BIOREFINE

Recycling inorganic chemicals from digestate derivatives

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Techniques for nutrient recovery from digestate derivatives

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PU	Public	x
PP	Restricted to other programme participants (including the Commission Services)	
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1 Glossary

AAD: Advanced Anaerobic Digestion

AD: Anaerobic Digestion

CHP-unit: Combined Heat and Power unit

DM: Dry Matter

DS: Dry Solids

g/l: Gram per liter

H: Hydrogen

H_2S: Hydrogen sulfide

K: Potassium

Kg/h: Kilogram per hour

kW : Kilowatt

kWe: Kilowatt-electric

kWh/m³: Kilowatt hour per cubic meter

kW th: Kilowatt-thermal

L: Liter

m³/d: Cubic meter per day

m³/h: Cubic meter par hour

Mg: Magnesium

mg/l: Milligram per liter

MF: Microfiltration

MW: Megawatt

N: Nitrogen

NH_3: Ammonia

NH_4-N, NH_4^+-N: Ammoniacal nitrogen nm: Nanometer

N-S fertilizer: Nitrogen-Sulfur fertilizer

N_{tot}: Total Nitrogen

P: Phosphorus

P_2O_5: Phosphorus concentration expressed in phosphorus pentoxide

RO: Reverse Osmosis

SBR: Sequencing Batch Reactor

t: Ton

t/d: Ton per day

t/h: Ton per hour

t/year or t/y: Ton per year

UF: Ultrafiltration

WWTP: WasteWater Treatment Plant

2 Introduction

Anaerobic digestion (AD) is a well-established method for the treatment of organic waste streams and the generation of biogas for the production of renewable energy. The main products for AD are biogas and digestate.

Digestate is the solid/liquid material produced by AD of organic feedstocks. Digestate is a mix of microbial biomass and undigested material. AD and gasification converts around 15 % of the feedstock to gas and 85 % remains as organic solid/liquid material. Characteristics of digestate are: high content of nitrogen (2.3 - 4.2 kg/tonne), phosphorus (0.2 - 1.5 kg/tonne) and potassium (1.3 - 5.2 kg/tonne) and as a consequence, digestate has value as an organic fertiliser (Chambers, 2011).

Similar to animal manure, there exists a large range of techniques suitable for the processing of digestate. An overview commonly used digestate processing techniques, including nutrient recovery techniques, is given in the inventory 'Techniques for nutrient recovery from digestate' published by Lebuf et al. (2013) under the Interreg IVB project ARBOR. In this brochure, nutrient recovery techniques are defined as techniques that (1) create an end-product with a higher nutrient concentration in comparison to the raw product or (2) separate the envisaged nutrients from organic compounds with the aim to create an end-product that can be used by the chemical or fertiliser industry (Lebuf et al., 2013). This report focuses on existing case studies of different nutrient recovery techniques from digestate or digestate derivatives.

It must be kept in mind that the composition of digestate is variable and depends on the input materials that are digested. Consequently, the processes described in this report will not always show the same efficiency.

3 State-of-the-art nutrient recovery techniques from digestate

Different techniques can be applied to digestate to extract nutrients. The following techniques mainly refer to pilot plants. These installations must cope with the variability of digestate composition. This high heterogeneity is a constraint which makes the upscaling of the processes more difficult.

3.1 Separation and thickening of the thin fraction

3.1.1 General description

The following scheme (**Figure 1**) shows the general description of the treatment scheme.

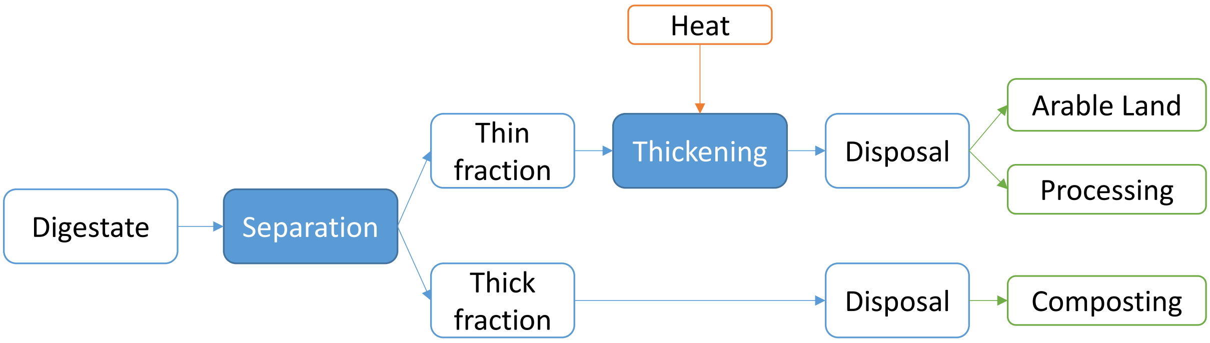

Figure 1: Scheme of the process of separation and thickening of the thin fraction of digestate.

Source: DLV InnoVision

All the raw digestate is forwarded to a separation step. The composition of the thin and thick fraction after this separation depends on the type of separation techniques applied.

Table 1 below gives an overview of the different compositions of both the thick and the thin fraction, right after the separation step, for 3 different separation techniques. These figures are calculated from both efficiencies found in literature and from practical experience.

Table 1: Composition of the thick and thin fractions of digestate after the separation step achieved through 3 different techniques.

			Centrifuge **with polymer**	**Screw press**	**Belt press**
Thin fraction	DM	%	3 %	8 %	3 %
	N_{tot}	kg/ton	5.0	5.7	3.9
	P_2O_5	kg/ton	1.0	3.8	0.5
Thick fraction	DM	%	35 %	26.6 %	24 %
	N_{tot}	kg/ton	7.5	4.5	9.4
	P_2O_5	kg/ton	14.1	5.6	11.6

Source: Beste Beschikbare Technieken (BBT) voor composteer - en vergistingsinstallaties; Verhoeven J.T.W., (2013) After the separation step, the thick fraction will not further be treated – it is disposed of in the actual format. This can be either an application on arable land, or in case this is not possible to a composting site which will further transform the thick fraction to compost.

The thin fraction will pass a thickening step in which water will be evaporated from the thin fraction. This is required in order to minimize the volume of thin digestate as much as possible as the more volume, the high the disposal costs.

The composition of the thin fraction after the thickening step is highly dependent of the amount of heat that can be used for the evaporation of the water. If we assume that about 3000 kW is to be used for evaporation purposes for a 60 000 ton/year installation, the final composition of the treated thin fraction will be close to the figures shown in **Table 2**.

Table 2: Composition of the thin fraction of digestate after the thickening step.

			Centrifuge **with polymer**	**Screw press**	**Belt press**
Thin fraction	DM	%	6.5 %	16.8 %	11.0 %
After evaporation	N_{tot}	Kg/ton	6.5	6.0	6.3
	P_2O_5	Kg/ton	2.6	8.0	1.7

Source: Beste Beschikbare Technieken (BBT) voor composteer - en vergistingsinstallaties; Verhoeven J.T.W., (2013)

This thin fraction will be used on arable land as a fertilizer.

3.1.2 Unit operations of separation techniques

Centrifuge

In a centrifuge, the non-soluble components are separated under influence of the centrifugal forces. The center of a decanter centrifuge (**Figure 2**) consists of a drum with a screw inside. Giving the drum a high rotational speed, a G-force develops. The basic operation of a centrifuge is based on the centrifugal force on the incoming stream, making the insoluble components heavier than water migrate to the wall of the drum. The screw inside the drum pushes the heavier parts in the opposite side of the stream direction making the thick stream further thickened.

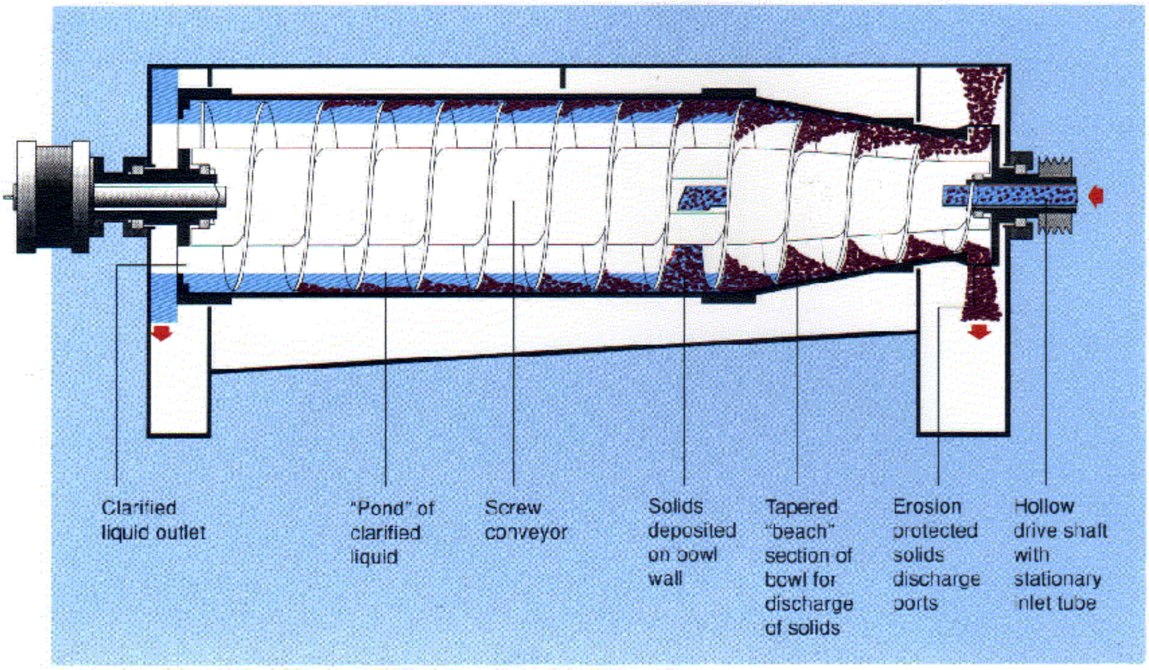

Figure 2: Scheme of a centrifuge, showing that the insoluble components heavier than water are separated at the right side while water is recovered at the left side.

Source: Poirier M.R. et al., (2002)

Screw press

Inside a screw press (**Figure 3**) a screw turns within a cylindrical perforated through with holes of 0.15 – 1.0 mm. Through these holes the thin fraction is separated from the incoming manure/digestate/mixture of both. The screw presses the thick fraction out of the machine (Verhoeven, 2013).

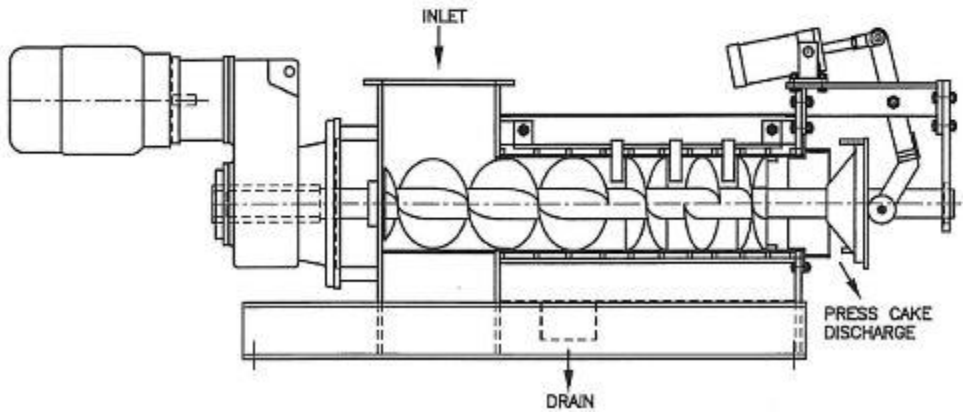

Figure 3: Scheme of a screw press, indicating the inlet, drain and cake discharge.

Source: VINCENT Corporation, (year not specified)

Belt press

In a belt press (**Figure 4**) the stream of manure or digestate is pressed between two conveyers. At least one of the two conveyers needs to function as a belt press. In most of the belt presses the bottom conveyer functions as a filter. The upper conveyer is typically a closed press belt which is pushed to the bottom belt with rollers (Verhoeven, 2013).

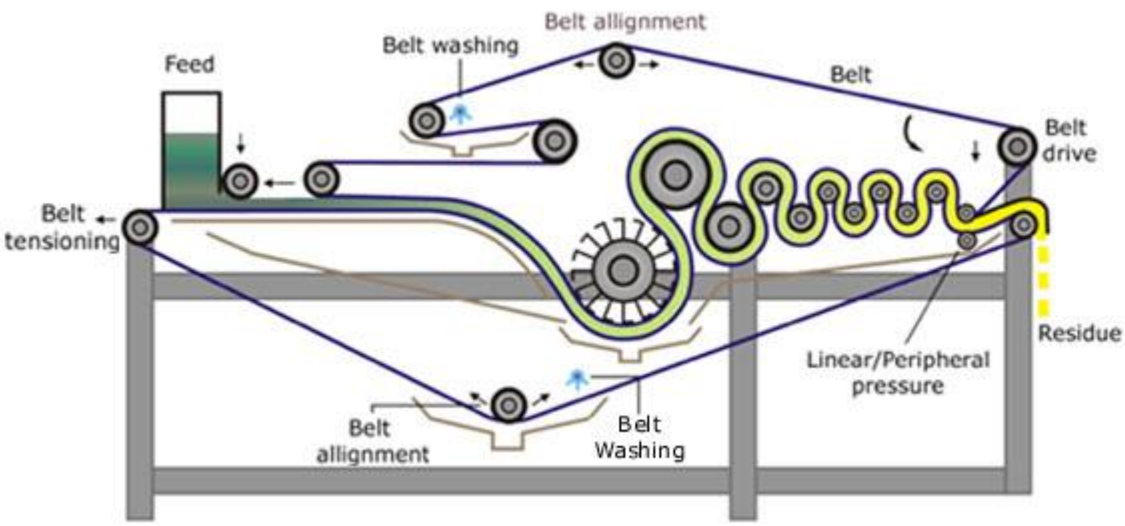

Figure 4: Scheme of a belt press.

Source: Schwarz Global Consulting, (year not specified)

3.1.3 Unit operations of thickening techniques

The regular application for the thickening of the thin fraction is in Flanders the use of a rotating wheel. This wheel (or multiple wheels – see **2** in **Figure 5**) will elevate thin layers of the material into the area where hot air (see **3** in **Figure 5**) is blown through. This contact of the hot air with the thin layer of the material will cause the required evaporation.

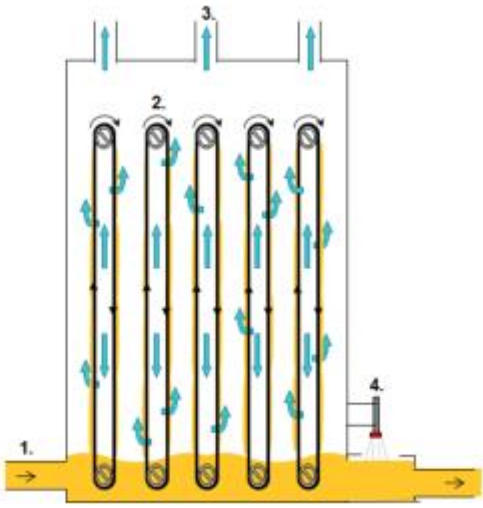

Figure 5: Schematic overview of a thickening system.

Source: Biogas Plus (year not specified)

3.1.4 Unit operations of air treatment

Blowing the hot air through the thin fraction of the material will cause a significant evaporation of the available ammonia. Apart from the ammonia, also other odour-components will be taken along with the waste air.

This waste air has to be treated before it is emitted into the air in order to prevent odour problems. The treatment required depends on the actual loading, but in normal circumstances at least a 3-step treatment is required. **Figure 6** provides an overview of the system.

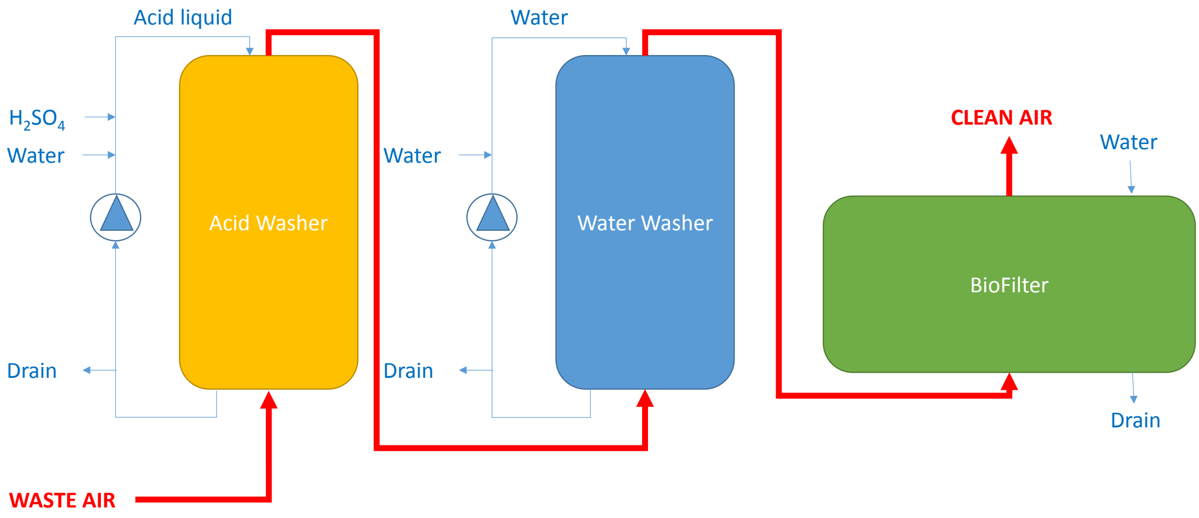

Figure 6: Schematic overview of the system of air treatment.

Source: DLV InnoVision

The process is composed of 3 steps:

- Acid Washing - by washing the air with an acid fluid (i.e. water combined with sulphuric acid) the ammonia present in the waste air flow will be washed out as ammonia sulphate.
- Water washing – after the acid washer the waste air is washed with water (high liquid / air ratio) in order to remove other components as dust and more acid components.
- Biofilter – as the final step of the air treatment the waste air will pass a biofilter – this biofilter consists of wooden particles and is moisturized on a regular basis. Bacteria are grown on this wood that will remove the remaining odorous substances from the waste air.

The drain of all the different steps is mostly mixed up with the thin fraction of the digestate for disposal on arable land.

3.1.5 Useful contact(s) for more information

DLV InnoVision (Belgium)

Email address: lies.bamelis@dlv-innovision.be

3.2 Drying of the thick fraction of digestate

3.2.1 General description

The scheme below shows the general description of the treatment scheme (**Figure 7**).

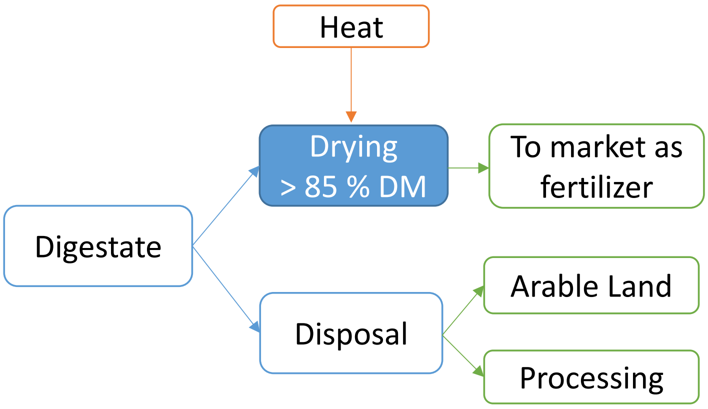

Figure 7: General overview of the process of drying of the thick fraction of digestate.

Source: DLV InnoVision

In this treatment scheme, there is actually only 1 treatment step: the raw digestate is dried as much as possible – consuming all the available waste heat from the cogeneration engine (CHP-unit).

The dried material should be > 85 %DM in order to be valuable on the market as a fertilizer. It is a stabilized product that can be stored – so it can be sold to the local market or even exported abroad. The raw digestate that is not treated can be disposed to arable land, or further processed. This further processing can either be on the site of the digester itself (mostly only separation in a thin and thick fraction) or in an external composting plant or a manure treatment plant.

3.2.2 Unit operations of the drying process

The most common application for drying of digestate in Flanders is the use of a belt drier. This type of drier has the benefit that it can work with a very broad range of heat – so also the lower temperature range coming from the CHP unit (55 – 65 °C) can be valorised in this type of drier. The disadvantage of this type of dryer – certainly if combined with lower temperature ranges – is that there is a large volume of air required to have an efficient process. All this air has to be treated in an air treatment system – which induces addition operation costs. **Figure 8** shows the structure of a typical belt dryer.

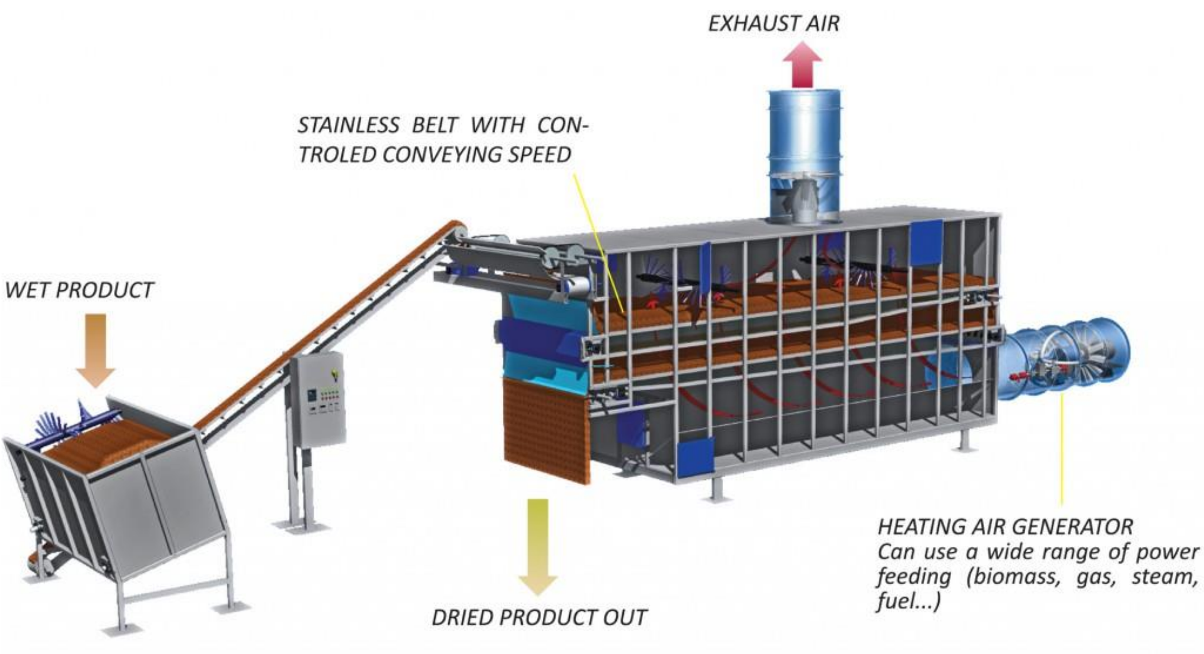

Figure 8: Structure of a typical belt dryer.

Source: Biogreen (year not specified)

In this type of drier, the wet product will be disposed over a rotating belt. Through this belt, hot air is ventilated – so the air is in direct contact with the material. As there is a significant recirculation of dried material to the point where fresh (wet) material is added, the material is in the dryer for quite a while.

There are other types of driers that do indirect drying where the hot air is not in contact with the wet material. This has the benefit that there is no need for an air treatment, though it requires higher temperature and a higher economic investment.

3.2.3 Unit operations of air treatment

The system is the same as under **3.1.3.** In addition to this, on the drying installation mostly a specific dust-remover is required for the removal of small particles from the waste air.

3.2.4 Additional comments

One of the most important issues of this type of treatment is the risk of fire in the dryer. Several installations in Flanders have had fire-issues that all started in the drier. The main issue is that in order to go from the digestate to the dried material, one has to pass a danger range from 40 – 55 % DM. Within this range a spontaneous ignition of the material (once it is let to pile up) is possible.

Therefore, it is always necessary to be very vigilant when shutting the drier down for maintenance or similar situations. At that moment some of the not completely dried matter will be left in the drier and can start a spontaneous combustion if the period is long enough. So when shutting down, one should always remove all the material present in and around the drier-installation.

3.2.5 Useful contact(s) for more information

DLV InnoVision (Belgium)

Email address: lies.bamelis@dlv-innovision.be

3.3 Separation, thickening of the thin fraction and drying of the thick fraction

3.3.1 General description

The scheme below shows the general description of the treatment scheme (**Figure 9**).

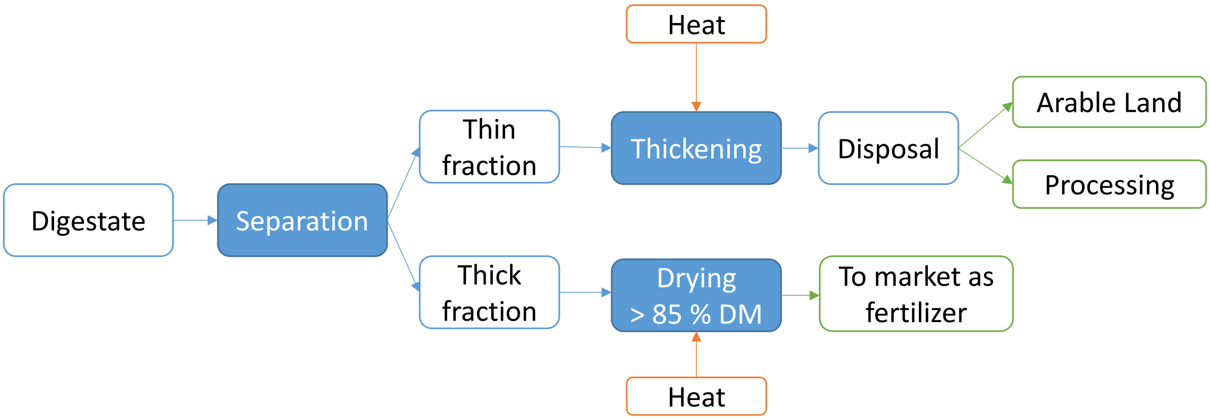

Figure 9: General description of the process of separation, thickening of the thin fraction and drying of the thick fraction of digestate.

Source: DLV InnoVision

This process actually is the combination of the different techniques described in **3.1.** and **3.2.** In this treatment scheme all the raw digestate will pass through a separation step. After that, a further processing of both the thin and the thick fraction is achieved on the site of the digester. For both processes, the waste heat from the CHP-engine is used. The plant owner can decide how much heat he converts to either thickening or the drying process. But under normal circumstances, first the heat will be applied to the drier system for drying all the available thick fraction. The remainder of the heat will then be consumed for thickening the thin fraction as much as possible.

The composition of the thin fraction throughout the process is given in **Table 3**. It is assumed that for a 60,000 ton installation, about 3 MW thermal can be applied for the drying and thickening. Of this 3 MW about 2 MW will be applied for the thickening and 1 MW will be applied for the drying of the thick fraction.

Table 3: Composition of the thin fraction before and after the thickening step.

			Centrifuge with polymer	Screw press	Belt press
Thin fraction	DM	%	3 %	8 %	3 %
	N_{tot}	kg/ton	5.0	5.7	3.9
	P_2O_5	kg/ton	1.0	3.8	0.5
Thin fraction after thickening	DM	%	4.5 %	14.5 %	5.4 %
	N_{tot}	kg/ton	4.5	5.2	3.1
	P_2O_5	kg/ton	1.8	6.9	0.8

Source: DLV InnoVision

3.3.2 Unit operations of the separation techniques

The unit operations are the same as in **3.1.**

3.3.3 Unit operations of the evaporation techniques

The unit operations are the same as in **3.1.**

3.3.4 Unit operations of the drying step

The unit operations are the same as in **3.1.**

3.3.5 Unit operations of the air treatment

The unit operations are the same as in **3.1.**

3.3.6 Useful contact(s) for more information

DLV InnoVision (Belgium)

Email address: lies.bamelis@dlv-innovision.be

3.4 Separation, thickening of the thin fraction and drying of the thick fraction combined with a biological treatment

3.4.1 General description

The process is the same than before (see **3.7.**). However, it is possible to improve it by implementing an aerobic treatment in the treatment scheme. This aerobic treatment allows reducing the nitrogen amount that is available in the thin fraction before it goes into the thickening step. This way it will be possible to dispose much more of the thin fraction on the same surface of arable land. This biological treatment will also "level out" the impact of the chosen separation technique, as it will be managed in order to meet a certain threshold at the outlet of the biological treatment.

The composition of the thin fraction throughout the process is given in **Table 4**. It also shows the composition of the material after the biological treatment. It is assumed that for a 60,000 ton installation, about 3 MW thermal can be applied for the drying and thickening. Of this 3 MW about 2 MW will be applied for the thickening and 1 MW will be applied for the drying of the thick fraction (as before).

Table 4: Composition of the thin fraction before and after the aerobic treatment.

			Centrifuge with polymer	Screw press	Belt press
After separation	DM	%	3 %	8 %	3 %
	Ntot	Kg/ton	5.0	5.7	3.9
	P205	Kg/ton	1.0	3.8	0.5
After biological treatment and thickening	DM	%	2.3 %		
	Ntot	Kg/ton	0.5		
	P205	Kg/ton	0.4		

Source: DLV InnoVision

3.4.2 Unit operations of the separation techniques

The unit operations are the same as in **3.1.**

3.4.3 Unit operations of the evaporation techniques

The unit operations are the same as in **3.1.**

3.4.4 Unit operations of the drying step

The unit operations are the same as in **3.1.**

3.4.5 Unit operations of the air treatment

The unit operations are the same as in **3.1.**

3.4.6 Unit operations of the biological treatment

The biological treatment that is applied in these types of installation is similar to the applications in a regular manure treatment system. The treatment chosen has to consist of a Nitrification – Denitrification set-up in order to assure the nitrogen removal. In Flanders both batch reactors (SBR) and continuous reactors are operational – all of them operating with activated sludge.

The excess sludge of these biological treatments can be fed to the digester for further valorisation.

3.4.7 Useful contact(s) for more information

DLV InnoVision (Belgium)

Email address: lies.bamelis@dlv-innovision.be

3.5 Solid/liquid separation and stabilization (Barkip plant)

3.5.1 General description

Scottish and Southern Energy (SSE) are working with Unicus scraped surface evaporators for digestate liquor treatment for liquid fertiliser production at the Barkip anaerobic digestion plant.

Barkip biogas facility is located in North Ayrshire and has a capacity to process up to 75,000 tons of food waste, manure and organic sludges/digestates. The scraped surface heat exchangers work by using the heat generated from the CHP process to concentrate the liquid fraction of the digestate to convert it into an organic fertiliser. Although this is the first time the technology will be utilised for digestate processing, the heat exchangers are well proven for other applications like pig manures (Waste & Resources Action Programme, 2012). The technologies employed are: two stage Thermophilic Anaerobic Digestion, centrifuge and scraped surface heat exchangers.

3.5.2 Unit operations

Barkip facilities comprises firstly an anaerobic digester which can treat up to 75,000 tons per annum of diverse organic streams, specially food waste, organic digestates and manure for the production of biogas. The biogas is stored and used in a CHP plant to provide heat and electricity within the installation. Excess energy is exported to the national grid. Other saleable products from the AD are digestate as soil conditioner and liquid fertiliser coming from AD liquors.

The acceptable feedstock for the plant are wastes from local farms, businesses, local authorities and commercial collections.

Waste delivery

Waste delivered to the site is off loaded into dedicated storage receptacles inside an enclosed reception building with exhaust ventilation venting to a bio-filter.

Anaerobic digestion

Anaerobic digestion process takes place in closed reactors with batch feeding for the production of gas, which is drawn off and stored prior to use.

Biogas purification

Biogas produced is transported via a gas scrubber to remove H_2S and NH_3 to reduce the potential of odour from the gases.

CHP plant

Biogas is burnt in CHP gas engines to generate heat and power. Remaining gas is sent to an emergency release via an 8 m high, enclosed flare.

Dehydration of digestate

The digestate resulting from the AD process is dewatered and separated by internally located decanter/centrifuges to produce a solid fibre and liquid concentrate.

Acidification of digestate liquor

Digestate liquor is then pre-treated with acid prior to evaporation within the scraped surface heat exchangers to prevent ammonia loss within the evaporator. The volume of acid dosed is dependent on the digestate and the desired retention. Within the evaporator the liquor is concentrated to approximately 20 % Dry Solids (DS).

Liquor concentration

The evaporator functions at temperatures between 50- 70 °C under vacuum. The heat required for the process is coming from the CHP plant. Barkip plant has evaporators that can treat 10,800 kg/h of digestate liquor and produce 1,565 kg/h of concentrate. For the Barkip application the heat exchanger tubes have been constructed from Duplex steel due to the high chloride content within the feedstock.

Mixing step

The concentrated liquor can be mixed with the separated solid fibre to obtain a solid fertiliser rich in nutrients and ready to export (Waste & Resources Action Programme, 2012).

The process scheme is shown in **Figure 10**.

3.5.3 Process scheme

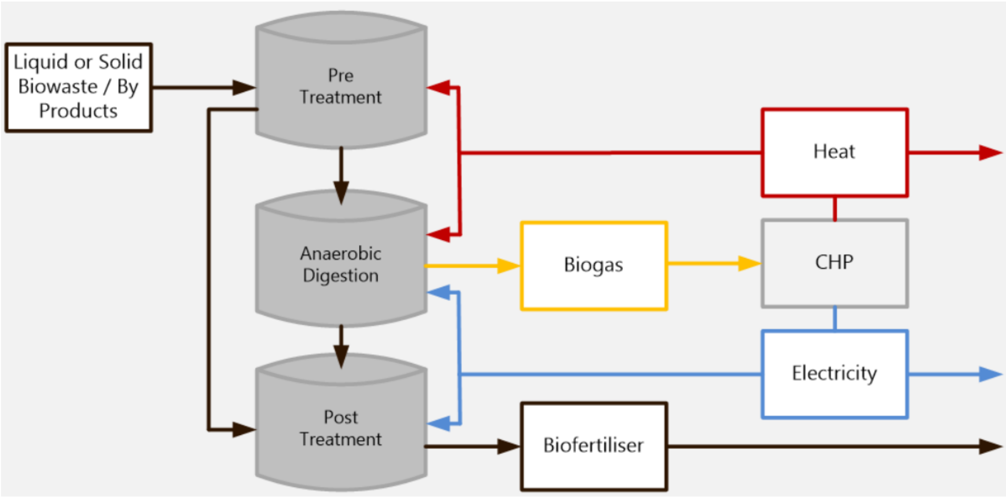

Figure 10: Anaerobic Digestion process used in the Barkip plant.

Source: Scottish and Southern Energy®

3.5.4 Useful contact(s) for more information

Scottish and Southern Energy (SSE) (United Kingdom)

Email address: see the website *www.sse.com*

University of Leeds (United Kingdom)

Email addresses: A.B.Ross@leeds.ac.uk, cndrr@leeds.ac.uk, M.A.Camargo-Valero@leeds.ac.uk

3.6 Pre-autoclaving and dehydration of digestate

3.6.1 General description

AeroThermal Group Ltd has been granted planning permission to develop a resource treatment and recovery facility at the site of Imerys Minerals Ltd at Lee Moor in South Devon.

AeroThermal's process consists in digestate autoclaving in a pressure vessel at constant temperature and pressure which helps to sterilise the digestate and at the same time break-down organic and lignin structures to reduce the volume by approximately 60 %. The autoclaving process helps to the removal of contaminants within the feedstock, improvement in biogas generation and the quality of the digestate.

Recyclable materials will also be recovered from the waste stream and the stable digestate, a by-product of the Advanced Anaerobic Digestion (AAD) process, will be used to help restore parts of the adjoining Lee Moor Quarry (Pell Frischmann, 2012). The main feedstock in the AeroThermal process is food waste for the generation of biogas as the central product by AD and digestate as subproduct, used as organic fertiliser.

3.6.2 Unit operations

The Lee Moor facility comprises two autoclave plants, screening and separation equipment, anaerobic digestion plant with associated buffer and digestate storage tanks, dewatering plant and a combined heat and power (CHP) plant (Barnes, 2012).

Loading into the autoclaves

Organic feedstock is loaded into two autoclaves in 10 ton batches. The autoclaves are fed up to 10 times per day via a system of conveyors from the weighing hopper.

Autoclaving step

Autoclaves are operated in alternating batch mode to recycle the steam from the unit that is being operated, to the autoclave that is starting a new cycle. With this procedure, steam utilisation efficiency is enhanced and there is also a reduction of steam released to the atmosphere. During autoclaving process, the reactor is rotated to allow a continuous mixed flow. Steam is injected inside the autoclave to reach 5.2 bar and 160 °C during 45 minutes.

Anaerobic digestion

Once autoclaving is finished, the temperature is returned to atmospheric conditions and ready to be placed in the anaerobic digester. Biogas will be produced by conventional AD process with mesophilic conditions at 28 days of retention time.

CHP plant

The biogas produced from AD enters to a CHP plant to produce up to 3.2 MW of electricity and 3.8 MW of heat. The electricity is suitable for being exported to the national grid while the heat is used to increase temperature of the autoclaves or the anaerobic digestion tanks. The technology enables the steam to be recycled between the two autoclaves, significantly reducing the amount of energy needed by the system.

Removal of contaminants

After autoclaving, some inorganic material and contaminants can be removed by mechanical separation providing a pasteurised and organic rich feedstock for AD.

Dehydration step

After AD, the digestate is dewatered in a centrifuge until reach a 25 % dry solids. Dewatered digestate is used for a restoration scheme at the Lee Moor quarry, which is located nearby. Digestate from the anaerobic digestion process is dewatered by a conventional centrifuge to obtain 25 % dry solids. The digestate fibre is used for land restoration at the Lee Moor quarry. On the other hand, liquors separated from the process are partially treated by dissolved air filtration (Barnes, 2012).

3.6.3 Useful contact(s) for more information

AeroThermal Group Ltd (United Kingdom)

Email address: see the website *www.aerothermalgroup.com*

University of Leeds (United Kingdom)

Email addresses: A.B.Ross@leeds.ac.uk, cndrr@leeds.ac.uk, M.A.Camargo-Valero@leeds.ac.uk

3.7 Drying and pelletizing (Biogas Bree case study)

In some cases owners of anaerobic digestion plants choose to dry the digestate in order to lower transport costs. The digestate is almost always dried using thermal energy from the CHP-engine.

3.7.1 Description of the technique

Because of legislative limitations for the use of digestate from animal origin in Flanders, the Biogas Bree plant has separate reactors for input from animal origin and from vegetable origin only. Two digestion reactors receive vegetable products. The digestate from these two reactors is fed to a post-digestion step after which it undergoes downstream processing. The end-product is used as a replacement for fossil based chemical fertilizers.

A third reactor, operated separately from the 'vegetable' reactors, is used for input from animal origin (including cow and pig manure), supplemented with smaller amounts of vegetable input materials (mainly molasses and sometimes maize). The raw digestate from this 'animal' reactor goes to a thermal drying step (**Figure 11**). The dryer uses heat from the Combined Heat and Power (CHP) engine. In 2014 the formation of digestate granules was accidentally induced after a slight adaptation in the drying process' parameters. After about a year and a half the Biogas Bree operators are able to control the granule formation using a 50 % recirculation of granular material. The grains have different particle sizes and are sieved accordingly and can be used as an organic fertilizer. At the moment they are mainly used in horticulture and not so much for agricultural purposes.

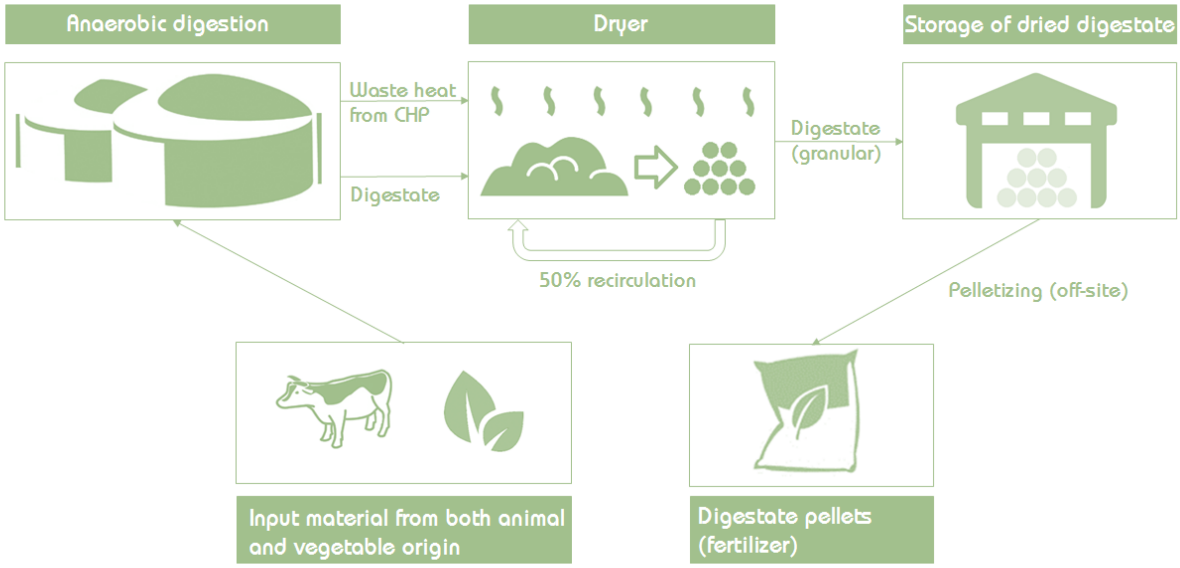

Figure 11: Schematic overview of the drying process with formation of granular material.

Source: Biogas-E

3.7.2 End-product

At the moment, the end product is dried granular digestate from different particle sizes. The particle size diameter is about 0.5 – 2 cm (order of magnitude).

3.7.3 Stage of development

The drying step is controlled in such way that the particle size distribution is quite stable. In the future it is the intention to pelletize the dried digestate to make the product more market conform for both private and professional uses. Certain fertilizer products (N, P, K) could be added to the pelletizer in order to be able to fabricate a product with a certain intended nutrient composition.

3.7.4 Useful contact(s) for more information

Biogas Bree bvba (Belgium)

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3.8 Membrane Filtration/Reversed Osmosis (Ampower case study)

3.8.1 Description of the technique

In pressurized membrane filtration the input stream is forced through the membrane by means of pressure. The input stream for membrane filtration can be either the liquid fraction of digestate from anaerobic digestion or a pre-processed stream, such as the condensate of the evaporator. Membranes are categorized and named based on pore size, different techniques have a typical range of pressure that can be applied, see Table 2.

Table 5: Different types of pressurized membrane filtration techniques.

Type of technique	Pore size (nm)	Pressure range (bar)
Microfiltration (MF)	> 100	0.1 - 3
Ultrafiltration (UF)	> 1	2 - 10
Reversed Osmosis (RO)	No pores	10 - 100

Source: Lebuf V. et al., (2013)

The product that is retained on the membrane is called concentrate. In a MF-concentrate suspended solids are retained, while UF can also retain macromolecules. There is a range of techniques available that can be used as a pre-treament for reversed osmosis in order to prevent clogging/fouling of the RO-membrane, including both MF and UF.

In the case of Ampower, the largest anaerobic digestion plant in Flanders, the digestate is first sent through a centrifuge to separate the solids from the liquid fraction. Polymers are added to induce coagulation and flocculation and increase separation. The solid fraction contains about 90 % to 95 % of the total input P and is dried with waste heat from the Combined Heat and Power (CHP) engine. The dried digestate is exported as a P-fertilizer, mainly to France. The liquid fraction is sent to a reversed osmosis (RO) installation. The permeate of RO is the ‘purified’ stream. The concentrate contains most of the input stream nutrients.

The biggest problem reported in membrane filtration is clogging and fouling of the membrane, which increases the hydraulic resistance. Waeger et al. (2010) investigated that pore is strongly correlated to particle size distribution which indicates the importance of a sufficient separation step before the RO. The efficiency of RO-membranes can decrease because (1) low-soluble salts can precipitate on the membrane surface (scaling), (2) suspended solids can adsorb to the membrane surface (fouling) or (3) bacteria can colonize the membrane (biofouling) (Lebuf et al., 2013). Ampower reduces blocking of the membrane pores by continuously dosing acid solutions to the RO-system, which is the most efficient way to reduce scaling and fouling. Regardless, after a certain amount of time the membranes need replacement. The system is represented in **Figure 12**.

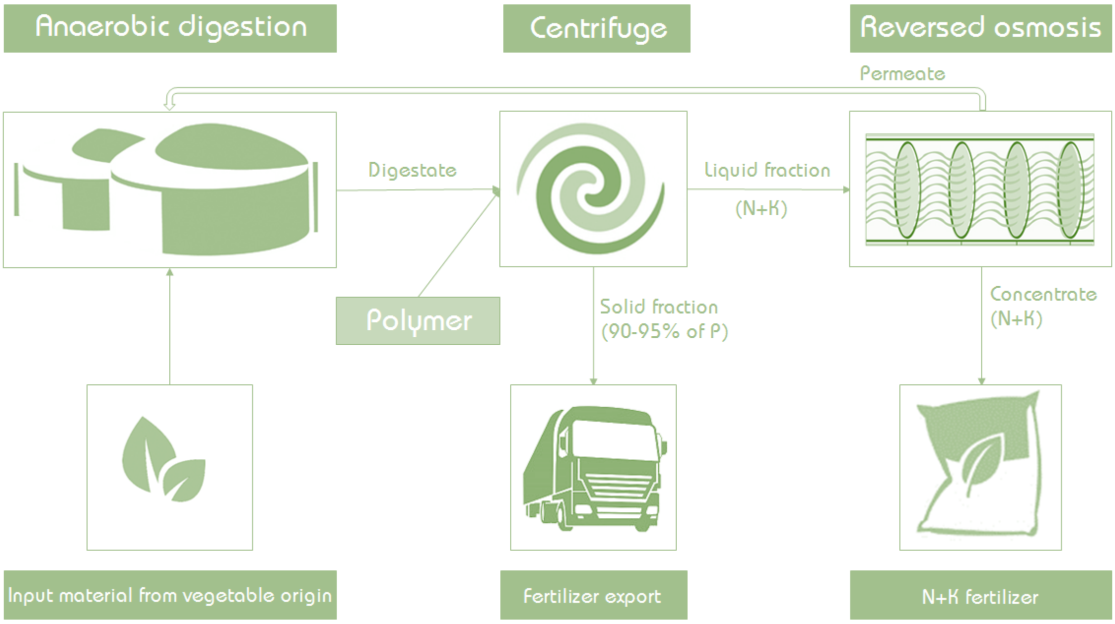

Figure 12: Schematic overview of the reversed osmosis process applied to digestate of vegetable origin.

Source: Biogas-E

3.8.2 End-product

Both permeate and concentrate can be considered as end-products of reversed osmosis, although they have different qualities. The **permeate** is quite clear and consists mainly of water and small ions. It can be discharged, if necessary after a 'polishing' step, or used as process water. Ampower uses the permeate (about 50 % of the input stream to the RO) to mix with the dry input streams for anaerobic digestion so that a desirable (lower) dry matter content is achieved for the 'wet' anaerobic digestion process. The **concentrate** is rich in nitrogen (N) and potassium (K) and is used as a fertilizer in agriculture on local scale. The system is shown in **Figure 13**.

Figure 13: Reverse osmosis installation at Ampower (©Biogas-e).

Source: Biogas-E

3.8.3 Stage of development

The RO technique works very well and is operated at a large scale installation in Flanders. However, the costs for chemicals are still quite high. Further investigations to reduce the need for chemicals are necessary.

3.8.4 Useful contact(s) for more information

Ampower bvba (Belgium)

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3.9 Evaporative-concentration: Process K-Révert, biogas plant "Pont Langlois" in Normandy (France)

3.9.1 General description

The biogas plant "SCEA Pont Langlois" is located in Condé sur Vire in Normandy. The A.D plant is at farm, producing biogas from 10000 t/year substrates, since 2013. Biogas is used in a 265 kWe CHP. Electricity produced is injected into the grid. Heat supplies the AD process, the farm warming and the digestate treatment. Heat avaible for digestate treatment is about 165 kW th. This plant produces 9600 t of raw digestate per year. Landspreading in this sector is limited; therefore a large part of digestate has to be exported at 30km in the neighbourhood of Caen. In order to make this A.D project possible, the digestate has to be treated in order to reduce the volume and therefore the transport costs. Moreover the exported digestate had to be solid to be accepted by the farmers around Caen. That's why digestate treatment was compulsory to allow the project feasibility.

The objectives of this digestate treatment were:

- Reducing the volume of digestate as far as possible by removing water of the digestate
- The treatment must keep all the fertilizing element in the by products
- Use less chemicals additives as possible
- Be automatized as much as possible
- Get a solid by product at the end of the the treatment
- Re-use the thermal energy of cogeneration

Digestate treatment consists of firstly separate liquid and suspended matter with a screw press. Solid fraction, which contains a large part of phosphorus, is stored. The liquid fraction, rich in ammonia and potassium, goes in a vacuum evaporator, ammonia is carried away by the distillate water. The residue of the vacuum evaporation is mixed with the solid of the screw press. The distillate water is post treated with a reverse osmosis after acidification to concentrate the NH_4^+ in a solution, although the purified liquid is send to the natural environment respecting the European law of February 1998. The treatment plant is shown in **Figure 14**.

Figure 14: Treatment plant of digestate in the biogas plant “Pont Langlois”.

Source: K-Revert

3.9.2 Unit operations

Screw press separation

The first step is a mechanical separation of the liquid and the suspended matter contained in digestate with a screw press. The screw press is eco-friendly, its electrical consumption is very low. Most important, no chemical additives are required. Production of liquid fraction is about 25 m^3/d. The solid part has a DM content of about 23 %.

Vacuum evaporation

Liquid digestate is sucked into the vacuum evaporator. The liquid is heated to 38°C thanks to the hot water of cogeneration. The water contained in the liquid phase turns into vapor carrying all the ammonia with it. The low temperature of evaporation is due to the high level of vacuum in the evaporator. The residue after evaporation is spread on the solid of the screw press, the final product (7.1 t/d) is handled as a solid (DM of 20 %) and exported at 30 km.

Acidification and reverse osmosis

The water charged in ammonia is acidified by a dosing pump with sulphuric acid in order to turn NH_3 in NH_4^+. The water is post treated by reverse osmosis producing 2 t/d solution of fertilizer (ammonium sulfate) which is used by the owner of the plant. The clean water produced is sent to the river (20 t/d). The process scheme of the treatment is shown in **Figure 15**.

3.9.3 Process scheme

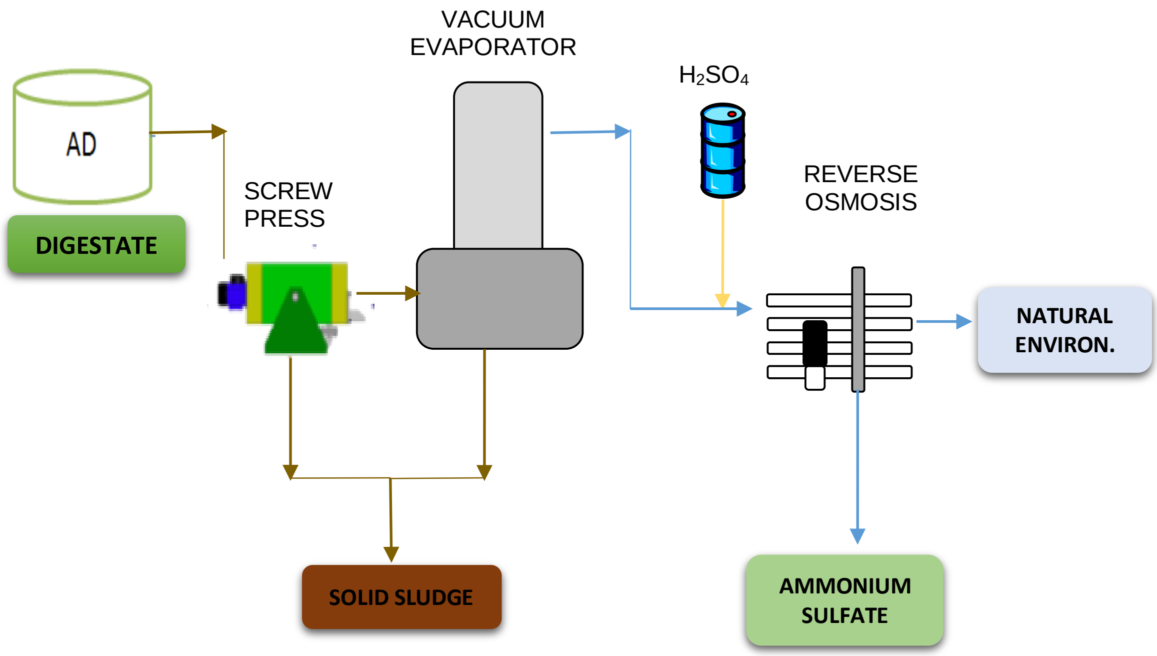

Figure 15: Process overview of the digestate treatment by K-RéVERT at SCEA Pont Langlois.

Source: K-Revert

3.9.4 Additional comments

The general catch rate of NH_3 in the raw digestate is about 98 %. The process allows the total recovery of fertilizing elements. The global electrical consumption is about 0.025 kWh/m^3 of digestate.

3.9.5 Useful contact(s) for more information

K-Revert (France)

Email address: Julien.brochier@k-revert.fr

3.10 Ammonia stripping and scrubbing : Plant Biogasyl, Les Herbiers (France)

3.10.1 General description

The biogas plant “BIOGASYL” is located in Les Herbiers, in the North West of France. It is a centralised plant which has been producing biogas from 25000 t/year substrates since 2008.

The composition of the mix of substrates is as follows:

- 57 % waste from food industry
- 29 % catering and retail waste
- 14 % slurry

Biogas is used in a 615 kWe CHP. Electricity produced is injected into the grid. Heat supplies the slaughterhouse located next door and the digestate processing.

This plant produces 23000 t of raw digestate per year. Land use possibilities in this sector are constrained; therefore a large part of digestate is being treated since 2009, with a process installed by *Europe Environnement*. The plant is shown in **Figure 16**.

Digestate is processed to produce on the one hand organic and mineral fertilizer that can be easily exported and sold and on the other hand waste sent to a waste water treatment plant. Digestate processing consists of firstly separate liquid and suspended matter with a centrifuge separator. Solid fraction, which contains a large part of phosphorus, is composted to satisfy the French standards NF U 44051. A part of the liquid fraction, rich in ammonia, goes in a stripping column. Evaporating ammonia is carried away by the air flow. The vicious air is then directed to a wash column to concentrate the NH_3 in a solution, although the purified liquid is directed to the WWTP. The process scheme is shown in **Figure 17**.

Figure 16: Picture of the plant "Biogasyl".

Source: AILE

3.10.2 Unit operations

Centrifugation

The first step is a mechanical separation of the liquid and the suspended matter with a centrifuge. The plant is equipped with a polymer dosing unit to optimize the performance of the centrifugation. 0.16 m^3 of polymer is added per m^3 of digestate. Production of liquid fraction is about 3.5 m^3/h with an amount of NH_4^+ above 3 g/L. The solid part has a DM content of about 25 % and is moved to the composting platform.

Ammonia stripping

A basic solution (whitewash) is added to the liquid fraction to promote a change in state from NH_4^+ ion in solution to NH_3 in the gas phase. The liquid to be treated is sprayed above a heat exchange surface composed by a polymer lining. Before treatment, the amount of NH_4^+ is about 3 to 5 g/L, and reduced to 0.2 g/L in the treated effluent.

Air scrubbing

The stripgas which is charged with ammonia is transferred to a wash column. This scrubbing column is connected to the acid dosage unit. A solution of nitric acid is sprayed above an exchange surface. Ammonia is absorbed by the acid solution and forms a solution of ammonium nitrate (NH_4NO_3) which could be ammonium sulphate if the acid used is replaced by sulphuric acid. The catch rate of NH_3 in the air is about 99 %. The ammonium nitrate solution produced contents 150 to 200 g/l of NH_4^+ (15-20 %) although the concentration in the effluent is less than 150-200 mg/l. This effluent is transferred to the WWTP.

3.10.3 Process scheme

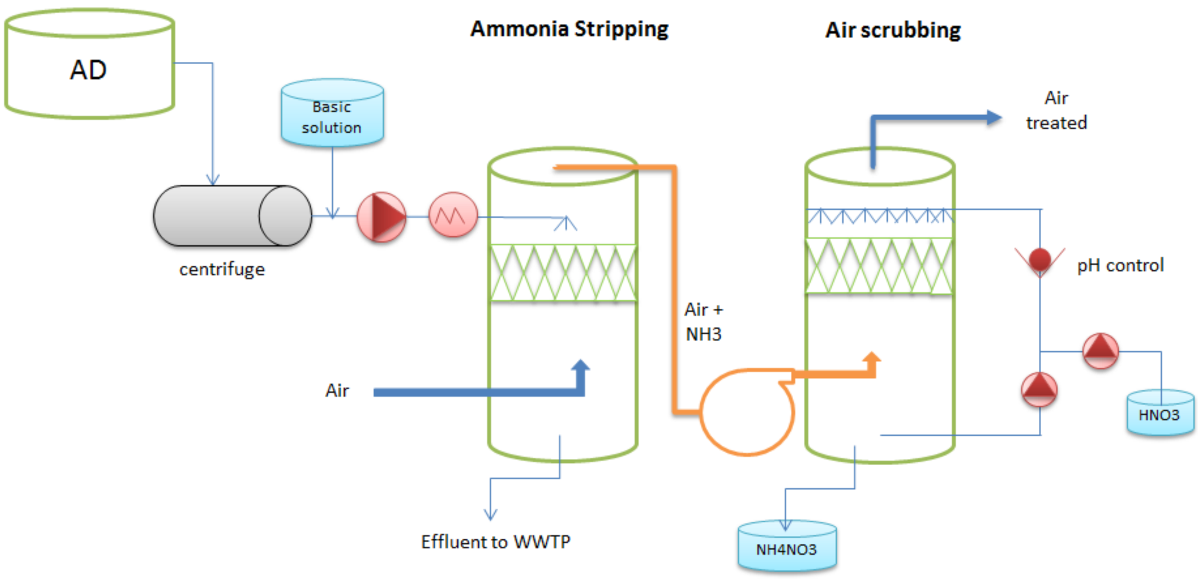

Figure 17: Process overview of the digestate treatment at Biogasyl (adapted from *Europe Environnement*).

Source: modified *from Debuchy C., (2011)*

3.10.4 Additional comments

The total abatement rate of ammonia is about 96 % depending on the N entrant concentration and optimisation of the unit operations.

3.10.5 Useful contact(s) for more information

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Bionerval (France)

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3.11 Ammonia stripping and scrubbing (Waterleau pilot installation)

Through a combination of stripping and scrubbing ammonia can be recovered from the liquid fraction of digestate which typically contains between 2 and 5 g/L NH_4-N. A pilot set-up for this process is being tested at the Waterleau NewEnergy plant in Ieper, Belgium. The company has already gained experience with classical ammonia stripping systems. The goal of the pilot is to find the most suitable system for digestate treatment and fertilizer recuperation, and to determine the optimal process parameters.

3.11.1 Description of the technique

In a first step ammonia is removed (stripped) out of the liquid fraction of the digestate by blowing air through the liquid stream in a tray stripper. In a second step the stripping gas charged with ammonia is put into contact with an aqueous sulphuric acid solution in a packed scrubber, resulting in ammonium sulphate. For the time being there have been no tests conducted with nitric acid solutions because of potential safety issues associated to the mixture. The pilot ran in a semi-continuous mode in which every hour 66 L of digestate is drained from the stripper unit which is then filled back with fresh influent (digestate). In aqueous solutions NH_3 and NH_4^+ concentrations are in equilibrium. Changes in temperature and pH can shift the equilibrium either to the left or to the right. Optimization of both temperature and pH was subject to a series of tests.

Influence of temperature

To determine the optimal temperature, three types of tests were performed after the start-up of the pilot:

(1) heating the digestate liquid fraction to a temperature of 50 to 60 °C,

(2) no heating of the digestate,

(3) application of maximum attainable temperature of the heat exchanger by increasing hot water flow.

Other parameters were kept constant both in the stripping and scrubbing unit including digestate flow, fan flow (connection between both units) and pH. The results showed that heating the digestate stream up to 50 to 60 °C should be sufficient to efficiently remove ammonia and recover ammonium sulphate in a time frame of three hours. If the stripper unit is not heated, there is low ammonia removal. Application of maximum attainable temperature of the heat exchanger did not show a higher ammonium recovery rate. This is against expectations, but due to local circumstances the temperatures that could be reached were lower than anticipated. The low recovery rate was caused by condensation in the pipeline that connects both units. It is expected that higher temperatures - to about 80°C - are attainable and will increase efficiency in an optimal design set-up. A schematic overview of the system is provided in **Figure 18**. The plant is shown in **Figure 19**.

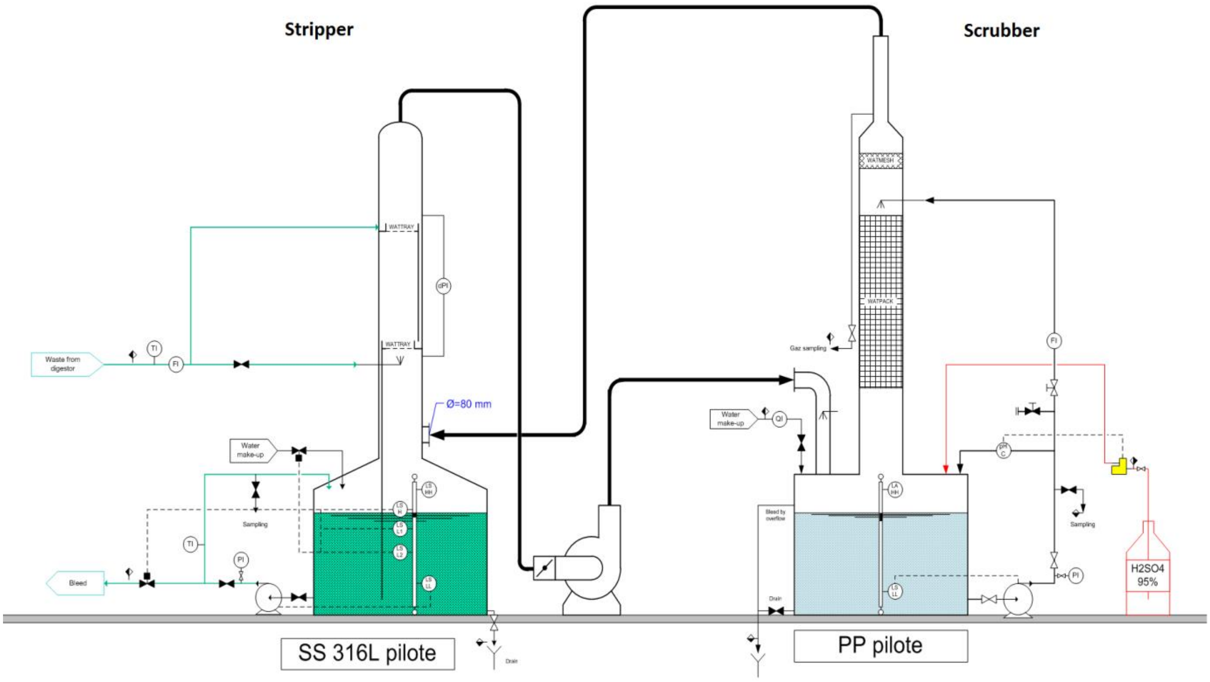

Figure 18: Schematic overview of the stripper-scrubber pilot.

Source: © Waterleau

Influence of pH

In a second testing phase the influence of increased pH was investigated. Two batch tests and one semi-continuous test showed that increased pH has a slight positive effect on stripping efficiency, compared to the first series of testing where temperature influence was tested. Both a higher temperature and pH have a positive effect on stripping efficiency.

3.11.2 End product

In the case of the Waterleau pilot the end product is ammonium sulfate ($(NH_4)_2SO_4$) which can be used as an **inorganic N-S fertilizer**. If the stripped ammonia would be brought into contact with – for instance – a nitric acid solution the end product is ammonium nitrate which can also be used as an inorganic fertilizer.

Figure 19: Schematic overview of the stripper-scrubber pilot.

Source: © Waterleau

3.11.3 Stage of development

The target of the pilot test was to determine optimal set-up and conditions for the stripper/scrubber system for digestate treatment with the highest ammonia removal efficiency and the best separation of useful fertilizer products.

An additional evaluation of temperature increase to about 75 to 80 °C is a potential next step in the pilot testing. At these temperatures nutrient recovery can be combined with hygienization of the fertilizer products. According to Bonmatí and Flotats (2003) it is possible to completely remove all ammonia from the liquid fraction, without any pH modifications, at a temperature of 80°C. This would imply that there is no need for the addition of pH-increasing chemicals to reach maximum stripping efficiency. By increasing the temperature, the solubility of heavier precipitates ($CaPO_4$, $MgPO_4$, etc.) increases accordingly. Some of these precipitation reactions will lead to the formation of useful fertilizer products but these reactions might also contribute to increased fouling of the system. In optimizing process parameters this is also something that needs to be taken into consideration. In a recent set of tests Waterleau used the stripping unit to strip raw influent during a hygienisation step to reduce the amount of nitrogen that goes into the digester unit. Initial results show that pH augmentation is also necessary to reach sufficient stripping.

Future efforts should also focus on reaching a maximum dry matter content of $(NH_4)_2SO_4$ at the scrubber outlet. This has a positive influence on transport costs and answers to a number of practical demands from fertilizer end users. A high dry matter content also induces crystallization of the $(NH_4)_2SO_4$ facilitating the harvesting process. In the current set-up crystallization could not be achieved. The main reason is that the units are developed to work in batches where the size of the scrubber unit is about 4 times the size of the stripper unit having a negative effect on $(NH_4)_2SO_4$-concentrations at the scrubber outlet.

3.11.4 Useful contact(s) for more information

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3.12 Phosphorus precipitation (Aquafin pilot installation)

The precipitation of orthophosphate as struvite (magnesium ammonium phosphate, $MgNH_4PO_4.6H_2O$) is one of the most common P recovery strategies. However, up to now this technique has mainly been applied on water instead of on sludge. At the WWTP of Leuven (120.000 inhabitant equivalent), operated by Aquafin n.v., a full scale phosphorus (struvite) recovery plant, developed and patented by © NuReSys, was used on digested sludge for the first time (Marchi et al., 2015). The plant is shown in **Figure 20**.

Figure 20: Picture of the pilot plant of struvite production designed by Aquafin.

Source: © Aquafin

3.12.1 Description of the technique

The digested sludge first passes a cutter. As shown in **Figure 21**, the precipitation process itself consists of a CO_2 stripper tank to increase the pH and a crystallization reactor in which $MgCl_2$ is dosed.

Subsequently, the sludge moves into the harvester which allows a partial separation of the crystals from the sludge by means of a cyclone. The retained crystals can either be harvested or recirculated to the reactor. After one year of operation an efficiency of 80 % of orthophosphate removal in the digested sludge between inlet and outlet of the struvite reactor was achieved. The process allows a maximum recovery potential of 15 % of the total phosphorus load of the plant (Marchi et al., 2015).

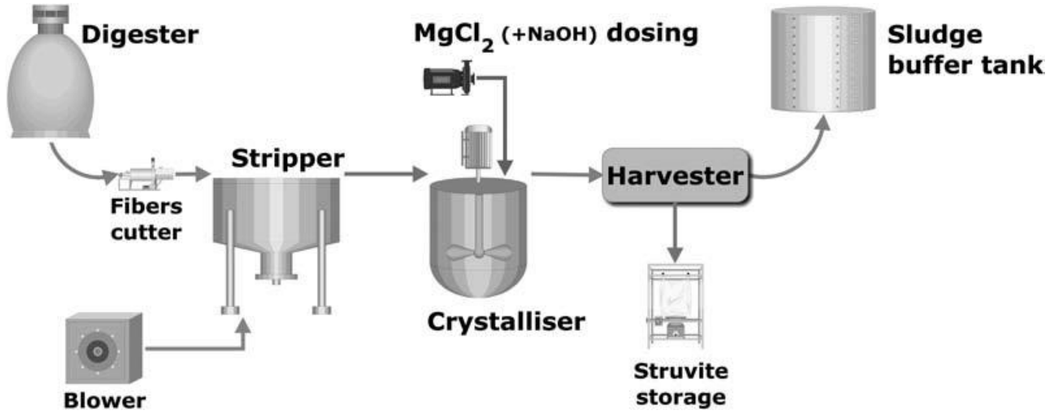

Figure 21: Schematic overview of the struvite precipitation process applied on digested sludge from the Aquafin WWTP.

Source: © Aquafin, Geerts S. et al., (2015)

Compared to an implementation on water phases, following operational benefits – specific to implementation on digested sludge – were identified within the first year of full-scale operation by Marchi et al. (2015):

(1) an average enhanced sludge dewater-ability of 1.5 % yielding energy savings when the sludge gets transported, mono-incinerated or dried afterwards (due to the diminished quantity of water to transport or evaporate);

(2) a reduced phosphorus content in the dried sludge pellets of about 5-10 % in case of subsequent drying. In Belgium the dried sludge can be valorised by the cement industry. A lower P-content has a positive influence on the hardening properties of the cement and would be considered as an improvement (Husillos Rodriguez et al., 2013).

Other operational benefits include:

(3) a reduced scaling: natural struvite precipitation in digested sludge lines are known to cause operational problems like pipe clogging and valve freezing, requiring regular and time-consuming pipe maintenance (Munch and Barr, 2001). The struvite process operation strongly decreases the orthophosphate concentration (and to a small amount the ammonium concentration) and thus reduces the speed of scaling downstream the process. The scaling reduction was not quantifiable during the first year of full-scale operation;

(4) a reduction of the P and N loads in the rejection waters from the dewatering that gets recycled to the water line. This allows for a decrease in aeration (needed for nitrification) and carbon source consumption (sometimes needed for the Bio-P removal and/or for denitrification). P in the recycled water was halved when applying struvite recovery from digested sludge. A reduction is also obtained with the implementation of struvite recuperation on rejection water from dewatering and not only when implemented on digested sludge.

3.12.2 End-product

The obtained end product is struvite ($MgNH_4PO_4.6H_2O$), a slow release P-fertilizer. By analyzing the elements N, H, Mg and P the ratio of $Mg:NH_4:PO_4$ could be calculated and compared with the theoretical ratio of struvite. It is clear that the produced struvite has a high purity, as the measured values approach the theoretical values (Marchi et al., 2015). The appearance of struvite crystals is shown in **Figure 22**.

Figure 22: Struvite crystals obtained through the process of Aquafin.

Source: © Aquafin

3.12.3 Stage of development

The main obstacle of the technique implementation on sludge is the difficult separation of the crystals from the sludge. Optimization research of the process should be further explored to increase the crystal size and thus improve the recovery rate. To date, the harvester at the Leuven WWTP could effectively recover around 25 % of the precipitated struvite. The reduction of the struvite scaling speed downstream the process is desirable, but the beneficial impact on pumps and dewatering devices should be evaluated on a longer term (Marchi et al., 2015).

Whether it is safe to use in agriculture should be evaluated by means of plant assays and phytotoxicity tests over a long period of time. Furthermore, this full-scale experience should contribute to determine the best way to recover P from municipal wastewater: from centrate, digestate or at the end of sludge life from incinerated sludge ashes. This is currently under investigation within the wastewater sector and needs further discussion (Marchi et al., 2015).

3.12.4 Useful contact(s) for more information

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4 List of useful contacts

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