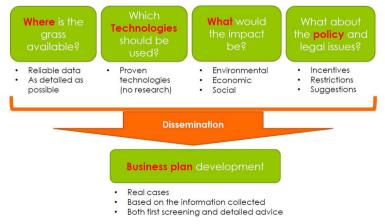


Figure 1: Overview of the information collected in the GR3 project

#### **PROJECTS OUTPUT**

The project's goal to inform involved stakeholders on the possibilities of grass digestion was done in different ways:

- A State of the Art technology overview was inventarised (available on www.grassgreenresource.eu)
- Reports on legal, ecological (LCA), social (job creation) and economical (CBA) studies were drafted
- Tools for predicting the profitability of grass digestion were designed
- A SWOT analysis on the digestion of grass was performed;
- Benchmarking Manuals were made for every group of involved stakeholders:
  - Managers of Natural areas / terrain managers
    - Managers of Rural areas / Local authorities
    - Biogas plant owners and operators
- Workshops were organised in every participating region
  - Including 2 technical workshops in Belgium and Italy
- Study tours were organised in Germany, Denmark and Belgium
- Final conferences were organised in participating member states
- All documentation is publicly available on the website

#### **RESULTS AND CONCLUSIONS**

Collecting all the information showed that there are quite some barriers that hinder the proper digestion of grass. For example, it is very difficult to assess the overall availability of grass residues – some region doesn't perform a proper collection of the grass residues (grass is left on site) what results in an overestimation of the available grass, while other regions don't have an adequate follow-up system, what resulted in an underestimation.

Also, the legal barrier for the digestion of grass residues is not to be underestimated – in some regions grass residues are considered waste, in other regions they aren't, what makes that the biogas plants in which they can be digested (agricultural vs. Industrial) is different. The uncertainty that is linked to this legal issue (waste or not?) should be resolved before heading towards broad scale grass digestion.

Nevertheless, at the end of the project, the project consortium could state that they supported the evaluation of more than 20 business cases what can be linked to the possible digestion of about 70 000-ton grass / year, and that about 25 000 ton of grass per year was to be digested at the end of the project. That latter number corresponds to a saving of primary energy of about 67 ton of oil equivalent (toe) per year, the production of 1670 toe/year of renewable energy and a reduction in greenhouse gasses of about 11 40 ton  $CO_2eq/year$ .

# PROJECT CONSORTIUM

pro|natura

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LNEG

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VENETO

# **BIOSURF - BIOmethane as SUstainable and Renewable Fuel**

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The objective of the BIOSURF project is to increase the production and use of biomethane (from animal waste, other waste materials and sustainable biomass), for grid injection and as transport fuel, by removing non-technical barriers and by paving the way towards a European biomethane market.

The project aims to:

- Analyse the value chain from production to use, based on territorial, physical and economic features (specified for different areas, i.e., biofuel for transport, electricity generation, heating & cooling);
- Analyse, compare and promote biomethane registering, labelling, certification and trade practices in Europe, to favor cooperation among the different countries and cross border markets based on the partner countries involved;
- Address traceability, environmental criteria and quality standards to reduce GHG emissions and indirect land-use change (ILUC), as well as to preserve biodiversity and to assess the energy and CO<sub>2</sub> balance;
- Identify the most prominent drivers for CO<sub>2</sub>-emissions along the value chain as an input for future optimisation approaches and to exchange information and best practices across Europe regarding biomethane policy, regulations, support schemes and technical standards.

Although biomethane used for transport falls under the RED, FQD and COM 2010/C 160/01, these documents do not provide the necessary details for handling upgraded biogas injected into the European natural gas network. The work within the BIOSURF project lead to the conclusion that a document issued by the European Commission would be required, while the issue cannot be resolved through separate national regulations. The core question is to recognise the European natural gas network as a single logistical facility with regard to injected biomethane. This approach would enable the cross-border transfer of volumes and mass balancing of biomethane in the European natural gas network. According to the concept elaborated by the BIOSURF consortium in cooperation with the existing national biomethane registries, a voluntary scheme is designed to perform the mass balancing of injected biomethane together with the transfer of sustainability characteristics attached to each consignment. **ERGaR**, the European Renewable Gas Registry, an association that seeks to establish and independent, transparent and trustworthy documentation scheme for mass balancing of biomethane distributed along the European natural gas network, was established in September 2016. ERGaR is completing its document package for the application to the European Commission for recognition under the

RED. Within this package, the functions and tasks for both the domestic and the European biomethane registries are set and the common rules are agreed.

The deliverables of the BIOSURF project contain the guidelines for the establishment of national biomethane registries, describe the documentation system for the issuance and exchange of European Biomethane Guarantees of Origin and several other issues related to the establishment of a European market for biomethane.

Since sustainable biomass is a limited resource, meta-analyses of respective potentials are crucial to pursue the right direction in the further development of the sustainable bioenergy sector. Three main biomass categories were of particular interest in the development of the survey report performed: animal waste (slurry and manure), other waste materials (municipal bio-waste and food/feed residues) and biomass residues (agricultural crop residues, byproducts from cultivation, harvesting and processing, residues from landscape maintenance and conservation, including pruning material and catch crops). The available amount of feedstock is of economic interest and the theoretical and technical feedstock potential was considered in national and international studies. Assuming that farmers and biogas plant operators are respecting "Cross Compliance" and "Best Practice" methods, dedicated energy crops as well as catch crops need to be part of a sustainable substrate portfolio for biogas production. In many countries (e.g. the UK and Italy), food waste is still landfilled because of missing separate food waste collection. An obligation for waste separation would help to tap this still unused potential and mitigate the other detrimental effects of landfilling biowaste.

The EU Commission has introduced sustainability criteria as part of the EU RED 2009/28/EC Directive (RED), including for example requirements regarding the GHG mitigation potential of biofuels. Some of the most important sustainability criteria at the European level, which are also used to evaluate the feedstocks for biomethane production, are: a) the mitigation of greenhouse gas emissions and b) the reduction of competition for land, food and feed resources, c) not reducing biodiversity and d) not changing land use if carbon stocks are reduced. Criteria are relating to: a) a general prohibition on the use of biomass from land converted from forests, other high carbon stock areas and highly biodiverse areas, b) a common greenhouse gas calculation methodology which could be used to ensure that minimum greenhouse gas savings from biomass are at least 35% compared to the use of fossil sources (rising to 50% in 2017 and 60% in 2018 for new installations) compared to the EU's fossil energy mix, c) the differentiation of national support schemes in favor of installations that achieve high energy conversion efficiencies and d) the monitoring of the biomass origins, taking into account Cross Compliance requirements for biomass cultivation.

Since the proof of fulfilment for these sustainability criteria has become a precondition for any promotion mechanism related to national biofuel quota systems, the individual calculation of GHG emissions has gained significant importance for biofuel producers as well as for certification schemes and auditors. The EU RED methodology defines the basic framework of the investigation by a clear definition of: a) the system boundaries (well-to-wheel), b) the allocation of by-products (based on the lower heating value of products and byproducts), c) the functional unit for the expression of the result calculated (per MJ biofuel), d) the life cycle impact assessment approach (GHG emissions), e) the characterisation factors for the conversion of greenhouse gases into CO<sub>2</sub>-equivalents, f) the reference value for the comparison and interpretation of the result.

EU RED Annex V includes three default values for biomethane (from municipal organic waste, from wet manure, from dry manure) which do not represent most of the existing biomethane concepts which are typically based on a combination of substrates.

According to EU RED 2009/28/EC, GHG emissions from the production and use of transport fuels (biofuels including biomethane) and bioliquids should be calculated as shown in Equation 1. According to this equation, the overall GHG emissions of a biofuel will be calculated considering both, the emissions from the various process steps (left-hand side of the equation) involved in its production and utilisation and the potential GHG-emission savings from different processes (right-hand side of the equation).

E = total emissions from the use of the fuel:				
$= e_{ec} + e_l + e_p + e_{td} +$	$e_u - e_{sca} - e_{ccs} - e_{ccr} - e_{ee}$			
GHG-Emissions from:	GHG-Emission savings from:			
$e_{ec}$ = the extraction or cultivation of raw materials $e_{I}$ = the carbon stock changes caused by land- use change	e <sub>sca</sub> = soil carbon accumulation via improved agricultural management e <sub>ccs</sub> = carbon capture and geological storage			
e <sub>p</sub> = processing	$e_{ccr}$ = carbon capture and replacement			
$e_{td}$ = transport and distribution	$e_{ee}$ = excess electricity from cogeneration			
e <sub>u</sub> = the fuel in use				

The overall calculation for the GHG emission factor of the main product biomethane can be completed as shown in Figure 1. The exemplary calculation illustrates the impact of an allocation of the upgraded digestate on the overall result of the main product biomethane. Without allocation of the digestate, the emissions associated with the production and distribution of the biomethane amount to  $26.5 \text{ gCO}_{2\text{equi}} \text{ MJ}^{-1}$ . Including the byproduct digestate into the calculations allows to allocate a small amount of those emissions to the byproduct and thus, reduces the emissions associated to the biomethane productions to  $19.8 \text{ gCO}_{2\text{equi}} \text{ MJ}^{-1}$ .

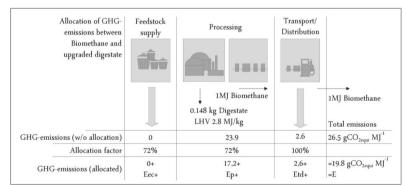


Figure 1: Exemplary GHG emission calculation including by-product allocation

In the context of the EU RED framework, when the biomethane produced from such waste materials or residues should be used as a transportation fuel, it is the question how these mitigation effects can be included in the calculation of the GHG mitigation potential of biomethane. Thus, the necessity for the calculation of individual values is typically higher for biomethane producers that are willing to sell their product as a transportation fuel.

Calculations on the GHG mitigation potential of manure (the most important agricultural byproduct) were performed during the project. The entire manure production, potentially available in the EU for manure processing, distributed in Member States, is estimated with 1.4 billion ton. In order to analyse the potential GHG savings from EU's manure production the first step was to gather numbers on the amounts of livestock in each EU Member State. Animal numbers were collected for the following: dairy cow (4500 kg milk/a), dairy cow (6500 kg milk/a), cattle < 1 year, cattle > 1 year, swine: breeding sows, swine: fattening pigs, horses, chicken (av. of hens and broilers), poultry (av. of turkeys, ducks, gooses, etc.). As a next step, the terms for different animal categories each, for calculating the emission factor (see Equation 2) were gathered from national values which are provided by Member States in their National Inventory Reports NIR.

#### Equation 2: Emission factor from manure management according to IPCC chapter 4 and 10

EQUATION 10.23  
CH<sub>4</sub> EMISSION FACTOR FROM MANURE MANAGEMENT  

$$EF_{(T)} = (VS_{(T)} \bullet 365) \bullet \left[ B_{o(T)} \bullet 0.67 \ kg \ / \ m^3 \bullet \sum_{S,k} \frac{MCF_{S,k}}{100} \bullet MS_{(T,S,k)} \right]$$

Where:

 $EF_{(T)}$  = annual CH<sub>4</sub> emission factor for livestock category T, kg CH<sub>4</sub> animal<sup>-1</sup> yr<sup>-1</sup>

 $VS_{(T)}$  = daily volatile solid excreted for livestock category T, kg dry matter animal<sup>-1</sup> day<sup>-1</sup>

365 = basis for calculating annual VS production, days yr-1

 $B_{o(T)}$  = maximum methane producing capacity for manure produced by livestock category *T*, m<sup>3</sup> CH<sub>4</sub> kg<sup>-1</sup> of VS excreted

 $0.67 = \text{conversion factor of m}^3 \text{ CH}_4 \text{ to kilograms CH}_4$ 

MCF(S,k) = methane conversion factors for each manure management system S by climate region k, %

 $MS_{(T,S,k)}$  = fraction of livestock category T's manure handled using manure management system S in climate region k, dimensionless

Using the emission factor described in Equation 2, on the one hand the real emissions of the mentioned animal categories per EU Member State were calculated. On the other hand, the potential energy that could be provided by anaerobic digestion of manure and the potential GHG emission savings per EU Member States were calculated. The calculation for potential GHG emission mitigation assumes that one third (33.3%) of the total amount of manure could be

realistically treated within the European Union. This way, a total of 56 Mio tons  $CO_{2equ}$  could be saved.

Additionally, the calculation of the GHG emissions of the six biomethane value chains were performed: a) biomethane produced from slurry, b) biomethane produced from straw, c) biomethane produced from a mixture of slurry and straw, d) biomethane produced from municipal organic waste, e) biomethane produced from a mixture of silage corn stover and catch crops, f) biomethane produced from a mixture of maize silage and catch crops. For each of the six pathways, a set of "scenarios" with different assumptions regarding the methodology have been calculated: a) no consideration of digestate and slurry credit in the calculations, b) internal use of digestate for fertiliser supply, c) digestate allocation, d) digestate credit, e) slurry credit min/max. Comparing all pathways considered in this report, the results range from 51% GHG mitigation (pathway based on maize silage and catch crops; ignoring the co-product digestate) to 202% GHG mitigation (pathway based on 100% slurry; digestate allocation and slurry credit max.).

The BIOSURF project has identified two main issues that can occur when applying the RED calculation methodology for individual calculations of biomethane production and use: a) allocation of the digestate from biogas/biomethane production because the biorefinery definition has been shown to be applicable to biomethane facilities (including the separation process) and b) emission savings from the fermentation of agricultural byproducts and organic residues and wastes. Such avoided emissions from can be included in the calculations under the term  $e_{ec}$ , the extraction or cultivation of raw materials. The magnitude of these effects can provide significant GHG mitigation potentials for biomethane based on slurry/manure.

For further results of the BIOSURF project, please go to the project website: <a href="http://www.biosurf.eu/en\_GB/downloads-and-deliverables/deliverables/">http://www.biosurf.eu/en\_GB/downloads-and-deliverables/</a>

#### IMPACTS OF THE BIOSURF PROJECT

Through its various actions, the results of the BIOSURF Projection will contribute both to proofing the sustainability of the biogas process when using sustainable substrates and also promoting the use of biomethane in whole Europe which will be made possible also via the European Renewable Gas Registry ERGaR.

#### **PROJECT PARTNERS**

- Istituto di Studi per L'Integrazione dei Sistemi Scrl (ISINNOVA) <u>http://www.isinnova.org/</u>
- European Biogas Association (EBA) <u>http://european-biogas.eu/</u>
- Arge Kompost Und Biogas Osterreich Verein (AKB) <u>http://www.kompost-biogas.info/</u>
- AGCS Gas Clearing and Settlement Ag (AGCS) <u>http://www.agcs.at/de</u>
- Cib-Consorzio Italiano Biogas E Gassificazione (CIB) <u>https://www.consorziobiogas.it/</u>
- Fachagentur Nachwachsende Rohstoffe E.V. (FNR) <u>https://international.fnr.de/</u>
- Magyar Biogaz Egyesulet (HBA) <u>http://www.biogas.hu/2/frameset</u>
- DBFZ Deutsches Biomasseforschungszentrum Gemeinnuetzige GmbH (DBFZ)
   <u>http://www.biosurf.eu/en\_GB/partners/#DBFZ</u>
- Association Technique Energie Environnement (ATEE), Club Biogaz <u>http://atee.fr/biogaz/</u>

- Renewable Energy Association Lbg (REA) <u>http://www.r-e-a.net/</u>
- Fachverband Biogas Ev (GBA) <u>http://www.biogas.org/edcom/webfvb.nsf/ID/DE\_Homepage</u>

#### **PROJECT DETAILS**

BIOSURF, a Horizon 2020 project funded by the Innovation and Networks Executive Agency (INEA) started in January 2015 and will run until December 2017. This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 646533.

PROJECT PARTNER COUNTRIES: Austria, France, Germany, Hungary, Italy, UK

**PROJECT COORDINATOR**: Stefano Proietti, Istituto di Studi per L'Integrazione dei Sistemi Scrl (ISINNOVA), <u>sproietti@isinnova.org</u>

WEBSITE: http://www.biosurf.eu/

# ERGaR – The European Renewable Gas Registry

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The European Renewable Gas Registry (ERGaR) was established on the 28<sup>th</sup> of September 2016, during the conference of the European Biogas Association in Ghent, Belgium. The association has been founded by nine members, representing national biomethane registries, biogas associations, and major biomethane industry players. In the meantime, 4 new members have joined and the interest towards this initiative is increasing. The aim of the association is to put in place the EU registry for renewable gases, like biomethane, once the voluntary documentation scheme for mass balancing of biomethane is approved by the European Commission.

The biomethane market is a steadily-growing in Europe. The number of biomethane plants in Europe increased significantly from 367 to 459 in 2015 (+25%), as shown in Figure 1. There are still many European countries that do not have any upgrading units installed. According to the data received from National Biogas Associations, 15 countries use upgrading techniques to produce biomethane. Germany has 185 biomethane plants and is therefore market leader for biomethane, as for biogas. The other 14 countries have a total of 274 biomethane plants. The two-digit growth of this sector maintains the trend established in previous years.



Figure 1: Evolution of the number of biomethane plants in Europe

The main purpose of the new association is to operate an EU-wide biomethane registry under the name European Renewable Gas Registry (ERGaR) non-profit international association (aisbl). Building on national registries the documentation system enables cross-border trade of renewable gases via the European natural gas network while preventing double sale and double counting. One important precondition is to establish an independent, transparent and trustworthy documentation scheme for mass balancing of biomethane distributed along the European natural gas network. The injected biomethane gets blended with natural gas in the pipelines and the methane molecules of fossil and renewable origin cannot be differentiated anymore. Hence, mass balancing with appropriate documentation is the only feasible solution to track the renewable fuel (biomethane) in the natural gas system.

The analytical preconditional work for setting up ERGaR has been carried out in the frame of two EU projects:

- Green Gas Grids (www.greengasgrids.eu) an IEE project
- BIOSURF (www.biosurf.eu) a Horizon 2020 project

The European Biogas Association played a leading role during these projects but also beyond their duration. The founding of the ERGaR was possible thanks to EBA efforts in summarising the relevant outcome of those projects, mobilising stakeholders and gathering political support for this approach. EBA members and EBA staff are supporting the establishment and the activities of ERGaR substantially.

The major task of a domestic biomethane registry is to generate confirmations for upgraded and injected biogas. This is done by so called Guarantees of Origin (GoO) which serve as a proof of quality and quantity of the produced biomethane volume. The registry has a responsibility towards participants of being a neutral and trustworthy settlement agency of GoOs. More specifically these responsibilities entail necessities such as registration process and preparation of a unified platform for the settlement process within the registry. This also includes the generation, inspection and transfer of GoOs when necessary. The biomethane registry should be an electronic account based system for the registration of biomethane quantities fed into the gas network, allowing the generation of a corresponding GoO, as well as the transfer of GoOs between registered account holders and subsidy agencies, among other institutions.

In the spirit of the RED the term "European Biomethane Guarantee of Origin (EBGoO)" is defined as the following: "EBGoO is an electronic dataset including information and attributes related to a specified biomethane consignment injected into the European natural gas network". European Biomethane Guarantees of Origin (EBGoOs) have a central role in virtual transfer of biomethane consignments along the European natural gas network.

The important first task of the ERGaR association is to elaborate the detailed procedures for performing the mass balancing of biomethane injected into the European natural gas system and to apply for the recognition of the voluntary scheme by the European Commission in accordance with the Renewable Energy Directive, Fuel Quality Directive and other related legislative documents.

#### **IMPACTS OF ERGAR**

Cross-border biomethane trade across Europe should be made possible via the European Renewable Gas Registry ERGaR. The designed mass balancing system is based on the recognition of the natural gas system operating on the European territory as a closed logistical facility. The application of a mass balance system allows the intrinsic "bio" value of the exported biomethane to be transferred without explicitly tracking the physical cross-border movements, widening the potential consumers besides the country of production.

Looking ahead, ERGaR expects other organisations (such as national biomethane registries, national biogas associations, natural gas industry partners, other stakeholders in the European biogas and biomethane industries) to join and to participate in the development of a documentation system applicable all over Europe.

#### **ERGAR MEMBERS**

- AT AGCS Gas Clearing & Settlement AG
- BE European Biogas Association (EBA)
- CH Swiss Association of Gas Industry (VSG)
- DE German Energy Agency (dena) Landwärme GmbH
- DK Energinet.dk NGF Nature Energy
- FR Gas Réseau Distribution France (GrDF)
- IT Consorzio Italiano Biogas (CIB)
- NL Vertogas STX Services B.V.
- UK Renewable Energy Assurance Ltd. (REAL)

#### DETAILS

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# GoBiGas – First Full-Scale Demonstration of Biomethane from Forest Residues

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#### SUMMARY

The Gothenburg Biomass Gasification (GoBiGas) project is a large demonstration of utilising forest residues for the production of a renewable fuel, in this case biomethane. The €164 million investment may unlock a substantial market and complement the valuable volumes of waste-based biogas from anaerobic digestion for transportation, industry, CHP and heating. The plant produced its first biomethane in December 2014 and succeeding in proving it possible to continuously use wood pellets over a period of two months. The plant is now expanded with a feeding system for wood chips in order to access less expensive renewable resources, while development of producing biomethane from wood chips continues.

#### PURPOSE AND APPROACH

Many sectors in Sweden, including power generation and heating, have moved from fossil fuels to more climate friendly alternatives since the 1970's. In contrast, transportation in Sweden is still almost entirely reliant on fossil fuels.

Biomethane is commonly produced today in many European countries by upgrading biogas from anaerobic digestion plants, where a variety of substrates (such as sewage sludge, food waste, manure and energy crops) are used. Waste-based biomethane is an excellent energy source, which can be used to generate either heat and power, vehicle fuel or to replace natural gas in industrial processes, with unparalleled environmental benefits. The potential for biogas from waste through anaerobic digestion is nonetheless relatively limited. In Sweden, this potential is estimated at 10 % of the current use of fuel in road transportation.

The potential in residues from the forestry industry is approximately six times larger in Sweden, but the high content of lignocellulose in forest residues makes it unsuitable for anaerobic digestion. Gasification as a production process is an alternative pursued by many renewable fuel options. One key aspect for each process route is the overall efficiency of the process, resulting in as much renewable energy as possible from the resource base. GoBiGas aims at proving an exceedingly high efficiency rate of transforming 65 % of the energy contained in the forest residues into high value biomethane. By utilising the plant's waste heat in the district heating grid of Gothenburg, the total energy efficiency of the plant will reach 90 % or more.

The forestry industry in Sweden and other countries is struggling, primarily due to the decreased use of paper in society. GoBiGas will prove a new opportunity of the industry to improve its competiveness and broaden its end markets.

Politicians will appreciate the development of a new technology that can provide society with a clean-burning fuel, derived efficiently from waste with the potential of replacing large amounts

of fossil fuels in the transportation sector. Åhman and Nilsson (2008) estimate that biomethane from forest residues can constitute 50 % of our need for fuel in the transport sector in 2030.

Biomethane is readily available in most parts of the southern half of Sweden and the fuel from GoBiGas can potentially be reached at any CNG filling station in Sweden. Due to the nature of methane, the biomethane from GoBiGas can potentially be sold to any European country on the natural gas grid. The market is currently under-developed and national agencies will have to work together to enable trade in biomethane over national borders, where the European Biogas Association's ERGaR project may lead the way.

#### THE SCIENTIFIC INNOVATION AND RELEVANCE

The production process at GoBiGas includes a number of stages, of which most are proven technology but the combination and application has been specifically developed for the project. The gasifier is fluidized with superheated steam and indirect gasification. In the gasifier, the fuel is converted into a so-called "synthesis gas". The heat required for the gasification reactions is supplied by the steam and hot bed material circulating between the gasification reactor and a combustion reactor. The heated bed material from the combustion reactor is recycled to the gasification reactor where it supplies heat to the gasification. The temperature in the gasification reactor is approximately 850 °C and in the combustion reactor about 930 °C.

After cooling the product gas is filtered in a bag house filter before being fed to a tar removal scrubber, where the gas is washed with bio-diesel and cooled to about 40 °C. After this the gas is further cleaned in several reactors filled with activated carbon and different kinds of catalytic material. The gas is washed in an amine wash where the  $H_2S$  is removed together with a large part of the  $CO_2$ . The almost  $CO_2$ -free gas is fed to four catalytic adiabatic methanation reactors connected in series. After the last methanation reactor the gas is dried to achieve a dew point low enough to allow feeding of the gas to the natural gas grid. The drying is done by adsorbing the water on molecular sieves. The produced biomethane is fed to a 35 bar transmission grid for natural gas.

The GoBiGas project aims at proving that biomethane can efficiently be produced in large amounts utilising forest residues. As the initial process has been proven, pellets are being substituted by chipped forest residues, which will bring down costs. By successfully running the plant 8,000 hours per year, 160 GWh of biomethane will be injected into the natural gas grid annually. This is enough to fuel 16,000 cars that drive 17,000 km/year. There are approximately 175,000 cars registered in the City of Gothenburg.

In addition, approximately 50 GWh of heat will be transferred to the district heating grid of Gothenburg. This is enough to heat the equivalent of 10,000 apartments. This increases the overall efficiency of the GoBiGas project and displaces the need for biomass in other heat generating plants.

When used as a fuel in transportation, the biomethane from GoBiGas will reduce the emissions of  $CO_2$  by 40,000 tons/year (according to the RED methodology).

The project will be evaluated over six years (2014-2019) in cooperation with its suppliers and report its results to the Swedish Energy Agency.

The project is highly replicable, and other projects are already underway (e.g. ENGIE's GAYA). Large amounts of forest residues are available across Europe, especially in Northern and Eastern Europe. Biomethane is easily and efficiently distributed throughout Europe through the existing natural gas grid. In order to realise this potential, additional countries must allow for the introduction of high-quality biomethane into the grids and national authorities must cooperate to enable cross-border trade of biomethane in these grids.

Natural gas and LNG is growing as alternative to fuel cars, trucks and busses and LBG-production is both supported by this growth but also offers an alternative to the fossil form of methane. The GoBiGas project goes hand in hand with the European Commission's expressed interest of quickly expanding the use of methane as a transportation fuel by 2020.

The project is the most ambitious demonstration ever attempted for efficiently producing a renewable fuel from forest residues. Previously tested in smaller scale for short periods, GoBiGas builds on these experiences to prove the technology at industrial scale, being the last step before commercialisation. This unique €135 million demonstration is an impressive attempt by a municipal energy company to make substantial progress toward a more sustainable future for Europe.

Joaquín Almunia (EU Commission Vice Chairman in charge of competition matters) is quoted saying, "The project is in line with the objectives for research and development, climate change and energy that the EU has mapped out for 2020. It is a question of a particularly innovative project."

Kyriakos Maniatis (DG ENER) says: "GoBiGas is a unique pioneer and a platform for developing the new advanced biofuels Europe so desperately needs."

The GoBiGas project also brings together a number of companies from different European countries in a cooperative project, which will further develop the Union's competitiveness in the field of renewable fuel production.

#### **RESULTS AND CONCLUSIONS**

Using the Wells-to-Wheels methodology developed by the Institute of Energy and Transport (one of the EU's Joint Research Centers), GoBiGas may become an example of excellence. Biomethane from the plant is estimated in resulting in 7-10 g CO<sub>2eq</sub>/MJ depending on when using pellets or chipped residues, which is far better than almost all alternatives. As biomethane is already distributed in natural gas grids in several EU countries and the use of methane in vehicles is quickly increasing in Europe as well, biomethane can be viewed as a drop-in fuel easily used by many current and a majority of future consumers. Of course, the biomethane from the GoBiGas project will also fulfil the sustainability requirements set out for renewable fuels in the Renewable Energy Directive.

Since its inception, the GoBiGas project has overcome several obstacles. Initially, production was halted when unexpectedly high levels of tars were detected in the synthetic gas. This made subsequent methanation impossible. With the assistance of researchers at the Chalmers University of Technology, a method was developed to activate the olivine sand in the gasifier in order to inhibit the formation of tars. This was an important step forward and will be a valuable experience for future gasification plants.

Following this achievement, producing the final methane product became closer and closer to be becoming a reality. Additional achievement in screw feeding, gasification chemistry and other areas enabled the project to successfully produce biomethane in December 2014. The biomethane was injected into the transmission grid for Natural Gas.

During 2015, 29 GWh of biomethane were produced at GoBiGas and injected into the gas grid. The majority of the biomethane was sold to the transportation sector making a 74 % reduction in climate emissions, according to the RED methodology.

During 2016, a new feeding system for wood chips has been constructed and new obstacles and achievements materialize on an almost daily basis.

# Recovery and Use of Nutrients, Energy and Organic Matter from Animal Waste (ReUseWaste)

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#### INTRODUCTION

Global livestock production has increased dramatically on a global scale, and changes in production systems have resulted in increased pollution of air, aquifers, surface waters and soil. A major sustainability concern is also the uncoupling of the sites of animal feed production and animal production, through the (mainly economic) driving forces specialisation, intensification and up-scaling. This leads to surplus amounts of animal manure in areas where livestock are produced, often in excess of what can be utilized for crop fertilisation (Sommer et al., 2013). Evidently it is necessary for the agricultural livestock industry to develop new environmental technologies to meet global challenges related to environmental impact and sustainability.

The *ReUseWaste* training network (funded by the Marie Curie program under EU-FP7) was therefore deliberately established with partners from some of the most livestock intensive regions of Europe in order to address this challenge. The objective of the *ReUseWaste* training network was to educate young scientists to rethink current, established manure management systems and apply new technology for improved and sustainable utilisation of the valuable organic matter and nutrient resources in manure. Therefore, the network has had a very strong focus on the training and career development of young scientists conducting relevant research within this field, who can then provide knowledge about and develop new technologies leading to improved soil, water and air quality.

#### PROJECT CONTENTS AND TRAINING APPROACH

*ReUseWaste* was structured in eight work packages to ensure a strong and committed focus on the key areas. Two work packages targeted scientific and complimentary training activities, respectively, whilst five work packages were dedicated to scientific research activities. These covered aspects pertaining to 1) manure characterisation, 2) treatment technologies and management, 3) energy and nutrient recovery, 4) land recycling and 5) synthesis of this knowledge across the manure chain. A separate work package was for overseeing management and dissemination activities of the network. Further project info can be found at <u>www.reusewaste.eu</u>

The *ReUseWaste* project reached its completion in early 2016 after a period of four years. The first phase of the project involved the initiation, establishment and consolidation of the *ReUseWaste* consortium such as setting up the management framework which could serve as the foundation for coordinating project activities, particularly recruitment of the 13 *ReUseWaste* fellows. These fellows formed the backbone of a training project such as *ReUseWaste* and

therefore considerable attention was given to finding ideal candidates following an extensive recruitment campaign.

The *ReUseWaste* training program, which all the fellows have followed, offered a multitude of different activities all aiming to improve their scientific and generic competences and to enhance their career prospects. The fellows' all developed and maintained individual research and career development plans to formulate their training needs and research objectives – these have at the end of the project been updated and developed further into career development plans for each fellow.

During the course of the training program, all fellows also embarked on a range of tailor made secondments together with partner organisations from the network. The secondments have served as an excellent opportunity for fellows to carry out joint research activities with other fellows from the network, but also gave them opportunity to acquire experience and training in a multitude of areas. In this way secondments have offered fellows a broader outlook and have developed their CV's and hence improved career prospects. Other highlights of the *ReUseWaste* training program have been the study-tours held in conjunction with the half-annual supervisor-fellow meetings. The study tours have given the fellows an exciting opportunity to observe technologies implemented in real life and to communicate with farmers, advisors, engineers and managers from the private sector, whilst the annual consortium meetings have continually served as an ideal opportunity to galvanise the collaborative spirit of the fellow group and to create joint learning and synergy.

#### **RESULTS AND OUTCOMES**

A measure of the outcomes of the *ReUseWaste* training program is the number and diversity of dissemination activities produced by the project. By the end of the project period in early 2016, *ReUseWaste* had produced 19 peer-reviewed scientific papers (in press or published), whilst a year later (Apr 2017) an additional 23 papers have been published or are in press, with further 10 manuscript currently in preparation or review, so a total of more than 50 peer-reviewed research papers will result from the project. *ReUseWaste* research has also been represented in 36 instances of conference presentations (poster or oral). Furthermore, the project has produced 25 research briefs, 16 newsletters and participants have been involved in more than 60 outreach activities.

Examples of individual scientific highlights of the research activities include:

- Fourier-transform mid-infrared photoacoustic spectroscopy was found to be a technique that can be successfully used for characterising very dark and opaque samples and potentially to replace various assays that are used for the determination of the usefulness of various organic wastes (Bekiaris et al. 2015 a,b,c, 2016; Bruun et al. 2017)
- The combined approach of slurry acidification and separation was found to drastically decrease harmful gaseous emissions whilst increasing the nutrient value of slurries (Regueiro et al. 2016a,b,c,d).
- Membrane technologies for solid-liquid separation of farm effluents were found to be a suitable technique for concentration of farm effluents and digestates, and membrane fouling could be mitigated (Camilleri-Rumbau et al. 2015, 2016).

- Successful gasification of poultry litter and pig manure can be done in updraft gasifiers; the data produced were furthermore used for waste gasification modelling using various approaches (Taupe et al. 2016; Pandey et al. 2015, 2016 a,b).
- The treatment of slurry by mechanical separation and additives can be utilised as a strategy to enhance recycling of slurry nutrients after field application or for utilisation in anaerobic digestion (Popovic et al. 2014; Owusu-Twum et al. 2017; Phuong et al. 2016).
- A combined acidification and drying treatment of solids from mechanical separation of animal slurry or digestate has been shown as effective for reducing ammonia emissions and increasing the fertilizing value of the final product, thus reducing the mineral fertilizing dependency of the agricultural sector (Pantelopoulos et al. 2016 a,b).
- Composting of solids from animal slurry separation with crop residues (cotton gin, maize stalks, green waste) was shown to be another promising option for upgrading the manure to a stable and more valuable fertiliser, with high C sequestration potential (Santos et al. 2016, 2017)
- Manure biochar showed completely different behaviour (in relation to soil GHG emissions, nutrient availability and crop growth) than standard wood biochar. Standard biochars were found not effective for reducing ammonia emissions from pig slurry (Subedi et al. 2015, 2016a,b, 2017)
- Nitrogen and GHG emission coefficients at each stage of the entire manure management chain have been determined and analysed. Lowering the protein content of feed and acidifying slurry are strategies that consistently reduce ammonia and GHG emissions in the whole livestock production chain (Hou et al. 2015, 2016, 2017 a,b).
- A stakeholder study revealed a high potential for farmer's increased use of organic wastes and treatment technologies, but also identified barriers and advantages perceived by farmers (Case et al. 2017).

The training was finalised by a Joint Scientific Workshop, "FIRe - *Innovative strategies to improve the recycling of energy, nutrients and organic matter from waste materials*", held together with several other related EU projects and networks (FIRe: FertiPlus, INEMAD, ReUseWaste and Biorefine Cluster Europe) in May 2015. For fellows, the FIRe event served as a training and dissemination event, and offered a chance for them to present and expose their research results to others and potential new employees, whether in the research community or private sector. The FIRe event was concluded in a plenary session focusing on key barriers and challenges to enhance organic waste recycling and a discussion of central future research priorities in the field. This concluded that we should focus on further exploring the potential of i) promising technologies such as manure acidification or separation for reducing emissions, ii) technology combinations, including mixes of co-substrates for energy and fertiliser production, iii) new analytical methods for prediction of important biological characteristics and potential value of waste biomasses, iv) identifying what the best scales are for implementation of various technology types in different contexts, and ultimately v) what types of incentives (economic and regulatory) are necessary and feasible to enhance organic waste recycling in Europe.

#### CONCLUSIONS

The core objective of the *ReUseWaste* project was the education of 13 young scientists to rethink current established manure management systems and develop new technologies for improved and sustainable utilization of the valuable organic matter and nutrient resources in manure. We feel that the project has fulfilled this objective. Their exposure to the comprehensive training and research program has paved the way for promising future research careers for all fellows, for the benefit of Europe. The reported scientific highlights of fellows' research activities, several also in collaboration with associated industrial partners, indicate that *ReUseWaste* has provided significant benefits for European research and agri-environmental technology sector.

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# II. Extended Abstracts – Research in Motion

# **Grass Supply Chains for Biogas Production**

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#### OBJECTIVE

The utilisation of energy crops for biogas production by anaerobic digestion can compete with food production and exerts a strong pressure on the environment (Basso et al., 2016). Consequently, alternative biomass sources, such as grass from non-cultivated areas, should be taken into consideration as an integrative feedstock for the biogas supply chain (Balimunsi et al., 2012).

Although today grass is not a common feedstock in the anaerobic digestion process, according to some authors (Prochnow et al. 2009; De Moor et al. 2013), it has good characteristics and it could be an integrative biomass in the biogas process.

However, one of the main problems that prevent its exploitation is the lack of appropriate technologies that could make its recovery sustainable under economic (Boscaro et al., 2015) and energy aspects (Boscaro et al., 2017). In particular, due to its low energy content, the logistic operations of grass seem to be one of the most important factors to solve for an energetic valorisation of such biomass (Athanasios et al., 2009; Pezzuolo et al., 2016).

The aim of this study is to evaluate different possible approaches to the logistic supply chain of grass harvested in riverbanks, focusing into its economic and energy aspects.

#### MATERIALS AND METHODS

#### Logistic systems

Three different logistic approaches for the supply of anaerobic digestion plants have been proposed and evaluated at four different supply distances from the biogas plant (5, 10, 20, 30 km), according to the mowing and harvesting systems proposed by Boscaro et al. (2015) (Figure 1).

In the scenario "a" (direct transport chain) and "b" (interrupted transport chain) the grass is managed as loose product. For this reason, a low transport density (assumed of  $180 \text{ kg/m}^3$ ) characterizes the grass.

On the other hand, in scenario "c" (transport in round bales), thanks to the compression action of the round-baler, grass presents a higher density (assumed of 330 kg/m<sup>3</sup>) with, as consequence, a reduction of the transport volumes.

For scenario "b", the distance to the temporary storage place was assumed to be of 1 km on average. A following loading operation in a trailer with a higher transport capacity is planned. Each system, agreeing to surveys among Italian operators, involves one or more tractors in order to complete different operations. In the present study, the following assumptions have been formulated:

- Scenario "a": Tractor 88 kW + Self Loading Wagon (25 m<sup>3</sup>)
- Scenario "b": Tractor 88 kW + Self Loading Wagon (25 m<sup>3</sup>); Tractor 60 kW + Hayfork loader; Tractor 100 kW + Trailer (40 m<sup>3</sup>)
- Scenario "c": Telehandler 73 kW; Tractor 100 kW + Trailer (40 m<sup>3</sup>)

The average transport speed was assumed, considering the Italian roads conditions and legislation, of 13 km/h.

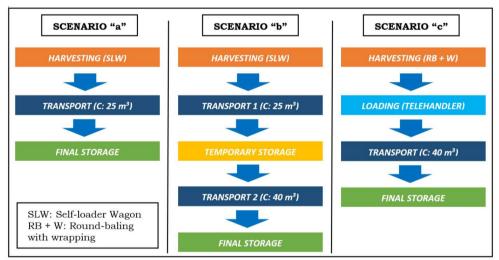


Figure 1: Logistic scenarios

Economic analysis

The economic analysis has been performed providing an economic value for each operation executed in every system. The unit costs per tons of fresh matter (f.m.) of the logistic process for the grass was calculated according the following equation:

 $C = \sum Su / C_t$ 

C= Unit cost of the operation (€/t f.m.)

 $\Sigma$  Su= Sum of the hourly costs of the tractors and equipment involved in the operation ( $\epsilon/h$ ) Ct= Transport capacity (t/h)

The hourly costs of machineries were computed according to the ASABE procedures (ASABE 2011), considering the purchase costs of the Italian price lists.

On the basis of previous experiments carried out in Germany (Pick et al., 2012), grass yield was assumed to be 6 t/ha (fresh matter) per cut, with a 25% dry matter content.

#### Energy balance

The energy comparison was evaluated based on the gross energy demand method (Slesser and Wallace 1981; Sartori et al. 2005; Pezzuolo et al. 2014), also including the energy value related to labour (Pezzuolo et al. 2017) (Table 1)

#### Table 1: Average energy content of the inputs required

Inputs	Energy required	Sources
Fuels (MJ/kg)	50.23	Biondi et al. (1989)
Oils (MJ/kg)	78.13	Carillon (1979)
Labour (MJ/h)	1.93	Pimentel & Pimentel, (1979)
Tractor (MJ/kg)	80.23	Hornacek (1979)

#### **RESULTS AND DISCUSSION**

Economic analysis

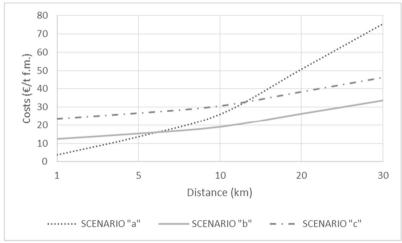


Figure 2: Economic analysis of different logistic scenarios

According to the economic analysis, the direct transport allows a reduction of transport costs in the case of short distances (Figure 2). However, the scenario "a" costs tend significantly to increase for long distances. Conversely, the interrupted transport chain appears to be more

convenient in the case of distances higher than about 6 km, with a tendency to keep the costs more stable than in the case of direct transport chain. On the other hand, the scenario "c" (transport in round bales) is never energetically advantageous compared to interrupted transport chain. In fact, the major costs due to the recovery of the product by telehandler negatively influence its economic balance.

#### Energy Balance

According to the energy balance, the direct transport chain requires the highest energy inputs (Figure 3). Indeed, the lower transport capacity negatively impacts on energetic efficiency. Conversely, the interrupted transport chain and the transport in round bales are the systems with a higher energy efficiency: their energy requirements are almost equal, and markedly lower than the direct transport chain.

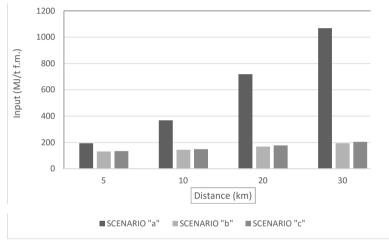


Figure 3: Energy balance of different logistic scenarios

#### CONCLUSIONS

Three different approaches of the logistic of grass for the feeding of anaerobic digestion plants have been evaluated under economic and energy aspects.

The results show that, under economic aspects, the direct transport chain seems to be the most convenient solution for the management of such material in short distances; conversely, for longer distances, the best solution appears to be the interrupted transport chain. The transport of grass in round bales always appears disadvantageous under economic aspects with respect to the interrupted transport chain.

On the other hand, the energy balance clearly highlights the higher efficiency of the interrupted and round bales transport scenarios while the direct transport system noticeably requires higher energy inputs.

In order to improve the performances of the interrupted transport chain, further investigations could be carried out to reduce the transport volumes in this system. Indeed, from preliminaries examines the reduction of volumes could reduce costs by nearly 30%.

#### ACKNOWLEDGEMENTS

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# Bioaugmentation of the thermophilic anaerobic biodegradation of lignocelluloserich substrates

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#### SUMMARY

Two stable, thermophilic mixed cellulolytic consortia were enriched from an industrial scale biogas fermenter. The two consortia, marked as AD1 and AD2, were used for bioaugmentation in laboratory scale batch reactors. They enhanced the methane yield by 22-24%. Next generation DNA metagenome sequencing revealed the predominant strains were *Thermoanaerobacterium thermosaccharolyticum*, *Caldanaerobacter subterraneus*, *Thermoanaerobacter pseudethanolicus* and *Clostridium cellulolyticum*. The effect of these strains, cultivated in pure cultures, was investigated with the aim of reconstructing the defined cellulolytic consortium. The addition of the four bacterial strains and their mixture to the biogas fermenters enhanced the methane yield by 10-11% but it was not as efficient as the original communities indicating the significant contribution by members of the enriched communities present in low abundance.

#### INTRODUCTION

Biogas production is one of the sustainable alternative technologies with the considerable benefit of being able to generate useful energy carrier from various raw materials of biomass origin including plants and plant residues. Biogas is formed anaerobically and it mainly consists of methane (55–70%) and carbon-dioxide (30–45%) (Tsavkelova et al., 2012). Various lignocellulose containing biomasses can be used for biogas generation, such as energy crops, agricultural and forestry residues, municipal and industrial wastes (Ho et al., 2014). The main component of plant tissues is lignocellulose. Lignocellulose is comprised of three types of biopolymers, i.e. cellulose, hemicellulose and lignin (Kovács et al., 2014). A family of various hydrolytic enzymes are needed for the complete decomposition of cellulose (Lee et al., 2002). Because of its recalcitrance, pretreatment techniques are frequently applied. Pretreatment methods can be physical (grinding, steam-explosion, liquid hot water, extrusion, irradiation), chemical (alkaline or acid pretreatment, wet oxidation, ozonolysis, oxidation by peroxides, ionic liquids), biological (fungal, microbial consortium, enzymatic) or combined pretreatment (Kovács et al., 2014). Bioaugmentation is a promising method to introduce adequate microorganisms into the biogas producing system and facilitate the target substrate degradation. Pure cultures or consortia have been tested (Kovács et al., 2014).

In the present study, two stable, thermophilic cellulose degrading bacterial consortia have been enriched separately from the same inoculum sludge. The composition and relative abundances

of the bacterial strains comprising the enrichment cultures were determined by Ion Torrent<sup>™</sup> whole genome DNA sequencing and the consortia and their most abundant components were tested for their bioaugmentation potential in laboratory AD fermentations.

#### MATERIALS AND METHODS

#### Experimental setup

#### Enrichment

The first enrichment step was carried out in 500 mL glass vessels. The vessels were filled with 400 mL sludge from a full-scale biogas plant operating at thermophilic temperature (55 °C) and were fed with  $\alpha$ -cellulose or glucose as the sole carbon source. The initial 1 g/L weekly substrate supply was increased gradually to 10 g/L in 10 weeks and then set at 6 g/L because of the elevated volatile fatty acid concentration. In the second enrichment phase the culture from the first phase was used as inoculum. In this set of experiments, the reactors contained 190 mL of inoculum and 10 mL of distilled water or *Caldicellulosiruptor saccharolyticus* culture (1.8x10<sup>7</sup> CFU/mL), respectively. The weekly substrate addition was 4 g/L of  $\alpha$ -cellulose.

To investigate the bioaugmentation effects of the enriched consortia and the pure cellulolytic strains 125 mL serum vials were used. The volume of the liquid phase was 60 mL comprising 50 mL inoculum sludge from the acclimated digestate from the industrial scale mesophilic reactor and 10 mL of the enriched consortium (2.6 x10<sup>8</sup> cells/mL) or sterile distilled water, which was later changed to 54 mL sludge and 6 mL (10 v/v%) of AD1 or AD2 consortium or pure bacterium culture (4.5 x 10<sup>8</sup> cells/mL), respectively. Controls received the indicated amount of sterile water.

In these experiments corn stover was used as substrate in a dosage of 8 g oDM/L.

**DNA** extraction

A modified method was used as described previously (Ács et al., 2015). The concentration of the purified DNA was measured spectrophotometrically (NanoDrop ND-1000 Technologies, Washington, DC, USA), and its integrity was determined by agarose gel-electrophoresis.

#### Next-generation DNA sequencing

Sequencing was performed using Ion Torrent PGM 316 chip (Thermo Fisher Scientific). The reads were analyzed and quality values were determined for each nucleotide. From the enriched consortium AD1 578,372 reads containing more than 119 million bp were identified, in the case of AD2 these values were 515,436 and 111 million, respectively. The average read lengths were 231and 246 bp. The individual sequences were further analyzed by using the MG-RAST software package (Wilkie *et al.*, 2014). The MG-RAST server computes results against several reference datasets (protein and ribosomal databases) as previously described (Wirth *et al.*, 2015).

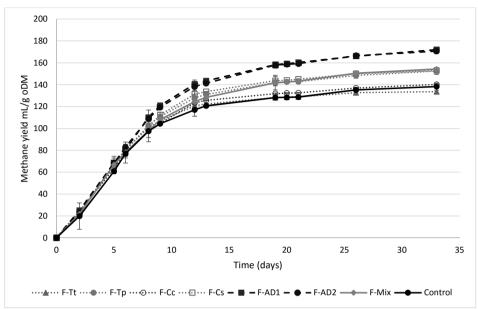


Figure 1. Effect of AD1, AD2, Thermoanaerobacterium thermosaccharolyticum (Tt), Caldanaerobacter subterraneus (Cs), Thermoanaerobacter pseudethanolicus (Tp), Clostridium cellulolyticum (Cc) and their mixture (Mix) on methane production compared to the control.

#### **RESULTS AND DISCUSSION**

#### Enrichment of cellulose degrading thermophilic consortium

Thermophilic (55 °C) temperature was selected for the enrichment because, as a general rule, at higher temperature the degradation is more efficient and the biogas yield is higher (Tsavkelova *et al.*, 2012). When the enrichment culture was fed with 10 g/L glucose, the VFA levels and particularly that of propionic acid, elevated to the alarming level of 2 g/L. Although the VFA content was above the inhibitory threshold, apparently it had no effect on the pH and the biogas production indicating a good buffering capacity of the system. Siegert and Banks (2005) found that the fermentation of glucose was slightly inhibited at VFA concentrations above 4 g/L and was more than halved above 8 g/L which indicated that the fermentation of glucose was less sensitive to inhibition caused by VFA. To monitor proper operation, the VFA contents and pH were measured weekly along with the  $\beta$ -glucosidase enzyme activity, which reflected the cellulose hydrolysis.

New fermentations were started using the enriched and the non-adapted sludge in order to examine if the first enrichment step was successful and further enrich the two isolated consortia. *Caldicellulosiruptor saccharolyticus*, a thermophilic bacterium possessing cellulase activity, was employed as positive control (Bagi *et al.*, 2007). *C. saccharolyticus* enhanced the biogas yield by 12 % in the non-adapted reactors but in the reactors containing our enriched first consortia the effect was negligible. Without the contribution of *C. saccharolyticus* the enriched consortia

yielded 14 % more biogas than the non-adapted one. Biogas yield from 1 g oDM was 463.4, 482.0, 411.6 and 456.1 N mL in the case of enrichment, enrichment supplemented with *C. saccharolyticus*, non-adapted and non-adapted supplemented with *C. saccharolyticus*, respectively. These values are 65.3-79.8% of the observed values of 604 and 630 N mL/g VS by Richards et al. (1991). The results corroborate that the adaptation of the thermophilic microbial community to cellulose was successful and our enrichment cultures brought about a similar bioaugmentation effect as *C. saccharolyticus* did. The consortia apparently remained stable in its biological activity. The two mixed cultures were marked as AD1 and AD2 and used in subsequent experiments.

#### Bioaugmentation effects of AD1 and AD2

In the subsequent experiments corn stover was used as substrate. C/N ratio of this substrate was 47.1. The dried corn stover was pretreated mechanically by grinding and sieving to <2 mm particles. Although the two enriched consortia were treated and handled separately, they showed a very similar bioaugmentation behavior enhancing the methane yield by 22-24 %. Methane yields were 167.1±0.1, 163.7±1.3 and 134.2±0.3 in case of bioaugmentation with AD1, AD2 and control, respectively.

In previous studies several attempts have been made to facilitate the decomposition of celluloserich substrates and increase biogas/biomethane yields by bioaugmentation. It is noteworthy that representatives of the orders *Thermoanaerobacterales* and *Clostridiales* were frequently found as key components of the enrichment cultures.

Metagenome analysis

Next generation sequencing of the whole DNA samples identified the predominating orders being *Thermoanaerobacterales* at 70% and 73% and *Clostridiales* at 10% and 11% abundances for AD1 and AD2, respectively. The good quality sequence data allowed the identification of the most abundant strains at species level. *Thermoanaerobacterium thermosaccharolyticum*, *Caldanaerobacter subterraneus*, *Thermoanaerobacter pseudethanolicus* and *Clostridium cellulolyticum* were identified as the most predominant strains with 46.99, 7.29, 3.62 and 3.42% relative abundance, respectively in case of AD1. Both AD1 and AD2 displayed essentially the same composition.

Our results may contribute to the development of a rationally designed and optimized stable microbial preparation for the reproducible facilitation of lignocellulose degradation. This is, however, not a simple task as the four strains, enriched in high abundance, were accompanied by strains of low abundance in the enrichment community.

The intriguing question is: to what degree the strains present in low richness contribute to the bioaugmentation effect if at all? To test this, the type strains of the four most abundant bacteria were purchased from the German Collection of Microorganisms and Cell Cultures and sterile pure cultures were grown in their respective media. The results, in case of *T. thermsaccharolyticum*, control, *C.cellulolyticum*, *T. pseudethanolicus*, *C. subterraneus*, Mixture, AD2 and AD1 are 133.6±1.3, 138.3±1.5, 140.2±1.1, 152.5±3.1, 152.9±2.2, 154.3±1.1, 170.6±2 and 172±0.7 mL, respectively, (Fig. 1). This indicates that addition of the four bacterial strains and their mixture (in the ratio matching the relative abundances) to the biogas reactors enhanced the methane yield in some cases. It is important to note that neither the individual pure cultures nor their mixture were as efficient as the original communities, AD1 and AD2. This is a strong indication of the active contribution of bacteria present in low numbers.

#### CONCLUSIONS

The two consortia, marked as AD1 and AD2, enhanced the methane yield from pure  $\alpha$ -cellulose and from corn stover by 22-24%. Next generation whole genome DNA sequencing revealed the main orders and most abundant species in AD1 and AD2. In line with earlier findings, members of the orders *Thermoanaerobacterales* and *Clostridiales* play important role in thermophilic lignocellulose decomposition. The composition and biological activities of the most abundant members in the two enriched communities were very similar. Several additional members, occurring in significantly lower numbers have also been identified. At this level a few differences between AD1 and AD2 were identified.

The mixture of the most abundant strains, containing the bacteria in the ratio corresponding to the enrichment community, did not achieve the same augmentation as AD1 or AD2 did, indicating additional contribution by the minor constituents of the enriched microbial community.

#### ACKNOWLEDGEMENTS

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## **Opportunities of Co-Digesting Manure with Grass**

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#### INTRODUCTION

Several researchers have reported higher biogas yields when co-digesting manure with other feedstocks compared to mono-digestion (De Moor et al., 2013 and Xie et al., 2011). The main reason behind this increase in biogas production is that in case of co-digestion a more favorable carbon nitrogen ratio (C/N) can be found. Manure has in general – due to its high nitrogen content - a ratio around 10 whereas the optimal value is around 25. Therefore, products that increase the C-concentration should be added to the feedstock mix in order to increase this ratio and the biogas production.

Besides that, co-digestion also has a positive effect of the macro- and micronutrient balance, the ammonium concentration, pH, and inhibitors/toxic compounds (Xie et al., 2011). When digesting cow or pig slurry with a dry matter (DM) content < 12 % it can be very useful to add more dry, fibrous products such as grass as it is necessary for the microbiological population to have some carrier material in the digester.

#### MATERIALS AND METHODS

An Excel model was developed to determine the biogas production of a single stage continuous stirred tank reactor (CSTR) digester with perfect mixing after steady-state. First the biogas potential of different grasses was estimated. The conditions of the field, when it was mowed and the storage method are the determining factors. Based on the biogas potential and the rate of production of all the different feedstocks, the biogas production of these feedstocks can be calculated. Different hydrolysis rates for different feedstocks were considered. To calculate the biogas production of the entire anaerobic digester, the residence time distribution was taken into account.

To determine the optimal feedstock an economic calculation was added, while also keeping an eye on several technical parameters. It is important to state that because of technical issues the maximal grass content in a wet digester should stay low. When the grass content does not exceed 20% of the input streams, wet digestion in a CSTR should work fine in regards with DM content and viscosity in the reactor (De Moor et al., 2013). A dry digester has no problems with the grass fibers. Grass must be processed in Flanders. This is why the biogas owner can ask, on average, a gate fee of 35 euro per ton but in some areas, it can rise up to 60 euro per ton. To get rid of the digestate the plant manager in Flanders will pay approximately 100 euro per ton DM.

#### RESULTS

For each feedstock, the profitability was assessed based on the purchase price, biogas production in the reactor and digestate cost after digestion. Grass has a high profitability when expressed in profit per ton fresh matter, higher than manure and comparable to maize. Especially when considering the high C/N ratio of this feedstock, more opportunities can be found. Low C/N ratios occur when digesting manure, animal slurries or kitchen waste and thereby causing ammonia inhibition. This results in a lower BGP and thus a suboptimal biogas production in the reactor. In order to optimize the C/N ratio for grass silage and pig manure, it should be fed to the digester in a 1:1 ratio. This result was also reported by Xie et al. (2011) as the best ratio for methane production. Adding grass to avoid ammonia inhibition can result in a 15 % higher BGP (Amon et al., 2006). This results in a more than 15 % increase of the total biogas produced in the digester because of the residence time distribution, leading to an even higher increase in profitability. Despite the economic and technical opportunities most of the cuttings are left on the field or are used to make compost. However, as calculated by Vos et al., (2014), if all cuttings in Flanders

used to make compost. However, as calculated by Vos et al., (2014), if all cuttings in Flanders would be processed and digested anaerobically, a  $CO_2$  reduction equal to 20.000 cars could be avoided and electricity for 13.000 households could be produced.

#### CONCLUSION

Digesting grass can increase the value of the process chain. However, often the operators of these anaerobic digestion plants are not aware of this potential and more importantly, they fear increasing costs when processing this material. It takes good management and some small changes in the supply chain in order to digest grass in an economically feasible way. Grass contaminated with sand or plastics or when it is already rotting, should not be put in the digester but when the area is free of litter and is mown around July, the product is desirable for anaerobic digestion after shredding.

#### ACKNOWLEDGEMENTS

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## Energy from Waste Biomass: Grass from Roadsides and Nature Management

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In Flanders, grass from roadsides or nature conservation areas is considered a problematic biomass waste stream. It cannot be used as fodder because of noxious weed species (e.g. Jacobaea vulgaris) and because the nutritional value is low. Therefore, farmers prefer cultivated grass of sufficient and guaranteed quality.

Grass residues from roadside management are obliged to be removed from the field and processed. Therefore, most of it is treated in compost facilities. Composting has an environmental impact greater than is generally assumed, more sustainable technologies are for example anaerobic digestion and IFBB.

Problems may occur during anaerobic digestion of grass such as: formation of floating layers in the reactor, increase in viscosity or accumulation of sand particles. These technical issues need to be addressed when feeding grass to biogas installations. De Moor et al. (2013) concluded that when the grass content does not exceed 20% of the input streams, wet digestion in a CSTR should work fine in terms of dry matter content and viscosity in the reactor. A model was designed to evaluate the economic and technical feasibility of grass digestion.

The IFBB approach is slightly different from anaerobic digestion of grass: first the grass is washed with warm water, then the washing water and intercellular liquid will be removed using a mechanical screw press. The liquid phase is sent to a fixed bed reactor where it is digested to produce biogas. The biogas is combusted and electricity is produced in the CHP unit. The solid part is dried using the residual heat from the previously mentioned CHP unit, and is subsequently pressed in briquettes as a storable fuel.

Based on our study, we found that thermochemical posttreatment of the solid fraction could further result in briquettes with better properties. When torrefaction was conducted at 300°C, no nitrogen or phosphorous was removed but the sulphur content in g/kg did decrease substantially. At 600°C also nitrogen removal was observed. However, because of the high carbon loss at that temperature, the overall nitrogen content in the char increased. Optimisation is required to make the process feasible.

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# Key Parameters for the Assessment of Biogas Yields from Manures in Commercial-Scale Biogas Plants.

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#### SUMMARY

To classify manures as feedstocks for biogas production, 360 manures samples (78 pig manure, 282 cattle manure) were collected in Germany in 2010 and 2011. Comprehensive analyses were made of relevant parameters. The commonly used method for determination of dry matter (DM) at 105°C caused evaporation of volatile fatty acids (VFA) and consequent underestimation of organic matter content (oDM). To avoid this error, the sum of oDM + VFA was defined as a new organic matter fraction: total organic matter (toDM). The parameter toDM had the best linear correlation with the biogas yields of manures and it may be used for a rough estimation of the biogas yields of manures. The results showed large variation mainly for pig manures which is why measurements of toDM and  $CH_4$  potential in batch assays are recommended for the planning of biogas installations.

#### INTRODUCTION

Liquid manure represents the major proportion of organic agricultural waste and is responsible for a large share of agricultural greenhouse gas emissions. Only small amounts are treated before land application or utilized for energy production. The energy yield to be expected when manures are used for biogas production can be found in official databases or can be measured for each specific case. Both these options have advantages and disadvantages. Often, variations among official data for the same type of manure can be as high as 29%, resulting in different economical scenarios when the figures are applied to commercial-scale biogas plans. On the other hand, biomethane potential tests are time-consuming. The present study provides essential information on manure characteristics to further extend the use of this agricultural residue for energy production.

#### MATERIALS AND METHODS

Over the course of 12 months in 2010 and 2011, 360 manure samples (78 pig manure, 282 cattle manure) were collected across the territory of Germany. At the farms, manures were stored in tanks that were not air-tight, not thermally insulated and were used also as storage for rain water. Storage times in these tanks before sampling were recorded. After collection, the samples were stored at 4 °C and analysed within two days at *bonalytic GmbH* (Troisdorf, Germany) for pH (electrode, DIN EN 12176 – S5), electrical conductivity (electrode, DIN EN 27888-C8), VFA (gas-chromatograph, house method), ammonium nitrogen (NH<sub>4</sub>-N, electrode), total nitrogen (tot-N, DIN EN ISO 11261), dry matter (DIN EN 12880 – S2a), organic matter (DIN EN 12879-S3a), macro and trace elements (DIN EN ISO 11885 – E22). For 263 samples of the 360 samples above (49 pig manure, 214 cattle manure), methane yield and kinetics of production were determined in batch

assays at 40 °C (DIN 4630). The chi-squared test was used to test the normal distribution of the CH<sub>4</sub> yields. The hydraulic retention time (HRT) needed in continuous stirred tank reactors (CSTR) to achieve 90% of the yields achieved in batch was calculated as 4 x  $T_{batch90\%}$ , where  $T_{batch90\%}$  is the time needed in batch to achieve 90% of the final yield.

#### RESULTS

Table 1 and Table 2 report the results (FM indicates the fresh matter).

		range	mean	median
DM	g/kg FM	6.7 – 138.1	40.9	37.1
oDM	g/kg FM	2.3 - 74.8	29.3	23.5
biogas yield	L/kg FM	0.7 – 28.0	8.3	7.4
	L/kg toDM	51 – 647	320	307
CH <sub>4</sub> content	volume %	54 – 69	60.7	60.1
CH4 yield	L/kg FM	0.4 - 19.3	5.1	4.7
	L/kg toDM	31 – 378	195	186
total VFA	g/kg FM	0.1 - 22.8	3.9	2.0
NH4-N	g/kg FM	0.9 – 7.5	2.9	2.9
tot-N	g/kg FM	1.2 – 9.3	4.3	4.2

#### Table 1: Measured values of pig manure samples

#### Table 2: Measured values of cattle manure samples

		range	mean	median
DM	g/kg FM	21 – 152	94	96
oDM	g/kg FM	11 – 137	76	77
biogas yield	L/kg FM	4.7 – 55.0	26.4	26.2
	L/kg toDM	115 – 595	340	338
CH <sub>4</sub> content	volume %	49 – 65	59	59
CH <sub>4</sub> yield	L/kg FM	2.7 – 32.7	15.6	15.5
	L/kg toDM	62 – 329	200	199
total VFA	g/kg FM	0.2 - 15.0	8.0	8.3
NH4-N	g/kg FM	0.4 - 3.3	1.8	1.8
tot-N	g/kg FM	1.2 - 8.0	4.0	4.1

The sum of oDM + VFA was defined as the new organic matter fraction toDM, Figure 1.

Manures were stored in tanks at the farms for 1 - 90 days (pig manures) and for 0 - 120 days (cattle manures), Figure 2.

#### DISCUSSION

The median was considered a better indicator than the average, because the measured CH<sub>4</sub> yields were spread on a broad range for both types of manures.

DM determination at 105 °C until constant weight causes VFA evaporation to an extend that depends on different parameters such as pH and salt concentration. The VFA evaporation results in underestimation of the oDM content. The CH<sub>4</sub> yield on oDM basis is calculated dividing the measured CH<sub>4</sub> yield on FM basis by the measured oDM. Therefore, an underestimation of the oDM content causes an overestimation of the CH<sub>4</sub> yield on oDM basis. This overestimation may threaten a commercial-scale biogas plant's economic viability, depending on the proportion of manure in its diet. Therefore, the new organic matter toDM was defined. Testing the linear correlation of the CH<sub>4</sub> yield against all other parameters, the best correlation was found with toDM (Figure 3 and Figure 4).

Considering the high number of samples and the broad territory where these were collected, the hypothesis of normal distribution for the samples and for their CH<sub>4</sub> yields was made and tested. The CH<sub>4</sub> yield on toDM basis confirmed normal distribution for both types of manures, while the CH<sub>4</sub> yield on oDM had normal distribution only for cattle manures. In the case of cattle manures the standard deviation of CH<sub>4</sub> yield on toDM basis was lower than the standard deviation of CH<sub>4</sub> yield on oDM basis, confirming that to use toDM instead of oDM improved data quality for both types of manures.

No correlation was found between storage time and biogas yields. Neither oDM nor toDM or VFA decreased during storage. This was probably due to temperature and oxygen supply being too low for significant conversion of organic matter. Dilution with rain water was negligible.

Most of the commercial-scale biogas plants being of the CSTR-type, the HRT time needed in CSTR to achieve 90% of the batch yield was estimated. For manure from dairy cattle, beef production, pig fattening and piglet rearing an optimum HRT was calculated of 62 days (d), 76 d, 64 d and 48 d, respectively (Figure 5). Within samples of cattle manure, significant variations could be identified between dairy and beef cattle manure. Within the pig manure samples, distinctions were apparent in yields between manure from porker and piglet production. Manure from beef production requires the longest HRT in CSTR because of the high content of lignocellulose (straw and fodder). For this type of manure, longer time in the biogas digester results in higher yield. Manure from piglet rearing requires the shortest HRT. After conversion of the easily-convertible oDM such as VFA, further residence in the biogas digester does not result in higher yield. However, the estimated HRT of all types of manures have high values for most of the existing

commercial-scale biogas plants. If the estimated HRT cannot be achieved, a biogas yield below 90% of the batch yield is to be expected.

High concentrations of Cu and of Zn were detected in pig manures. These concentrations can inhibit the biogas process.

Aside from a few exceptions, it could be concluded that most types of pig manure are unsuitable as a monosubstrate for biogas production owing to their low DM and low gas yields that often fail to even provide the energy required for raw manure to reach digester temperature. The low DM content of pig manures requires large digester volumes to achieve the planned HRT. Co-digestion with energy-rich feedstocks with low water content (such as glycerine, oil) may be needed to balance the economy of the biogas plant. Ammonia inhibition should be carefully considered for both types of manures. The biggest risks of ammonia inhibition are presented by pig manures as these are rich not only in organic nitrogen, but also in NH<sub>4</sub>-N. Most cattle manures were suitable as mono-substrates for biogas production.

Although toDM was identified as the most suitable parameter for estimating CH<sub>4</sub> yield of pig and of cattle manures, when used as a single parameter it can only give a rough estimation of the yield and does not take into account potential inhibitors such as ammonia, metals, disinfectants. The determination of the biomethane potential in batch tests and the analyses of other components such as trace elements and nitrogen are crucial for planning commercial-scale biogas plants.

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# Simple Pretreatment of Poultry Manure for Efficient Biogas Production and Co-Fermentation with Maize Silage

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#### SUMMARY

Water extraction of raw chicken manure elevated the carbon-to-nitrogen ratio 2.7-fold, i.e. from 7.48 to 19.81. The treated chicken manure (T-CM) became suitable for biogas fermentation as monosubstrate. Improved methane production was achieved in co-fermentations with maize silage (24% more methane) relative to T-CM monosubstrate. The standardized biogas potential assay indicated that the methane yields varied with the organic loading between 160 and 250 mL CH<sub>4</sub>/g organic total solid (oTS). Co-fermentation with maize silage was sustainable in continuous anaerobic digestion for at least 4 months.

#### INTRODUCTION

The poultry industry is growing rapidly along with human consumption, which results in large quantities of animal wastes to be treated. Inappropriate management of manure may cause numerous undesirable consequences such as odor problem, attraction of rodents, insects and other pests, release of animal pathogens, groundwater contamination, surface water runoff, deterioration of biological structure of the soil, etc. (Kocak-Enturk et al., 2007). NH<sub>3</sub> and greenhouse gases, CH<sub>4</sub> and CO<sub>2</sub>, emitted from the waste storage units cause air pollution problems (Yetilmezsoy and Sakar 2008). Anaerobic digestion (AD) is a commonly employed process for treating animal manure and biogas production from manure is widely studied and practiced (Mackie and Bryant 1995, Huang and Shih 1981, Nishio and Nakashimida 2007, Kovács et al., 2014).

Chicken manure (CM) is generally considered a problematic substrate for AD (Güngör-Demirci and Demirer 2004). CM has high nitrogen content, which is in two main forms: uric acid and undigested proteins, representing 70% and 30% of the total organic nitrogen, respectively. AD of these components is accompanied with the production of inhibitory concentrations of unionized  $NH_3$  and  $NH_4^+$  ions (Chen et al., 2008, Salminen and Rintila 2002). Accumulation of the toxic products does not allow fermentation at higher total solids (TS) loadings. The recommended substrate concentration is less than 5% TS and a decrease in biogas production rate has been observed when TS was further increased (Sakar *et al.*, 2009, Kelleher *et al.*, 2002).

The digestibility of nitrogen-rich wastes could be improved by mixing them with substrates of high carbon content, thereby improving the C/N ratio (Ahring *et al.*, 1992, Kaparaju *et al.*, 2002, Wang *et al.*, 2012). Co-digestion has important benefits, including the balancing of the macro and

micronutrients, pH, inhibitors/toxic compounds and dry matter. C/N ratios of 20:1 - 30:1 provide optimal digestion, stable pH and low concentrations of free  $NH_3$  and total  $NH_4^+-N$  (Wang *et al.*, 2012).

In the present study, the removal of the majority of water-soluble inorganic and organic nitrogen compounds from CM by water extraction at ambient temperature was tested, which is a simple and inexpensive method. The insoluble particulate fraction, separated by centrifugation or simple sedimentation, became suitable substrate for biogas generation. Furthermore, co-digestion of CM with maize silage and corn stover was employed to improve the substrate C/N ratio.

#### MATERIALS AND METHODS

#### Substrates and inoculum

CM was collected from a commercial broiler poultry farm (Hungerit Corp.) located at Csengele, Hungary. The free-range poultry houses use wheat straw bedding. Water extraction comprised of soaking 5g CM in 100 mL tap water at room temperature followed by separation of the liquid and solid phases by centrifugation (10,000 rpm for 3 min). The solid fraction was air dried and stored at -20 °C. This treated chicken manure (T-CM) was used in most AD experiments.

Corn stover was obtained from University of Szeged, Faculty of Agriculture. Maize silage came from the biogas plant of Zöldforrás Ltd., Szeged, Hungary. The AD inoculum was collected freshly from the same industrial biogas plant operated with a mixture of pig slurry and maize silage at mesophilic temperature. CM and corn stover was milled and sieved with an electric grinder (Retsch SM 100, Haan, Germany).

Parameters of the biogas substrates raw chicken manure (CM), pretreated CM (T-CM), maize silage and corn stover are presented in Table 1.

Parameter	СМ	T-CM	Maize Silage	Corn Stover
Organic total solids (oTS) [g]	84.1	83.80	95.11	94.32
Total solids (TS)[g]	92.6	95.87	29.32	93.72
Carbon/Nitrogen ratio	7.5	19.8	45.3	52.5
Particle size [mm]	<2	<2	<10	<2 <10

#### Table 1: Parameters of the substrates used

#### Batch fermentation

Experiments were carried out in 160 mL reactor vessels (Wheaton glass serum bottle, Z114014 Aldrich) containing 60 mL liquid phase at mesophilic temperature (37±0.5 °C). All fermentations were done in triplicates. The inoculum sludge was filtered to remove particles larger than 1 mm and was used according to the VDI 4630 protocol (VDI 2006). Each batch fermentation experiment lasted for 30 days in triplicates.

#### Fed-batch fermentation

The working volumes of custom-made reactors were 5 L, the headspaces were 1 L, the continuously stirred tank fermenters (CSTR) were designed and constructed by Biospin Ltd, Hungary (Kovacs et al., 2013). The AD experiments were performed at  $37\pm0.5$  °C. Inoculum sludge came from the effluent of an operating biogas plant (Zöldforrás Ltd, Szeged, Hungary) and was incubated in the laboratory CSTR for 7-10 days to exhaust its residual biogas potential. Afterwards the reactors were fed daily with the specific substrates/mixtures until biogas production became stabilized. The biogas production measurement started from week 5 after the beginning of daily feeding.

#### Carbon-to-nitrogen ratio (C/N)

To analyze C/N, an Elementar Analyzer Vario MAX CN (Elementar Group, Hanau, Germany) was used. The equipment operates using the principle of catalytic tube combustion under an  $O_2$  supply at high temperatures (combustion temperature: 900 °C, post-combustion temperature: 900 °C, reduction temperature: 830 °C, column temperature: 250 °C). The components were separated from each other with the aid of specific adsorption columns (containing Sicapent (Merck, Billerica, USA), in C/N mode) and determined in succession with a thermal conductivity detector. Helium served as carrier and flushing gas.

#### <u>NH4+-N</u>

For the determination of  $NH_4^+$ -N content, the Merck Spectroquant Ammonium test (1.00683.0001) (Merck, Billerica, USA) was employed.

#### Phosphate measurement

Total phosphate content of chicken manure supernatant was measured by the standard 4500-P E ascorbic acid method (Standard Methods for the Examination of Water and Wastewater, SMWW 4000-6000).

#### **Biochemical oxygen demand determination**

To measure the biochemical oxygen demand of chicken manure supernatant (CMS) a 5-day BOD test was applied (OxiTop OC 110, Wissenschaftlich-Technische Werkstätten GmbH). In the parallel 500 mL BOD-sample bottles 0.5 mL of microorganism culture and 43 mL of CMS solution were placed. The results were read after 5 days in mg  $O_2/L$ .

#### VOAs/TAC

5 g of sample was taken for the analysis and diluted to 20 g with distilled water. The measurements were carried out with Pronova FOS/TAC 2000 Version 812-09.2008 automatic titrator (Pronova, Berlin, Germany).

#### Gas chromatographic analyses

The CH<sub>4</sub> content was determined with an Agilent 6890 N GC (Agilent Technologies) equipped with an HP Molesive 5 Å (30 m × 0.53 mm × 25  $\mu$ m) column and a TCD detector. The temperature of the injector was 150 °C and application was made in split mode 0.2:1. The column temperature

was maintained at 60 °C. The carrier gas was Linde HQ argon 5.0, with the flow rate set at 16.8 mL/ min.

### **RESULTS AND DISCUSSION**

#### The water extraction of CM

CM has a high biogas potential, but due to its high N-content the C/N ratio is only about 5–10. This leads to inhibition of the methanogenic community (Yenigün and Demirel 2013).

A simple water extraction pretreatment removed significant portion of the nitrogen content of the solid material, which decreased from 53.75 mg/kg to 21.99 mg/kg. The process increased the C/N from 7.5 to 19.8 (Table 1), which was close to the optimum range of 20-30 (Yadvika et al., 2004). After the pretreatment the phosphate, nitrogen and BOD contents of the supernatant (CMS) were 10 mg/L, 21.3 mg/L and 5.9 g/L, respectively. The C/N ratio of CMS was 4.7. The water extraction technology may therefore be considered a suitable approach to pretreat CM as biogas substrate.

#### Batch AD of pretreated and untreated CM using two organic concentrations

According to previous experience, at higher TS loadings, i.e. >50 g/L, raw CM cannot be subjected to AD efficiently (Bujoczek et al., 2000, Dalkilic and Uruglu, 2015). Batch measurements at two lower organic load concentrations were tested using twice the substrate concentration prescribed by the standard VDI test protocol (VDI 2X) (VDI 2006), which contained 16.26 g oTS/L of CM and 15.65 g oTS/L of T-CM; and VDI 4X, which contained 32.52 g oTS/L and 31.29 g oTS/L of CM and T-CM, respectively.

The reactor fed with T-CM produced 209.5 mL  $CH_4/g$  oTS, which is considerable if one takes into account that the "golden standard" maize silage yields around 250-280 mL  $CH_4/g$  oTS under the same conditions. The difference between the T-CM and raw CM was about 24% in favor of the T-CM substrate. Doubling the substrate concentration decreased specific methane production.

#### Batch co-fermentation of T-CM and maize silage

Improvement of the efficiency of CM fermentation frequently invokes co-fermentation of the manure (Chen et al., 2008, Kaparaju et al., 2002, Heiermann et al., 2007). To ensure the stability of the fermentation, the organic loadings were decreased to VDI 1X and VDI 0.5X. Methane yields of co-digestion did not reach those of maize silage in the control reactors. Nevertheless, the mixed substrates yielded more  $CH_4$  than mono-fermentation of T-CM indicating the benefits of co-fermentation. The VDI 0.5X fermentation yielded 260.2 mL  $CH_4/g$  oTS, this specific activity slightly lower than that of VDI 1X.

#### Fed-batch co-fermentation of T-CM and maize silage

To test the stability and sustainability of co-digestion with maize silage we studied the system in semi-continuous fed-batch fermentation. The experiments lasted for 16 weeks and consisted of two parts. During the first 8 weeks, the dosage of the organic matter was 0.5 g oTS/L/day and the OLR was increased to 1.0 g oTS/L/day for the second 8 weeks. In the case of co-fermentation, the ratio of maize silage to T-CM was 1:1 on oTS basis. The objective was to allow a longer period for the adaptation of the microbial community to the substrate. Figure 1 shows the cumulative methane yields. It was apparent that the methanogenic consortium became accustomed to the

increased OLR and CH<sub>4</sub> yields did not change significantly from week 9. CH<sub>4</sub> productivity of mono-, and co-digestion of T-CM did not reach the estimated yield assuming additive biogas production from the two substrates. This suggests that maize silage did not efficiently improve the decomposition rate and yield of the substrate mix. Apparently, the microbes chose to degrade maize silage from the daily dosage of mixed substrates and left some of the T-CM untouched. The ratio of VOAs/TAC was around 0.15-0.20, which indicated a balanced biogas fermentation. Not surprisingly, mono-fermentation of T-CM resulted in the highest NH<sub>4</sub><sup>+</sup>-N levels. The ammonium level started to accumulate and reached 3.6 g/L at the end of the experiment (Salminen and Rintala 2002, Nielsen and Angelidaki 2008). Co-digestion of T-CM with maize silage lowered the NH<sub>4</sub><sup>+</sup>-N levels as expected. In the reactors fed with maize silage:T-CM=1:1 ratio (at oTS) NH<sub>4</sub><sup>+</sup>-N attained 2.3 g/L at week 16, which should have provided a suitable environment for the biogas producing community and could ensure a stable long term biogas fermentation. Nevertheless, the biogas yields did not improve appreciably upon addition of maize silage. This suggests that the C/N ratio may not be the best indicator of system operation when CM is a major substrate component in AD.

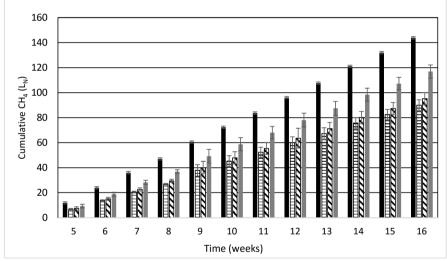


Figure 1: Cumulative biogas productions in fed-batch AD reactors using various substrate compositions: maize silage (black columns), the solid fraction of water extracted chicken manure (T-CM) (horizontally striped columns), co-fermentation of maize silage and T-CM (diagonally striped columns), and the estimated yield assuming additive biogas productivity of the co-fermentation partner substrates (grey columns)

#### CONCLUSIONS

Water extraction successfully increased the C/N ratio of chicken manure from 7.45 to 19.81 and AD of the solid fraction became sustainable when the reactors were fed with T-CM monosubstrate. In the batch reactors about 27% more CH<sub>4</sub> was produced from T-CM relative to raw CM. Co-digestion of T-CM with maize silage increased further CH<sub>4</sub> production presumably

due to the improved C/N. Corn stover efficiently replaced maize silage in batch co-fermentations, which may have important ramifications for practical application.

Fed-batch fermentation corroborated that T-CM was a suitable monosubstrate in sustained biogas fermentation. An increase in OLR from 0.5 to 1 g/L/day did not perturb the system significantly. Interestingly CH<sub>4</sub> yields of T-CM and co-fermentation with maize silage were very similar, in spite of the lower ammonium ion concentration brought about by the introduction of maize silage. This may indicate that there are components in T-CM, which hinder its AD even under acceptable C/N conditions. A conceivable consequence of this effect could be that the microbial consortium consumed first the easily biodegradable substrate component, which was maize silage was supplied daily, thus the microbes were not compelled to digest much of T-CM. This assumption is in line with the batch fermentation results but needs further experimental verification.

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# Wastewater Stabilisation Pond Train System as Ecotechnological Tool for Bioregenerative Resource Recycling: Showcasing an Indian Experience

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#### INTRODUCTION

Wastewater generated from diverse sources such as municipalities and industries contains a cocktail of both beneficial and harmful chemical substances. Continuous generation and discharge of wastewater into the natural system is creating imbalance among the dissolved materials and adversely affects the ecology of recipient water bodies. Typical "package plant" or municipal sewage treatment plants are capital - and energy-intensive in nature and needs technical expertise to operate. In developing countries, such plants are not often considered as an economically viable option for treating sewage. Nonetheless, wastewater treatment should not also allow the nutrient-enriched wastewater down the drains and is desired to accomplish the return of nutrients and water to productive use closing the nutrient loop (Reed and Brown, 1995). Here we can draw inspiration from natural wetlands which are regarded as 'nature's kidney' or 'nature's liver' owing to their actions as sediment sinks, filters and sponges for nutrients and toxins. The removal of pollutants of these systems relies on a combination of physical, chemical and biological processes that naturally occur in wetlands and are associated with vegetation, sediments and their microbial communities. As highly biologically active ecosystems, wetlands transform many pollutants at a high rate due to their high biological activity, and have increasingly been used for wastewater treatment (Andersson et al., 2005; Vymazal, 2011). They act as bio-filters through a combination of physical, chemical and biological reactions which result in the reduction of organic, nutrient and microbiological loads (Brix 1998; Vymazal, 2011). Treatment wetland systems exemplify systems of low-cost, low-tech, low maintenance and minimal energy demanding system and therefore, can be promoted as appropriate ecologically-based solutions to wastewater problems. In recent decades, there has been a major shift in reclamation strategies from costly conventional engineering technology to environmentally sound, low-cost, sustainable eco-technology using living machines such as probiotics, macrophytes, herbivorous fishes, mollusks, hypolimnetic aeration, and treatment wetlands. Another important way of utilizing wastewater in economy driven activities is aquaculture where income of essential elements such as nitrogen, phosphorous, organic carbon,

etc. from sewage can be converted into biomass. Wastewater-fed aquaculture therefore, offers means to treat wastewater with integrated material-flow recycling and generation of fish biomass.

Kalyani is a modern town in West Bengal, India, established in 1945 with a well-planned underground sewage system. A sewage treatment plant was constructed in 1993 under the Ganga Action Plan (GAP) receiving  $17\times10^6$  L d<sup>-1</sup> of domestic wastewater from about 82.000 inhabitants of the township. Of the total amount of sewage generated per day, about  $11\times10^6$  L is treated through conventional treatment plants, whereas the remaining  $6\times10^6$  L enters into a series of stabilization ponds (WSP) before being discharged into the river Ganga. Despite the demonstrated ability of treatment wetlands to treat effectively domestic and agricultural/industrial sewage wastewater as an ecotechnogical tool, it has not been applied as widely as it deserves. The present study was undertaken in sewage treatment pond series in from April 2007 to March 2008 to assess the potential of treatment wetlands for removal of pollutants and bioregenerative reclamation of nutrients.

#### MATERIALS AND METHODS

The WSPs employed in the present study fall under three different categories: two anaerobic (26 m x 52 m x 2.5m), two facultative (64 m x 150 m x 1.5 m) and four maturation ponds (52 m x 156 m x 1 m). All ponds were arranged in succession finally connected to a canal falling into the river Ganga. The sewage covered the distance of 826 m from inlet to the final outlet of the sewage treatment farm maintaining respective average retention time of 1.127 days in anaerobic ponds, 4.8 days in facultative ponds and 5.408 days in the maturation pond. Two anaerobic ponds remained fully loaded with water hyacinth. The last four maturation ponds were used for culture of both carp and tilapia but only tilapia in facultative ponds.

Surface water was collected from five sites with two sub sites of one meter distance - inlet of anaerobic pond (AP<sub>in</sub>), outlet of anaerobic pond (AP<sub>out</sub>), outlet of facultative pond (FP<sub>out</sub>), outlet of first maturation pond (MP<sup>-1</sup><sub>out</sub>) and outlet of fourth maturation pond (MP-4<sub>out</sub>), regularly between April, 2007 and March, 2008 and different water quality parameters namely pH, total alkalinity, dissolved oxygen (DO), orthophosphate (OP), turbidity, organic carbon (OC), total solids (TS), total dissolved solids (TDS), total suspended solids (TSS) and chemical oxygen demand (COD) of anaerobic, facultative and maturation ponds were estimated following standard methods (APHA, 2005). Fecal coliform (FC) and total coliform (TC) bacteria were counted following the Multiple Tube Fermentation or Most Probable Number (MPN) Technique (APHA, 2005). The data were subjected to appropriate statistical tests.

#### **RESULTS AND DISCUSSION**

Water temperature ranged from 15.6 to 35 °C. pH of water gradually increased from inlet (6.7 – 7.08) to outlet (7.6 – 8.3). Appreciable increase in pH from the anaerobic pond to the final outlet is due to  $CO_2$  uptake by algae during photosynthesis and increase in oxygen concentration in the water. The values of total alkalinity in water ranged from 89-119,113-147, 122-177.5, 136-188, 167-220 mg L<sup>-1</sup> in AP<sub>0</sub>, AP<sub>0</sub>, FP<sub>0</sub>, SP1<sub>0</sub> and SP4<sub>0</sub>, respectively. Total alkalinity increased from anaerobic pond to final outlet of maturation pond. Seasonal variations in total alkalinity were clearly discernable exhibiting highest and lowest values during summer and winter, respectively. The inlet of the anaerobic pond showed total absence of oxygen. There was a gradual increase in

DO concentrations to as high as 15 mg  $L^{-1}$  at the outlet of the facultative pond. This was then followed by a rising trend with some fluctuations along the effluent gradient. The concentrations in the final outlet were 6.32-11.2 mg  $L^{-1}$ . Seasonal variability showed the higher concentrations of DO during summer and minimum during winter period. The major increase occurs in the facultative pond, due to the rapid change from anaerobic to shallow and oxygenated conditions that remained for almost 5 days. The presence of higher level of oxygen is known to be fundamental for a good quality of the water, for the survival of fish in the maturation ponds without health risks for human consumption and for the removal of pathogenic bacteria in a process called photo-oxidation (Maynard et al., 1999). COD ranged from 437-902, 386-621, 235-379, 160-191, and 138-160 mg L<sup>-1</sup> at AP<sub>i</sub>, AP<sub>o</sub>, FP<sub>o</sub>, SP1<sub>o</sub> and SP4<sub>o</sub>, respectively. There was a gradual decreasing trend in COD values with 74-82% decline from inlet to outlet. The concentration of OC in water varied, 27.5-55, 21-48, 16-41, 11-29 and 6-14 mg L<sup>-1</sup> in AP<sub>i</sub>, AP<sub>o</sub>, FP<sub>o</sub>, SP1<sub>o</sub> and SP4<sub>o</sub>, respectively showing 70-78% decline during the period of study. Overall seasonal variation of organic carbon was in the order: summer>monsoon>spring>winter. The average concentration of ammonium nitrogen in water ranged, 2-3.45, 1.8-2.92, 1.32-2.5, 1.04-2.1 and 0.5-0.95 mg L<sup>-1</sup> in AP<sub>i</sub>, AP<sub>o</sub>, FP<sub>o</sub>, SP1<sub>o</sub> and SP4<sub>o</sub>, respectively showing an overall decline of 75-85%. The concentration declined by 40-45 % through the facultative ponds followed by 29-40% decline through the maturation/stocking ponds. The mean concentrations of nitrite nitrogen in water ranged from 0.02-0.219, 0.18-0.27, 0.1-0.29, 0.04-0.123 and 0.013-0.055 mg L<sup>-1</sup> in AP<sub>i</sub>, AP<sub>o</sub>, FP<sub>o</sub>, SP1<sub>o</sub> and SP4<sub>o</sub>, respectively. The mean concentrations of nitrate-nitrogen ranged 0.158-0.31, 0.2-0.36, 0.172-0.33, 0.158-0.25 and 0.09-0.16 mg L<sup>-1</sup> in AP<sub>i</sub>, AP<sub>o</sub>, FP<sub>o</sub>, SP1<sub>o</sub> and SP4<sub>o</sub>, respectively. The variation overall seasonal trend of followed the order: summer>autumn>monsoon>spring>winter. Concentrations of OP of water ranged 0.025-0.54, 0.24-0.51, 0.2-0.47, 0.18-0.29 and 0.1-0.27 mg L<sup>-1</sup> at AP<sub>i</sub>, AP<sub>o</sub>, FP<sub>o</sub>, SP1<sub>o</sub> and SP4<sub>o</sub>, respectively. The counts of heterotrophic bacteria ranged from 2.4-180, 3.2-256, 1.95-340.5, 1.25-145 and 0.3-72.5 x 10<sup>5</sup> mL<sup>-1</sup> in AP<sub>i</sub>, AP<sub>o</sub>, FP<sub>o</sub>, SP1<sub>o</sub> and SP4<sub>o</sub>, respectively. There was a steady decline in counts of heterotrophic bacteria down the sewage effluent gradient from the anaerobic pond to outlet of last maturation pond showing a decline of 87% but with varied rate within 400m. The major removal mechanism for nitrogen in treatment wetlands is nitrification-denitrification. Oxygenation is often the limiting step for nitrogen removal. Nitrate removal in anaerobic ponds is dominant over the other ponds which were fully covered with water hyacinth and loaded with huge organic matter that may limit the oxygen diffusion to the system and causes anaerobic sediments; this condition favored the denitrification process. Also, the supply of organic carbon raises the total heterotrophic activity, which consumes oxygen and thus indirectly favors denitrification by lowering oxygen concentrations in the sediment (Arheimer and Wittgren, 1990).

Both fecal coliform and total coliform bacteria were present in the influent at higher concentrations than in the final effluent. Fecal coliform concentrations were reduced by two orders of magnitude, from 10<sup>6</sup> to 10<sup>4</sup>, which is consistent with the previous observation (Bhowmik et al., 2000). Total coliform bacteria data are quite variable, decline was observed along the effluent gradient, from 10<sup>7</sup> to 10<sup>5</sup> MPN 100 mL<sup>-1</sup>. Facultative (87%) and maturation ponds (75%) performed quite successfully in the removal of fecal coliform, while less efficiency was found for the total coliform bacteria (30% in FP; 61% in MP). The processes involved in pathogen removal is the result of an intricate interaction between physical (sedimentation,

mechanical filtration, aggregation, inactivation by UV radiation), chemical (oxidation, exposure to biocides excreted by plants or other microorganisms, adsorption to organic matter and the biofilm, adsorption to solids, entrapment in the root system and gravel or soil bed matrix) and biological processes (antibiotic activity of root exudates, predation by nematodes and protozoa, attack by lytic bacteria or viruses, retention in biofilms, natural biofilters of bacteria (Kaseva, 2004). In a tropical climate area with high insolation and water temperature, inactivation by ultraviolet radiation and oxidation are likely to play an important role in bacterial die-off as well as sedimentation and entrapment in the root system of water hyacinth.

A three-step biological treatment system comprising three subsystems in the form of two anaerobic ponds in the first step, two facultative ponds in the second step and four stocking ponds in the third step was responsible for sharp reduction of organic carbon (70-78%), ammonium nitrogen (75-85%), nitrite nitrogen (67-78%), nitrate nitrogen (55-64%) and phosphorus (50-58%) during the course of study which shows better removal efficiencies compared to Shilton's (2005) report of up to 30 – 70 % N and 20 – 40 % P in facultative lagoons. Depletion in nutrient contents along the sewage effluent gradient was related with the gradual decline in the counts of heterotrophic bacteria. Because of distinct spatial variability in the counts of heterotrophic bacteria coupled with downstream decline in organic load, the responses of these bacteria along the effluent gradient further served as functional indicators of reclamation of the system in the present investigation. The specially designed facultative ponds having aerobic condition at the surface and anaerobic at the base were the most dynamic in the train in the reclamation process due to their enhanced microbial activities and development of intense algal productivity which triggered further reclamation of effluents by providing aerobic condition (Faulwetter et al., 2009); major reclamation occurred in this subsystem before being passed into the next subsystem of fish growing ponds, primarily responsible for transforming organic wastes into fish biomass through the complex multi-channel grazing-detritus food chain. This is of considerable significance in tropical developing countries, where production of fish protein from such low cost organic wastes is highly promising.

The ecotechnology applied in the Kalyani model of WSP train for wastewater reclamation showed that first-stage anaerobic ponds were involved in removal of high volumetric rate of organic load; the secondary facultative ponds accomplished the second-stage biological treatment; and maturation ponds provided the sludge polishing service and tertiary treatment for further removal of pathogens. As a sustainable ecotechnology WSPs train systems offer: (i) low energy, (ii) oxygenation of the upper water layer via movement of air and natural wave action; (iii) aeration via algal photosynthetic activity; (iv) pH buffering via carbonate / bicarbonate system; (v) natural nutrient uptake and reduction, (vi) solar disinfection and (vii) aquaculture. A major implication of the study is such WSPs tied with aquaculture can be strongly recommended as a reclamation strategy for municipal sewage which would not only escape expenditure for mechanical treatment but also generate profit through production of fish biomass from sewage. Hence with cumulative treatment effect of multiple ponds in series such WSPs have been championed as an effective, low-cost, nature-based, acceptable, appropriate and sustainable ecotechnological alternative to high-cost, energy intensive conventional wastewater treatment systems particularly in the developing countries.

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# Combining Phototrophic Polyhydroxybutyrate Production by Cyanobacteria with Anaerobic Digestion for Providing Nutrients and Utilizing Residual Biomass

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In recent years, the interest in producing high value compounds (Chew et al., 2017) or biodegradable polymers (Singh et al., 2016) with algae/cyanobacteria steadily increased. Reasons for this are, in case of fossil based and persistent plastics, the rising environmental pollutions (Lechner et al., 2014), which can be avoided by using biodegradable materials like poly-ß-hydroxybutyrate (PHB). Currently, PHB-production is based on heterotrophic bacteria (Panda et al., 2006) using organic carbon sources from agricultural crops. As alternative and to avoid competition with food and feed production cyanobacteria, metabolising PHB from CO<sub>2</sub> e.g. from exhaust gas, can be used (Drosg et al., 2015). Due to the comparable low PHB concentrations accumulated by cyanobacteria (Drosg et al., 2015) it is necessary to increase the economic efficiency of photoautotrophic PHB production. A possibility is to use effluent streams (e.g. digestate) instead of fertilizers to provide nitrogen and phosphorous sources (Morales-Amaral et al, 2015) and to recycle process water of algae/cyanobacteria cultivation (Sing et al. 2014). Other options are to gain further high-value products from the cyanobacterial biomass (e.g. pigments, proteins; Koller et al. 2014) or to recycle residual biomass into an anaerobic digestion process.

Considering all these options a biorefinery concept was designed, where digestate and process water are used as nutrient source for cyanobacteria cultivation, additional products are gained and the residual biomass as well as the produced methane (CH<sub>4</sub>) and carbon dioxide (CO<sub>2</sub>) are utilized. Literature (Bhati et al. 2016) and previous experiments (Meixner et al. 2016) already demonstrate that effluent streams can be used as nutrient source for producing PHB with cyanobacteria. The current issue of research is to investigate further high-value products as well as the biochemical methane potential of residual biomass.

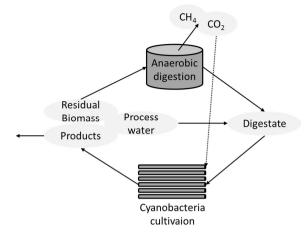


Figure 1: Combining phototrophic PHB production with cyanobacteria and anaerobic digestion to a biorefinery

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# The Fate of Heavy Metals in Anaerobic Digestion Process

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#### INTRODUCTION

As a direct consequence of environmental legislation (e.g. EU Nitrate Directive 91/676/EEC), farmers in regions with intensive livestock production need to find alternative ways to deal with their manure surplus. Increasingly, anaerobic co-digestion (AD) plays an important role in processing animal manure in such regions. This results in biogas as a renewable energy source and digestate as a nutrient rich fermentation residue. When digestate is applied to arable land as a bio-fertilizer, most of the micronutrients are fully utilized, as they are essential for plant growth. However, some trace elements, such as Cu and Zn, have elevated concentrations in digestate, due to their addition to livestock feed as metabolic micronutrient. As such application of digestate could lead to Cu and Zn accumulation in agricultural soils and a permanent threat for humans by entering the food chain.

Currently, digestate is being considered as potential candidate for End-of-Waste status. Accordingly, the concentration level of Cu and Zn in digestate must remain between the legal limits of 100-200 mg/kg dry matter and 400-600 mg/kg dry matter, respectively (European Commission, 2008). In practice, however, the elevated Cu and Zn levels in pig slurry as a feedstock for anaerobic digestion, pose a risk to exceed the currently proposed End-of-waste criteria. The aim of this study is to simulate concentrations in digestate for essential elements like Cu and Zn, based on their loading in natural products such as manure.

#### MATERIAL AND METHODS

The basic principle for the simulation model is that up-concentration of heavy metals in digestate can be predicted based on biogas potential (i.e. mass reduction) and initial concentrations (i.e. dry matter (DM), Cu and Zn content) in feedstock. As a result, four different approaches for a simulation model were proposed and validated with input data received from physicochemical and biogas batch test analyses of digestate from co-digestion (BP(A)) and mono-digestion (BP(B)) processing units, as follows:

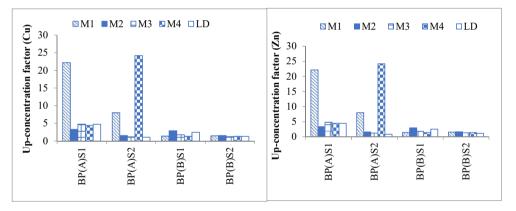
- M1 Biogas Yield Approach: mass reduction is determined by knowing the biogas potential and converting it on mass basis by using biogas mass conversion factor that was set at 1 Nm<sup>3</sup> = 1.1 kg of biodegradable mass. The biogas mass conversion factor is based on the weight of biogas, which mostly contains 52% methane, 47% carbon dioxide and 1% other, primarily sulphide.
- **M2 Biodegradation Factor Approach:** mass reduction is based on biodegradation factor (*bdf*) for food waste (*bdf*=81%; Zhang et al., 2007) and animal manure (*bdf*=45%; El-Mashad and Zhang, 2010).
- **M3 Dry Matter Approach:** mass reduction is based on the difference between DM content of feedstock and digestate, and this difference is used as an approximate value for *bdf*.

• **M4** - **Power Generation Approach:** mass reduction is based on power generation potential of each biogas plant where the latter is used to calculate biogas yield and hence the mass reduction factor. BP(A) had an electrical efficiency of 40%, whereas BP(B) had an electrical efficiency of 22.5%.

While BP(A) performs anaerobic co-digestion of pig manure (15%) and organic biological waste produced by food industry (85%), BP(B) performs anaerobic mono-digestion of cattle manure. Sampling at biogas plants (BP) has taken place at 2 sampling moments, S1 and S2, with one month difference in between.

#### RESULTS

The simulation models calculated approximate values of mass reduction factor reflecting the concentration of Cu and Zn in digestate as compared to results obtained from physicochemical analysis. The difference between these concentrations gives an up-concentration factor which indicates how many times heavy metal concentration in digestate is higher as compare to input fed to digester.



# Figure 1: Comparison of differences in up-concentration factor between M1, M2, M3, M4 and laboratory data (LD) for Cu and Zn concentrations. BP(A): co-digestion; BP(B): mono-digestion; S1: sampling moment 1; S2: sampling moment 2

As shown in Figure 1, a high difference between the proposed models can be observed with codigestion due to the variability in feeding patterns that is highly dependent on the market availability of the feedstock. Lower differences are notable with mono-digestion since the same input, cattle manure, is fed to the digester. The compatibility factor between simulation models and laboratory data ranged in following order: M3 > M2 > M1 > M4. The M1 and M4 were the least similar to LD with regard to BP(A). As expected, M3 scored the best since it takes into consideration the DM of the output. However, this will not be the case in practice, therefore the biodegradable approach seems currently the most promising aproach.

#### CONCLUSION

The results indicate that Cu and Zn concentrations were higher in digestate from co-digestion units where pig slurry is used as feedstock. Thus, the simulation result is highly influenced by the input stability (co-digestion versus mono-digestion). In order to minimize this impact, focus on individual input streams and their characteristics is needed. Mass reduction factor, in general, proved to be a good indicator of metal concentration in digestate.

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# Nutrient Recovery from Wastewaters as High-Valued Hydroponic Fertilizer and Non-Fertilizer Products using Hybrid Ion Exchange Nanotechnology

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#### SUMMARY

Current nutrient recovery practices for phosphorus (P) from wastewaters carryout precipitation of pollutant P to make struvite/brushite or carryout liquid/solids separation that focuses on the solids bound P. Either approach results in low-grade, slow-release, land applied fertilizer. Residual dissolved nutrients not captured are more harmful in the environment and act as biocatalysts contributing to the ever-increasing frequency and extent of harmful algae blooms. RD&D efforts have demonstrated that commercially available Hybrid Ion Exchange resins (HIX-Nano), which remove naturally occurring arsenic from drinking water, can remove soluble reactive dissolved P and nitrate. The HIX-Nano is regenerable, which lowers overall product usage cost. Moreover, regeneration is the key to recovery and reuse. Custom regeneration chemistry can convert regenerant solution of eluted nutrients into liquid N-P-K fertilizer and non-fertilizer products with greater market value.

#### INTRODUCTION

Current nutrient recovery practices for phosphorus from wastewaters carryout precipitation of total phosphorus (TP) to make struvite/brushite, or carryout aggressive liquid/solids separation that focuses on the solids bound P. Either approach results in low-grade, slow-release, land applied fertilizer. A review of operating costs of each of these nutrient recovery technologies used on multiple continents at Water Resource Recovery Facilities (WRRFs) indicates that the value of such recovered products cannot compete with commodity synthetic fertilizer and the cost to recover P on a pound basis is greater than its value as P in the market place (SenGupta and Cumbal, 2007). Unless there are other redeeming benefits, such as operations and maintenance cost savings by preventing struvite precipitation or enhanced biosolids management, these precipitation approaches are favorable at only the largest scale WRRFs. Moreover, the unreacted dissolved phosphorus (DP), albeit generally less than 10% of the total phosphorus available in these wastewaters subject to struvite or calcium phosphate processing, is instantly bioavailable and significantly contributes to the ever-increasing frequency and extent of harmful algae blooms (HABs) in fresh water bodies, e.g., Lake Erie, Everglades, Jordan Lake. The adversity of small amounts of DP (relative to the total phosphorus in these wastewater streams) serving as biocatalysts for HABs birthed the sustainable and cost-effective concept to produce and apply manufactured inorganic chemical catalysts made from recovered pollutant P as non-fertilizer phosphoric products with significant market value in Oil & Gas and the Energy Storage sectors.

This research aims to develop a new, disruptive approach to nutrient recovery, which focuses on the residual soluble reactive nutrients (nitrogen and phosphorus), using adsorptive nanomaterials that carry out more than removal/recovery/partial reuse of these pollutants. In total, this novel approach using commercial available products provides "5Rs" of sustainable treatment of soluble reactive pollutant nutrients, i.e. Removal, Recovery, Reconcentration, Reuse and Recycle. Because effluent soluble reactive nutrients are captured and ultimately reused, the approach is supplementary and complementary to the existing nutrient recovery precipitation processes.

#### MATERIALS AND METHODS

Hybrid ion exchange (HIX) nanotechnology has been developed and commercialized to remove trace levels of arsenic (10-500 ppb) from drinking water in the US and several countries. HIX Nanomaterials are conventional anion exchange resin beads dispersed with bound nanoscale Fe(III), or more recently, with Zr(IV) particles (Figure 1; Padungthon et al., 2015). In this research, performed at Lehigh University, HIX-Nanomaterials were found to be extremely effective in capturing phosphate, nitrate and selenium by adsorptive mechanisms because dissolved oxyanions/nitrate/phosphate chemistry in the aqueous phase is very similar to that of arsenic. This strong selectivity makes dissolved nutrient pollutant removal from wastewater or freshwater at low or high effluent concentrations easy to implement. HIX-Nano consists of a regenerable media and the resin material can withstand the rigors of multiple regenerations, where multiple reuse of "refreshed" media lowers the overall production cost. No noticeable drop in sorption capacity has been recorded after prolonged use.

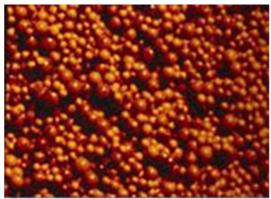
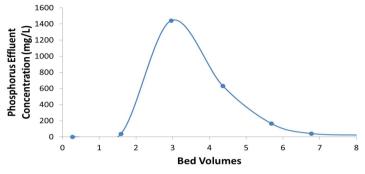


Figure 1: Picture of HIX-(Fe)Nano resin beads

#### **RESULTS AND DISCUSSION**

The key to nutrient recovery using HIX-Nano is regeneration of the spent media. The HIX-Nano capture efficiencies and recoveries (desorption) have been researched and evaluated. The conventional practice is to use a weak alkaline solution of caustic soda (NaOH). However, Na has little nutrient value. A representative elution curve (Figure 2) shows the NaH<sub>2</sub>PO<sub>4</sub> concentration

profile (as P) vs. bed volumes alkaline rinse (NaOH) for regenerant P solution desorbed from HIX-(Zr) Nano. Similar results are generated using HIX-(Fe) Nano.





However, if ammonium hydroxide or potassium hydroxide (KOH) or both are used as the alkaline rinses to generate commercially viable liquid fertilizer solutions, the resultant fertilizer solution (N-P-K) can be customized to the extent of: X%- P% - Y%, where X can be 0–10% (N) and Y can be 0–10% (K) as long as X + Y < or = to 10%. Hence, there will be an unlimited combination of customized N-P-K formulations possible. A typical end-product after recovery and reconcentration is an N-P-K liquid fertilizer, 3-0.28-3, used for hydroponic lettuce growing.

Current research looks at the recovery and further concentration of HIX-Nano regenerant solutions for manufacturing of non-fertilizer products. Leading candidates, based on high-purity ammonium phosphate solutions made from recycled pollutant nutrients phosphate (and nitrate) are: fluidized catalytic cracking (FCC) Catalyst - Phosphate Zeolite; Li-ion Battery Cathode Material - LiFePO<sub>4</sub>, including novel nanostructured material; and, Heterogeneous Biodiesel Catalyst - K<sub>3</sub>PO<sub>4</sub>.

Moreover, research is being performed on the development of an advanced integrated biological-physicochemical unit process model for the HIX-Nanotechnology, within the framework of the generic nutrient recovery model (NRM) techno-economic library developed by Vaneeckhaute (2015). The latter includes anaerobic digestion of organic waste for generation of biofuels (biogas) and transformation of organic pollutant nutrients N and P into inorganic, soluble nutrients now capturable by HIX-Nano and hence, integrated into NRM updates. The current model updates will help guide the most cost-effective product line mix of fertilizer and catalytic chemicals for more rapid acceptance by the end-users of such "green" products.

#### CONCLUSION

Hybrid ion exchange (HIX) nanotechnology has successfully been applied at pilot scale to recover nutrients from a variety of wastewaters. A typical resulting product after recovery and reconcentration is an N-P-K liquid fertilizer, 3-0.28-3, used for hydroponic lettuce growing. Current research targets the recovery of nutrients as non-fertilizer products with significant market value in Oil & Gas and the Energy Storage sectors. Moreover, a new generation biological-

physicochemical mathematical process model is being developed for HIX-Nano in order to optimize and control bio-product quality and process performance in a cost-effective way.

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# Energetic Valorisation of Cheese Whey using UASB Technology: a case-study

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#### SUMMARY

This study was focused on energetic valorization of cheese whey using UASB anaerobic digestion; whey represents a strongly polluted stream, which needs to be properly treated. In particular, the mountain area of Friuli-Venezia Giulia region was selected as a case-study, due to the presence of a number of cheese factories and a UASB reactor, actually inactive, located in municipal WWTP of Tolmezzo (Ud).

The results showed that first and second cheese whey were characterized by extreme COD value, up to 105 g/L, significant solid matter presence (6.63-7.44% TS) and high phosphates concentrations (527-530 mg/L); a major difference between the matrixes was lipids concentration, that was significantly higher in first whey (33.0 g/L versus 2.4 g/L). Elemental analysis highlighted high C (35.8-43.1%) and low N (1.0-2.4%) concentrations of whey solid matter. A useful dilution of whey (1.50 v/v) was used in continuous tests, that were executed both on laboratory and pilot-scale: the results showed excellent COD removal, as high as 84.7%, and a methane production of 0.193  $Mm^3 CH_4/Kg COD_{removed}$ , that boosts for anaerobic valorization of this substrate.

#### INTRODUCTION

Cheese whey is one of the main cheese factories sub-products, and is a highly polluted stream, not amenable to be treated in conventional municipal treatment plants; in fact, it can create serious problems of organic burden (Janczukowicz et al., 2008). Dairy effluents are typically characterized by high biodegradability (BOD<sub>5</sub>/COD ratio of 0.4-0.8) and COD concentration as high as 100 g/L; organic matter is mainly due to the presence of milk carbohydrates and proteins, such as lactose and casein (Rivas et al., 2010). Usually, biological and chemical-physical processes are used to treat these effluents (Kushwaha et al., 2010); however, when biological processes are not fully controlled, lactose and casein decomposition can generate odors and attract insects (Rivas et al., 2010).

#### MATERIAL AND METHODS

All the analyses were carried out according to Standard Methods for Examination of Water and Wastewater (APHA, 2005). In particular, the following parameters were analyzed: TS, VS, tCOD, sCOD, alkalinity, NH<sub>3</sub>-N, PO<sub>4</sub><sup>3-</sup>, SO<sub>4</sub><sup>2-</sup>, VFA, TKN, pH, macromolecular analysis (carbohydrates, proteins, lipids), elemental analysis (C, H, N, S). Cheese whey was withdrawn from local facilities, and first whey was collected and analyzed separately from second whey; granular sludge was taken from a full-scale IC reactor.

Continuous tests were first performed on a lab-scale UASB reactor (operating volume of 11.5 L). Flow rate was fixed, in order to obtain an HRT of 30.2 h. Mean OLR was calculated as 0.88 Kg  $COD/m^3d$ .

The pilot-UASB reactor was composed of: influent tank (1000 L volume), pre-acidificator (40 L volume), provided of a stirrer and a pH-controller (with set-point at 7.0), UASB column (60 L volume), hot water recirculation system (which maintained the reactor at the T of 37±2 °C) and biogas registration unit (µFlow, Bioprocess control). As for operating conditions, mean OLR was calculated as 0.81 Kg COD/m<sup>3</sup>d, up-flow velocity was 0.06 m/h and HRT was set at 40 h.

As for substrate characteristics, for all continuous tests (both on laboratory and pilot scale), cheese whey was diluted with tap water in a proportion of 1:50 v/v.

#### RESULTS

Chemical-physical characterization underlined the high COD concentration of both first (105.0 g/L) and second whey (81.8 g/L); the high VS/TS ratio (85.2-90.4%) indicated general biodegradability of solid matter. pH was slightly acidic (5.5-5.8); phosphates concentration was significant (527-530 mg P/L), while ammonia was quite low, in particular in second whey (3.2 mg/L). TKN concentration was higher in first whey (332 mg/L) than second whey (28 mg/L). VFA concentration was 41 mg/L in first whey, while it was negligible (1 mg/L) in second whey. Sulphates were detected only in second whey (55.5 mg/L).

As for macromolecules, high carbohydrates concentration was found both in first (27.5 g/L) and second whey (43.8 g/L), while lipids were mainly present in first whey (33.0 g/L). Protein concentration was generally low in both substrates (0.16-1.8 g/L).

Finally, as for elemental composition, a high carbon content was present mainly in first whey (43.1%), while N was found in little percentage in both matrixes (1.0-2.4%).

Parameter	First whey	Second whey
tCOD <sup>a</sup> (g/L)	105.0	81.8
sCOD <sup>b</sup> (g/L)	68.6	62.5
TS <sup>c</sup> (% w/w)	7.44	6.63
VS <sup>d</sup> (% w/w)	6.73	5.64
VS/TS (%)	90.4	85.2
Alkalinity (mg CaCO₃/L)	1297	1153
рН	5.5	5.8
PO <sub>4</sub> <sup>3- <i>e</i></sup> (mg P/L)	530	527
SO <sub>4</sub> <sup>2-<i>f</i></sup> (mg/L)	<2	55.5
NH <sub>3</sub> -N <sup>g</sup> (mg N/L)	44.1	3.2
TKN <sup>h</sup> (mg N/L)	332	28
VFA <sup>i</sup> (mg/L)	41	1
Carbohydrates (g/L)	27.5	43.8
Proteins (g/L)	1.8	0.16
Lipids (g/L)	33.0	2.4

Table 1: Chemical-physical characterization results of first and sec	ond cheese whey
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Parameter	First whey	Second whey
C (% w/w)	43.1	35.8
H (% w/w)	5.8	5.4
N (% w/w)	2.4	1.0
S (% w/w)	0.0	0.0

<sup>*a*</sup>= Total Chemical Oxygen Demand; <sup>*b*</sup>=Soluble Chemical Oxygen Demand; <sup>*c*</sup>=Total Solids; <sup>*d*</sup>=Volatile Solids; <sup>*e*</sup>=Phosphates; <sup>*f*</sup>=Sulphates; <sup>*g*</sup>=Ammonia; <sup>*h*</sup>=Total Kjeldahl Nitrogen; <sup>*i*</sup>= Volatile Fatty Acids

A first series of tests were performed using a lab-scale UASB reactor; mean influent COD concentration was 1.11 g/L, even if some fluctuation occurred in this parameter (in the range of 0.73-1.50 g/L), due to the extreme heterogeneity of the substrate. Good tCOD and sCOD removal were observed, with mean values of 69.0% and 72.7% respectively; mean  $CH_4$  production rate was 63 NmL/h.

Pilot-UASB tests results were better than that obtained at lab-scale: while influent COD concentration was  $1.31\pm0.49$  g/L, mean COD removal was  $84.7\pm8.3\%$ , leading to a mean effluent COD of 0.18 g/L. Mean CH<sub>4</sub> production was 323 NmL/h, corresponding to a specific methane production of 0.193 Nm<sup>3</sup> CH<sub>4</sub>/Kg COD<sub>removed</sub>.

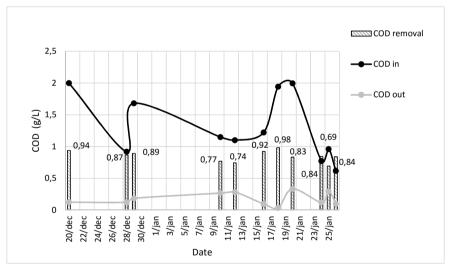


Figure 1: Influent and effluent COD concentration (in g COD/L) and COD removal from pilot UASB tests on cheese whey

# DISCUSSION

Cheese whey characterization results were consistent with other literature studies (Carvalho et al., 2013; Erguder et al., 2001; Kalyuzhnyi et al., 1997); however, from scientific papers a high variability in cheese whey characterization emerges, in particular in COD (range of 50-102.1 g/L) and pH (3.8-6.5) (Carvalho et al., 2013).

Erguder et al. (2001) found a highly acidic pH of 3.44-3.92 in cheese whey, while COD concentration was 55.3-74.5 g/L (lower than that of the present study). Moreover, a lower phosphates concentration of 124 mg/L was reported.

According to the classification proposed by Carvalho et al., 2013, based on substrate pH, the analyzed whey falls in an intermediate range between acidic whey (pH<5) and sweet whey (pH=6-7). Little data are available in literature related to elemental composition and macromolecular compounds of cheese whey; in Kalyuzhnyi et al., 1997, a lower lipid concentration of 0.4-5.7 g/L and a higher protein concentration of 2.3-33.5 g/L is reported, if compared to the actual results. As for operating conditions of pilot-UASB tests, in literature most of the experiments were conducted at 35-37 °C, HRT ranging from 2 to 7 days (Prazeres et al., 2012; Karadag et al., 2015). OLR were generally higher than that used in the present study, with a maximum value of 31 Kg COD/m<sup>3</sup>d (Gutierrez et al., 1991).

Erguder et al. (2001) treated diluted cheese whey using UASB technology, and they obtained an excellent COD removal of 95-97%, with a high methane yield of 0.424 m<sup>3</sup> CH<sub>4</sub>/Kg COD, significantly higher than the yield obtained in this work (0.193 m<sup>3</sup> CH<sub>4</sub>/Kg COD), using a HRT of 2.1-2.5 days. They reported effluent COD of 1.7-2.7 g/L, and OLR applied was as high as 22.6-24.6 g COD/L day; moreover, a high methane content of 77% was founded in biogas.

Blonskaja and Vaalu (2006) treated undiluted cheese whey in a contact reactor, followed by a UASB column: they obtained 98% COD removal, using a 2.5 days HRT, and a high system stability was highlighted. In their work, cheese whey exhibited similar characteristics to the present work, with TS in the range of 5.7-7.1%. and pH of 3.8-6.3, even if COD was lower (60.3-66.7 g/L). They obtained a gas production of 0.2-18.5 L/d (78% CH<sub>4</sub>).

In conclusion, this study can be regarded as a full characterization of first and second whey, followed by pilot-UASB tests, that showed the feasibility of valorization of cheese whey in a narrow area, such as the mountain area of Friuli-Venezia Giulia region. This can lead to an improvement in whey management, also through collaboration with cheese factories, in order to select the best option for the treatment of this substrate. Also co-digestion with other high-loaded substrates, such as pulp & paper wastewater and OFMSW leachate, is being under consideration, for enhancement of biogas yield and optimization of operating conditions.

#### ACKNOWLEDGEMENTS

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# Two-stage Anaerobic Digestion: Towards Pipeline-Quality Biogas Production

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#### SUMMARY

Nowadays, the demand for renewable energies has driven the efforts of scientists around the world to find sustainable technological pathways using biomass as source of energy which eventually can replace traditional fuels. In this study, we evaluated the biogas production potential of two-stage anaerobic digestion as this configuration gives higher biogas production and increases COD removal. A strengths, weaknesses, opportunities and threats (SWOT) analysis was performed in order to evaluate the 2-stage AD system. Also, the current problems are summarized and several future development directions for enhanced biogas production are pointed out.

#### INTRODUCTION

Over the recent decades, the world has been looking at renewable fuels as result of greenhouse gas pollution and the depletion of traditional fossil fuels. The demand for renewable energies has driven the efforts of scientists around the world to find a sustainable technological pathway using biomass as source of energy which eventually can replace traditional fuels. Anaerobic digestion is becoming an efficient system not just for treatment of wastes but for the generation of pipeline-quality biogas. Different types of biowaste have shown to be suitable for such purpose. Besides gas production, anaerobic digestion procedures, resulting in a sustainable closed loop for biogas production. Of course, two-stage process requires further optimization through the analysis of microbial and process dynamics, as some two-stage biogas plants are already operating.

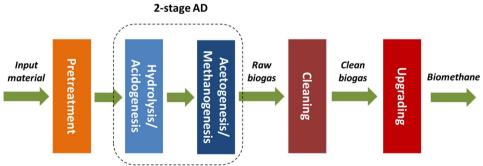


Figure 1: The scheme presents the 2-stage AD process configuration

Current research is underway to improve further the two-stage anaerobic digestion by investigating the microbial ecology and process conditions changes of each stage separately. The existing results show that two-stage anaerobic digestion is a promising technology providing higher methane yield and increasing solids removal. Therefore, the objective of this study is to review and compare the single and two-stage anaerobic digestion and assess their performances from different substrates.

SWOT analysis was performed in order to identify the strengths and weaknesses of the technology and the opportunities and threats in the environment. Having identified these factors strategies can be developed which may build on the strengths, eliminate the weaknesses, exploit the opportunities or counter the threats. The advantage of SWOT analysis or the TOWS matrix is its attempt to connect internal and external factors to stimulate new business strategies.

#### MATERIAL AND METHODS

This study is reviewing the single and two-stage anaerobic digestion according to literature data. It compares their performances in terms of biogas/methane yield and COD/VS removal. SWOT analysis is applied in order to evaluate the 2-stage process configuration by capitalising its strengths, overcoming weaknesses, exploiting opportunities, and countering threats. Moreover, it is about identifying the most important issues, setting priorities, appraising the options, and taking action.

#### RESULTS

Based on literature data, performances in terms of biogas/methane yield and COD/VS removal are given in Fig. 2 and Fig. 3. The most significant contributions of biogas are from oily food and cattle manure. The feedstock mainly dictates the quality of the products such as biogas, anaerobic surplus sludge and the necessity of effluent post-treatment at the end of the digestion process. The degradation rates of waste organic matter can vary significantly with the substrate composition, e.g. protein-, carbohydrate-, and fat content.

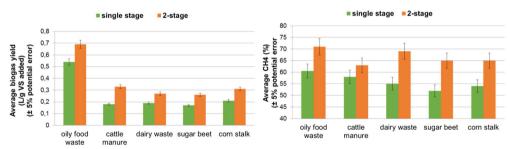
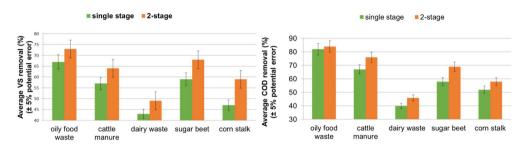
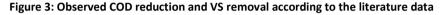


Figure 2: Observed biogas and methane yields according to literature data





Although fats require high retention times due to their poor biodegradability, they provide the highest biogas yields (Fig. 2). Fig. 3 shows that the VS and COD removal percentages of different feedstocks. Depending on the chemical and physical composition of the waste, the resulting overall degradation rate of organic matter varies between less than 20% to over 90%. The results from the Strengths, Weaknesses, Opportunities, and Threats (SWOT) analysis are provided by the matrix scheme and can be seen in Fig. 4.

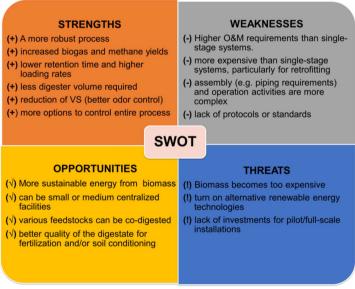


Figure 4: A SWOT matrix for 2-stage anaerobic digestion

# DISCUSSION

SWOT analysis is a very useful tool to help business strategies. Through the analysis, we conclude that the 2-stage AD system can be optimized further. Because of lower retention times, which facilitate higher loading rates, these systems require less digester volume to handle the same

amount of input volume than single-stage systems. Different studies reported that reduction of VS and COD can be achieved, providing also better odor control. From technical viewpoint, different configurations can be made in order to address foaming as well as short circuiting of solids is reduced through the separation of stages and optimization of retention time in each stage. However, the operation and maintenance requirements are higher and more complex than single-stage systems. The system can be more expensive than single-stage systems, particularly for retrofitting.

To sum up, biogas is a promising biofuel that offers sufficient characteristics compared to conventional fuels and the existing results show that two-stage anaerobic digestion is a promising technology providing higher methane yield and increasing solids removal. There is room for further progress via optimization so that 2-stage system makes up an emerging economic competitiveness. Furthermore, recent metagenomics studies focus on microbiological performance of 2-stage AD process using advanced microbial population analytical tools e.g. proteomics, genomics, Next Generation Sequencing.

#### ACKNOWLEDGEMENT

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# Comparison of Biogas Upgrading Systems with different Biomethane usage Paths and decentralised Biogas Usage in CHP Units

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#### SUMMARY

Biogas upgrading can have advantages from GWP perspective in comparison with decentral biogas usage especially if no heat usage on side of the biogas plant is possible. Whether advantages exist depends on effectiveness of the upgrading system (methane leakage, energy demand), national electricity mix and chosen reference system by including (by-) product substitutes in the assessment such as electricity production. In most cases, biomethane use in new CHP units has to be preferred. The lower the GWP of the national electricity production mix is the easier biogas upgrading can generate advantages compared with decentral biogas usage.

#### INTRODUCTION

Biogas production mostly takes place in rural areas. This has great advantages for the input and digestate logistic. But there is the disadvantage, that often no sufficient heat sinks are available. This leads to inefficiency in energy use. In Germany, according to the DBFZ survey, 45 % of the surplus of heat is used for e.g. district heating, heating of buildings, greenhouses, stables and drying (DBFZ, 2012).

One solution can be upgrading biogas to biomethane, which can be fed into natural gas grid and used where it is needed. Table 1 shows current state of biogas upgrading in selected European countries.

Country	Biogas Plants [unit]	Biogas Upgrading Plants (Fed In) [unit]	Upgrading Capacity [Nm³/h]	Gas filling stations [unit]
Austria	421	10 (7)	2,000	203
Belgium	119	0	0	15
Croatia	12	0	0	0
Denmark	137	1 (1)	180	4
Finland	34	5 (2)	959	18
France	256	3 (2)	540	149
Germany	9,066	120 (118)	72,000	904
Hungary	58	1 (0)	0	0
Ireland	22	0	0	0
Italy	1,264	1 (0)	540	903
Luxembourg	31	3 (3)	894	7

Country	Biogas Plants [unit]	Biogas Upgrading Plants (Fed In) [unit]	Upgrading Capacity [Nm³/h]	Gas filling stations [unit]
Sweden	187	53 (11)	16,800	190
The	211	16 (16)	6,540	150
U.K.	265	3 (3)	1260	40

# MATERIAL AND METHODS

The ecological assessment was performed based on the standardisation for life cycle assessment (DIN EN ISO 14040). System boundaries start from raw biogas delivered from biogas plant<sup>1</sup> and end with the utilisation unit e.g. CHP-unit, gas filling station or boiler. Energy and material inputs were taken into account, whereas infrastructures were excluded except for the gas grid. As far as possible data from existing biogas upgrading plants were collected. Further background data, up- and downstreaming process data were taken from the Ecoinvent database and completed with literature data. The impact assessment focused on global warming potential (GWP). For the biogas upgrading the pressure water scrubbing technology was chosen, which is most common in Europe (FNR, 2013).

# RESULTS

The substitution of natural gas with upgraded biogas doesn't have any advantages compared with a decentralised CHP unit with 40 % external heat usage. Due to high electricity consumption for upgrading biogas countries with low carbon dioxide emissions of their national electricity production can result in little benefits for GWP. Regarding GWP France has very low emissions per kWh<sub>el</sub> so biogas upgrading is more efficient there. If assumed that new CHP-units are built for the usage of upgraded biogas there will exist little benefits for upgrading in nearly all countries. Here benefits for substitution of gas heating and national electricity mix have been considered.

# DISCUSSION

The calculation shows that Biogas upgrading can have advantages from GWP perspective in comparison with decentral biogas usage especially if no heat usage on side of the biogas plant is possible. Whether advantages exist depends on the effectiveness of the upgrading system (methane leakage, energy demand), the national electricity mix and the chosen reference system. In most cases the biomethane use in new CHP units has to be preferred. In all cases the lower GWP for the national electricity market the higher the advantages of upgraded biogas. So upgrading makes more sense if the national electricity mix has low GWP. Beside these environmental benefits, the natural gas grid can be looked like a gas storage. So upgraded biogas that is fed in the natural gas grid is stored, electricity and heat can be produced on demand. This results in more flexibility in energy production and lower dependence on natural gas imports.

<sup>&</sup>lt;sup>1</sup> The biogas plant is not part of the analysed system

# ACKNOWLEDGEMENT

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THRÄN D., PERSSON T., DANIEL-GROMKE J., PONITKA J., SEIFFERT M., BALDWIN J., KRANZL L., SCHIPFER F., MATZENBERGER J., DEVRIENDT N., DUMONT M., DAHL J., BOCHMANN G. (2014). "Biomethane: Status and Factors Affecting Market Development and Trade". IEA Bioenergy; 2014.

# Liquid Fraction of Digestate as good as Chemical Fertiliser? – Observations from a Three-Year Field Experiment

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# INTRODUCTION

To safeguard water quality and protect waters against agricultural pressures, a comprehensive framework of EU legislation has been introduced (ex. Nitrate Directive 91/676/EC and Water Framework Directive 2000/60/EU) and translated to member states action plans. In Flanders (Belgium), restrictions are imposed on both nitrogen and phosphate rates coming from livestock manure. While the maximum allowed nitrogen rate is 170 kg N/ha/year (European Commission, 1991), phosphate application rates are quite lower, ranging from 40 to 70-90 kg  $P_2O_5$ /ha/year for P-saturated area and areas with a low phosphate binding capacity (Amery and Schoumans, 2014), respectively. This makes it impossible to fill the maximum allowed rate for nitrogen entirely by livestock manure. Therefore, despite of existing manure surpluses, expensive and energy consuming synthetic fertilizers are utilized to meet crop nutrient requirements. Nitrogen recovery and thus valorization of remaining N in these manure surpluses could however decrease the need for synthetic fertilizers.

In Flanders, livestock manure is often treated by anaerobic co-digestion, resulting in valuable biogas and digestate as a side stream that needs further processing. As a first step often mechanical separation is applied resulting in a P-rich solid fraction and a P-poor, NK nutrient-rich liquid fraction (LF). The latter, could have great potential as a replacement of synthetic N and K fertilizers in P-saturated areas. To evaluate the nutrient use efficiency of LF, a three-year maize field trial (2011-2013) was conducted in Wingene, Belgium. During these field trials, the impact of LF on soil quality and maize production was determined as compared to conventional fertilization regime (livestock manure + synthetic fertilizers).

#### MATERIAL AND METHODS

Based on the soil characteristics and crop demand, the advice given on fertilizer requirements was formulated at 150 kg effective N ha<sup>-1</sup>, 180 kg K<sub>2</sub>O ha<sup>-1</sup> and 30 kg MgO ha<sup>-1</sup> in 2011, and 135 kg effective N ha<sup>-1</sup>, 250 kg K<sub>2</sub>O ha<sup>-1</sup> and 60 kg MgO ha<sup>-1</sup> in 2012 and 2013. Three different experimental treatments were tested in four replicated subplots (n = 3) spread in the field in order to minimize the potential influence of variable soil conditions on the results (Table 1). The size of individual subplots amounted to 9 m x 7.5 m.

Treatment 1 presents the reference scenario in which only animal manure and synthetic fertilizers (N,  $K_2O$ ) were used. In treatment 2, animal manure was converted into digestate through anaerobic co-digestion and digestate mixtures were spread to the field, with (2011 and 2013) or without (2012) the addition of synthetic fertilizer. While in treatment 1  $P_2O_5$  was the limiting factor for manure application, in treatment 2 N became the limiting factor, as the ratio of  $P_2O_5$  over effective N is in general lower for digestates compared to animal manure. Finally, in treatments 3 LF of digestate was added in combination with animal manure as a replacement for synthetic N.

Table 1: Overview of three different experimental	reatments (n=3) for three consecutive years
(2011 - 2013).	
	Digestate /I F

TRT	Year	Synthetic start N	Synthetic N	Animal manure	Digestate/LF digestate mixture	LF digestate	Synthetic K₂O
1	2011	х	х	х	-	-	х
	2012	х	х	х	-	-	х
	2013	х	х	х	-	-	х
2	2011	х	х	-	х	-	х
	2012	-	-	-	х	-	-
	2013	-	-	-	х	-	х
3	2011	х	-	х	-	х	х
	2012	х	-	х	-	х	х
	2013	х	-	х	-	х	х

# RESULTS

Results showed that the highest biomass yields were obtained when the LF was used as a P-poor fertilizer in addition to livestock manure, as compared to a reference scenario (Figure 1). The only exception was 2012 when all treatments, including the reference, have resulted in lower biomass yield due to exceptional weather conditions. The NO<sub>3</sub>-N residue in the soil profile (0-90 cm) between 1<sup>st</sup> of October and 15<sup>th</sup> of November was below the legal limit of 90 kg/ha, except for 2011 when approximately 40% of the NO<sub>3</sub>-N measurements in West Flanders exceeded the allowable level due to exceptional dry spring and wet summer. In the following two years, all NO<sub>3</sub>-N values were below the legal limit, resulting in no significant differences over a three-year trial (Figure 2).

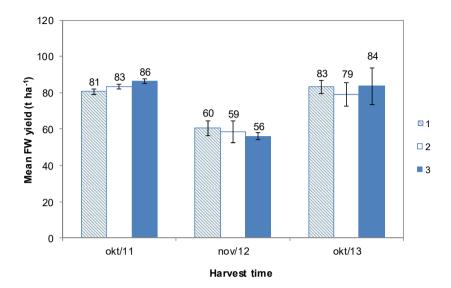


Figure 1: Fresh weight (FW) biomass yield (t ha-1) at harvest time for the three different fertilization treatments

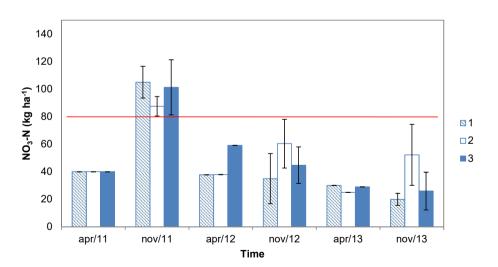


Figure 2: NO<sub>3</sub>-N residue (kg ha<sup>-1</sup>) in soil layer 0-90cm at harvest time for the three different fertilization treatments in time. The line indicates the maximum allowable level of nitrate residue in soil (80 kg NO<sub>3</sub>-N ha<sup>-1</sup> for sandy soil in 2012 and 2013) between October 1<sup>st</sup> and November 15<sup>th</sup> according to Flemish environmental standard. In 2011, the maximum allowable

# level of nitrate residue in soil was 88 kg $NO_3$ -N $ha^{-1}$ . Error bars indicate standard deviations (n=3)

# CONCLUSIONS

The results indicate that LF has a potential to be used as a substitute for synthetic fertilizers, allowing farmers to meet crop nutrient requirements by good economic and environmental farming practice. Unfortunately, use of LF is currently being hampered by legislative issues and perception. We expect that this research can act as a catalyst for recognition of LF as a valuable fertilizer within the European legislation.

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EUROPEAN COMMISSION (1991). Directive of the Council of 12 December 1991 concerning the protection of waters against pollution caused by nitrates from agricultural sources (91/676/EC). Official Journal of the European Communities L375, pp. 0001-0008.

# The Issues of Nitrogenous Components during Anaerobic Digestion

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#### SUMMARY

Anaerobic digestion is widely used as a source of renewable energy. The aim of the ReciDigest project is to investigate if treatment of the effluent of the digester with an advanced oxidation process (AOP) and its recycling to the digester can improve the overall biogas yield. As the AOP most likely will give rise to the oxidation of  $NH_4^+$  to  $NO_2^-$  or  $NO_3^-$  the fate of the latter in the digester was investigated. This study clearly demonstrates that  $NO_2^-$  and  $NO_3^-$  are denitrified during the subsequent digestion process resulting in the formation of nitrogen gas. Lab scale experiments demonstrated that this nitrogen issue can be better solved by removing the  $NH_4^+$  present in the effluent by means of a strip and scrub process prior to the application of an AOP.

#### INTRODUCTION

Anaerobic digestion of organic waste is generally considered as one of the most sustainable technologies to produce renewable energy (Khalid *et al.*, 2011). Due to the presence of rigid and not readily biodegradable material, a long residence time is required in the digester (15 - 30 days). Even so, often only 50 % of the total organic dry matter is converted into biogas (Park *et al.*, 2005). This limited conversion efficiency in combination with the reduction of governmental grants have placed the Flemish biogas sector under significant pressure. In the ReciDigest project, it is investigated if treatment of the effluent of the digester with an advanced oxidation process (AOP), such as ozonation or peroxidation, and recycling it to the digester can improve the overall biogas yield. Besides the low conversion efficiency, high ammonium contents are often a problem as they inhibit methanogenesis. The use of an AOP is expected to give rise to the oxidation of ammonium to nitrite or nitrate and such can circumvent the ammonium problem. The main objective of this study is therefore to investigate the behavior of these nitrogenous components during the digestion process. A second objective is to find out whether it is possible to remove the ammonium from the digestate to simplify the nitrogen issue. From this perspective, a strip and scrub process is developed and tested at lab scale.

#### MATERIALS AND METHODS

#### Origin of the digestate and substrate

On April 18, 2016 25 L of substrate and 50 L of digestate were obtained from Mydibel NV, a potato processing plant. The digestate comes from a mesophilic digestion (operating temperature of 37

°C). The substrate is the biomass that is fed to the digester and comprises potato peels, rejected fried products, etc. Prior to characterization, both the substrate and digestate were blended.

# Biogas experiments performed in batch mode

Figure 1 shows the configuration in which the biogas experiments were performed in batch mode. The digestate (2400 mL) and the substrate (100 mL) are introduced into an Erlenmeyer flask of 3 L (1). Chemical products may or may not be added to this mixture (see Table 1). This flask is placed in an incubator with an operating temperature of 37 °C. The flask is connected to a recipient filled with 0.01M  $H_2SO_4$  (2) by means of a gas-tight tubing. The volume of (bio)gas formed is measured by the pressure driven displacement of the  $H_2SO_4$  solution from recipient 2 to the graduated recipient 3.



Figure 1: Configuration used to perform the biogas experiments

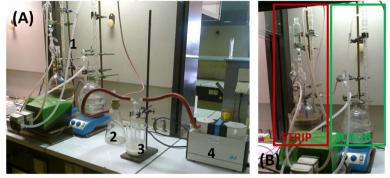
Several experiments were carried out to investigate the effect of nitrite or nitrate in combination with extra ammonium on the volume of biogas formed. An overview of the experimental conditions is given in Table 1.

rable 1: Summary of the experimental conditions of the mesophilic blogas experiments			
Objective	Concentration of chemicals added		
Reference	-		
Effect of NO <sub>2</sub> -	2.5 g.L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup> -N (NaNO <sub>2</sub> ) + 2.5 g.L <sup>-1</sup> NH <sub>4</sub> +-N (NH <sub>4</sub> Cl)		
Effect of NO₃ <sup>-</sup>	2.5 g.L <sup>-1</sup> NO₃ <sup>-</sup> -N (NaNO₃) + 2.5 g.L <sup>-1</sup> NH₄ <sup>+</sup> -N (NH₄Cl)		
Effect of NaCl	10.4 g.L <sup>-1</sup> NaCl		

For the reference experiment, no chemicals were added to the mixture of digestate and substrate. To study the effect of NaNO<sub>2</sub> or NaNO<sub>3</sub> in combination with NH<sub>4</sub>Cl the chemicals were dissolved in the digestate. The addition of NaNO<sub>2</sub>/NaNO<sub>3</sub> in combination with NH<sub>4</sub>Cl inevitably results in the addition of NaCl into the system and therefore also in a change of the ionic strength. In the last experiment, an equimolar amount of NaCl was thus added to the digestate to examine its effect on the digestion process.

#### SETUP FOR AMMONIUM REMOVAL BY STRIPPING & SCRUBBING

The removal of ammonium was carried out through a strip and scrub process at lab scale as illustrated in Figure 2. Figure 2A shows the complete setup used for the ammonium removal in which four parts can be distinguished. With a vacuum pump (4) air is drawn through the stripping and scrubbing part of the setup (1). The vacuum is regulated by a valve (2) and a cold finger (3) is used to protect the vacuum pump against the gas vapours formed.



# Figure 2: Picture of (A) the complete setup and (B) detail of the strip and scrub process at lab scale

Figure 2B shows a detailed picture of the stripping and scrubbing parts of the setup in which Vigreux columns provide an exchange surface between gas and liquid phase in both parts. In the stripping part, the supernatant of the digestate is continuously stirred using a magnetic stir bar and continuously pumped over a Vigreux column using a peristaltic pump. The ammonium present is volatized as ammonia by increasing the temperature (to 50 °C) and the pH to 8.5 using 4 M NaOH. The air that is drawn through the system takes the ammonia from the stripping part to the scrubbing part, which initially contains 1 L  $H_2SO_4$  1 M. The sulphuric acid is also continuously stirred and pumped over a Vigreux column resulting in the formation of  $(NH_4)_2SO_4$ .

#### ANALYTICAL METHODS

The pH of the samples was measured with a pH meter from Mettler Toledo. Total Organic Carbon (TOC) was measured with a TOC Analyzer (TOC- $V_{CPN}$ ) from Shimadzu. Ion concentrations were measured with an ion chromatograph from Metrohm.

#### **RESULTS AND DISCUSSION**

#### Characterisation of the digestate and substrate

The substrate has the following characteristics: pH 3.9,  $NH_4^+$  0.2 g.L<sup>-1</sup> N and TOC 46 g.L<sup>-1</sup> C. The digestate has a pH of 7.6, an ammonium concentration of 1.8 g.L<sup>-1</sup> N and a TOC concentration of 0.9 g.L<sup>-1</sup> C. Both the substrate and the digestate do not contain nitrite and nitrate (below detection limit of ion chromatograph).

The analysis of the digestate clearly shows that the digestion process results in a significant decrease in TOC. The substrate indeed contains a lot of starch (demonstrated by the high TOC value), derived from the potato products, which is very readily biodegradable. The residual

concentration of TOC is probably due to lignocellulosic structures present in the potato peel. In addition, an increase of ammonium is observed because of the release of the organically bound nitrogen during digestion. As mentioned, the digestate does not contain nitrite and nitrate and to simulate their oxidative formation by AOP and effect during subsequent digestion they were added as shown in Table 1.

#### Mesophilic biogas experiments

Four biogas experiments were carried out under different experimental conditions (see Table 1). Figure 3 shows the volume of (bio)gas produced as a function of time for these 4 experiments.

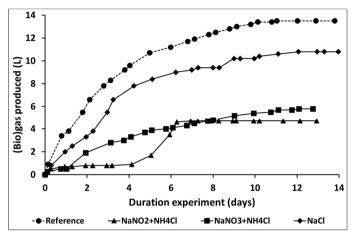


Figure 3: Volume of (bio)gas produced as a function of time for 4 different biogas experiments

The reference experiment results in approx. 14 L of biogas after 14 days of incubation. An obvious inhibitory effect is noted for the 3 other experiments, certainly for nitrite and nitrate. In addition, Table 2 shows the results of the analysis of the digestates of the 4 completed biogas experiments.

	рН	NH4 <sup>+</sup> -N	NO <sub>2</sub> N	NO <sub>3</sub> <sup>-</sup> -N	
	[-]	[g.L <sup>-1</sup> N]	[g.L <sup>-1</sup> N]	[g.L <sup>-1</sup> N]	
Ref	7.4	1.8	0	0	
Ref + NaCl	7.5	1.7	0	0	
Ref + NaNO <sub>2</sub> & NH <sub>4</sub> Cl	8.6	4.2	0	0	
Ref + NaNO <sub>3</sub> & NH <sub>4</sub> Cl	8.3	3.9	0	0.3	

Table 2: Analytica	I results of the f	our completed	biogas experiments
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The ammonium content remained unchanged for the 4 experiments (value for NaNO<sub>2</sub> and NaNO<sub>3</sub> is higher due to an extra 2.5 g.L<sup>-1</sup> NH<sub>4</sub><sup>+</sup>-N). No nitrite was detected, even for the experiment in which 2.5 g.L<sup>-1</sup> NO<sub>2</sub><sup>-</sup>-N was added. For the experiment with NaNO<sub>3</sub> only 0.3 g.L<sup>-1</sup> NO<sub>3</sub><sup>-</sup>-N was measured afterwards. Moreover, the manipulations with NaNO<sub>2</sub> and NaNO<sub>3</sub> clearly resulted in a

higher pH. Both aspects indicate the occurrence of a denitrification reaction. In the case of nitrate, the reaction is as follows:  $2NO_3^- + 10e^- + 2H^+ + 10\{H\} \rightarrow N_2 + 6H_2O$ .

Following Avogadro's law, 5 L and 4.4 L N<sub>2</sub> gas should be formed in the experiment with NaNO<sub>2</sub> and NaNO<sub>3</sub>, respectively. These calculated volumes agree with the registered volumes during both experiments (see Figure 3). Nitrite and nitrate are thus denitrified during anaerobic digestion resulting in (1) consumption of electrons that could otherwise be used for the biogas production and (2) the production of N<sub>2</sub> gas.

# Ammonium removal via stripping & scrubbing

To prevent the oxidation of ammonium to nitrite/nitrate by AOP, we recommend that ammonium would be removed prior to the use of the AOP. In this way, the nitrogen issues are avoided during the subsequent digestion process. For this purpose, a strip and scrub process was developed (see Figure 2) to remove the ammonium from the supernatant of the digestate. During a first preliminary test, we could remove 89 % of the ammonium present in the supernatant during the stripping part. Simultaneously, 80 % of the stripped ammonia was absorbed in 1 M  $H_2SO_4$  during the scrubbing part resulting in the formation of  $(NH_4)_2SO_4$ . The advantages of the strip and scrub process are threefold: (1) denitrification of nitrite/nitrate will not occur during digestion as these components cannot be formed during the AOP; (2) reduction of the ammonium content is favorable in view of its potential inhibitory effect and (3) the removed ammonium is recovered as  $(NH_4)_2SO_4$  which can be used as a nitrogen fertilizer.

# CONCLUSION

The ReciDigest project investigates if the overall biodegradability of the digestate can be enhanced by applying an AOP on the effluent. However, this is expected to result in the oxidation of ammonium to nitrite or nitrate. This study clearly demonstrates that nitrite and nitrate are denitrified during the subsequent digestion process resulting in the formation of nitrogen gas. The denitrification reaction is negative due to (1) the consumption of electrons and (2) in more general terms the production of a less pure biogas. This nitrogen issue can be solved by removing the ammonium present in the digestate prior to the use of the AOP by a strip and scrub process. The process ensures that the ammonium is stripped from the digestate and is recovered as  $(NH_4)_2SO_4$ .

#### ACKNOWLEDGEMENT

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# Greenhouse Gas Mitigation and Renewable Energy Production through Farm-Scale Anaerobic Digestion – Potential for Flanders and the European Union

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The development of farm-scale anaerobic digesters is recently gaining more interest due to its ecological and financial benefits. Such installations allow the conversion of proprietary biomass to biogas and digestate and thus contribute to the mitigation of greenhouse gas (GHG) emissions by avoiding uncontrolled emissions during long-term biomass storage (Marañón et al., 2011; Mesa-Dominguez et al., 2015; Miranda et al., 2015). The produced biogas, usually burnt in a combined heat and power (CHP) unit, will provide electricity and heat for farmers to become (partly) self-sufficient in their energy demand. However, most of the current farm-scale installations only use cattle slurry as input. Other agricultural sectors can still not benefit from the (partial) fulfillment of the energy requirements this technology can offer while rising energy prices become a more and more determining cost. Within the context of the Pocket Power project a sector scan for agricultural subsectors is ongoing for the region of Flanders, Belgium. This sector scan explores the possible transfer of the positive experiences with small-scale anaerobic digestion of cattle slurry to other agricultural streams (e.g. pig manure, crop residues). By means of data from the BIOSURF project, funded by the Horizon 2020 research and innovation program of the European Union, the results obtained for Flanders will be extrapolated to an overall potential for Europe. The BIOSURF project itself aims to describe opportunities and potential environmental burdens associated with biomethane production and consumption (Kirchmeyr, 2016).

The sector scan for Flanders is based on a multi-criteria analysis in which not only the amount of available biomass on the farm and the technical feasibility to digest new feedstocks will be important but also the legal restrictions, economic and ecological impact. The coherence of the different criteria will be crucial to decide on the sectors with the highest potential for a farm-scale digester. Using the emission factors researched from National Inventory Reports (NIR) for different animal categories and the number of livestock in EU Member States from FAO (2015) and EUROSTAT (2015), real emissions and energy potentials per animal category are calculated and extrapolated for the European Union in the BIOSURF project. The results can be used to

estimate a potential for small-scale anaerobic digesters across Europe while taking the unique specialties of farm-management in each region into account and incorporating the differences in the overall analysis.

The calculation of GHG emissions described in the EU Renewable Energy Directive (RED) lacks options to include the various specialties of biomethane compared to conventional liquid biofuels. Providing an alternative calculation model with accurate statistical values at European level thus highlights the importance of this study. Furthermore, quantifying the potential for farm-scale digestion in Europe will help (potential) end-users, policy makers and constructers in their decision making. Moreover, by investigating specific trends in Europe, countries could learn from each other on how to improve their farm-management to fully benefit from this technology.

#### ACKNOWLEDGEMENTS

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FLANDERS INNOVATION & ENTREPRENEURSHIP



The objective of the BIOSURF PROJECT (BIOmethane as Sustainable and Renewable Fuel) is to increase the production and use of biomethane (from animal waste, other waste materials and sustainable biomass), for grid injection and as transport fuel, by removing non-technical barriers and by paving the way towards a European biomethane market. This project has received funding from the European Union's Horizon 2020 research and innovation programme.

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# Molecular Analyses of Microbial Communities Reveal an Optimization of the Anaerobic Fermentation Process for Improved Biogas Production during Calcium-Nitrate Treatment

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#### SUMMARY

The production of renewable energy in industrial scale is among the primary research topics worldwide. In this context, the anaerobic digestion of volatile organic solids such as maize silage, manure and organic waste is used for the industrial production of biogas as a resource of renewable energy. One of the primary goals of biogas production plants is the optimization of the fermentation process towards a high-quality biogas. The most valuable component of highquality biogas is the clean-burning methane ( $CH_4$ ), whereas hydrogen sulfide ( $H_2S$ ), a side product of the anaerobic fermentation process, is unfavorable. Therefore, a major aim in the optimization of the anaerobic fermentation process is to maximize CH<sub>4</sub> production while minimizing H<sub>2</sub>S production. Empirical observations and reports of industrial plant operators repeatedly confirmed an improvement of biogas quality after dosage of a calcium-nitrate  $Ca(NO_3)_2$  solution to the anaerobic fermentation process. Therefore, we sampled control fermenters and fermenters treated with Ca(NO<sub>3</sub>)<sub>2</sub> and analyzed the structures and functions of microbial communities using molecular methods. We found a shift in community structure after treatment with  $C_{a}(NO_{3})_{2}$  to the benefit of denitrifiers and methanogens and at cost of sulfate reducing bacteria (SRB). Furthermore, quantitative real time PCR experiments revealed a significantly increased denitrification and methanogenesis activity, while sulfate reduction was notably reduced in most cases. We here propose a mechanistic concept for the mode of action of  $Ca(NO_3)_2$  as a tool to optimize the anaerobic fermentation process for the production of improved biogas quality.

#### INTRODUCTION

The conversion of biomass such as manure, maize silage and organic waste into biogas is an anaerobic process mediated by a complex microbial community. The process can be divided schematically into four phases: hydrolysis, acidogenesis, acetogenesis and methanogenesis. In the first phase particular organic polymers, such as carbohydrates, lipids and proteins, are hydrolyzed into sugars, fatty acids and amino acids, which are further degraded into intermediates like volatile fatty acids (VFAs), acetate, alcohols, carbon dioxide (CO<sub>2</sub>) and hydrogen (H<sub>2</sub>) during the acidogenesis and the acetogenesis. In the last phase, methane (CH<sub>4</sub>) is generated either from acetate (acetoclastic) or from hydrogen and carbon dioxide (hydrogenotrophic) (Klang et al. 2015). Besides methanogenesis, numerous other microbial processes are involved in the anaerobic fermentation of organic material. These include among others denitrification, which produces nitrogen gas (N<sub>2</sub>), nitrite (NO<sub>2</sub><sup>-</sup>) or laughing gas (N<sub>2</sub>O), and

also sulfate reduction, resulting in hydrogen sulfide ( $H_2S$ ). While methane is a very valuable component in biogas based on its energy efficiency and its environmental compatibility after combustion, hydrogen sulfide is highly toxic and impairs methane production (Schieder et al. 2003) and also the health of fermenter operatives in case of leakages (Truong et al. 2006). Furthermore, hydrogen sulfide is highly corrosive causing notable economic damages especially in combined heat and power stations (CHPs). Therefore, expensive measures are usually necessary to remove hydrogen sulfide from biogas prior to conversion. As a result, one major aim in anaerobic digester operation is to maximize the production of methane while minimizing the production of hydrogen sulfide. Empirical observations and reports of industrial plant operators repeatedly confirmed an improvement of biogas quality after dosage of a calcium-nitrate Ca(NO<sub>3</sub>)<sub>2</sub> solution to the anaerobic fermentation process. These improvements included (i) increased yield of methane, (ii) reduction in hydrogen sulfide production, (iii) significant substrate savings, (iii) improved viscosity and (iv) a more robust plant operation.

In this study, we aimed to reveal the biological effect of  $Ca(NO_3)_2$  treatment on microbial community structures and their metabolic activities to infer a mechanistic concept for the effect of  $Ca(NO_3)_2$  on the anaerobic fermentation process.

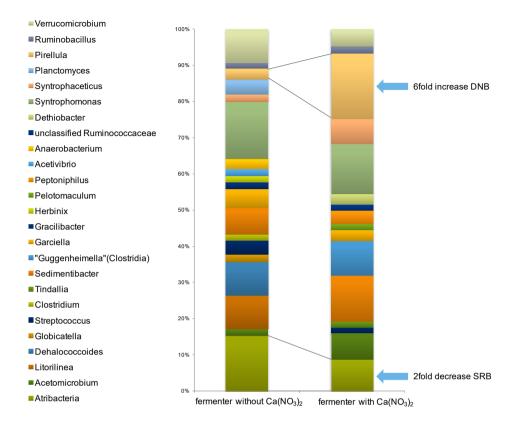
# MATERIALS AND METHODS

Samples were taken from fermenters treated with  $Ca(NO_3)_2$  and control fermenters without  $Ca(NO_3)_2$  of operating biogas plants. DNA and RNA were extracted from samples using MoBio's PowerSoil RNA extraction kit with the DNA accessory kit according to the manufacturer's instructions. We amplified taxonomic marker genes for bacteria (V3-V4 fragment of the 16S rRNA-gene using primers and protocol described in Klindworth et al. 2013) from the extracted and purified DNA and subjected the obtained gene fragments to Illumina MiSeq sequencing. Millions of obtained gene data were then analyzed according to standard computational data processing pipelines (Caporaso et al. 2010). For metabolic activity, we amplified functional genes involved in denitrification (*nosZ*, PCR primers and protocol see Pereyra et al. 2013), methanogenesis (*mlas*, PCR primers and protocol see Steinberg and Regan 2009) and sulfate reduction (*dsrA*, PCR primers and protocol see Lefèvre et al. 2010) from the extracted, purified and transcribed RNA in a quantitative real-time PCR assay.

#### **RESULTS AND DISCUSSION**

#### **Community structures**

In all samples analyzed, we found shifts in community structures. It was conspicuous that in most samples these shifts (species turnover) included an increase in the relative proportion of denitrifying bacteria, while the relative abundance of sulfate reducing bacteria decreased (Fig. 1).



# Figure 1: Changes of bacterial community structure in a control fermenter and a $Ca(NO_3)_2$ treated fermenter exemplary showing shifts in bacterial groups involved in sulfate reduction (SRB – sulfate reducing bacterium), and denitrification (DNA – denitrifying bacterium) Community function

Relative gene expression showed a shift in the metabolic profiles of the investigated microbial communities in time-series experiments. The results below (Fig. 2) show exemplary for other plants and fermenters a significant increase in genes involved in methanogenesis (*Mlas*) and in denitrification (*nosZ*). In most cases, sulfate reduction to sulfide decreased notably after  $Ca(NO_3)_2$ -treatment (results not shown).

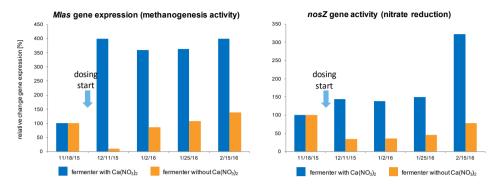


Figure 2: Relative gene expression level for genes involved in methanogenesis (left) and denitrification (right). Non-treated fermenters (orange) are compared with treated fermenters (blue) from November 2015 until February 2016. Treatment commenced after first sampling date. Values from first sampling date were set to 100% as benchmark

Based on the data obtained from genetic analyses, we propose the following mechanistic concept as one of the possible mode of actions of calcium-nitrate in anaerobic digesters:

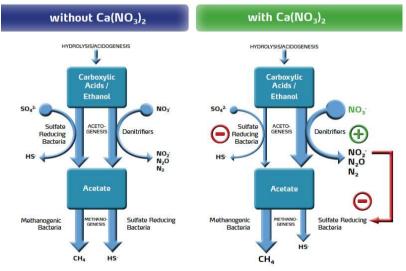


Figure 3: Simplified conceptual concept for a possible mode of action of  $Ca(NO_3)_2$  in anaerobic fermenters. Denitrifying bacteria benefit from the additional supply of preferred energy source (calcium-nitrate) for the decomposition of organic substrates (carboxylic acids and alcohols). Therefore, denitrifiers are able to outcompete sulfate reducing bacteria, which utilize the same substrates. Furthermore, nitrite as a product of denitrification inhibits the ATP-Sulfurylase, an enzyme needed for the conversion of sulfate into sulfide (Carlson et al. 2015)

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# Microbial Conversion of H<sub>2</sub> and CO<sub>2</sub> into Liquid and Gaseous Energy Carriers

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# INTRODUCTION

A growing need of green energy led to the development of immense wind parks and solar farms all over the world. As these technologies are highly weather dependent (Díaz-González et al., 2012), the demand for large scale energy storage is greater than ever. Hydrolytic hydrogen is often suggested as a storage medium for excess electricity arising during peak productions (Agbossoi et al., 2004). However, hydrogen is difficult to store and cannot be distributed using the existing structure of the natural gas grid (Bossel and Eliasson, 2003). The *Hydrofinery* project tackles these problems by a microbial conversion of H<sub>2</sub> together with CO<sub>2</sub> in gaseous and liquid energy carriers. The investigated process cascade (Figure 1) is split into two parts. The first part aims at a conversion of H<sub>2</sub> and CO<sub>2</sub> to acetate, a storable intermediate, by taking advantage the Wood-Ljungdahl pathway of homoacetogenic clostridia. Acetate serves as a substrate in the subsequent steps, which on the one hand includes the production of acetone, butanol and ethanol (ABE) using ABE-clostridia, and on the other hand methane, using methanogenic archaea. Furthermore, the direct production of bio-methane from H<sub>2</sub> and CO<sub>2</sub> by hydrogenotrophic methanogens is subject of investigation.

As acetate producing organisms the homoacetogenic clostridia model strains *Acetobacterium woodii* (DSM-1030) and *Moorella thermoacetica* (DSM-2955) were compared with regard to productivities and final acetate concentrations. Beyond that cells were immobilized to compare their performances in continuous fermentations. *Clostridium acetobutylicum* (DSM-792) and *Clostridium beijerinckii* (DSM-6422) were investigated for their ABE productivities. As for hydrogenotrophic CH<sub>4</sub> production, *Methanosarcina barkeri* (DSM-800) and *Methanobacterium formicicum* (DSM-3637) are subject of ongoing investigation. In addition, *Methanosarcina barkeri* is capable of methane production from acetate, thus acting as model organism also for the acetoclastic pathway.

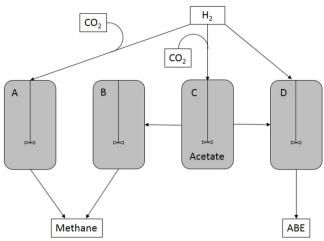


Figure 1: Schematic overview of processes. A: Direct conversion of  $H_2$  and  $CO_2$  into  $CH_4$ . B: Conversion of acetate into  $CH_4$ . C: Conversion of  $H_2$  and  $CO_2$  into acetate, which serves as a substrate for B and D. D: Conversion of acetate into ABE

# MATERIALS AND METHODS

All organisms were purchased from DSMZ and adapted to cultivation media in 100 mL serum bottles with 50 mL working volume. Methanogens are cultivated on minimal media with substrate gas (80% H<sub>2</sub>, 20% CO<sub>2</sub>) in the headspace. Homoacetogenic clostridia were grown on a minimal medium with a mixture of 40% H<sub>2</sub> and 10% CO<sub>2</sub>, besides nitrogen, as substrate gas. ABE-clostridia were cultivated on media containing glucose (60 g/L) and yeast extract (1 g/L). Productivities were determined in pH controlled fermentations carried out in 1 L fermenters (Multifors II, Infors HT, Switzerland). In order to prevent cells to be washed out during a continuous process, *Acetobacterium woodii* was immobilized on a woven piece of linen, which proved a suitable immobilization material during continuous fermentation in acidic broth. Continuous fermentation of *A. woodii* was carried out in 1 L fermenters with a working volume of 700 mL. The dilution rate was calculated on basis of growth rates of previous experiments. A media flow rate of 0.048 L/d was set.

#### RESULTS

Methanosarcina barkeri and M. formicicum have been cultivated successfully on minimal media and are to be investigated concerning productivities. Acetobacterium woodii not only produced ~25 % more total acetate in pH controlled batch fermentation than M. thermoacetica (21.6 g/L and 17.1 g/L, respectively), but also reached higher acetate productivities with 1.9 g/L/d and 1.4 g/L/d, respectively. Based on these results, A. woodii was chosen for continuous fermentation. In continuous mode productivities between 0.86 g/L/d and 1.02 g/L/d were reached. ABE yields of C. acetobutylicum and C. beijerinckii were within the range of 8.5 g/Land 14 g/L, respectively, per 60 g/L glucose.

#### DISCUSSION

Although yields of methanogens have not been determined yet, hydrogenotrophic biomethanation is considered a promising technology to biologically convert electrolytic H<sub>2</sub> into CH<sub>4</sub>, a substitute for natural gas (Rachbauer et al., 2016). For the production of acetate as intermediate metabolite from H<sub>2</sub> and CO<sub>2</sub>, *A. woodii* proved to be a suitable organism and was successfully immobilized on a low-cost surface, thus enabling a continuous fermentation process. Acetate was constantly produced over a period of 35 days, which sets the basis for an industrial scale production process. The addition of previously produced acetate into the ABE-fermentation step as to further increase ABE yields will be tested in future experiments. Finally, a two-stage continuous fermentation with the production of acetate by homoacetogenic clostridia in the first stage and ABE in the second stage will be established. This innovative process poses potential for low carbon biofuel/biochemical production on the basis of CO<sub>2</sub> rather than biomass. After this first proof-of-concept, investigations on scale-up and industrial implementation are still required and will be subject to future research.

#### ACKNOWLEDGEMENTS

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# Power-to-Gas in Fed-Batch Reactors Using Biogas Digestate Catalyst

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# INTRODUCTION

The most rapidly developing and spreading renewable technologies worldwide include the conversion of wind energy and direct solar energy (photovoltaics) to electricity. In view of the discontinuous electricity production by these technologies, coupled with fluctuating utilization, severe electricity storage problems arise. A likely solution of this emerging setback is conversion of electricity to alternative energy carriers (Kovács *et al.*, 2014) or chemicals (ElMekawy *et al.*, 2016). Hydrogen (H<sub>2</sub>) can be generated via electrolysis of water, a well-known and efficient process, however, technologies to store and transport H<sub>2</sub> are underdeveloped at present. Methane (CH<sub>4</sub>) is an obvious next candidate. Biogenic CH<sub>4</sub> production takes place during anaerobic degradation of organic matter in biogas reactors, swamps, ruminants, termites, etc. (Kovács *et al.*, 2014). Hydrogenotrophic methanogens form CH<sub>4</sub> by reducing CO<sub>2</sub> with H<sub>2</sub>. An additional advantage of the biological conversion of electricity to CH<sub>4</sub> is offered by coupling the process with CO<sub>2</sub> mitigation.

In the present study, reactors containing AD effluent supplied with daily dispensing of  $H_2$  gas, were studied in order to partially overcome the  $H_2$  solubility problem. Several operational conditions were tested under mesophilic conditions and efficient CH<sub>4</sub> productivity was attained.

#### MATERIAL AND METHODS

#### The fermentation system

The total volume of the reactors was 160 mL (Wheaton glass serum bottle, Z114014 Aldrich). The reactors routinely contained 40 mL inoculum from the mesophilic industrial biogas plant Zöldforrás Ltd., Szeged, Hungary. The inoculum was used according to the VDI protocol (VDI Handbuch 2006). The daily H<sub>2</sub> dosage was  $0.81 \pm 0.16$  mmol, unless indicated otherwise. The reactors were sealed with butyl septa and aluminum crimps and were made anaerobic by N<sub>2</sub> gas exchange of the headspace (5 min). Following the daily gas composition analysis by gas chromatography (GC), the gas phases of the reactors were degassed by purging with N<sub>2</sub> (Messer nitrogen 4.5) for 5 min and the internal pressure was adjusted to atmospheric level. H<sub>2</sub> and CO<sub>2</sub> were injected manually and daily into the gas phase with disposable plastic syringes according to the experimental protocol. The amount of the injected gas was verified by GC. The reactors were incubated in a rotary shaker at 37 °C.

#### Analytical methods

Organic acid spectra, gas composition, total organic solids and process parameters were measured as described elsewhere (Szuhaj *et al.*, 2016).

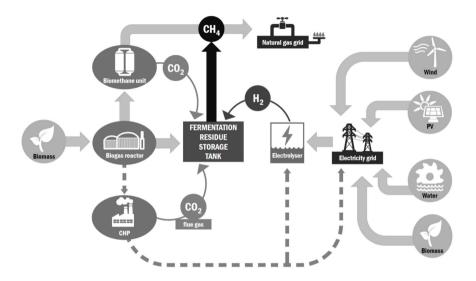


Figure 1: Proposed novel P2G scheme involving the AD fermentation residue storage tank as bio-CH<sub>4</sub> reactor, which converts  $CO_2$  from biogas or flue gas and H<sub>2</sub> from electrolysis by renewable electricity

#### **RESULTS AND DISCUSSION**

The duration of these fermentations was 80 days to test for sustainable CH<sub>4</sub> production. The reactors were supplied with the optimal daily dosage of 0.81 mmol of H<sub>2</sub>. CH<sub>4</sub> evolution progressed steadily until day 28, but dropped sharply afterwards. A warning sign of system failure was noticed already on day 27, when measurable residual H<sub>2</sub> was detected in the headspace. As shock therapy, massive CO<sub>2</sub> injection (25 mL) was dispensed into the reactors following the daily dosage of H<sub>2</sub> on day 31. All of this CO<sub>2</sub> disappeared from the gas phase within 24 hours, indicating that the system was indeed severely depleted of CO<sub>2</sub>/bicarbonate. The daily CO<sub>2</sub> dose was then gradually decreased to the stoichiometric volume, i.e. approximately 0.25 mol of CO<sub>2</sub>/mol of H<sub>2</sub> per day. The system responded positively, as exhibited by the restoration of CH<sub>4</sub> production on day 32 accompanied by a gradual decrease of residual CO<sub>2</sub> levels in the gas phase. Daily CO<sub>2</sub> injection (25 mL) was resumed on day 47. CH<sub>4</sub> production returned to the previous level, all of the injected daily H<sub>2</sub> and CO<sub>2</sub> were consumed within 24 hours and this continued for an additional month (Szuhaj *et al.*, 2016).

#### CONCLUSIONS

The biological route of the power-to-gas process, has been recognized and tested in laboratory and scale-up works (Luo *et al.*, 2012, Bassani *et al.*, 2015, Lee *et al.*, 2012, Reuter 2015). In our approach, the fed-batch fermentation technique was selected to increase the contact interaction between the gaseous substrate and the biocatalyst methanogens.

Consumption of the greenhouse gas  $CO_2$  by the process is an additional benefit of the P2G technology from an environmental point of view. A proper feeding routine in the fed-batch system leads to a sustained high rate of  $CH_4$  formation and the process may operate efficiently for an extended period of time.

A general strategy can be proposed (Fig. 1) on the basis of the results reported above to utilize the microbial community formed in the biogas reactor for the efficient conversion of  $H_2$  to  $CH_4$ as part of the P2G principle. At the center of the projected strategic alliance comprising either of the methods yielding renewable electricity and biogas technology are the hydrogenotrophic methanogens present in the biogas effluents. They convert  $H_2$ , which is produced from excess electricity by electrolysis, to  $CH_4$ . The proposed novel strategy places biogas technology into the hub of the renewable energy production and utilization network.

The potential economic advantages consequent from the scheme (Szuhaj *et al.*, 2016) are numerous. First, the microbial community present in the biogas effluent can be directly exploited for the efficient conversion of  $H_2$  and  $CO_2$  to  $CH_4$ . Second, this biological catalyst is continuously formed at the biogas plants at no additional cost. Third, the microbial community participating in the process is well organized and able to carry out the task under various environmental conditions very efficiently. Fourth, the process is easily manageable, and the microbial community flexibly tolerates the "turn-on" and "turn-off" situations. Fifth, the product is practically pure bio- $CH_4$  needing no further purification. Sixth, the process also accomplishes a  $CO_2$  sink and therefore directly contributes to  $CO_2$  mitigation.

The biogas installations may therefore complement their current operation by becoming bio-CH<sub>4</sub> producers and improve the economy of their technology without substantial additional investments.

#### ACKNOWLEDGMENTS

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# Biomass to SNG via Woodroll<sup>®</sup> process – Results from syngas preparation for catalytic conversion

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#### SUMMARY

The study reports on results from pilot tests of producer gas cleaning to a level required for a catalytic conversion of product gas to Synthetic Natural Gas (SNG). The product gas is generated by the WoodRoll® biomass gasification process. The producer gas cleaning is based on reactive adsorption in a ZnO bed at 400°C. The pilot tests confirmed 95% reduction of the sulfur content keeping it at a level below of 100ppb, indicating the active role of ZnO. A hydrogen cyanide reduction of 64% was verified by the condensate composition.

#### INTRODUCTION

Cortus Energy has developed a patented gasification technology, Woodroll<sup>®</sup>. Gasification of biomass is based on decomposing the biomass into two parts in a pyrolysis process: a gaseous part – pyrolysis gas, and, a solid part – char. Existing gasification technologies gasify the tar rich pyrolysis gas with air. The formed gas is diluted with nitrogen and requires a harsh cleaning, mainly for tar compounds, before it can be used. The cleaning is expensive and results in a reduced yield. The solid char is combusted to provide energy to the process. WoodRoll turns biomass into gas in a different manner, eliminating the tar impurities within the process. The process is divided into three (3) main steps: (1) drying, (2) pyrolysis and (3) gasification. The pyrolysis gas generated during step two (2) includes tar components which is further incinerated, and the heat generated from the incineration process is used to drive the gasification process. The clean carbon rich char is milled into a fine powder that is mixed with steam. Heat from the pyrolysis gas combustion is transferred indirectly via tubular burners to the steam-char mixture which initiate the chemical reactions that forms the product gas. The result is a gasification process that from a wide range of different types of biomass generates a clean, tar-free and energy-rich syngas with the highest efficiency. See Figure 1 for a basic illustration of WoodRoll<sup>®</sup>.

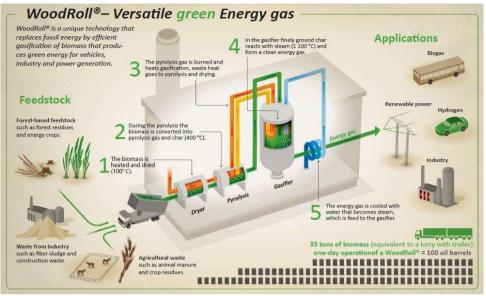


Figure 5 WoodRoll<sup>®</sup> gasification plant

WoodRoll's product gas composition is stable but can be varied by tuning the steam and char ratio. A typical dry gas composition is in the range of:  $H_2 = 54 - 60$  vol%, CO = 30 - 40 vol%, CO<sub>2 =</sub> 5 - 15 vol% and CH<sub>4</sub> = 1 - 3 vol% with a yield of biomass to syngas of 80% (if the moisture level of incoming biomass is 35% or lower). The product gas can be used to many applications including catalytic reforming to SNG. However, for catalytic processes some unwanted species such as H<sub>2</sub>S, HCl, NH<sub>3</sub>, particles or similar must be removed. Sulfur must be kept on a very low level as it reduces the life-time of a nickel-based catalyst commonly used for the methanation (C.H. Bartholomew et.al). During pyrolysis, 60 - 75% of the sulfur depart with the pyrolysis gas that is incinerated (Knudsen 2004). Between 25 to 40% retains in char which is gasified, the sulfur present in the char will end up in the syngas mainly in form of H<sub>2</sub>S. For nitrogen, 40-50% ends up in char mainly in form of ammonia (Lang et.al, 2006). Most of the impurities are removed from the product gas via basic separation units existing in the WoodRoll® system: filtration and drying (condensate). Meaning contaminates such as ammonia, alkali metals and halogens, and to a large extend  $H_2S$  are stripped out from the product gas. However, for catalytic processes the sulfur components needs to be further reduced. Numerous processes are available for low temperature adsorptive desulfurization. Advantages in processing, notably the energy conservation and capital savings, make high temperature desulfurization attractive. Reactive adsorption is superior to ordinary physical adsorption because it involves interaction of the adsorbent with the sulfur compound which is stronger than van der Waals interaction, increasing the desulfurization efficiency (Hernandez & Ralph, 2003). Manganese and zinc oxides appears to be most prominent in the temperature range 300-600°C as they can maintain very high desulphurization performance, could regenerate via oxidation, and no carbide formation was reported (Slimane & Abbasian, 2000). The selection of the adsorbing material for sulfur capturing was based on the

compromise between the available product gas temperature and effectiveness and availability of the material. ZnO was found to be most prominent solution for sulfur removing from the product gas at a temperature around 350-500°C.

Current study focus on a newly developed gas cleaning system. The purpose of the gas cleaning is to condition (clean) the WoodRoll<sup>®</sup> product gas to a level suitable for feeding into a downstream catalytic process converting it into SNG. The main target for the gas cleaning is to remove sulfur in a ZnO adsorption column to a safe level below 0.1 ppmv. Bark was used a reference feedstock and the tests were conducted during the fall of 2016.

#### MATERIALS AND METHODS

The gas cleaning system with a capacity of 4-5Nm<sup>3</sup>/h of dry gas is presented in Figure 2.

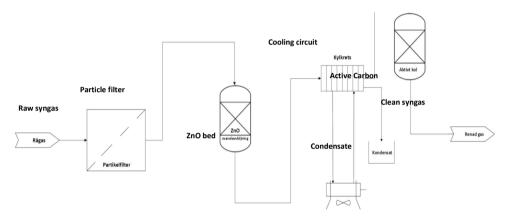


Figure 6 Gas cleaning system

The gas composition was measured by gas chromatography using a Thermo scientific C2V microGC calibrated for  $H_2$ , CO, CO<sub>2</sub>, CH<sub>4</sub>, N<sub>2</sub>, O<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>2</sub>, and 1-C<sub>4</sub>H<sub>8</sub>.

Hydrogen sulfide, ammonia and hydrogen cyanide was determined using a wet method. According to the method,, gas bubbles through impinger bottles filled with solutions that trap the compounds controlled sampling flow (~1 l/min) over a sampling time of 20 min. A 0.01 M HCl solution was used to capture NH<sub>3</sub>, while for HCN and H<sub>2</sub>S, a 0.1 M KOH solution and a 2% zinc acetate, 1% NaOH solution werewere used respectively. The solutions where determined by means of spectrophotometry for both H<sub>2</sub>S and HCN and NH<sub>3</sub> by ion chromatography. In addition, elemental analysis of the adsorbent (ZnO) before and after test was conducted.

After the condenser, downstream the adsorption reactors, dry gas was analyzed for remaining  $H_2S$  using a Galvanic's 903 online  $H_2S$  analyzer with a digital colorimetric sensor which measures the rate of darkening caused by the reaction of  $H_2S$  on white tape impregnated with lead acetate in accordance with ASTM methods D4084-94, D4323-97 and D4468-95. Linearity and repeatability of ±1% on ranges up to 0-2000 ppm. Detection limit <10ppb.

#### **RESULTS AND DISCUSSION**

The product gas composition was found to be within the following range (dry gas):  $H_2 = 55 - 60$ vol%, CO = 28 - 35 vol%, CO<sub>2</sub> = 8 - 12 vol% and CH<sub>4</sub> = 1 - 3 vol% with increasing yield of H<sub>2</sub> and CO<sub>2</sub> for a higher steam-to-fuel ratio. This gas allows to generate methane with a yield of 85-91% dependently of technology (Biacchi, 2015). The gas composition is in line with previous Cortus' studies conducted in a pilot scale of 0.5MW with several different types of biomass. From these studies, no significant effect depending on type of fuel was found to have an impact on the gas composition, but rather operating parameters for gasification played a greater role. [Amovic et al. 2013]. The measurement of H<sub>2</sub>S using a wet method indicated the sulfur level to be below detection limit for all tested cases. It was expected to determine the H<sub>2</sub>S from the raw gas sampling but the wet method for the raw gas of 50 ppm sulfur seems not to be accurate enough using current procedure. Increase of the sampling time could be a way to go forward with this method. Nevertheless, the expected concentration of H<sub>2</sub>S in the raw gas based on thermodynamic calculation for the tested fuel for the given flow should be 50.2 ppm. It was also found out that some part of the sulfur was captured within the ash in the filter before reaching ZnO bed. Although a saturation point of capturing H<sub>2</sub>S in the ZnO-bed could not be reached, the changing of color from pale white to gray of the adsorbing material was observed. The chemical analysis of the bed material before and after use confirmed an increase by 30% in the concentration of sulfur after 11-hour exposure. In addition, the analysis of condensate collected after the desulfurization showed a ~95% reduction in sulfur content referred to the expected sulfur input for mass balance calculation. The average presence of H<sub>2</sub>S in the dry gas was below 100 ppb, measured via continuous analyzer, which was satisfactory for requirement of the catalytic process. An interesting observation was found when analyzing NH<sub>3</sub> and HCN presence in the gas before and after ZnO bed. Their initial concentration in the raw gas was 1010 and 2.2 ppm, respectively. After 3 hrs. of operation the measured NH<sub>3</sub> in the gas did not exceed 20 ppm while an almost constant value of ~900 ppm was attained at longer times on stream indicating 50-fold reduction and showing that material's NH<sub>3</sub> sorption capacity was almost reached. Similar trend was observed for HCN where at the beginning of the test, when bed was still not saturated, the concentration of HCN was below 0.1 ppm and after 3<sup>rd</sup> hour of operation it significantly increased to ca 1.5 ppm (15 times). ZnO was saturated to 90% with respect to  $NH_3$  and to 84% with respect to HCN capacities, respectively.

#### CONCLUSIONS

WoodRoll<sup>®</sup> delivers a clean product gas which after implementing the gas cleaning system proposed in this paper can be used for catalytic reforming to SNG with a yield of up to 91%. The sulfur content in the output gas can be kept below 0.1 ppm using ZnO adsorbent at 400°C. A 15-fold and 50-fold reduction in hydrogen cyanide and ammonia was also observed, respectively, using the same material. Preliminary regeneration test indicated that the material can be regenerated; however, further investigation is needed to find optimum regeneration conditions and to assess its performance after several regeneration cycles. Optimization of this process is also recommended.

Based on the results of gas cleaning system, the pilot test of coversion of product gas to SNG are expected be conducted during spring 2017.

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# Comparison of Biogas Plant Financial Indicators between different Regions as a Mean to deduce the Impact of regional Framework Conditions

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The level of financial support needed in order to exploit a profitable biogas plant differs from region to region. It makes an interesting exercise to compare this level of financial support needed in different European countries. The European FP7-INEMAD project allows to gather and interpret this information through its consortium members. The year of reference is 2014.

The INEMAD consortium was asked to define financial parameters for representative biogas plants in their country. In order to align and compare these plants a common method was found based on the Flemish certificate support measure, where the financial gap that is needed to get the required return on investment for the technology is taken into account. This is an interesting comparison method as it leaves out all direct support measures. This financial gap calculation was done for the different types of biogas plants. To allow a comparison in between countries (regions) and technologies, a distinction was made on the level of investment cost, operational cost and profit. Due to the large variation in financial indicators for the different cases, a further classification of costs was limited to a distinction between maintenance costs, biomass cost and electricity and heat profit. The comparison was done for one or more types of plants in Belgium, Denmark, The Netherlands, Bulgaria, Croatia and Germany.

Belgium has a relative high investment cost due to the post treatment processes that are obligatory in regions with high nutrient pressure. These investments pay off by reaching a similar level of operational costs due to a lower deposit cost of the digestate. The profit range of a plant is highly influenced by the useful application of the residual heat as is done in the Netherlands and Germany by injecting residual heat into a heat grid.

In general German plants need lower levels of support (if expressed per  $MWh_e$ ) due to their beneficial ratio between energy content and price for its feedstock. In Germany, the price of the input material represents one of the key components. In the beginning of the biogas area (early 2000) the prices for crops like maize and wheat were very low. Since then the prices have increased putting pressure on the profitability of German plants. The investment cost for the technology are manageable due to the great number of biogas plants.

Bulgaria describes the Han Bogrov plant from the Sofia Municipality, receiving household waste and green waste with a downstream composting plant. As this is the first digester of its kind, the

investment costs are high. The plant aims to cover its cost through the high-energy price and selling the compost at favourable market prices.

Unlike other countries, Denmark describes the case of a *centralized biogas* plant that sells it biogas in large quantities to a third party. This is a different approach from the smaller *decentralized plants* who apply the biogas locally in a CHP unit. Based on this case, we see that the financial support needed to invest and exploit the centralized biogas plant is similar to the decentralized concept, if expressed per  $MWh_e$ .

# Social Acceptability of Innovative Nutrient Management Strategies to Recycle Nutrients

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#### INTRODUCTION

Specialization, both on farm and at regional level, is a common trend in European agriculture, replacing traditional mixed farming systems. This farm specialization co-evolves with an increased reliance on external inputs, such as nutrients (in the form of e.g. fertilizer) and energy (Pretty and Bharucha, 2014), in turn leading to additional environmental pressure (Hodgson et al., 2005; Pretty and Bharucha, 2014; Kleijn et al., 2015). Within regions consisting of mostly specialized farms, nutrient recycling becomes a crucial element when striving for agricultural sustainability. INEMAD (Improved Nutrient and Energy Management through Anaerobic Digestion), an European FP7-project, searched for innovative strategies in reconnecting livestock and crop production to reduce the reliance on external inputs. The processing sector, which uses primary products to produce renewable energy and green fertilizers, is assigned an important role as a third party to close the nutrient cycle (www.INEMAD.eu).

During the INEMAD project, several new strategies to restore the energy and the nutrient cycle have been developed. Adoption rates of innovative strategies depend a.o. on the technical and economic feasibility of the strategy and on the regulatory and institutional conditions in a specific country or region (Stonehouse, 1995). Another important, and often underestimated, aspect of adoption is social acceptability, defined here as the extent to which a certain strategy is perceived as acceptable or useful by the end-user, and possibly the wider community. Strategies that lack social acceptability are unlikely to persist, even if they are profitable and considered as a technical improvement (Clawson, 1975).

Our objective is to determine and evaluate factors influencing social acceptability of the strategies developed in INEMAD, and hence the willingness to adopt these strategies. We first present the theory of planned behavior (TPB), a socio-psychological theoretical framework. We discuss the main elements of the theory, and present a qualitative research approach based on this framework, followed by the main results, and a brief conclusion.

#### ANALYTICAL FRAMEWORK

The TPB has already been proven useful in predicting and explaining various individual human behaviors, including technology adoption in agriculture (Ajzen, 1991; Wauters et al., 2009; Sutherland, 2011; Home et al., 2014). It is a standardized and repeatable framework, allowing a comparative study in different countries (Burton et al., 2004). According to the theory of planned behavior, the individual behavior, in this case implementing a new strategy concerning nutrient recycling, is predicted by the intention to perform a certain behavior, which in turn reflects three motivational influences; i.e. i) attitude, ii) subjective norm and iii) perceived behavioral control.

Attitude refers to the favorable or unfavorable evaluation an individual has of a specific behavior, based on the beliefs about the outcome when performing that behavior. Subjective norm is determined by the perceived social pressure to perform the behavior, and the degree to which an individual cares about the opinion of a referent person. Perceived behavioral control is determined by the perceived ease or difficulty to successfully perform the behavior and is influenced by past experiences and factors that facilitate or obstruct the execution of the behavior. The contribution of each motivational influence in predicting the intention is not always equal and varies from case to case. However, it can be stated that if the aforementioned motivational influences are assessed more positively, the intention to behave increases, hence leading to a higher likelihood that an individual will perform a certain behavior. This all under the condition that the person has sufficient actual behavioral control (Ajzen, 1991) (Figure 1).

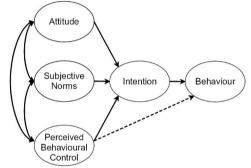


Figure 7: The Theory of Planned Behavior (Ajzen, 1991)

#### METHODOLOGY

The theoretical framework was used in a qualitative research approach. The present research is exploratory and identified relevant factors and concepts related to social acceptability. Data was collected through focus groups, organized in all INEMAD partner countries, where stakeholders had the possibility to share and discuss their ideas, experiences and beliefs about the strategies and expected outcomes.

All focus groups started with a brief presentation from the research team to inform stakeholders about the innovative strategies. Strategies were divided in three groups, i) the use of bio-based fertilizers, ii) transport of manure (products) and iii) post treatment techniques of manure. During a feedback session the social acceptability of these strategies was addressed. A protocol with sample questions ensured a similar evaluation method in all countries. Feedback session reports were coded and analyzed according to the different elements of the framework, and relevant concepts for the three groups of strategies under discussion were identified.

#### **RESULTS AND DISCUSSION**

An overview of the concepts influencing intention, and thereby possibly determining behavior, related to the use of bio-based fertilizers, to the transport of manure, and to post-treatment techniques of manure is presented in Table 1. For attitude, benefits and disadvantages describe the expected or believed, respectively positive and negative, outcomes of applying the specified

strategies. Subjective norm is influenced by persons or institutions stimulating or impeding the implementation of the strategy and perceived behavioral control consist of factors stimulating or limiting the execution of the specified strategy at the moment as well as facilitating factors during the implementation process.

From Table 1, we can summarize the most important factors or people influencing the intention to implement strategies, as discussed during the focus groups. In all three types of strategies, the government plays a dual role. On the one hand they are considered as a potentially useful partner in the process, as they can offer different types of stimuli (e.g. financial support) to support the implementation of innovative strategies. On the other hand, they are often seen as an impeding factor (e.g. by not providing a suitable legislative framework). Absent or immature markets are another common factor hindering implementation. Perceived behavioral control and thus intention would increase by tackling these factors.

The use of bio-based fertilizers and post-treatment techniques of manure are both stimulated by broader society, while this is not mentioned for transport of manure. Reason can be that awareness for the first two strategies is increasing since these strategies are performed on local scale, often aimed at environmental improvement, thereby raising public interest. Society stimulates these strategies, but also suffers somewhat from the NIMBY-syndrome. A disadvantage for both aforementioned strategies are high (investment) costs, since they often require often new materials or techniques. The lack of public interest for the strategies associated with manure transport might be explained by the regional level of organization and the extra burden associated with additional transport. A believed benefit for stakeholders, common to both the use of bio-based fertilizers and transport of manure, are the associated positive effects on nutrient recycling and the environment. Although post treatment techniques of manure also can have this positive impact, this was not mentioned by the stakeholders, as a believed benefit. This may be due to the fact that this impact is often indirect since it is mostly followed by one of the other types of strategies.

More specific disadvantages include, for post-treatment techniques of manure, the fact that they often stay on a theoretical level, and, despite promising results, lack practical implementation. As such, research is potentially a stimulating factor, but this should be further improved by an increased dissemination of research results to practitioners. For transport of manure there is the issue of different regulations across countries, as this involves the actual transfer of nutrients across European borders.

Overall, there is a clear need for understandable information, directed at different stakeholders, to increase adoption of innovative techniques (Sutherland, 2011; Home et al., 2014). To further increase awareness and acceptance of research results, stakeholders can be involved at different stages during the research or development process. (McCown, 2002). Besides increasing acceptance, stakeholder involvement has the added advantage of including a diversity in perceptions and knowledge from relevant non-scientific sources (van de Kerkhof, 2006).

#### CONCLUSION

Our results show a wide range of both general and more strategy-specific influencing factors. One of the main general influencing factors is the government. Different government services have an important role to play in stimulating the different types of strategies, either by offering financial support, providing a legislative framework or through creating or stimulating markets. Research is a second factor, necessary to determine or confirm the efficiency and effectiveness of the different strategies. Results of this research can be useful for other researchers developing similar strategies, for the government as advice to stimulate these innovative nutrient strategies and for the industry to strengthen contacts with the government and researchers. Finally, sufficient attention and effort should be directed at good communication strategies, to inform stakeholders, both on a theoretical and practical level. This is preferably an interactive process, where different stakeholders types communicate with each other, during the different stages of research, development and implementation.

It should be noted that this was an exploratory study, based only on responses given during the focus groups in which we tried to identify factors clarifying the social acceptability of some specific nutrient management strategies. Furthermore, the strategies under discussion may be relatively new, so stakeholders' opinions on them are possibly limited. Hence generalization in other contexts has to be avoided. The research has however unraveled some concepts which can be a start for future research.

#### Table Concepts determining behavior related to the use of bio-based fertilizers, transport of manure and post treatment techniques of manure

	Attitude	Subjective norm	Perceived behavioral c	ontrol
The use of bio- based fertilizers	Benefits:         • Socio-economic benefits for society, environment and farmers         • Clean source of energy         • Higher nutrient content, good quality manure         • Additional source of income         • Nutrient recycling         Disadvantages:         • High investment costs         • Uncertain product stability         • Marketing is difficult and time consuming	Stimulate: • A growing portion of farmers are replacing traditional with environmentally friendly technologies • Biogas associations and associations are working on ecological products • Public interest is increasing Impede: • Lack of information (farmers, industry and general public) • Legislation • Urban areas, "people smell with their eyes"	Stimulate:         • High prices of artificial fertilizers         Limit:         • High costs         • No legal framework         • Complex administrative procedures         • No financial support mechanisms         • Poor information         • Too expensive for small farms         • Information about application date         • No market         • Competition for raw materials	Facilitate:         • More information         • Financial support         • Legislative framework         • Linkages in the production chain         • Building regional clusters         • The right shape (granulated or pellets)         • A reliable nutrient content (especially N)         • A user-friendly product (right shape/form (granulated or pellets), no smell, can be easily applied)
Transport of manure	Benefits: • Recycling of nutrients on EU- level • Lowers costs • Better soil fertility <u>Disadvantages</u> : • Veterinary risks • Different regulations across countries	Stimulate: • Livestock producers (NI, Fr) • Market ("receivers" of manure in manure shortage areas • Politicians (Ge, Fr) Impede: • Government (Wal-Be) • Manure surplus areas in Germany/France	Limit: • Immature market • Market not uniform across Europe • No legislative framework • Political ideologies	<ul> <li>Facilitate:</li> <li>Support of politics and government</li> <li>Financial support</li> <li>Legislative framework</li> </ul>
Post-treatment techniques of manure	Benefits: • Useful • Interest from the market Disadvantages: • Some techniques exist only in theory, have no practical application • Cost	Stimulate: • Research • The government (especially during development) • Society Impede: • Lack of clear policies	Stimulate: • Increasing pressure on agricultural sustainability Limit: • High costs • No market • Transport • No legislative framework	Facilitate:         • Financial support         • Open markets         • New technologies         • A profitable end product         • Legislative framework         • More information

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# Comparison of different Digestate Processing Systems Concerning environmental Efficiency

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#### SUMMARY

There are regions in Europe with high livestock density, which means high quantities of manure with organic nutrients but insufficient areas to applicate these nutrient amounts. On the other side, there are large areas with a high nutrient demand for plant cultivation. There are several options to treat manure with the aim of saving emissions and solve transport distance problems simultaneously. This assessment will make a contribution towards further optimization of manure treatment systems with focus on climate change, eutrophication and acidification potential. The conducted life cycle assessment shows that manure treatment is not better than conventional manure handling in any case. From an overall ecological view treatment, options that separate manure or digested manure with downstream composting can be an optimal way of manure treatment. Further positive aspects are the increase of transport distances in consequence of composting or drying processes.

#### INTRODUCTION

The aim of manure treatment systems is to reduce nutrient content or to create a more concentrated product to come to a more effective and efficient nutrient management. Further advantages are that manure storage can be avoided and the products can be handled easier than raw manure. Manufactured manure products can be transported to areas with nutrient demand to substitute mineral fertilizers.

Here beside traditional manure handling (storage and application), ten different treatment opportunities have been considered. The scenarios have been evaluated regarding the effects for the impact categories global warming potential, eutrophication potential and acidification potential.

#### MATERIALS AND METHODS

Analysing the environmental impact of different manure treatment technologies with the aim to identify treatment procedures with extraordinary environmental impacts the Life Cycle Assessment (LCA) tool was used. This approach was performed according to the methodology described in the ILCD Handbook for LCA (EC JRC, 2010) and the (DIN EN ISO 14040/44).

System boundaries starts with delivering raw (cattle) manure and ends with the application of (treated) manure. Table shows the analysed scenarios. Scenario s00 is defined as baseline scenario, s01 - s04 treat manure in different ways like separation, composting drying or biological treatment (nitrification/denitrification), whereas S05 - s10 consider also manure in same ways but first manure is pre-treated in digestion. The assessment focuses on the environmental impacts due to the treatment of 1 ton raw cattle manure (FU). The relevant impact assessment categories to answer the research questions are global warming potential, eutrophication

potential and acidification potential based on ReCiPe assessment method (Goedkoop et al., 2008).

Life cycle inventory data is based on literature data. Data for Infrastructure as well as up- and downstreaming processes were taken from the Ecoinvent database (Weidema et al., 2013; Nemecek & Kägi, 2007).

The use of manure and products of manure treatment as organic fertilizer can avoid the use of conventional mineral fertilizer products and conventional produced electricity and heat. Further transport distances can be expanded by drying and composting the solid fraction of manure and digestate. These effects are taken into account as benefits.

No.	Product	Process st	teps				
s00	raw manure				storage	transport	application
01 raw manure		separation	liquid fraction	storage	transport	application	
			solid fraction	composting	transport	application	
s02 raw manure		separation	liquid fraction	storage	transport	application	
			solid fraction	drying	transport	application	
s03 raw manure		separation	liquid fraction	boil.treatment	transport	application	
			solid fraction	composting	transport	application	
s04	c04 row monuto		separation	liquid fraction	boil.treatment	transport	application
s04 raw manure	raw manure			solid fraction	drying	transport	application
s05	raw manure	digestion				transport	application
s06	raw manure	digestion			drying	transport	application
s07 raw manure	raw manuro	digestion	separation	liquid fraction	storage	transport	application
	raw manure			solid fraction	composting	transport	application
s08	raw manuro	digestion se	separation	liquid fraction	storage	transport	application
sub raw manure	raw manure			solid fraction	drying	transport	application
s09 raw manure	raw manuro	digastian	constration	liquid fraction	boil.treatment	transport	application
	ugestion	separation	solid fraction	composting	transport	application	
S10 raw ma	raw manuro	w manure digestion	separation	liquid fraction	boil.treatment	transport	application
	aw manure			solid fraction	drying	transport	application

#### Table scenarios of analysed digestate treatment paths

#### RESULTS

The following figures show the advantages and disadvantages of manure treatment in comparison to the baseline scenario for global warming potential, eutrophication potential and acidification potential.

Global warming potential

Manure treatment is definitely a way to save climate change emissions. Except for scenario s03 and scenario s04 all scenarios are better than the conventional manure application. Especially with the avoided storage emissions, the emissions can be halved at least. Heat demand to dry digestate in s06 results in high emission, although the use of renewable energy sources emission can be reduced by a factor of 7, but expenditure for drying wet digestate is still very high.

Biological treatment of manure and digestate due to dinitrogen monoxide emissions of nitrification and denitrification process produces high emissions as well. Only 1 % of nitrogen is lost in dinitrogen monoxide in the biological treatment but with a global warming potential of 298 kg  $CO_2$ eq per kg  $N_2O$  the effects are enormous. The application of treated manure or digestate leads to further benefits especially if manure is digested with upstream separation and composting of the solid fraction.

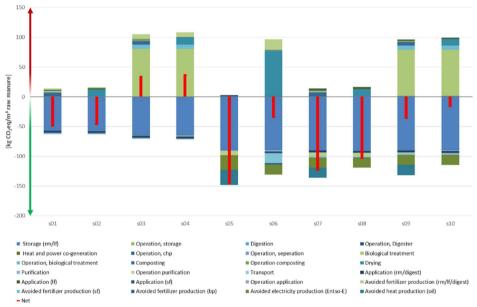
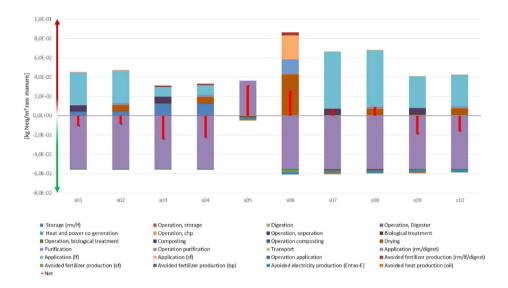


Figure 8: Advantages and disadvantages of manure treatment in comparison to S00 / results of environmental impact assessment – climate change (Red line : net results)

#### Eutrophication potential

Figure 9 shows emission saving potential for eutrophication potential of several treatment options. Especially the scenarios s05 - s08 with high emission saving of global warming potential results in more emissions in the category eutrophication in comparison to the baseline. The other scenarios (s01, s02, s03, s04, s09, s10) can reach emission reduction. Eutrophication is mainly influenced by nitrogen emission, so that the largest source of emissions are manure or digestate application. Field emissions can be avoided through drying or composting of solid fraction whereas application of digestate leads to higher emissions than the application of raw manure. Best results can be reached by biological treatment of the liquid fraction of manure but also of the liquid fraction of digestate.



### Figure 9: Advantages and disadvantages of manure treatment in comparison to s00 / results of environmental impact assessment – eutrophication potential

#### Acidification potential

The results for the impact category acidification potential in comparison to the baseline scenario s00 are comparable to the category eutrophication. Application processes are the largest emitters in this impact category. However, drying raw digestate also leads to high acidification potential. Emissions can be avoided by biological treatment of the liquid fraction and drying as well as composting the solid fraction.

#### DISCUSSION

The assessment shows that manure treatment is definitely a good way to save emissions focused on global warming potential as well as eutrophication and acidification potential. Especially through the avoided storage emissions, the emissions can be halved at least. Scenarios with highest emission saving potential in a single category lead to impacts in the other categories. Focused on all three categories only the scenarios s01, s02 and s09, S10 save emissions overall. For global warming potential, digestion can be a good solution to reduce emissions, for eutrophication as well as acidification potential, biological treatment has to be preferred, so that a combination of both treatments is the best opportunity to handle manure environmental friendly. Generally, the use of organic fertilizer is an essential opportunity to close nutrient cycles as well as saving finite and fossil resources. Thus, processes with high nutrient losses respective nutrient destruction should be avoided as far as possible. Perhaps the export of composted or dried products in regions with nutrient sinks can be a better solution. That means that the considered treatment options describe several opportunities to avoid emissions.

#### ACKNOWLEDGEMENT

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# III. Policy Recommendations based on Research & Development

# Meeting the Paris Agreement Objectives requires a Forward-Looking Circular Economy Approach

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In December 2015, the Paris Agreement was signed by world leaders, with the aim of limiting global temperature to 2 degrees centigrade and preferably 1.5 degrees centigrade compared to pre-industrial levels. As suggested by the post-2015 development Agenda, implementation strategies need to be based on a holistic approach, tackling not only CO<sub>2</sub> emissions, but also other substances with high Global Warming Potential, first and foremost **methane** (CH<sub>4</sub>), looking at these goals keeping in mind the broader international context. Improved waste management and Anaerobic Digestion (AD) can effectively be at the core of the strategy to substitute natural gas, conventional electricity and fossil oil derived fuels.

The international (and European) post-2015 climate and energy framework has been set out. The Paris Agreement has indisputably affirmed the principle of Shared But Common Responsibilities (SBCR), showing that OECD Members and emerging economies have finally embraced the reality that industrialised countries - responsible for decades of CO<sub>2</sub> emissions need to drive the efforts towards climate change mitigation and adaptation. In particular, countries responsible for the historical bulk of emissions - mostly EU Member States - will need to be the first ones taking concrete action at all levels, investing in a relatively short period of time on climate change mitigation and adaptation. As such legal framework is now in place with a 2050 horizon, the implementation strategies for European countries are now to be defined both at EU- and Member States-level. Besides, the 2030 Agenda for Sustainable Development, which includes the 17 United Nations Sustainable Development Goals (SDGs) with their 169 targets, establishes an additional ambitious layer to achieve global sustainability. If on the one hand the Paris Agreement establishes the global climate targets in the decarbonisation process, the SDGs are supposed to provide a comprehensive list of global actions to take a holistic approach towards a deeper look at the root causes of social and environmental unsustainability. The SDGs are interlinked and indivisible, meaning that if SDG 2 aims at achieving sustainable farming, SDG 7 clean energy for all and SDG 13 environmental protection, a comprehensive and new, multidimensional strategy is required at global level. Especially with a view on the 2030 horizon, it seems unlikely to achieve these objectives without a coordinated action, as the first requirement is to break down silos to ensure a far-reaching policy-making process. The further positive development is represented by business, which are finally seizing the opportunity offered by technologies likewise AD, contributing to SDG 8 ("promote inclusive and sustainable economic growth") as well.

It is not only about carbon emissions reduction: it is about our environment, our society and our economy. As shown by the *Clean Energy for All Europeans* package of the European

Commission, renewable energies and energy efficiency are at the core of the current strategies to reduce carbon emissions, although the climate goals are unlikely to be achieved only through renewable energies deployment, but **also require a coordinated waste management system and well planned circular economy strategies**. Therefore, **a comprehensive climate strategy should not only be based on CO<sub>2</sub> emissions reduction, but also on harmful methane emissions mitigation**. There is a robust climate change impact of methane and CO<sub>2</sub> deriving from poorly managed waste: within ten years, dumpsites could be responsible for up to a tenth of manmade greenhouse gases, hindering the SDG 13 progresses. It is clear that a business opportunity (in line with SDG 8) emerges from these synergies, allowing CH<sub>4</sub> emissions reduction while supporting more sustainable farming, clean energy production, job creation and environmental protection, perfectly in line with the SDGs. The international community has established the framework: it is now the moment for countries and stakeholders to seize the opportunity to achieve a sustainable, inclusive growth, within planetary boundaries.

AD is one of the key technologies to replace fossil-based energy, and its potential for deployment is far greater than it is today. AD is a technology with proven significant GHG emissions reduction over the last 20 years, which is highly adaptable to the type of feedstock used. Its potential is far from being fully exploited: AD can mitigate a significant amount of GHG emissions reduction in manure management, solid waste disposal on land, waste and wastewater handling and agricultural waste burning which represents at least 20% of the global methane emissions (see table below). Traditional manure management involves open storage and spreading on lands, releasing more than 3% of global methane emissions. AD could mitigate those emissions, while producing energy and recycle nutrients via spreading the newly produced digestate. AD also helps to reduce the number of pathogens in the digestate and allows a higher agronomic efficiency (Lukehurst et al 2010). The EU BIOSURF project estimates that 1.4 billion tons of manure are potentially available for manure processing in the EU alone (Kirchmeyr, 2016). Concerning solid waste disposal on land and municipal biowaste, the circular economy strategy of the European Commission (EC 2015) aims to phase out the landfilling of biodegradable biowaste and introduces higher overall recycling targets and a ban on the incineration of source separated municipal waste – measures that encourage the digestion of biowaste. Integrating AD on the site of Waste Water Treatment Plans (WWTP) allows to stabilise sludge, reduce dry matter content and generate electricity, thereby strengthening the economy of these facilities. This widely recognised technology is well established and is regarded as 'a major and essential part' of modern WWTP (Guangyin et al., 2017). However, only 7.5% of WWTP were using AD to treat sewage sludge in Europe in 2013 (EBA 2016), allowing further significant improvements. The potential amount of feedstock for energy use, representing the proportion of the technical potential after satisfying other existing projected competing uses for the same feedstock, could mitigate at least 3.4% of global methane emissions if anaerobically digested.

Sector	Shai	re of annual	Potential emission
Sector	methan	e emissions	mitigation via AD
	Fugitive emission (solid fuels, oil, gas)	33.19%	-
Energy	Residential and other sectors	3.38%	-
	Other	0.13%	-
	Subtotal Energy	36.70%	-
Industry	Subtotal Industry	0.21%	-
Transport	Subtotal Transport	0.18%	-
Fires/slash and	Subtotal Fires/slash and burn	4.85%	-
burn			
	Enteric fermentation	27.50%	-
Agriculturo	Manure management	3.16%	1.04% <sup>1</sup>
Agriculture	Rice cultivation	10.31%	-
	Subtotal Agriculture	40.97%	1.04%
	Solid waste disposal on land (incl.	7.97%	1.59% <sup>2</sup>
	landfill)		
	Wastewater handling	8.55%	0.64% <sup>3</sup>
Waste	Waste incineration	0.02%	-
	Other waste handling	0.09%	Unknown
	Agricultural waste burning	0.43%	0.14%4
	Subtotal Waste	17.80%	2.38%
All sectors	Total	100.00%	> 3.42%

#### Table 1: Share of global emissions of methane in 2008 (data from (JRC 2011))

<sup>1,4</sup> based on BIOSURF assumptions (Kirchmeyr, 2016)

<sup>2</sup> Based on the IPCC Global default value for methane recovery rates (Rajaram et al. 2011)

<sup>3</sup> Extrapolated from the percentage of WWTP using AD on site in Europe (EBA, 2016)

An economy that benefits from moves towards environmentalism. The products generated by AD – biogas, biomethane and biofertiliser – have the ability to substitute fossil energy, circulate nutrients and provide adequate waste management, in addition to a more sustainable agriculture: based on feedstock potential and with the right policies in place, by 2030, the industry could produce renewable energy equivalent to approximately 10% of the EU's current natural gas consumption, for use in electricity generation, heating/cooling and as a transport fuel (GGG 2013). In a nutshell, if we can achieve environmental benefits, exporting technology and contributing to an inclusive economic growth, it seems that the advantages can truly support the post-2015 global Agenda.

#### POLICY RECOMMENDATIONS

An historical chance emerges from the policy interlinkages generated by AD. Without a doubt, resource-efficiency is an essential objective to ensure a decided move towards a more circular

economy, reducing EU dependency from resources depletion, and strengthening *inter alia* European geopolitical advantages. Positively, there have been interesting advancements tackling the issue through a mature technology such as AD, which allows producing sustainable electricity and heat, preserving at the same time our environment and creating jobs in rural areas. However, strategic policies supporting industrial symbiosis, along with a more sustainable agricultural system, including **livestock and manure management**, should be at the core of a holistic approach towards global climate and energy goals, and a major step towards the SDG. **2050** targets are approaching fast but there is an outstanding news: long-term solutions have already emerged.

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#### EU Waste Policies: Getting the Right Measures to Recycle Biowaste

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#### INTRODUCTION

Europe is a net importer of energy and primary raw materials. In 2014, the European Union (EU) imported 53.5% of its energy from a hand full of countries headed by Russia, at an estimate cost of more than  $\leq 1$  billion a day or  $\leq 400$  billion a year. In addition, the EU imports of primary raw materials reached  $\leq 72$  billion in 2015 and are still increasing, headed by metalliferous ores, agricultural products, paper and mineral fertilisers. In this difficult economic and geopolitical context, it is crucial for an international actor with limited resources like the EU to set measures encouraging domestic renewables production, resource efficiency and recycling. In this respect, biowaste and organic residues are a valuable and mostly untapped resource of energy and nutrients.

This policy paper will deal with existing EU provisions for waste management, evaluating their effectiveness as drivers for resource efficiency. In particular, the focus will be placed on the organic fraction of municipal solid waste, commonly known as biowaste, which can be efficiently recycled via composting and anaerobic digestion into organic fertilisers and renewable energy. Biowaste constitutes a very substantial share of the EU's total municipal waste, approximately a third, and therefore it is crucial for overall resource efficiency. Besides resource efficiency, it is important to move away from the landfilling of biowaste, which currently contributes to at least 1,6% of the world's total anthropogenic greenhouse gas emissions into the atmosphere.

### TWO KEY PRINCIPLES IN EU WASTE LEGISLATION: THE WASTE HIERARCHY AND LANDFILL DIVERSION

Waste legislation is an important part EU environmental legislation, which has been revised in several occasions since the 1970s. The application of many of these measures has been very effective in increasing recycling rates and reducing uncontrolled landfill sites, where new member states which joined the EU in the 2000s now have to catch up with old member states that have been implementing EU waste rules for decades. Several member states have been taken to court by the European Commission for non-compliance of binding waste objectives, including several recent cases, evidencing that implementation of waste rules is taken seriously. The two most relevant pieces of legislation for biowaste treatment currently in place are: (1) Waste Framework Directive (WFD) 2008/98/EC setting the definitions, recycling targets and general principles for municipal waste management; (2) Landfill Directive 1999/31/EC setting rules for landfill sites and a landfill site diversion target for biowaste.

At the core of the WFD is the waste hierarchy (see Figure 1 below), where national legislation shall give priority to resource efficiency, starting from the upper part of the inverted triangle with waste prevention, reuse and then recycling, where the lower levels should be avoided as much

as possible including recovery operations (e.g. energy recovery from incineration) and in particular disposal via landfilling. This hierarchy gives a clear signal to resource efficient solutions, where materials stay in the system and are used in a circular way as much as possible. For the case of biowaste, preferred measures include better prevention of food waste by the improvement of distribution systems and the creation local food banks.



Figure 1: EU Waste Hierarchy (European Commission website)

Following prevention, two techniques are recognised for recycling biowaste under EU legislation: composting and anaerobic digestion. This gives a clear signal to national authorities and investors to favour these two technologies. Commission Decision 2011/753/EU, a secondary piece of legislation related to the WFD, establishes that they can be used to count towards national recycling targets under the main condition that the produced compost or digestate is used on land for fertilising purposes. In contrast, the utilisation of biogas from anaerobic digestion without the use of digestate for agronomic purposes, is classed lower in the hierarchy as an energy recovery operation. This condition is an effective way of boosting resource efficiency, although the rules could be more clearly stated in the main text of the WFD which was under revision in early 2017. Additionally, rules related to EU waste legislation should be better adapted for recyclers, in particular concerning fertilisers to ensure that digestate and compost can leave their waste status and be used on land, which in turn will enable waste managers to fulfil the WFD requirements for biowaste recycling.

Another important principle in EU legislation that concerns biowaste resource efficiency is landfill diversion. Under article 5 of the Landfill Directive, biodegradable waste capable of decomposing such as "food and garden waste, and paper and paperboard" had to be reduced by 2016 under 35% of the share by weigh compared to the reference year of 1995. This measure has been very successful at reducing methane emissions from Europe's landfill sites and at making biodegradable waste available for processes higher up in the hierarchy such as recycling. In view that most EU countries have either succeeded or are on their way to matching the required 35%

landfill diversion, it is essential to reinforce resource efficiency by setting a lower landfill diversion target for 2030 of no less than 10% with reference to 1995. As more biodegradable material continues to be diverted from landfills across Europe, future EU legislation should ensure that it does not go straight into incinerators for energy recovery. To ensure that resource efficiency is maintained, an incineration ban should be placed on all recyclable biodegradable material including biowaste.

#### THE IMPORTANCE OF RECYCLING TARGETS AND SEPARATE COLLECTION

While the waste hierarchy ranks measures and technologies in terms resource efficiency, the waste target is the tool to ensure that member states comply with this principle. Article 11 of the WFD sets a mandatory target for recycling and re-use of at least 50% of all household waste by 2020. To ensure the constant quality of the materials destined for recycling and reuse, article 11 sets an equally important mandatory requirement for household separate collection by 2015 for the following waste streams: paper, metal, plastic and glass. According to the latest official waste implementation report, recycling and compliance rates across the EU are generally encouraging but results are mixed from one country to another: seven EU Member States had already reached or exceeded the 50% 2020-target for recycling by the end of 2012; seven had achieved over 40% recycling by the end of 2012 and were well on track to meet the 2020 goal; the remaining EU countries, mainly new members, still have to catch up by making significant investments and changes to national legislation, while some of them are likely to need a transition period beyond 2020 to fulfil the 50%.

The mentioned four waste streams with separate collection duties currently have the highest recycling rates in Europe, which constitute the lion share of the material that is either recycled or reused under the WFD target. In contrast, biowaste does not have a separate collection obligation. As a result, in most cases it is mixed with the residual fraction of municipal waste, which introduces contaminants that makes it unfit for agronomic use and therefore not eligible for recycling. The lack of mandatory separate collection, together with the relatively low 50% recycling target, are key factors in the comparably high rates of biowaste incineration and landfilling. To solve this imbalance, it is essential to introduce a separate collection duty for biowaste ensuring the stream remains clean to then be digested or composted. In view of the steady increase of recycling rates of member states in the EU, the waste target should be updated beyond 2020, increasing it to 70% by 2030. These two changes would give a clear signal to investors and waste managers to systematically recycle the biowaste fraction.

The WFD and the Landfill Directive have both been important drivers for resource efficiency and with the right reforms they could have a radical effect over the coming decade on the recycling rates of biowaste. This success in the household sector should be replicated elsewhere, by extending principles like the waste hierarchy and recycling targets to other areas. The WFD and Landfill Directive targets should extend their scopes in particular to the industrial and commercial sectors where large quantities of mostly homogeneous biodegradable waste can be recycled.

#### THE IMPORTANCE OF LEAVING THE WASTE STATUS FOR RECYCLED MATERIAL

On the one hand, waste legislation can be a key driver to source clean waste streams and encourage recycling. On the other, it can act as a barrier if the recycled material does not cease being waste in the legal sense. Waste substances are subject to strict health and safety restrictions, can only be transported and handled under specific conditions by certified operators and more importantly cannot be used or sold as products unless an authorised transformation processes exists and is carried out. To avoid unmercenary burdens, it is important to first clearly define what is considered waste and what is not, and secondly to ensure that recyclable waste substances have clear processing criteria under which they cease being waste to become products or useable secondary raw materials.

It is important to make a distinction between substances with a certain level of risk or variability common to waste, such as biowaste, from safer organic residues mostly from plant origin with no marked risks, such as pulp from sugar beet or spent grain from breweries. Article 5 of the current WFD allows for by-products of a production process not to fall under the waste category, as long as the substance's use is certain and does not lead to adverse environmental or health effects. While this provision is in place since 2008, EU Member States have for the most part not integrated it in their own national waste legislation. This results in that many organic materials in the agroindustry which could be eligible by-products fall or may fall under the waste category. In turn, this brings unnecessary regulatory burdens and costs for industry using these residues and potentially discourages new investors. To avoid this, it is important to strengthen the by-product article in the WFD, requiring member states to actually make use of this provision. In addition, the Commission could prepare specific guidelines on the kind of organic materials that could be regarded as by-products.

Having an end-of-waste point is essential for recyclers, as it sets the transformation parameters and/or quality and safety criteria under which a waste is no longer considered as such, but instead it becomes a product or a raw material. This is set under article 6 of the WFD on End-of-waste criteria and then technical secondary legislation is developed on specific recycling parameters for several waste streams. There are existing EU decisions on end-of-waste including various metals and glass which have been very useful for recyclers of these materials. For biowaste, however, the technical discussion to have EU end-of-waste criteria stalled for years and none could be set under the WFD. This gap in legislation has been a big drawback for organic recycling. At the moment, several EU countries have their own diverging national end-of-waste criteria, undermining the creation of a European market for organic fertilisers. In other countries, there is still a lack of national criteria, where either decisions to use organic fertiliser are taken on a case by cases basis or their use is virtually impossible.

There are positive developments in the current revision of the EU Fertilisers Regulation 2003/2003, to include the quality and safety requirements for compost and digestate, which includes technical processing requirements such as temperature profiles and retention times for anaerobic digestion, as well as limit values for heavy metals and other pollutants. If the various conditions of this future directive are met by recyclers, then organic materials including source

separated biowaste will be able to leave the waste status and become a fertilising product that can be transported, used and sold freely across the EU's single market. By-products from the agroindustry are also eligible. Giving EU-wide product status to digestate and compost will lift many regulatory burdens and strengthen consumer trust in recycled materials. Nonetheless, this is not a substitute for biowaste end-of-waste criteria, which are still necessary to produce and use products or raw materials other than for soil fertilisation that the Fertilisers Directive will no cover, such as biochemicals, bioplastics and precursors for algae growth. Therefore, the discussion should be relaunched to set biowaste end-of-waste criteria as soon as possible under the European Commission's Circular Economy strategy.

#### CONCLUSION

Ambitious and targeted waste legislation can act as an important driver for resource efficiency, by imposing targets on public authorities and by creating a stable legal framework for investors in recycling. Without detriment to health and safety, waste legislation should also be both clear and flexible to ensure that useful streams can either stop being waste, or in the case of by-products not become waste in the first place.

Organic recycling technologies are well established and still developing fast. At the same time, crucial EU revisions are underway on waste and product legislation. With the right ambition and incentives in place, the coming decade could be a turning point for organic recycling in terms of large scale deployment across the EU. The waste industry and decision-makers should work together to get there.

# Economic Opportunities for the German Biogas Market within the Framework of the EEG 2017

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On the 1st of January of 2017, the German Renewable Energy Act (EEG, for its German acronym) came into force. The mechanism to support the development of renewable energies in Germany changed with this amendment for the first time in the history of this law since the year 2000 from fixed feed-in tariffs to a tendering model. These requirements create new challenges for plant operators and public authorities, who are involved with the implementation.

In the future, only the amount in cent (ct) per kilowatt-hour (kWh) is decisive for the acceptance of a given bid. The maximum bidding price for biogas plant operators who want to achieve a follow-on regulation is 16.9 ct/kWh. For new biogas plants the maximum bidding price is 14.88 ct/kWh, except for small scale plants based on manure with a maximum installed electrical capacity of 75 kW (kilowatt).

In the following, the impacts of the EEG 2017 on the established plant stock and the future expansion of the installed electrical capacities of biogas plants are shown.

The German Biogas Association has examined the impacts on different types of plants which differentiate from each other in terms of feedstock used, installed electrical capacity, location and thus available heat distribution potential. It has to be discussed if a competition between the different types of plants is possible within the framework of the EEG 2017.

The electricity generation costs or Levelized Costs of Energy (LCOE) were calculated for different types of plants under varying influencing parameters. LCOE for a small-scale plant with an installed electrical capacity of 150 kW  $_{\rm el}$ , a plant of the medium performance segment with a rated power of 400 kW $_{\rm el}$  and a large-scale plant with 1,000 kW $_{\rm el}$  were calculated and compared.

The results showed that a competition between the various types of plants can only be possible in a limited framework by means of a joint tender. The LCOE of larger plants are below those of smaller ones. Therefore, only under estimated optimized operating conditions is possible for plants with a lower installed electrical capacity to compete with such large plants. In view of this, the expansion/maintenance of the electrical power in the biogas sector is expected to be in the segment of larger concepts, which would jeopardize one of the central objectives of the EEG, the diversification of actors in the energy sector.

Additionally, the anaerobic digestion of bio-waste will suffer from significantly changed framework conditions. For example, plant operators who use separately collected bio-waste from households for biogas production have to be successful in two different tendering rounds. On one hand, the bid to obtain the authorization for the waste collection, and therefore the feedstock acquisition. And on the other hand, the bid in the framework of the EEG, to ensure a long term economically perspective.

However, it must be noted that in most cases the maximum bids are well below the actual electricity production costs. Therefore, the EEG 2017 offers reasonable perspectives to the biogas industry in Germany, but it requires considerable improvements.

### IV. In the Spotlight – Awarded Research

#### Nominee - Tolpe Award 2017 GENIAAL - from Manure to green Minerals and Clean Water

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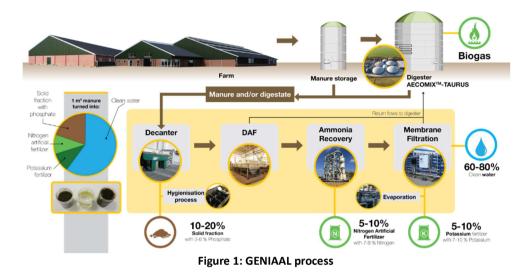
#### INTRODUCTION

Several countries in Western Europe and North America with farmers and manure processing facilities are challenged to manage their manure or digestate. Digestate is the solid/liquid fraction after digesting manure into biogas. Manure consists for more than 90% out of water and low concentrations of minerals. Therefore, the value of manure is very low and transport costs are high. However, pig, cow and chicken manure can be turned into heat and electricity and recover resources such as water and fertilizers.

#### TOTAL SOLUTION FOR MANURE

Nijhuis GENIAAL is a total solution for manure and turns raw manure or digestate (the solid/liquid fraction after digesting manure into biogas) into separated phosphate, nitrogen and potassium fertilizer and clean water. The principle of GENIAAL is based on producing valuable bio fertilizers and green minerals from raw manure and digestate and will help for example to contribute by solving the manure- and phosphate surplus in Western Europe based on an innovative, cost saving and sustainable way.

The first step of GENIAAL is the separation of manure or digestate (the solid/liquid fraction after digesting manure into biogas) into a solid and liquid fraction with a decanter without using polymer. The second step is treating the liquid fraction with flotation technology where a large part of the total suspended solids will be removed from the liquid fraction. The third step is treating the liquid fraction with an ammonia stripper and recovery system, producing ammonium based nitrogen fertilizer. The fourth step is treating the liquid fraction with membrane filtration followed by evaporation into organic potassium fertilizer and clean water.



#### **RESOURCE RECOVERY TO ACHIEVE REGIONAL BALANCED FERTILIZATION**

GENIAAL is a "Resource Recovery Center" and produces 10-20% in volume of phosphate rich solid fraction with 3-6 % of phosphate, 5-10% in volume of liquid nitrogen fertilizer with 7-8% of ammonium sulphate, 5-10% in volume of organic potassium liquid fertilizer with 7-10 % of potassium and 60-80% of clean water. This clean water can be used as cattle feed water or discharged to surface water. The produced green minerals ensure regional balanced fertilization by dosing sufficient individual nitrogen, phosphate or potassium fertilizers for pasture land or arable crops. This will reduce the rinse of fertilizers into ground and surface water and reduces the use of the chemical fertilizers.

#### **GREEN MINERALS WITH VALUE**

Long term pilot research has shown that GENIAAL is a reliable and feasible solution for 50.000 ton of manure per year or more and has a lower manure and digestate treatment price in comparison to the current manure treatment prices in the Netherlands and Belgium. Based on the results of the pilot research and the interesting business case, Groot Zevert Digestion in the Netherlands has given Nijhuis the order to build the first full scale GENIAAL installation for 100.000 manure or digestate (manure from approx. 60-80 farmers). The installation will be operational at the beginning of 2018.



Figure 2: GENIAAL build at manure digestion plant Groot Zevert, the Netherlands

Several manure-treating companies are paying a lot of money to get rid of their digestate. With the GENIAAL solution, the treatment cost for digestate will be lowered and manure digestion becomes financially more feasible. The return on investment for GENIAAL is in most cases between 3 and 6 years due to the reduction in digestate with 60-80% and the specific revenues for the produced green minerals.

#### SUSTAINABLE PROCESS

GENIAAL is a sustainable process because no by-products and no waste products will be produced, it is an energy positive solution in combination with digestion and reduces the CO<sub>2</sub> footprint due to lower production of chemical fertilizers and reduction in manure or digestate transport. The produced organic rich phosphate fraction, liquid nitrogen fertilizer and the liquid potassium fertilizer can reduce the import of artificial fertilizers for pasture land or arable crops and can reduce the export of surplus manure. The volume of manure or digestate will be reduced by 60-80%, because this will be discharged as clean water. GENIAAL is a total solution to achieve value with manure and create a circular economy.

WEBSITE: www.nijhuisindustries.com

#### Winner - Tolpe Award 2017

#### Manure Valorisation – Turning a Problem into a Commodity

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#### INTRODUCTION

Land application of animal manure and digestate supplies agricultural soil with valuable and essential nutrients and organic matter, which help to meet crop nutrient requirements and to maintain soil fertility. Nevertheless, livestock production in some parts of Europe (e.g. Flanders, North-West Germany, and Holland) has experienced significant changes in the last decades like intensification and regional conglomeration generating significant amounts of surplus manure in regions where it cannot be efficiently used. Hence, current manure management practices results in environmental problems and economic disadvantages for the farmers.

In the EU-funded project BioEcoSIM an innovative technology to valorise pig manure was developed and demonstrated in real farm environment: This circular economy approach integrates a series of modules to transform manure into stable and marketable soil improvers and mineral phosphorus (P) and nitrogen (N) fertilisers easy to handle, process and transport.

#### METHODOLOGY

Plant nutrients were extracted from pig slurry using a technology developed consisting on a series of modules that can be used independently or as a whole system according to regional legal aspects and context (Figure 1).

- <u>Conditioning of raw manure</u> to dissolve P found in the solid manure matrix into the liquid manure fraction and avoid ammonia losses during storage. This conditioning is carried out through an acid leaching of manure.
- <u>Two step solid-liquid separation</u> of the acidified manure. In the first step, a solid manure fraction is separated without any additives for further processing as soil improver with low P concentration at an organic fertilizer processing facility or dried on site. In the subsequently separation step, the liquid fraction is further separated through a microfiltration to obtain a particle-free liquid for further P and N recovery.
- <u>Drying of the solid manure</u> fraction with superheated steam in case waste heat can be obtained on site. This process is up to 80% more energy efficient than conventional air driers and has the advantage that pathogens are fully destroyed during this process.
- <u>Phosphorus recovery</u>: Once solids have been removed from the liquid phase a P recovery step takes place. In this stage, phosphate salts (struvite and other phosphate salts, e. g. calcium, magnesium, and potassium magnesium phosphates) are crystallised at pH values between 7

and 8.5 without the addition of magnesium salts. The P fertilisers are then separated from the liquid and dried either in situ or at a fertiliser processing facility.

- Nitrogen recovery: Ammonia contained in the liquid fraction diffuses then across a gas • tubular membrane and is recovered as ammonium sulphate.
- Blending: the solid manure fraction, poor in NPK but high in organic matter will be blended with the recovered mineral fertilisers to create designed organic fertilisers with the exact nutrient ratio to meet specific crops demands.
- The remaining liquid contains only traces of P and N, but is rich in potassium and is ideal for • irrigation purposes. Alternately, in sites with waste heat, this liquid could be vacuum evaporated at low temperatures (~60°C).

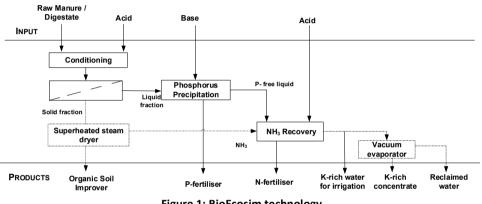


Figure 1: BioEcosim technology

Moreover, the integrated sustainability of BioEcoSIM and three State-of-Art (SoA) systems (long distance transport, manure separation and manure drying) were assessed using economic costbenefit analyses, Life Cycle Assessment and social statistical polls.

#### RESULTS

Within BioEcoSIM, it was demonstrated that manure could be transformed in a continuous process into stable and marketable soil improvers products easy to handle, process and transport. Every hour the BioEcoSIM pilot plant processed 50 kg of pig manure to about 500 g of mineral phosphate fertilizer, 500 g mineral ammonium fertilizer, as well as 2 kg organic soil improver. >99% of the total P in manure was solubilized during the acidification step. The manure was then successfully separated, to obtain a solid organic fraction poor in P ( $P < 0.7 \pm 0.2$  %) and a particle-free solution with high P concentrations (450 - 1200 mg/l P in manure). P-fertilizers were then precipitated and separated from the liquid fraction. These P-fertilizers were rich in nutrients, P<sub>2</sub>O<sub>5</sub> (22.5 ± 1.8 %), N (5.5 ± 3.4 %), K<sub>2</sub>O (3.8 ± 2.6 %) in DM. The N was recovered as pure ammonium sulphate (18% N).

Extensive investigations in greenhouse and field studies in two different locations (temperate -Germany and Mediterranean – Spain) have shown that the recovered mineral fertilizers can be used directly in agriculture as readily available fertilizers and the biomass yield achieved with

BioEcoSIM fertilizing products is comparable to commercial available mineral fertilizers such as triple-superphosphate and ammonium sulphate (Ehmann, 2017).

Moreover, a BioEcoSIM system processing 20.000 tonne manure per year performed better in both environmental and economic terms than the three compared SoA systems. BioEcoSIM had lower costs per ton of processed raw manure ( $\in$ 15) compared to SoA systems ( $\in$ 17 to  $\in$ 25). Plants processing annually 100.000 tonne manure had even lower costs ( $\notin$ 9- $\notin$ 13/t). In addition, BioEcoSIM contributed especially to the reduction of climate change, eutrophication, acidification and particulate matter formation. BioEcoSIM has good opportunities for social appreciation with farm-scale plants, limited regional transports and substantiated environment-friendliness claims. This makes BioEcoSIM also more favourable on social impact compared to SoA systems.

#### DISCUSSION

BioEcoSIM is an innovative concept since it offers a real manure valorisation. Our technology **1**) is **less expensive** than current approaches for manure disposal or treatment, **2**) is **automatized** and easy to use to avoid expensive labor costs, **3**) has a **robust operation** to avoid maintenance costs, **4**) is **modular**: according to the different manure fertilizing and disposal restrictions across Europe different modules of the plant can be selected, **5**) **recovers marketable products** (mineral fertilizers and soil improvers) from manure, which can be used directly in agriculture as readily available fertilizers and humus-forming substrates, and **6**) **better on all aspects of sustainability** than three state-of the-art manure management approaches. If 30 BioEcoSIM plants are sold after five years, we will be able to valorized 2.3 millions of surplus manure, produced 27.000 tonnes of phosphorus salts, 28.000 tonnes of ammonium sulphate and 61.000 tonnes of soil improvers. This will mean savings of 345.000 tonnes of CO<sub>2eq</sub>.

BioEcoSIM is a business-driven approach with excellent potential for quick deployment and market take-up. BioEcoSIM is at a mature development stage since it has been successfully validated and demonstrated at a pilot scale of 50 kg/h for over 15 months in a farm located in Kupferzell, Germany. Currently, a full-scale prototype with 1 tonne/h is being built to demonstrate the conditioning, solid-liquid separation and P-recovery modules. The techno-economic effectiveness of the modules will be validated in 2017 and the core members of BioEcoSIM consortium aims to introduce it to the market by 2018.

#### ACKNOWLEDGEMENTS

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#### Nominee for EBA Conference Poster Award 2016

# Techno-Economic Analysis of Biogas Plant Repowering Measures – Effects of an Extended Digestate Storage Capacity

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#### INTRODUCTION

The first biogas plants in the state of Baden-Wuerttemberg (BW), Germany, will drop out of the 20-years support scheme beginning in 2021 and the majority is soon reaching their halftime (see first half Figure 2). Rising substrate and operating costs, new conditions of the energy system, new fertilizer regulations, as well as the end of life of major components require changes in the biogas plant setup. Additionally, from the sustainability aspect, an improvement of the energy efficiency and a reduction of the ecological impact are favorable.

All these modernisation, cost reduction and improvement measures can be summarized under the term "biogas repowering". It describes technical and conceptual measures, independent of the location in the process chain, changing the original plant setup and increasing the energy as well as economic efficiency.

This study focuses exemplary on repowering measures resulting from ongoing changes in the fertilizer regulation in Germany. These changes may lead to an extension of the digestate storage capacity. Either caused due to not enough spreading land linked to the biogas plant or due to the expanded blocking periods for spreading organic fertilizers (Rauh 2017).

Therefore, the analysed measures are designed either to supply storage capacity or to reduce the need for it. For each measure, further investments and adjustments are required. In this context, the following questions arise:

- What effects result by the implementation of repowering measures regarding technical, economic and ecological aspects?
- How do development scenarios for the existing plants in BW look like if the measures (or combination of measures) with the lowest demand for an additional support scheme are implemented? It is assumed that due to the high production costs for electricity and heat (see Figure 1) biogas plants will not be able to operate cost-covering under the current market conditions without a support scheme. Likewise, no new major installations are expected.

#### MATERIAL AND METHODS

To conduct a detailed technical, economic and ecological analysis, a new model was created using MATLAB. Due to the heterogeneity of the existing plants, the assessment and identification of promising repowering measures is conducted on the individual plant level. As primary data input, the model uses the annual substrate feed mix and mass for each plant in BW. This data is derived from a substrate analysis of the EEG plant transaction data (TransnetBW 2016). It also delivers

important parameters like the installed electric capacity. Based on the location of the plants also regional parameters like the substrate costs are considered. The model boundary is the substrates supplied to the plant and the energy supplied to the grid.

In the first step, the model calculates the plant setup as well as the energy and mass balances for each plant in its original state using additional input and assumptions from an operators survey (Härdtlein et al. 2013). Using cost-functions for different components and other type of costs (e.g. substrate costs) the production costs based on the net present value method are calculated. All relevant greenhouse gases (GHG) emitted on site are covered, since they also have influence on the energy. Combined with specific GHG emission linked to the production of the substrates the overall GHG emissions can be calculated.

In the second step, the effects of the implemented measures are assessed using key performance indicators such as the fuel utilisation rate or the levelized cost of energy (LCOE).

If an extension of the digestate storage capacity will be necessary, different measures are possible, adding extra costs in most cases. Table 2 lists the analysed measures.

For a consistent comparison, each measure has to supply a total storage capacity for liquid digestates of nine months. In the reference case, the capacity is six months. In the case of measures reducing the volume of liquid digestates the remaining storage demand is supplied by additional storage tanks. Further benefits of a reduction in volume or changes in solid liquid phase ratio are also accounted for by spreading costs. Other benefits like changes in the nutrient composition of the digestate fractions leading to different spreading limitations, or synergetic effects with other measures, e.g. an increased gas storage volume for a flexible operation mode, are not accounted so far.

Measure	Description	Added storage capacity [m³]*	Added total cost [M €]*	Mean net fuel utilisation rate [%]
Add. storage 1	Gastight cover if the HRT is below 150d	827,151	36.47	60.09
Add. storage 2	Always with a gastight cover	827,151	101.19	60.53
Separation	Separation by screw press into a liquid and solid phase + Addition of storage tank for the reduced liquid phase	306,315	75.21	60.10
Thickening	Direct drying of liquid phase in a drum or belt dryer with remixing up to a dry matter content of 12%** + Addition of storage tank for the reduced liquid phase	365,731	108.86	46.85

### Table 1: Overview of measures dealing with an extended digestate storage capacity and total values calculated for the biogas plants in BW (nplants,data= 852)

Measure	Description	Added storage capacity [m <sup>3</sup> ]*	Added total cost [M €]*	Mean net fuel utilisation rate [%]
Vacuum Evaporation	Separation + treatment of liquid fraction by vacuum evaporation with a concentrate dry matter content of 15%** + Addition of storage tank for the reduced liquid phase	76,500	214.88	46.60

\*Sum over all plants; \*\* only unused available heat by the CHP unit is considered

# **RESULTS AND DISCUSSION**

Figure 1 shows the LCOE according to different size classes of the biogas plants in BW, calculated for further operation of 10 years. One can see that they are dominated by demand-related costs, which consist mainly of costs for substrates and auxiliary energy. It also shows that the costs of most plants are higher than the cap in the biding process for a follow-up promotion in the newest revision of the German renewable energy act (EEG 2017).

Figure 2 shows the mean additional specific production costs according to plant size for each measure listed in Table 2. Adding an open storage tank is the most economical solution for almost all plants. In general, there is high variation of the economic effects and smaller plants would face the highest impacts. In regard to technical aspects, gas-tight storage tanks will result in the highest mean net fuel utilisation rate and also likely lead to the lowest on-site emissions. The lowest demand for the total added storage capacity for all plants can be achieve by vacuum evaporation (see Table 1).

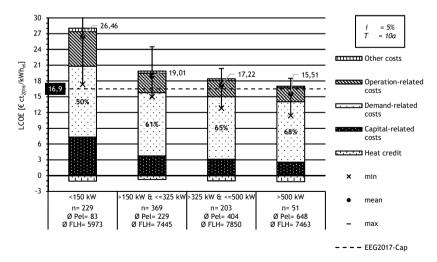


Figure 1: LCOE for existing biogas plants, considering salvage values and heat credits

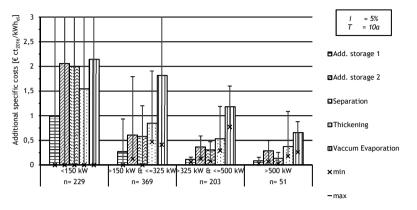


Figure 2: Additional specific costs of different measures - Mean values for different size classes

Assuming that all the plants with a LCOE below the EEG 2017 cap of 16.9 ct/kWh<sub>el</sub> will continue their operation for another ten years, the capacity and number of the biogas plants in BW will develop according to the Figure 3. It compares this with the reference scenario that assumes a shutdown for every plant after 20 years.

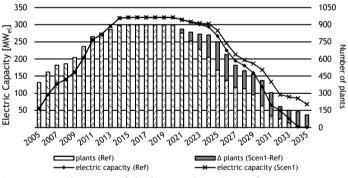


Figure 3: Development of the biogas plants in BW from 2005 to 2035

# CONCLUSION

Looking at the current development path, many biogas plants in BW will need to shut down and the structure will chance towards plants with a bigger capacity. Current changes in the German fertilizer regulation might aggravate this situation by inducing additional investments and costs. Again, smaller plants are affected more severely. This could contribute to and accelerate the structural change.

However, for many plants these investments present dead-end investments with no further use after the end of the biogas production, especially in the case of the storage tanks at farms without any livestock. Moreover, for more than a third of the plants the remaining operation time is

shorter than the period under review of 10 years. The additional specific costs for these plants will be higher and most likely lead to an early shutdown due to uneconomical conditions if there is no prospect of a feasible follow-up operation.

It remains to be analysed how further repowering measures and changes of the framework conditions might change this development.

### ACKNOWLEDGEMENT

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# Nominee for EBA Conference Poster Award 2016 Power-to-Gas by Biomethanation– From Laboratory to Megawatt Scale

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# SUMMARY

The share of renewable energy on total energy production has been rising. Wind and solar produce electricity intermittently and generally independent of demand. Bulk energy storage by an integrated Power-to-Gas system is the most promising solution to address the need for such challenge. In the Power-to-Gas process, low-cost and stranded electricity from wind and solar is utilized to convert carbon dioxide into methane, the principal component of natural gas (Sterner 2009, Götz 2015).

This renewable gas can then be injected into existing natural gas pipeline infrastructure and stored for days, weeks and even months without losses. It can also be used in the transportation sector or in industrial processes.

Electrochaea is using a proprietary microorganism for its Power-to-Gas technology, which is based on biological methanation. These microorganisms, so called methanogenic archaea, have been specifically developed to convert  $CO_2$  and  $H_2$  to  $CH_4$  at very high efficiency (Hafenbradl, Hein 2015).

The Power-to-Gas technology has now been applied at 1MW industrial scale with the BioCat Project. Further scale up towards 10MW is already in the planning phase.

### INTRODUCTION

Two basic chemical reactions are required to produce  $CH_4$ . The gaseous energy substrate  $H_2$  is produced by electrolysis (1).  $H_2$  together with  $CO_2$  (from biogas or other industrial waste streams) is then converted into  $CH_4$  (2). This methanogenesis is mediated by the biocatalyst employing a unique set of enzymes (Ferry 1998).

- (1) Electrolysis:  $4 H_2 O \rightarrow 4 H_2 + 2 O_2 + Heat$
- (2) Methanation:  $CO_2 + 4 H_2 \rightarrow CH_4 + 2 H_2O + Heat$

The application of the Power-to-Gas technology is driven by two general aspects:

- Storage of energy in form of methane directly into the existing gas grid. For this application, high flexibility of the biocatalyst to allow intermitted operational cycles is required. Fast response during shut-down/start-up is critical.

 Reduction of the CO<sub>2</sub> footprint to support international commitment and need to reduce net CO<sub>2</sub> exhaust and to reduce fossil fuel consumption. Biogas upgrading is one of the applications that can be addressed with the Power-to-Gas technology. A highly robust biocatalyst and efficient bioreactor system are the prerequisites to produce high quality biomethane in a long-term operation.

Both aspects have been addressed at Electrochaea in small laboratory reactor scale, and have consecutively been scaled to a 1 MW industrial bioreactor. The critical experiments to address all potential applications of the technology will be shown here.

# MATERIALS AND METHODS

Electrochaea's Power-to-Gas process is using a proprietary strain of methanogenic archaea, feeding on hydrogen and carbon dioxide, while producing exclusively methane at gas grid quality. The process has been optimized in laboratory scale (as described in detail in Martin 2013) and has been scaled to a 1 MW<sub>el</sub> plant at the so called BioCat Project.

Figure 1 shows the schematic design of the plant, which is located at a waste water treatment plant in Denmark. Hydrogen, which is produced by two 0.5  $MW_{el}$  electrolysers, is being mixed with raw biogas from the waste water treatment plant at a 4:1 ratio of H<sub>2</sub>:CO<sub>2</sub>. After mixing, the gas is being injected into the reactor at 9 barg. The optimum process temperature is 62°C. The heat which is generated by the exothermic biomethanation reaction is utilized at the waste water treatment facility. The archaea in the stirred tank reactor convert the carbon dioxide content to methane.

### RESULTS

The Electrochaea biocatalyst is a robust microorganism, which efficiently produces  $CH_4$  in a stable and continuous manner (Figure 1). More than 98%  $CO_2$  are converted over long periods of more than 400 hours of operation.

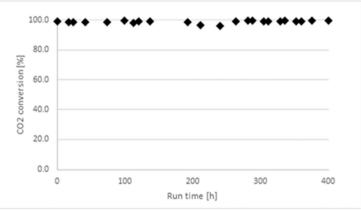


Figure 1: Stable methanation and conversion efficiency demonstrated in a laboratory reactor

The biomethanation bioreactor can be shut down for various time intervals. The instantaneous response following the start-up is independent on the duration of shut-down. Such fast response

will allow intermittent energy supply and accommodate load following schedules with high flexibility. Longer shut down periods of several weeks have also been tested (not shown here) and have no influence on the response to start-up based on the feeding with  $H_2$  and  $CO_2$  or biogas (Figure 2).

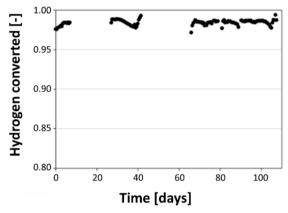


Figure 2: Fast response to fluctuating gas supply in a laboratory bioreactor

To address the application of biogas upgrading, the biocatalyst has been exposed to typical biogas mixtures. The volume of methane in the CO<sub>2</sub> gas stream was gradually increased, up to 60%. The supply of such high content of CH<sub>4</sub> in the feed-gas stream has no effect on the conversion efficiency (Figure 3) of the biocatalyst and provides evidence that there is no product inhibition reaction. In addition, various potential impurities of raw biogas or other industrial gas streams have been tested. In particular, the H<sub>2</sub>S, which is often found in high concentration in raw biogas, has no effect on the efficiency of the biocatalyst (Figure 4). This renders biological methanation superior to non-biological CH<sub>4</sub> synthesis such as the Sabatier process (Che 2013).

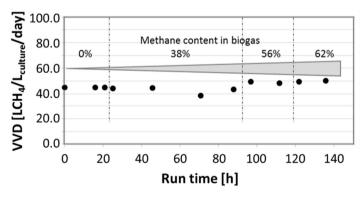


Figure 3: Efficient methanation using biogas in a laboratory bioreactor

Contaminant	Tolerant	Somewhat Tolerant
Hydrogen Sulfide (H <sub>2</sub> S)	٧	
Sulfate (SO <sub>4</sub> <sup>2-</sup> )	V	
Sulfite (SO <sub>3</sub> -)		V
Nitrogen Oxides (NO <sub>x</sub> )	V	
Ammonia (NH <sub>3</sub> )	V	
Oxygen (O <sub>2</sub> )		V
Carbon Monoxide (CO)	${f V}$ (<20% by vol.)	
Ethanol (C <sub>2</sub> H <sub>6</sub> O)		V
Particulates	V	

Table 1	Tolerance	to impurities
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### Results at BioCat 1 MW scale plant (Figure 5)

The conversion rate from  $CO_2$  to methane is stable and typically >98% methane content in the product gas is achieved. In the BioCat process 50 Nm<sup>3</sup>/h of methane and 320 kW/h of heat in total are produced. The product gas from the top of the reactor is ready for grid injection after drying and cleaning. The BioCat plant is fed with raw biogas, a mixture of  $CO_2$  and methane without requiring previous separation.

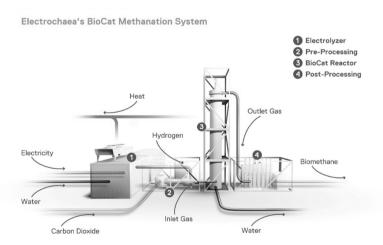


Figure 5: BioCat Power-to-Gas plant in Avedøre, Denmark

### DISCUSSION

The results show that Electrochaea's biological catalyst is capable of converting  $CO_2$  from different sources such as but not limited to biogas into grid quality methane very robustly and efficiently. Compared to non-biological CH<sub>4</sub> synthesis such as the Sabatier process the biological

catalyst is much more tolerant regarding H<sub>2</sub>S and other potential contaminants in the biogas. As the reaction time of the archaea is very fast, the process is very suitable for an electricity infrastructure with many volatile power plants such as wind power plants. The key features of Electrochaea's Power-to-Gas technology are:

- Rapid reactor ramp-up/-down time (<40s)
- Instant biocatalyst recovery after system idling allows high partial load flexibility
- High methane production rate is independent of biogas CO<sub>2</sub>/CH<sub>4</sub> ratio
- Raw biogas is currently used to generate grid quality gas in the BioCat Project (www.biocat-project.com)
- Lower CAPEX due to use of raw biogas
- Different biogas composition can be used for biomethanation
- Highly robust biocatalyst

Finally, the ability to scale-up the process to several MW opens the option for large scale decarbonisation.

### ACKNOWLEDGEMENT

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# Winner of EBA Conference Poster Award 2016

# Underpinning green energy: Reference standards and methods for the measurement of siloxanes in biogas

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### INTRODUCTION

Siloxane impurities within biogas can cause extensive damage to gas processing equipment if not removed effectively. A European specification is being developed that will impose a maximum limit value on the total silicon content of biogas in order for it to be safely utilised [1,2]. This requirement for accurate measurements of total silicon at low amount fractions is what has driven advances in measurement methods as part of a European project [3].

### **MATERIAL & METHODS**

### Gas Mixture Preparation

High accuracy, traceable gas reference standards containing L2, L3, D4, and D5 siloxanes were prepared gravimetrically. The mass of each component added to the cylinder is quantified using high-precision balances. The addition process has been refined through over five years of research at NPL.



Figure 1: Injection of pure siloxane into transfer vessel prior to high-accuracy weighing

### **Homogenisation**

After the addition of the final mixture component, the cylinder is heated and rolled for several hours to ensure the gas mixture is homogenous.

### Validation

The siloxane reference standards are validated using high precision techniques, to ensure they meet quality requirements.

### Speciated siloxanes method

Individual siloxanes are quantified using a Gas Chromatograph-Mass Spectrometer with flame ionisation detection (GC-MS-FID).



# Figure 2: Capillary column GC-MS allows for the quantification of siloxanes at ppb levels

#### Total silicon method

A novel technique is being developed at NPL that will measure the total silicon content of a biogas reference mixture at ppb amount fractions. The principal relies on removing the lighter biogas molecules from a gaseous sample and then measuring the silicon content of the remaining siloxanes. This is achieved through use of a Gas chromatograph coupled to an Inductively Coupled Plasma Mass Spectrometer (GC-ICP-MS).



Figure 3: GC-ICP-MS setup for the measurement of total silicon in gaseous reference samples

### RESULTS

The results of mixture validations prove that ppb level siloxane gas standards can be produced with k=2 uncertainties ranging from 1-8% relative. Higher uncertainties are associated with heavier molecules and lower amount fractions. Stability of siloxane components within a gas mixture have been shown to vary by species, with L3 being the least stable molecule within gas cylinders.

# CONCLUSIONS

The developments in preparation and analytical methodology over 5 years of research at NPL have provided insights into the accuracy and stability of siloxane reference gas standards. These standards will provide valuable support the growing biogas industry by improving the accuracy of siloxane quantification. Future work would look to address the stability of siloxanes within cylinders and further improve the accuracy of the standards.

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- [3] EMRP Metrology for Biogas project <u>http://projects.npl.co.uk/metrology-for-biogas/</u>

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