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WOW!

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SUBJECT: WPT1 Designing value chains for carbon based elements from wastewater

A1 STATE OF THE ART REPORT

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Content

1   Introduction

Wastewater contains valuable substances that could be used as raw materials for biobased products. However, in North-West Europe (NWE) this potential is hardly exploited yet. This results in loss of valuable materials and increased CO_2-emmissions and use of natural resources.

There are market opportunities for raw materials from wastewater, but for this, supply (wastewater treatment plants (WWTPs)) and demand (from industry) need alignment. This calls for a transition: at WWTPs a shift is needed from treating wastewater to producing valuable materials. Market parties need to regard wastewater as a valuable source instead of 'dirty unsafe water' and policies should better fit this new circular practice.

To capitalise on these opportunities and work towards a circular approach on wastewater, WOW! will:

- Show the economic potential: by identifying the most promising value chains from wastewater,
- Demonstrate the technical feasibility: in 3 pilots recovery and upcycling techniques for cellulose, lipids and polyhydroxyalkanoates (PHA) will be optimised, while tailoring the product specifications to market needs,
- Lower policy barriers: policy barriers for market uptake of recovered materials will be identified and action plans developed.

This State of the Art Report is the deliverable 1.1 of the work package (WP) T1 Activity 1. This WP focusses on the market potential of carbon based elements (CBEs) from wastewater. The objective is to identify viable value chains for 5 carbon based products from wastewater: PHA-bioplastic, biodiesel, bio-oil, biochar, and acetic acid.

This report contains analyses on the state of the art of current supply and market uptake of materials from wastewater in NWE. Analyses comprise an overview on past (best practices) and current attempts to reuse CBEs.

First, data for typical wastewater characteristics in the different countries are summarised in NWE. As a basis for later calculations, the population connected to the WWTPs in each country was then determined.

Then the very limited existing value chains on other marketable wastewater-products were analysed. These include power (biogas), heat (both on the WWTP and in the sewer system), phosphorus, nitrogen, extrapolymeric substances and cellulose.

Afterwards the target substances in WOW! were discussed in more detail. The production from non-wastewater resources is described as well as the state of research for the recovery of the substances from wastewater.

This report concludes with an initial estimation of which quantities of the five CBEs can be produced in the future at WWTPs in NWE. In the following market potential study, these figures will be investigated in detail and will be compared with the demand for PHA, biodiesel, bio-oil, biochar and acetic acid from the North-West European Market.

2   Wastewater characteristics

2.1   Wastewater Composition

The wastewater composition in the different North-West European (NWE) countries varies due to differences in user consumption, portion of industrial loads and kind of sewer system. Table 2-1 shows the specific load design values in the related countries. In Germany and Luxembourg the design value is based on an 85 %-percentile value. In the Netherlands the mean values are multiplied by the factor 1.25 to get peak loads for dimensioning the biological reactor.

Table 2-1:   Design values for specific load in inflow, North-West Europe

Parameter	\ Country Unit\	DE	BE	FR	FR	LU	NL	NL	UK	NWE
definition design load	-	85%			Mean	85%	Mean	125%	Mean	
specific load COD	g/(PE·d)	120	123	120	110	120	106	133	120	**121**
specific load BOD	g/(PE·d)	60*	43		50				60	
specific load TS	g/(PE·d)	70	70	60	55	70	43	54	65	**63**
specific load N	g/(PE·d)	11	13.2	15	11	11	9.5	12	10	**12**
specific load NH4	g/(PE·d)								8	
specific load P	g/(PE·d)	1.8	2	4	1.5	1.8	1.5	1.9	2.1	**2**
flow	l/(PE·d)	175	150	120		175-200	125	156	150	**150**

* used until 2015

2.2   Cellulose

For the wastewater constituents cellulose and lipids which are in focus of the WOW! project only little data are available. These components contribute to the parameter COD, however there are no standard operational analytical methods, thus they and are not regularly measured at wastewater treatment plants. Therefore, only a few theoretical estimates can be given.

Cellulose in wastewater mainly originates from the use of toilet paper. Therefore, the screening material is rich in fibrous cellulose material. These fibres are part of the hardly biodegradable COD-fraction and represent a corresponding share of the COD load in wastewater. Van Loosdrecht (2017) and Ruiken et al. (2013) report a toilet paper usage of 10 – 14 kg/(P·a). There may be differences in the usage of toilet paper from country to country. According to Kirstgen (2016) for Germany the usage is 15 kg/(P·a). Only half as much is used in Spain, twice as much in Sweden. The average for the EU-26 is 13 kg/(P·a) (Kirstgen, 2016). Kirstgen (2016) assumes that 89 % of toilet paper consists of cellulose. This leads to a specific cellulose load in wastewater of approximately 31,7 g/(P·d).

With a COD of 1.19 g O_2/g cellulose (Kirstgen, 2016), a specific COD load of 37.7 g/(PE·d) from toilet paper results. Taking the mean value of 121 g COD/(PE·d) for NWE from Table 2-1 into account, about 30 % of the inflow COD of wastewater are built by cellulose. This is according to Ruiken et al. (2013), who report that 25 – 30 % of the COD in the inflow of the wastewater treatment plant is based on the cellulose fibres from the toilet paper.

2.3 Lipids

Lipids are a collective term for completely or at least largely water-insoluble (hydrophobic) natural substances. They can be divided into seven groups: Fatty acids, triglycerides (fats and fatty oils), waxes, phospholipids, sphingolipids, lipopolysaccharides and isoprenoids. The main source of lipids in domestic wastewater are faeces and food remains (Lebek, 2003). There are a number of publications on the concentration of fats and lipids in wastewater, which come to different conclusions. On the one hand, this may be due to the examination method and the solvent used. On the other hand, industrial discharges may have had an influence. In addition, the investigations took place in different countries, so that different eating habits may have influenced the results.

According to Koppe and Stozek (1986), a specific fat load of 2 to 6 g/(PE·d) can be assumed. The investigations by Quemeneur and Marty (1994) took place at three wastewater treatment plants in France (ether extraction and gas chromatography). They indicate a load of long chain fatty acids (LCFA) of 4 to 8 g/(PE·d). They also made a differentiation of the individual fatty acids: the most common long chain fatty acids are oleic acid (32 % of total LCFAs), palmitic acid (30 %) and stearic acid (15-20 %) (Quemeneur and Marty, 1994). The composition of the fatty acids collected in various phases of urban wastewater treatment is also shown later in (Figure 4-2). The largest proportion of lipids in wastewater consists of triglycerides and only a small proportion of free fatty acids (Paris, 2004).

Raunkjær et al. (1994) (CCl_4 as extracting agent and infrared spectroscopy) obtained significantly higher results in investigations at four sewage treatment plants in Denmark. Fat, oil and grease (FOG) therefore represents about 30 % of COD in wastewater influent of a municipal wastewater treatment plant which corresponds to a value of approx. 36 g COD/(PE·d) at 120 g COD/(PE·d). With a conversion factor of 2.03 g COD/ g FOG, the authors calculate a specific population load of 13 to 16 g FOG/(PE·d). Tanaka et al. (1991), on the contrary, indicate only a lipid content of about 19 % of COD, i.e. about 11 g FOG/(PE·d). FOG is partly retained in the grease trap of the wastewater treatment plant, but also reach the preliminary clarification and the activated sludge tank, which is why they are also found in primary and excess sludge.

2.4 Recovery potential from wastewater in NWE

In order to calculate the potential of cellulose and lipids that can be theoretically recycled from wastewater the number of population connected to the urban wastewater collection system (Table 2-2) and of population equivalents to the wastewater treatment plants (Table 2-3), resp. in North-West Europe are used. In this consideration, Ireland and Switzerland are neglected as they are not involved in the project and only an estimation of the population equivalents is available.

Table 2-2: Population connected to the urban wastewater collecting system in NWE (Eurostat, 2018)

Country	Population [P]	Year of data
Belgium	10,180,000	2013
Germany	77,464,000	2013
France	54,139,000	2014
Luxembourg	563,000	2015 (estimated)
Netherlands	16,799,000	2015
United Kingdom	60,822,000	2010
total	219,967,000	

Table 2-3 lists the total load in the influent (PE) to wastewater treatment plants based on a specific BOD_5-load of 60 g BOD_5/(PE·d). In contrast to Table 2-2, Table 2-3 also includes the industrial share of wastewater pollution which amounts to about 21.9 % of the total connected PE to wastewater treatment plants. For Belgium, the values are not coherent, however, this fact might be caused by the large number of commuters (this counts also for Luxembourg) to neighbour countries in combination with only little industrial loads or by data failures.

Table 2-3: Total generated load (PE) based on 60 g BOD_5/(PE·d) for 2014 (eea.europe, 2019)

Country	Total generated load (PE)	Rate of connection to sewer system [%]
Belgium	9,243,830	99,77
Germany	109,928,131	98,14
France	72,466,152	100,00
Luxembourg	625,031	99,28
Netherlands	18,229,830	100,00
United Kingdom	70,973,675	99,48
Total	281,466,649	

The difference between population connected to sewer system and population equivalents in the WWTP inflow is of high importance as the composition of household and industrial wastewater might differ significantly also concerning load of cellulose or lipids.

Table 2-4 shows statistical data for the amount of disposed sewage sludge in the NWE area (Destatis, 2018; Eurostat, 2018b; Wensink, 2018), that can be used for proving the above mentioned data. For Germany and the Netherlands only the states part of the NWE area are considered. For France no such data is available. Ireland and Switzerland again were neglected.

Table 2-4: Sum of sewage sludge production in NWE (Destatis, 2018; Eurostat, 2018b; Wensink, 2018)

Country	Sewage sludge (Mg DM/a)
Belgium	157,200
Germany	1,340,501
France	961,520
Luxembourg	9,156
Netherlands	299,907
United Kingdom	1,136,700
Total	**3,904,984**

For municipal wastewater conditions a specific sludge production as primary and excess sludge of about 60 to 70 g DM/(PE·d) (DM – dry matter) can be assumed. This is reduced by aerobic or anaerobic stabilisation to about 40 to 50 g/(PE·d) or 15 to 20 kg DM/(PE·a), resp. taking an average value of about 45 g DM/(PE·d), the total sludge production p.a. given in Table 2-4 yields to a connected population equivalent of about 240 mil PE which confirms the total values given by Table 2-2 and Table 2-3.

3 Existing value chains – Best practice

This chapter gives an overview about the existing value chains for carbon based elements (CBE) from wastewater. The Technology Readiness Level (TRL) evaluates the technological maturity during the acquisition process. Figure 3-1 shows the TRL for the existing value chains in the different countries and the status of their implementation in a simplified view. The TRLs are based on a scale from 1-9, with 1 representing basic technology research and 9 the most mature technology in phase of system testing, launching and operation. More detailed information about implemented processes and examples are listed in the appendix.

\ Country Value chain \	DE	FR	NL	UK	BE	LU
Power (biogas)	green	green	green	green	green	green
Heat (WWTP)	green	green	green	green	green	green
Heat (sewer system)	green	green	yellow	green	green	green
Phosphorus	green	yellow	green	green	green	yellow
Nitrogen	green	yellow	yellow	yellow	blue	yellow
Extrapolymers	blue	blue	yellow	blue	blue	blue
Cellulose	blue	blue	green	blue	yellow	blue
PHA	orange	blue	yellow	yellow	blue	blue
Lipid	orange	yellow	blue	blue	blue	orange
Pyrolysis	orange	blue	blue	yellow	blue	orange

blue	no application
orange	lab scale (TRL 1-4)
yellow	pilot scale (TRL 5-7)
green	large scale (TRL 8-9)

Figure 3-1:   Existing value chains in the various countries and status of their implementation

In this concern the extraction of PHA only from wastewater is meant and for pyrolysis only the use of this process for treatment of cellulose is considered. For production of biodiesel from lipids from wastewater already a large scale process exists in the UK. The company *Argent Energy* blends skimmed floating material from primary settlings with other FOG wastes and converses this to biodiesel. However, this approach is quite different from the one under investigation in the WOW! project. Therefore, in Figure 3-1 the TRL-value was set to "no application".

3.1 Power and heat

Wastewater contains energy in different major forms (Meda et al., 2012):

- chemically bound energy
- thermal energy
- potential energy

The energy in wastewater is primarily chemically bound in the organic contamination. One major route to recover chemically bound energy from wastewater is to convert the produced sludge into biogas by anaerobic digestion (AD). In combined heat and power (CHP) plants electrical energy can be generated and the heat can be used for heating up of raw sludge and to cover the heat demand of the digester and operational buildings (Holmgreen et al., 2016). As shown in Figure 3-1, the recovery of power and heat on WWTPs is state of the art in all NWE countries considered. Currently, the focus in energy recovery is turning from energy efficiency to energy neutrality and to having a production that exceeds consumption (Holmgreen et al., 2016).

The kinetic energy content of used water usually is negligibly small (Holmgreen et al., 2016), especially because of only low hydraulic gradient in the existing sewer system. Also the potential energy on wastewater treatment plants is normally not used because of little hydraulic differences to the receiving water and high dynamic flow conditions. Thermally energy is only used in some cases when the local frame conditions (flow, temperature, pipe diameter) allow the use of higher water temperatures by operation of heat pumps (Kroiss and Svardal, 2009).

3.1.1   Power from biogas

Wastewater contains about 120 g COD/(P·d). The fate of this chemically bound energy is illustrated in Figure 3-2, as an example for one specific preliminary clarification time and one specific sludge age. The numbers will vary depending on the constellation. In the primary settling tank, high-energy primary sludge is separated and fed to digestion. In the activated sludge stage, most of the organic material is degraded by the metabolic processes of the microorganisms and only a smaller, inert part remains in the effluent. Approximately 25 % of the organic matter is fed to the digestion by the produced excess sludge. A part of the organic input remains in the digested sludge, about 27 % is transferred into the biogas.
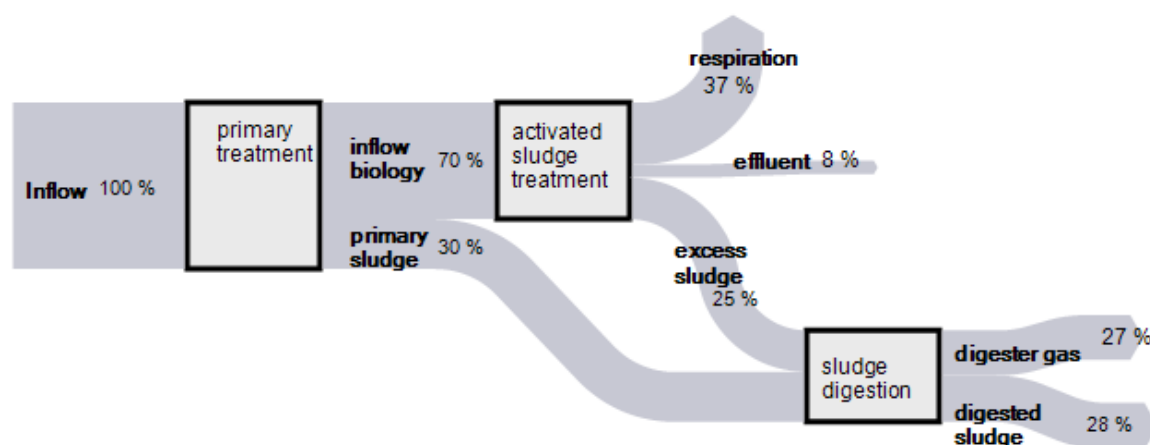
Figure 3-2:   Fate of the chemically bound energy in a wastewater treatment plant as an example (after Svardal, 2014)

Biogas from sewage treatment plants consists predominantly of methane (60-65 %) and carbon dioxide (30-40 %) as well as small concentrations of nitrogen, hydrogen sulphide and other trace gases. Biogas yield and composition depend on a variety of process conditions such as reactor circulation, substrate and process temperature, feeding strategy and substrate composition (DWA-M 363, 2010), which is influenced

by the wastewater composition and the wastewater treatment process. The biogas yield ranges from 3.5 l/(PE·d) for aerobically stabilised sludge up to 25 l/(PE·d) for plants with large pre-treatment and low sludge age (DWA-M 363, 2010).

Biogas production through anaerobic digestion normally converts only the readily biodegradable portion of the organic carbon. There are ways to enhance the degraded fraction by pre-treatment. This so-called disintegration breaks the bacterial cells of the activated sludge, thus releasing the cell contents and making them available for the anaerobic bacteria which produce biogas. Technologies for pre-treatment include thermal pressure hydrolysis, sonication, mechanical disintegration and electrical pulse treatment (Holmgreen et al., 2016). Another possibility to increase the biogas yield is the co-fermentation of easily biodegradable raw materials.

The following possibilities to use biogas are technically mature and have proven themselves in practice (DWA-M 363, 2010):

- Utilisation in gas boilers,
- Utilisation in stationary gas engines, usually as CHP units (combined heat and power unit),
- Utilisation as fuel for the operation of motor vehicles,
- Feeding into a natural gas network (after preparation),
- Delivery of the biogas to third parties for utilisation

Aggregates that have seldom been used for biogas utilisation so far:

- Gas turbines (conventional and micro turbines),
- Fuel cells.

The most common application is the conversion of biogas into electricity by means of CHP units. (DWA-M 363, 2010). The resulting heat is used on-site (see chapter 3.1.2).

Based on a calorific value for methane of 3.49 kWh/(kg COD), the inhabitant-specific energy content of the wastewater is calculated to about 150 kWh/(PE·a). Only about one third of this theoretical value can be used as primary energy via the gas produced in digesters (see Figure 3-2). With modern CHP units, approx. 17 kWh/(PE·a) of electrical energy and approx. 27 kWh/(PE·a) of thermal energy can be generated from the biogas (Hansen and Kolisch, 2017).

The expression energy self-sufficient wastewater treatment plant is used when the annual average amount of energy produced by the wastewater treatment plant is equal to or greater than the amount required. This does not mean that no energy is supplied to the wastewater treatment plant. The first step on the way to an energy self-sufficient wastewater treatment plant is the optimisation of energy consumption. Further requirements are a high amount of biogas with a high methane content and a high electrical efficiency of the CHP plant (Lindtner, 2011). However, only a few WWTPs are known to be able to cover their energy requirements in the annual average solely with the help of the energy contained in the wastewater due to favorable boundary conditions.

3.1.2 Heat at WWTP

Heat is mostly generated at the wastewater treatment plant through the operation of CHPs. Some plants use boilers for heat production. Another way to generate heat is to operate a sludge incineration plant (SIP). In addition, wastewater contains energy in form of heat which can be utilised via heat pumps.

The most common method for using the energy content of wastewater typical for wastewater treatment plants above a certain size is anaerobic digestion of the sludge with production of biogas and subsequent conversion of the biogas into electricity in CHPs. The heat generated in CHPs can be used for heating the raw sludge and the digester as well as for other heating purposes (see also the fact sheets in chapter 8.4.2 and 8.4.3). The degree of self-sufficiency of a sewage treatment plant in terms of heat depends, for example, on the plant size (as mostly only larger plants operate an anaerobic digestion of the sludge), the wastewater composition and COD load and the insulation standard. The heat generated is usually sufficient to cover most of the heat demand throughout the year. Only at low outside temperatures it might be necessary to cover the heat demand with a boiler for a short period of time.

If a sludge incineration plant is operated at the wastewater treatment plant, it is possible to supply external consumers via a local heating network (see section KA Buchenhofen, chapter 8.4.3). However, considerations must be made in this context in order to make appropriate use of the high excess heat generated in summer.

Figure 3-3:   Combined heat and power unit and sludge incineration plant at WWTP Buchenhofen, Germany

Wastewater provides a relatively high temperature level throughout the year. Depending on the location, the wastewater reaches temperatures of 10 to 15 °C (DWA-M 114, 2018) even in winter, when the heating demand is largest. Heat can generally be extracted from wastewater at three different locations. Either directly in the building in which the wastewater accumulates, in the raw wastewater in the sewer system (see chapter 3.1.3) or in the treated wastewater in the effluent of the wastewater treatment plant (DWA-M 114, 2018). Heat extraction from the sewer is limited due to the efficiency of the biological stage at the wastewater treatment plant, esp. nitrification process. However, in many cases the temperature doesn´t drop down significantly because the surrounding soil works also as a heat exchanger. In case of heat extraction after the wastewater treatment plant, a considerably larger temperature difference can be obtained (DWA-M 114, 2018). Currently, there are only a few examples of internal wastewater heat

utilisation wastewater treatment plants. The reason for this is that many wastewater treatment plants with anaerobic sludge treatment have plenty of surplus heat from the use of biogas by CHPs. It may make sense to sell the heat generated in the CHPs at a temperature level of 85 °C to external customers and to supply the digesters efficiently with heat from the wastewater at a low temperature level (DWA-M 114, 2018).

3.1.3 Heat from the sewer system

The advantages of installing heat exchangers (see Figure 3-4) in public sewers are that large quantities of wastewater and thus large amounts of heat can be used. In several cases installation can be carried out in such a way that the transport routes are economical in terms of investment and heat loss (DWA-M 114, 2018). The effects of lowering the temperature of the wastewater on the purification capacity of the downstream wastewater treatment plant must be taken into account. Cooling down by 1 °C reduces the activity of nitrifying bacteria by approx. 10 %, so that the demand for aerobic aeration tank volume increases by 10 % (Kroiss and Svardal, 2009). However, it is assumed that heat extraction is not critical with regard to the cleaning performance of the wastewater treatment plant as long as it does not reduce the wastewater temperature resulting in the inlet to the wastewater treatment plant by more than 0.5 °C (DWA-M 114, 2018).

Figure 3-4:   Heat exchanger elements during installation (Butz and Müller, 2010)

So far, the use of heat from wastewater is not very widespread. For further dissemination, various cities such as Stuttgart or Hagen (DE) have commissioned potential studies to develop the basis for future wastewater heat utilization in the urban area. The core results of the studies are plans in which the general limit criteria for the economic use of wastewater heat are graphically presented. These are, for example, a minimum flow in the sewer, which should not be undercut at night, a minimum sewer diameter and a maximum distance of the object to be supplied from the sewer. Similarly, the required heating load should not be too low for economical operation (Stuttgart, 2019; WBH Hagen, 2019). For this reason, the technology has so far mainly been used for larger units such as residential complexes, office buildings and schools.

An innovative energy concept including a wastewater heat utilisation system was implemented during the construction of an IKEA sales branch (21,000 m² sales area) in Berlin in 2010 (Berlin, 2019). The largest wastewater heat recovery unit in Europe is currently being built in Stuttgart. The approximately 850

apartments, hotels as well as service and commercial businesses in the new NeckarPark district are to be heated and cooled with the energy of the urban wastewater by means of heat exchangers and cascaded heat pumps via a low-temperature local heating network (Baden-Württemberg, 2018).

An example from the UK (Scotland) is the Borders College in Galashields. Catering for over 6,000 students, the campus contains a range of buildings including the modern main building. They installed a heat recovery system to support the entire campus by connecting it to the town's sewer networks. The first installation of its kind in the UK, it now supplies the campus with the majority of it's annual heating demands of around 1.9 GWh/a (Scottishwater, 2019).

3.2 Phosphorus

Phosphorus is a non-renewable resource that is essential as a nutrient for humans, animals and plants. In Europe the recovery of phosphorus has received increasing attention from politics and science in the last years. Among other reasons this is due to the importance of phosphorus for food production and the fact that Europe is highly dependent on imports. This led to the inclusion of phosphorus to the EU-list of the 20 most critical raw materials in 2014 (EUKOM, 2017).

As can be seen in Figure 3-5 around 90 % of the phosphorus load entering a wastewater treatment plant equipped with state of the art technology is bound in sewage sludge. Depending on the technique applied for phosphorus removal the nutrient in the sludge is bound mainly chemically (in the case of chemical phosphorus precipitation by iron or aluminum dosing) or mainly biologically (in the case of enhanced biological phosphorus removal). In the latter case a significant share of the phosphorus is carbonised during the usually subsequently applied anaerobic digestion of the sludge and therefore is transferred into the sludge liquor that results from dewatering of the digested sludge. If chemical phosphorus precipitation is applied the amount of phosphorus that re-dissolves during anaerobic treatment is significantly lower.
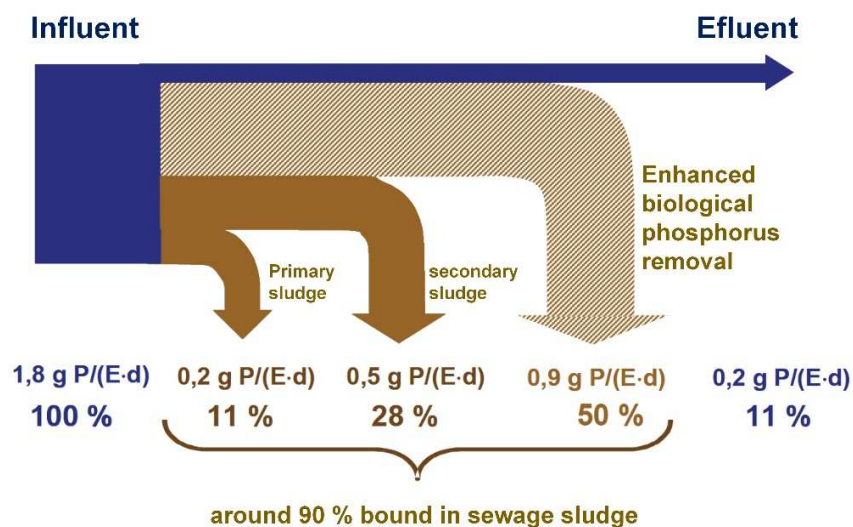
Figure 3-5:   Fate of phosphorus in wastewater treatment plant (Schaum, 2007) modified

In order to use this valuable raw material, the sewage sludge is partly used as fertilizer in agriculture. However, sewage sludge contains besides nutrients also pollutants such as heavy metals and organic trace substances. In Germany for example, therefore, only sewage sludge that complies with the limit values of the Sewage Sludge Ordinance and the Fertiliser Ordinance are allowed be used in agriculture. In 2015, approx. 24 % of German sewage sludge was used in agriculture, 64 % was thermally disposed (DESTATIS, 2016). A paradigm shift is currently taking place in Germany. After a transitional period, sewage treatment plants ≥ 50,000 PE and with ≥ 20 g phosphorus/(kg DM) in the sewage sludge will be obliged to recover phosphorus.

Phosphorus can be recovered from wastewater or process water (e.g. dewatering water) or from the solid residues of the wastewater treatment process (e.g. sewage sludge and sewage sludge ash). Which

technology is suitable depends on the type of phosphorus elimination in the wastewater and the amount of phosphorus which should be recovered.

Until 2018 more than 40 phosphorus recycling processes have been developed which use different wastewater derived substances or process streams as raw material. There are great differences between the processes regarding level of development (lab-, pilot-, full-scale), percentage of recovered phosphorus, type of recycling product and specific costs for the recycled phosphorus. Table 8-1 in the appendix shows an overview of phosphorus recovery facilities in Europe already in operation or under construction (Kabbe and Kraus, 2017).

This state of the art-report for North-West Europe presents the two as of today most mature recycling routes which are already implemented in full scale or are about to be implemented in full scale in the near future

- Struvite crystallisation from wastewater, especially sludge dewatering liquor (several plants are operating)
- Phosphoric acid production from sewage sludge ash (full scale plants are planned but not in operation yet)

The basics of each recycling route are briefly presented and value chain design as well as market supply and uptake are discussed.

3.2.1 Struvite crystallisation from sludge liquors

Recovering phosphorus by struvite (also called magnesium-ammonium-phosphate MAP (NH_4MgPO_4) precipitation mostly uses sludge liquors (or similar liquid process streams) from wastewater treatment plants with enhanced biological phosphorus removal and anaerobic digestion of the sludge. Nevertheless largescale application for struvite production from digested sewage sludge from plants with chemical P-precipitation are running. Therefore additional treatment steps like acidification and complexation of the metal-ions are necessary. The phosphorus from the fluid fraction can be recovered by adding a Magnesium source and rising the pH-value which triggers the reaction of magnesium, phosphorus and nitrogen, present in the liquor, to form solid struvite. The resulting struvite crystals can be removed by solid-liquid separation. Apart from the recovery of phosphorus the technically induced struvite crystallisation has several operational benefits for the wastewater treatment process:

- Improved sludge dewaterability (2 % – 5 % increase in dry solids)
- Reduced clogging of pipes and scale on sludge treatment machinery due to uncontrolled struvite precipitation
- Reduced phosphorus back-contamination of the treatment plant
- Reduced flocculant consumption

In North-West Europe today there are around 25 full scale struvite recovery units operating, which are recovering about 12,000 Mg of Struvite per annum containing 1,500 Mg of phosphorus in municipal and industrial waste water treatment plants (Kabbe and Kraus, 2017a)

There is a good market uptake of the produced struvite since it has a well proven fertilizing effect as a slow release phosphorus-nitrogen fertilizer. It is either directly applied to agricultural land or used for further processing in the mineral fertilizer industry (Knackfuß, 2016). When it comes to the sales price that can be

obtained there is a huge range which is mainly due to regional market conditions and product quality. Issues discussed regarding the latter are organic matter content that is associated with possible odour problems and potential contamination with organic pollutants like e.g. pharmaceuticals.

Struvite recovery from waste water can be economically feasible if apart from the revenues generated by the struvite sales the savings connected to the operational benefits in the waste water treatment process are taken into account (see above). Revenues from struvite sales by itself usually don't cover the necessary investment and operating costs for the phosphorus recovery unit, since the obtainable market price is too low, which is a consequence of the low price level for mineral fertilizers.

A drawback of phosphorus recovery by struvite crystallisation from sewage sludge generated from wastewater treatment plants using chemical phosphorus precipitation - which is the case for many plants in North-West Europe especially Germany – is the high technical effort for P- dissolution and complexation. Using just the sludge water leads to relatively low phosphorus recovery rates ranging from 5 % to 40 % of the phosphorus load of the influent.

There are several technology providers for struvite crystallisation processes. As an example the PEARL™ process for struvite recovery in municipal waste water treatment plants implementing enhanced biological phosphorus removal can be named (see chapter 8.1). Another prominent example for a struvite process for waste water of industrial origin is the REPHOS™ process that recovers struvite of high purity from industrial waste water like produced e.g. in the dairy industry.

Another example for recovery as MAP is Airprex-process applied in Berlin-Wassmannsdorf (see appendix and chapter 8.4.4).

3.2.2 Phosphoric acid production from sewage sludge ash

Conventional phosphoric acid is produced by sulphuric acid digestion of phosphor rock. The mineral acid is an important basic chemical that serves as precursor for a wide range of products. Important applications are e.g. mineral fertilizer production and industrial applications like metal treatment or production of flame retardants.

The conventional phosphoric acid production from phosphor rock causes significant environmental impacts because mining of the phosphate ore leads to high land-, water- and energy-consumption. Further, the resulting phospho-gypsum, the pollutant containing by-product of the production process, has to be landfilled in most cases. Therefore, phosphoric acid plants often are located in countries with low environmental standards. In North-West Europe only one phosphoric acid production facility exists that processes rock phosphate of extraordinary purity and that additionally implements a special process step for phosphor-gypsum purification so that the by-product can be used as building material.

Apart from the usually occurring described negative impact related to the production process of phosphoric acid the application of mineral phosphate fertilizer can lead to pollution of agricultural land as many rock phosphates contain significant amounts of contaminants like e.g. cadmium and uranium which to a large extent are transferred into the produced acid and the mineral fertilizer produced from it respectively.

Due to the problems related with conventional phosphoric acid production on the one hand and the various possible applications of phosphoric acid and the large world market on the other hand phosphoric

acid production from sewage sludge ash is a promising approach. In the past the implementation of several recycling processes, which were tested at lab- or pilot scale, stalled because of low economic effectivity, which often was due to uncertainty regarding market uptake and / or obtainable price of the resulting recycling product. Furthermore mono-incineration of sewage sludge is a prerequisite.

Regarding the potential supply of ash-based recycling phosphoric acid a good database exists for Germany. Here as of today, the sewage sludge ashes from mono-incineration plants produced annually contain about 19.000 Mg of phosphorus which would cover more than 12 % of the national demand for mineral P-fertilization. Due to the paradigm shift in sludge disposal in Germany (see above) it seems very likely that in future more sludge will be incinerated resulting in case of mono-incineration in an increasing potential for phosphoric acid produced from sewage sludge ash.

Currently, different sewage sludge ash based recycling processes producing phosphoric acid have been developed, e.g.:

- REMONDIS TetraPhos™-process (REMONDIS Aqua)
- EcoPhos-phosphoric acid-process (EcoPhos)
- Phos4Life™-process (ZAR & Técnicas Reunidas)

The progress of implementation differs between the processes. For the TetraPhos™-process (see chapter 8.2) full scale implementation is currently prepared at the waste water treatment plant Hamburg although so far all experiences are based on a half-technical scale pilot plant. The EcoPhos-process and the Phos4Life™-process are tested at pre-industrial scale at present.

It is difficult to assess the competitiveness of phosphoric acid produced from ash with conventional phosphoric acid, since only few public information of production cost is available. It seems likely though, that from a purely economic point of view conventional phosphoric acid production has advantages over the recycling-based approach. This is due to economies of scale that can be achieved in the large scale phosphoric acid plants and in particular because the substantial costs caused by the massive environmental damage associated with phosphate rock mining and acid-production are not considered in the price formation process of the phosphoric acid manufacturer. If these costs, that today are borne by society, would be internalized it seems very likely that the value chain based on recycling would be competitive or even advantageous. Thus, to provide a level playing field for ash- and rock phosphate-based phosphoric acid production, similar environmental regulations should apply. Today there are only very few phosphoric acid plants in Europe. Most of them are operating in countries where rock phosphate is mined like e. g. China, Morocco or Tunesia. As of today it seems unlikely that environmental regulations will change in these countries in the near future.

3.3 Nitrogen

So far, the recovery of phosphorus has been the focus of interest in nutrient recovery from wastewater and sludge, as its global fossil reserves are limited. However, the production of nitrogen fertilizers is energy-intensive. The production of nitrogen fertilizer from atmospheric nitrogen requires approx. 11 kWh/(kg N) of primary energy (Zessner, 1999).

The removal of nitrogen from wastewater usually takes place biologically in wastewater treatment plants through the processes of nitrification and denitrification. The nitrogen is on one hand converted into elementary nitrogen and passes into the atmosphere. On the other hand, organic nitrogen compounds are bound in the biomass and thus fed to the sludge treatment. During digestion, up to 50 % of the organic nitrogen is dissolved as ammonium (Siegrist, 1996). This results in an internal nitrogen return load of about 1.5 g N/(PE·d) or 15 to 25 % relative to the total nitrogen in the inflow to the biological stage (DWA Working Group AK-1.3, 2004). The process water is therefore suitable for the elimination respectively recovery of nitrogen. Here, biological processes and stripping processes have so far proven themselves on a large scale (Grömping, 2012).

3.3.1 Biological processes

For the treatment of process water, all biological nitrogen conversion processes (such as nitrification/denitrification, nitritation/denitritation and deammonification) can be used (DWA-M 349, 2017). The process of deammonification is becoming increasingly widespread because it offers the greatest savings potential (energy, substrate, sludge disposal) compared to the other biological nitrogen elimination processes (DWA-M 349, 201) and is currently regarded as the most economical digestion water treatment process (Liebi, 2014). Various configurations have been implemented on an industrial scale, e.g. variants of the SBR process (batch operation), processes with suspended or granulated sludge, in biofilm and in a floating bed. Currently, more than 100 plants are in operation worldwide (Lackner et al., 2014). In biological process water treatment, nitrogen is not recovered. However, energy consumption in the main stream for nitrification and the need for carbon for denitrification are reduced.

3.3.2 Chemical/physical processes

One way of recovering nitrogen from the bypass of the process water is by stripping. This means the expulsion of volatile components from liquids by contact with a gas phase. In a first step, ammonium must be converted into the comparatively volatile ammonia. The position of the dissociation equilibrium is determined by the temperature and the pH value, whereby high temperatures and high pH values shift the equilibrium to the side of ammonia. After pre-treatment, pH adjustment and heating, the stripping process usually takes place in packed columns by irrigation of the process water via the filling elements with simultaneous gassing in counter-current flow. When stripping ammonium from the process water, this gas phase can be either air or steam. Nitrogen can be recovered by subsequent condensation or absorption or eliminated by oxidation. The majority of the stripping plants carried out in Germany are plants with downstream acidic washing. They use acids to regenerate the ammonia-laden stripping air in an absorber column by combining the ammonia in the air stream with the acid to form the corresponding salt (Grömping, 2012). By stripping and absorption with sulphuric acid, for example, ammonium sulphate of fertilizer quality can be produced (see fact sheet of WWTP Kloten Opfikon chapter 8.4.5). A disadvantage is the susceptibility of the stripper columns to clogging, which necessitates extensive pre-treatment for particle removal. Due to precipitation, frequent chemical cleaning of the stripper column or heat exchanger is also necessary (Lauterböck et al., 2019).

Membrane contactors are a recent method for carrying out extraction, stripping or absorption processes. Both fluids are separated by a porous hydrophobic membrane (Melin and Rautenbach, 2007). The membrane itself has no special properties such as selectivity for separating the substance mixture, but serves as a pure exchange surface between two phases. The material transport is diffusive. The driving force for mass transfer is a concentration gradient of the transferring component (Melin und Rautenbach, 2007). For the recovery of nitrogen from process water, the wastewater loaded with ammonia flows on one side of the membrane, while an acid flows on the other side, which binds the ammonia after it has passed through the membrane. This eliminates the nitrogen from the process water and, for example, when sulphuric acid is used, it is obtained as ammonium sulphate, which can be used as an approved fertilizer. During a 2-week test operation with a pilot plant at the Münster-Coerde main wastewater treatment plant, it was shown that membrane contactors can eliminate approx. 90 % of the ammonia content. The first two years of operation of a large-scale plant in Münster are to be scientifically accompanied. Among other issues, the operational safety, the efficiency, the economic efficiency, a suitable pre-cleaning as well as the cleaning of the membranes will be investigated. Advantages over stripping and adsorption processes include reduced space requirements and lower energy consumption. A value of 0.2-0.3 kWh/kg $N_{eliminated}$ is expected (LANUV, 2019; FH-Münster, 2019).

Further approaches for the recovery of nitrogen are e.g. ion exchanges (IEX). Ion exchangers are materials that can absorb positive or negative ions from a solution and release other ions with the same charge. The deposition of ions is reversible so that the initial state can be reached again by regeneration (Ender, 2015). To recover nitrogen from process water, it is passed through filters containing the exchange material (e.g. zeolites), which selectively adsorb ammonium. Ion exchangers in connection with process water have so far been tested in experiments and pilot plants, and large-scale industrial applications are also known. The regeneration of the filter material takes place via saline solution or caustic soda solution and requires a complex treatment of the regeneration liquid. Ammonium can be removed from the regeneration chemical by various methods such as MAP precipitation (Liberti et al., 1986) or membrane contactors (Sancho et al., 2017). Regeneration via electrodialysis is described in Morck's dissertation (2011) and tested in a semi-industrial pilot plant. This is an electrochemical membrane separation process for the selective separation of ions from solutions. A concentrated ammonium sulphate solution is produced in the concentrate of the electrodialysis cell. Further technology combinations are conceivable in order to increase the efficiency of the recovery and, for example, to achieve a concentration as pre-treatment of the material flows.

New ion exchange media with higher absorption capacity are being researched within the framework of the EU project SMART-Plant. Currently, a demonstration plant is operated at Cranfield University, which is fed with 10 m^3/d process water. The aim is the removal and recovery of ammonium and phosphorus from process waters based on Ion Exchange as well as the optimization of regeneration cycles while maintaining a high sorption capacity. Two different ion exchange media will be used, capable of removing ammonia and phosphate from the process water. Mesolite is used in the ion exchange process to remove ammonia (smart-plant.eu, 2019). Mesolite is a chemically modified mineral. With 45-55 mg NH_4-N/g it has a higher ammonium exchange capacity than naturally occurring zeolites (Mackinnon et al., 2003). Phosphorus is removed with a hybrid anion exchanger. The adsorbers are regenerated with NaCl or NaOH. Ammonia is recovered with membrane contactors. Here ammonium sulphate is produced. Phosphate recovery from the concentrated regenerate is achieved by precipitation by adding hydrated lime as calcium phosphate (Guida et al., 2019).

3.4 Extrapolymeric Substances (EPS)

A novel raw material can be obtained from the sludge granules formed during the Nereda® sewage treatment process. This is a cleaning technology in which the sludge remaining after cleaning does not form flakes but rapidly settleable granules. A special biopolymer with multifunctional properties can be obtained from this granulate: Kaumera Nereda® Gum (formerly Neoalginate).

The combination of Kaumera Nereda® Gum with another raw material changes its character. Kaumera is an amplifier and connector of features. For example as part of light bioplastics. Kaumera can retain water, but can also repel it. This enables a wide range of applications, for example in agriculture, horticulture and the concrete industry (Kaumera, 2019). One example is a reduction in the leaching of fertilisers in agriculture, which enables crops to better absorb fertilisers. They are more resistant and grow better. Its water-repellent properties also make Kaumera an excellent coating for concrete floors.

The Regional Dutch water authority Rijn en Ijssel is building a factory in Zutphen, which will extract Nereda® Gum from the granules treating wastewater of the Royal Friesland Campina dairies in Borculo and Lochem starting in spring 2019. There are other Nereda® treatment plants in the Netherlands, but they process domestic wastewater and do not yet have an extraction plant like the one in Zutphen. The Regional Dutch water authority Vallei en Veluwe and other partners are working together on a plant that will also make it possible to extract these biopolymers from domestic wastewater (efgf, 2019) (see chapter 8.4.6).

The recovery of these alginate-like biopolymers from wastewater takes place within the National Alginate Development Programme. In this programme the water authorities Vallei en Veluwe and Rijn en Ijssel, the Stichting Toegepast Onderzoek Waterbeheer (STOWA), the Royal Haskoning-DHV engineering consultancy and the Delft University of Technology work closely together.

3.5 Cellulose

Cellulose is the main component of plant cell walls and thus the most common organic compound. It is an important raw material for paper production but also, for example, for the chemical industry. Paper is made from lignin- and cellulose-rich wood. By removing the lignin content, pulp can be produced which is used for the production of higher quality paper and also for toilet paper and tissue paper.

With the toilet paper, considerable amounts of cellulose get into the sewage (see chapter 2.2) and can be found in the wastewater treatment plants. There are currently various efforts to recover this cellulose at the wastewater treatment plant and process it for various purposes. Applying a finescreen or drum filter in the mainstream of a WWTP delivers cellulosic screenings as product. This is a mixture of cellulose, hair, fats, organics and contaminants. This material is also the feedstock for the pyrolysis of WOW!

As part of the BMBF research project E-Klär, a very fine screen and a screenings treatment with optional washing and dewatering were implemented. In the fibre-rich screenings, the usable fibre content was determined for reuse in the paper industry. Fibres with a length of less than 200 µm are of secondary importance for the paper industry. Therefore, the lowest possible fines content is desirable. Particles different from fibres were also found in the samples examined. The results only allowed a very rough assessment of the amount of usable fibres in the screenings. According to this, reuse in the paper industry is theoretically possible. However, it remains to be investigated whether they can be separated under technical conditions in such a way that fibres of sufficient length can be recovered which are not separated

by bonding, spinning, etc. in the sorting of the paper machine (Palmowski and Pinnekamp, 2018). Due to various imponderables and possible hygienic concerns of consumers, the production of activated carbon from screenings was investigated as an alternative recycling method. In laboratory tests, activated carbon was produced from the screenings by means of hydrothermal carbonisation and chemical activation. Subsequently, the adsorption capacity of this activated carbon was investigated in stirring tests with water from the effluent of municipal wastewater treatment plants with regard to SAC_{254} (spectral absorption coefficient at 254 nm). This correlates well with the micropollutant concentration (Anumol et al., 2015, Zietzschmann et al., 2014). As a result, the activated carbon did not achieve the same elimination performance as the reference carbon at the same dosage. With a higher dosage, however, the same elimination can be achieved as with the reference carbon at a lower dosage. There is a need for further research to optimize the parameters in the production of activated carbon. It is expressly pointed out that, in addition to the wet process of hydrothermal carbonisation pyrolysis (dry process) must also be investigated (Palmowski and Pinnekamp, 2018). Furthermore, material balances have to be prepared in order to assess the self-sufficiency of a wastewater treatment plant with regard to the supply of activated carbon from screenings for the elimination of micropollutants (Benstöm et al., 2018).

In another research project (EU project smart plant) the pilot plant is in operation since June 2017: At the WWTP Geestmerambacht in the Netherlands, a dynamic fine screen is to recover up to 400 kg of cellulose fibres per day from the raw wastewater. The cellulose is then separated from the cellulosic screenings and processed at the wastewater treatment plant into a clean, dried and disinfected product that is marketable. The process to achieve the separation is called Cellvation®. On the one hand, this can relieve the burden on the wastewater treatment plant as the cellulose fibres can cause operational problems and have to be disposed of via the sewage sludge. Therefore, removing the fibres at the beginning of the wastewater treatment process leads to energy and cost savings. On the other hand, the products obtained can be used in the construction industry and as fillers for composite materials.

In the Netherlands, cellulose is already recovered on an industrial scale at several wastewater treatment plants (e.g. Aarle-Rixtel, Blaricum, Beemster and Geestmerambacht) (see chapter 8.4.7). Energie- en Grondstoffenfabriek (EFGF), a network organization set up by the water authorities, takes care of the valuable load of wastewater. At present, the cellulose obtained is disposed of as waste, as it only has the legal status of a waste product. The supplier of a product is responsible for its legal status. A working group is working on quality assessment and an assessment framework. The search for possible applications for the recovered cellulose is also underway. It can, for example, be used as a drip inhibitor in asphalt or as a raw material for insulating materials in construction (efgf, 2019). A market application was successfully demonstrated as part of the project "Van Zeefgoed naar Asphalt" (VAZENA; From screening materials to Asphalt) in Jeltsum near Leeuwarden. Here, a bicycle fast track was opened in 2016. This cycle lane already contains cellulose fibres as drip inhibitors. This guarantees the homogeneity of the asphalt mix during production, transport and processing. Cellulose based asphalt is frequently used in the Netherlands.

4 Current attempts to reuse materials from wastewater

4.1 Polyhydroxyalkanoate (PHA)

Polyhydroxyalkanoates (PHA) are naturally occurring water-insoluble and linear biopolyesters that are formed by many bacteria as reserve substances for carbon and energy. These biopolymers are biodegradable and are used in the production of bioplastics.

Until today, supply of PHA from wastewater has not yet been realized in industrial scale and consequently, a market uptake of PHA from wastewater does not occur yet. Nevertheless, a future industrial production from wastewater is promising considering environmental and technical aspects.

The production of PHA in pure cultures has already been discovered in 1920s (Lemoigne, 1926). From the 1970s on, PHA is industrially produced from carbon substrates like sugar, starch or corn (Plackett, 2011). Research on PHA production with low cost substrates in mixed microbial cultures from activated sludge followed from the 2000ers (Serafim et al., 2008). Exclusive usage of mass flows of a wastewater treatment plant with primary sludge as low cost substrate and mixed microbial cultures from activated sludge was studied by Pittmann and Steinmetz (2013 and 2014). An up-scaling process continued to pilot-scale PHA production from wastewater, where the first pilot studies were set up in 2011 (Morgan-Sagastume et al., 2014).

Bioplastics in general are used in an increasing number of markets, for example packaging, catering products, consumer electronics, automotive, agriculture/horticulture and toys, textiles and a number of other segments. Packaging remains the largest field of application for bioplastics with almost 65 % (1.2 mil tonnes) of the total bioplastics market in 2018 (European Bioplastics, 2019).
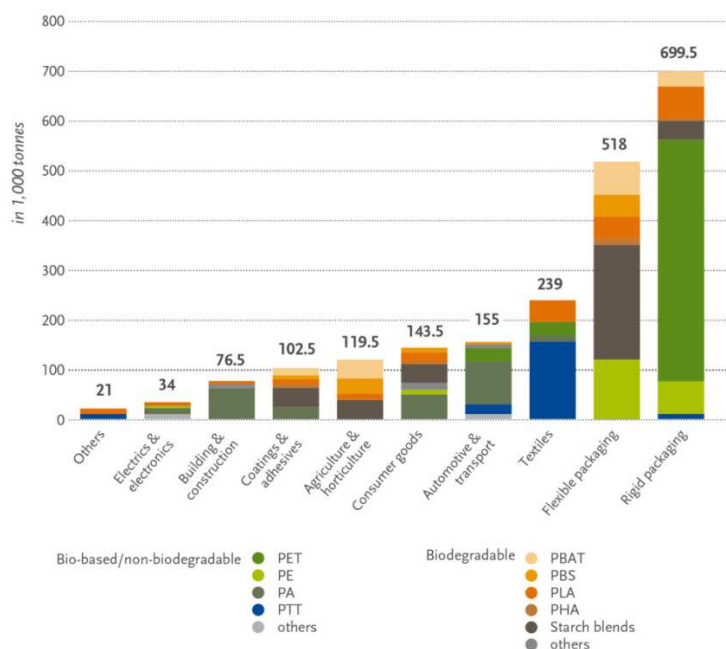

Figure 4-1   Global production capacities of bioplastic 2018 (by market segment) (European Bioplastics and nova-Institute, 2018)

PHAs have comparable material properties to the conventional polymers polypropylene (PP) and polyethylene (PE) (Plackett, 2011). The more rigid versions of PHAs such as polyhydroxy-butyrate-co-valerate (PHBV) have properties close to styrenic polymers (PS or ABS). Additionally, they are biodegradable, which opens new fields of application. PHAs are biodegradable under natural conditions, such as both aerobic and anaerobic and at low as well as high temperature. PHAs are also the only biodegradable polymer that is able to biodegrade rapidly in water environment (Wang et al., 2018).

PHAs are currently used for consumer goods, flexible packaging and rigid packaging (see Figure 4-1). According to Bugnicourt (2014) PHAs can be applied e.g. in packaging or for agricultural foils. Natureplast (2019) completes that due to their good thermomechanical properties PHA is widely used for the production of biodegradable coffee capsules. Other applications are mainly related to their fast biodegradation kinetic. The evolution of regulations ordering quickly compostable products for more and more applications (single use objects, packagings, etc.) requires the use of such raw materials. Many development projects are thus in progress using PHA for this purpose. Another main market for PHA is agriculture/ horticulture items where again good biodegradability properties are needed and a specific end-of-life behavior is sought.

Due to their water-purifying properties, PHA pellets are also used in fish tanks to reduce the phosphorus and nitrogen content. PHA is used as a carbon source by bacteria that also need phosphorus and nitrogen for growth. The bacteria are removed either by being eaten by other tank inhabitants or by protein skimmers. According to Natureplast (2019), this application is currently one of the main markets for PHA. One manufacturer of these biopellets is TL Reefs (TLREEFS, 2019).

From an environmental perspective, the biodegradability of PHAs contributes to a minimization of future plastic pollution at the source (Burgstaller et al., 2018). Moreover, the usage of waste water as raw material for the PHA production process is resource-preserving and cost-efficient. It has neither an essential demand for land nor is it based on fossil fuels.

4.1.1 PHA production from non-wastewater resources

A supply of PHA from wastewater for industrial purposes does not exist yet (see also Figure 3-1). A broader view on the topic is applied to gain information. Consequently, the following sections aims to give an overview of PHA producers from non-wastewater resources and feasibility results for PHA production from wastewater from pilot-scale experiments.

Recently, several companies produce PHAs from starch, sugar and oil (Bugnicourt, 2014). Table 4-1 displays PHA-producers in Europe and their PHA-trade names and raw materials. Apart from Europe PHA production facilities are mainly in the USA (e.g. Yield10 Bioscience, formerly Metabolix Inc. and Danimer Scientific), in China (TianAn) and in Japan (Kaneka Corporation).

Table 4-1:   PHA-producers in Europe, their PHA-trade names and raw materials

Company	Country	Trade name	Raw material	Link
Biomer	Germany	Biomer®	Renewable resources	http://www.biomer.de
Bio-On	Italy	MINERV-PHA™	agricultural co-products or waste	http://www.bio-on.it

The company Bio-On produces PHA from renewable sources in an amount of about 1,000 Mg/a. They are planning to set up its second bioplastic production plant for medical and bioremediation purposes. Bio-on has also set up a new company named Bio-on Plants for the increasing demand of PHAs.

Danimer Scientific is a manufacturer of biopolymers. The company has patented its novel biopolymer Nodax mcl-PHA. It stores PHA polyesters for energy and is biodegradable in air, soil, and water. This property widens its applications in various industries. The company produces a wide range of polymers to support various applications. TianAn Biologic Materials produces PHBV. The company production capacity is 2,000 metric tonnes per year. The company has patented its water-based extraction technology for PHBV production. Kaneka is a Japanese plastic producer of PHA. Kaneka's PHBH bioplastics is made from plant oil and fat, and other ingredients. An investment of 2.5 billion yen (more than 20 mil. €) will be made to their Takasago plant to increase its production to 5,000 Mg per year.

4.1.2 Research on wastewater derived PHA

Pilot-scale studies of Morgan-Sagastume et al. (2014), Bengtsson et al. (2017a) and Bengstsson et al. (2017) and potential estimations for PHA-production from wastewater of Pittmann and Steinmetz (2016) are promising for a future industrial production in North-West Europe (Table 4-2). Pittmann and Steinmetz (2016) estimated an annual production capacity of 1.4 kg PHA/(PE·a) by using primary sludge of municipal WWTP as substrate and Bengstsson et al. (2017) even assessed a yield of 4 – 5 kg PHA/(PE·a). With these values the possible supply of PHA from wastewater in NWE can be calculated (see chapter 5).

For a meaningful comparison between studies it is important to use the same benchmarks, comparable approximation parameters and to report quality and quantity parameters for all production steps. Till now, as Bengstsson et al. (2017) has already remarked, the calculation basis for yields differs in literature. Moreover, the determination of the parameter of biomass content is an approximation. Pittmann and Steinmetz (2016) refer the biomass content to the cell dry weight (CDW), whereas Morgan-Sagastume et al. (2014) refer it to the volatile suspended solids (VSS). This leads to relatively higher values as only part of the cell dry weight are volatile suspended solids. As analytical methods can differ, it is crucial to pay attention to the applied analytical methods to receive a meaningful comparison. The composition of PHA is determined by the type of carbon substrate (Madison and Huisman, 1999). Consequently, both, composition of volatile fatty acids from the first production step and the composition of the PHA should be reported.

Table 4-2:   Location, scale and production parameters of PHA production from wastewater in North-West Europe

Citation	Location	Scale [l]	Operation period	Yield [kg PHA/(PE·a)]	Maximum intracellular PHA content
Pittmann and Steinmetz (2013, 2014)	Stuttgart, Germany	Lab 4 – 15	alternating	1.4	28.6 % CDW[a]
Morgan-Sagastume et al. (2014)	Brussel, Belgium	Pilot 500 – 1000	August 2011 – May 2013	n/a	0.34 gPHA/gVSS[b]
Bengtsson et al. (2017a)	Leeuwarden, Netherlands	Pilot 500 – 800	October 2013 – May 2014	n/a	0.49 gPHA/gVSS[b]
Bengstsson et al. (2017)	Bath, Netherlands	Pilot 500 – 1200	June 2015 – March 2016	4 – 5	0.39 gPHA/gVSS[b]

[a] Cell Dry Weight; [b] Volatile Suspended Solids

As there is not yet an industrial production of PHA from wastewater, currently a market uptake of PHA can only be observed for production processes apart from wastewater. This market uptake already demonstrates that PHA can generally be taken up by the market.

As the demand for bioplastics is increasing, the market is predicted to be continuously growing (European Bioplastics, 2019). At comparable quality and price, a market uptake of PHA produced from wastewater is likely but needs to be investigated and demonstrated.

So far, PHA from waste water is only produced in lab and pilot scale in North-West Europe, without a continuous production. As PHA from other sources than wastewater is provided on the market, it is probable that there will also be a market for PHA from wastewater – as long as the product has comparable material properties to conventionally produced PHA.

4.2 Lipids

Lipids (fat, oil, grease; FOG) often represent an issue already when wastewater is being collected and transported, and thereafter when being treated in wastewater treatment plants (Williams et al., 2012). Applying British statistics (SevernTrent, 2016) to a larger scale, the annual costs related to cleaning of sewers and removing of blockages caused by FOG were calculated to 1 € per PE, meaning over 500 mil € per year are necessary for maintenance of pipelines in the whole EU. Estimating the follow-up costs for the remaining FOG being treated in WWTPs is more difficult. Lipids have detrimental effect on oxygen transfer to microorganisms, resulting in a decline of microbial activity (Henkel, 2010). Additionally, adsorption of lipids to biomass decrease the ability of sludge to settle resulting in bulking and/or foaming. FOG (not-dissolved, particulate (>0.45 µm) and prevailing portion) can easily be separated already at the inlet (sieving/grease trap), mixed with cellulosic wastes, paper, pieces of wood and other light materials. However, not all WWTPs are equipped with the oil-water separator and the residues need to be properly treated and disposed. An efficient valorisation of this waste, e.g. turning it into biodiesel (fatty acid methyl esters; FAMEs) would eliminate the costs referred to its disposal, and additionally, might generate a net profit.

Lipids are less responsive to biodegradation than other organic substances such as sugars and amino acids (Chipasa and Mędrzycka, 2006). However, overall literature considers lipids to be readily removed (even at high loads) especially in activated sludge. Loehr and Roth (1968) showed that biodegradability of lipids >C12 (long chain fatty acids, LCFAs) in wastewater increases with decreasing carbon chain length and increasing degree of unsaturation of carbon chains. Low substrate utilization rates of LCFAs are expected because they are found in treated wastewater effluents, usually >0.3 g/l (Dignac et al., 2000).

In chapter 2.3, a rough estimate was already made of how many lipids are in the influent of a municipal wastewater treatment plant. Considering 16 g/(PE·d), as Raunkjær et al. (1994) report, about 584 t FOG/a from a WWTP sized for 100,000 inhabitants could be collected. Such an amount cannot be ignored, and if efficiently separated, it could represent an exploitable resource for biofuel production, an important and desired step towards the circular economy (European Commission, 2015, 2016). The current recovery and reuse of wastewater-FOG is limited to biogas production in digesters. The residual (majority) of the lipid potential in sewage is currently not utilized, it is being disposed (incineration, direct agriculture application or composting) (Hudcová et al., 2019), which brings additional cost to the WWTPs. Even though FOG has been mentioned several times as a possible starting raw material for producing biodiesel, it has been scarcely studied in practice.

4.2.1 Biodiesel, composition and characteristics

Today, biodiesel is produced by processing vegetable oil or animal fat. These feedstocks are expensive and to some extent part of the ongoing food vs. fuel discussion. Biodiesel processes commonly use base-catalysed transesterification, where triacylglycerol reacts with 3 molecules of methanol to form three FAME molecules (Samios et al., 2009) because it achieves high conversion within couple of hours at low operating temperatures. However, if the free fatty acid content exceeds 1 %, acid-catalysed transesterification takes place. Although this process is much slower, it prevents soaps formation hindering otherwise base transesterification and downstream purification (van Gerpen, 2005).

Generally, highly unsaturated fatty acids are expected to be more prone to oxidation, hydrolysis and lower gel formation than their saturated counterparts. Longer saturated fatty acids are excellent for biodiesel

production, whereas unsaturated fatty acids are great for cold weather biodiesel production, therefore mixture is desirable (Gustone, 2004).

Biodiesel can directly be used in conventional engines. Owing to its unique characteristics – having higher cetane number (quality and performance of fuel, ignition speed), lubricity, positive ethanol fuel energy balance (net energy gain), higher flash point (more stable to autoignition), compatibility with the existing fuel distribution infrastructure and being free of Sulphur – biodiesel is a promising renewable fuel offering a partial substitution to the non-renewable petroleum-derived diesel fuel (Aghbashlo et al., 2016; Hajjari et al., 2014). In addition, biodiesel emits less by 20 % of unburned hydrocarbons, 30 % of CO, and 50 % of smoke compared to diesel fuels (Datta and Mandal, 2016).

4.2.2 Research on wastewater-derived lipid biodiesel

Lots of experiments and scientific research have been performed regarding lipid separation and transesterification at different stages of wastewater treatment. So far, no pilot set up has been tested in combination with the enrichment of lipids in microthrix parvicella and subsequent extraction.However, the approach by Argent Energy (see chapter 3) must be considered. Additionally, just a few of articles (Chi et al., 2018; di Bitonto et al., 2016; Dufreche et al., 2007; Mondala et al., 2009; Olkiewicz et al., 2014; 2012, Pastore et al., 2015, 2013; Patiño et al., 2018; Wang et al., 2016) present both, biodiesel yield from urban wastewaters (extracted FAMEs/dry sludge weight*100) and its acid composition defining quality, thereby suitability for biodiesel production.

As lipids are readily removed by mixed microbial populations in WWTPs, total FAME yield showed decreasing trend along the treatments: almost 60 % originated from EWC (sludge from oil-water separator), 9-15 % from primary sludge, 6-23 % from scum (floatation tank), and 0.5-6 % from the secondary sludge; from blended (65:35 mixed primary and secondary sludge) and stabilized sludge (after anaerobic digestion), there was only one paper (Olkiewicz et al., 2012) reporting 10.9 and 1 %, respectively. At first glance, fatty acid composition collected from various WWTPs in different countries and stages of treatment did not propose a substantial difference (Figure 4-2); the content of palmitic (C16:0) and oleic acid (C18:1) were maximal, with stearic (C18:0) and palmitoleic acid (C16:1) behind. However, analysis of variance revealed the type of wastewater, meaning the stage of treatment samples were taken, to be highly significant for some acids.
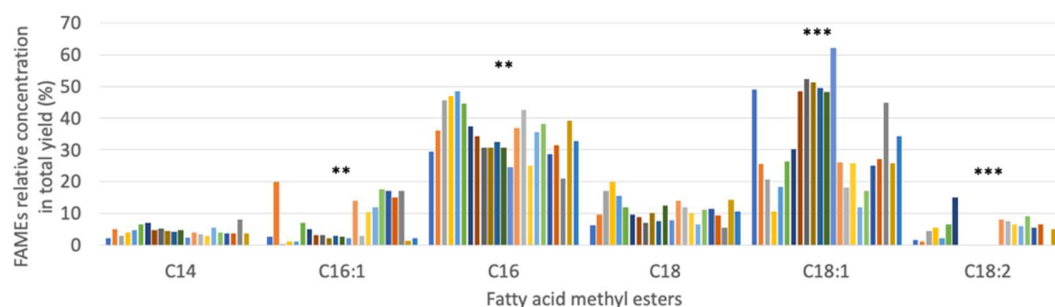

Figure 4-2:   Composition of fatty acids in total FAMEs yield in samples collected at different stages of urban wastewater treatment (EWC from oil-water separator, primary sludge, scum, secondary, blended and stabilized sludge, 1., 2.-6., 7.-13., 14.-21., 22., 23. Column respectively). ANOVA significance levels <0.01 and <0.001 are depicted with two and three asterisks, respectively

4.2.3 Current use of wastewater derived biofuel in the NWE region

Until today there is no relevant use of biofuel that is produced from wastewater or sewage sludge in the NWE region (see also Figure 3-1). However, there is a lot of research regarding this topic. In Figure 4-3 the most promising routes that are in the center of ongoing research for biofuel production from wastewater are presented. All of the routes produce biodiesel respectively FAME.
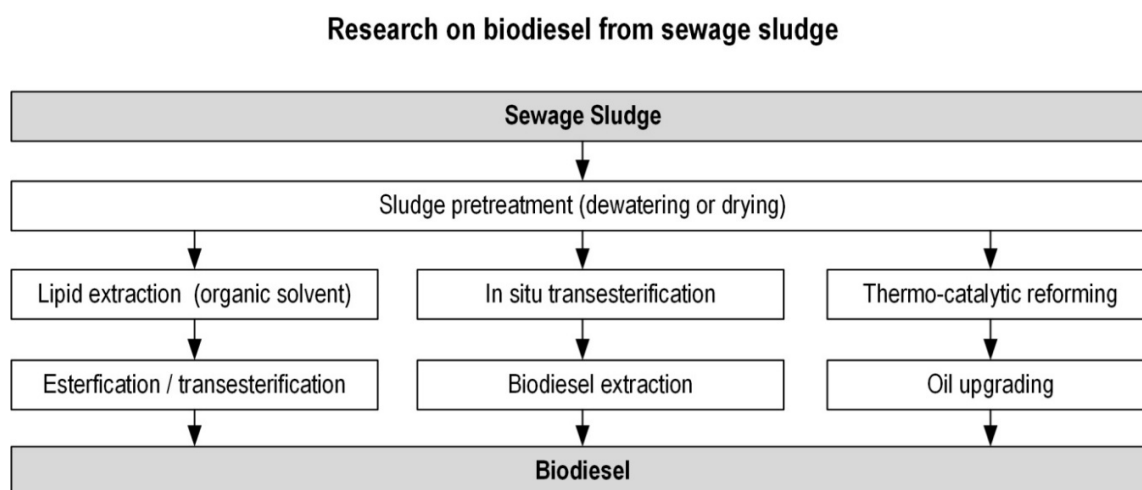
Research on biodiesel from sewage sludge

Figure 4-3:   Schematic presentation of Biodiesel production routes based on sewage sludge that are in the center of ongoing research

As in most production routes water hinders the process the water content of the sludge has to be reduced by suitable measures prior to processing. Here a combination of mechanical dewatering and thermal drying is applied. Thermo-catalytic reforming can accept sewage sludge with a water content of up to 30 %. The other routes need dry sludge matter.

In the production route of lipid extraction and subsequent esterification/transesterification in a first step the lipid is extracted from the sewage sludge using organic solvents or supercritical CO_2. Subsequently after removing the solvent from the extracted lipid fraction by suitable measures like e.g. distillation the lipids are converted into biodiesel by direct esterification or transesterification of triglycerides and free fatty acids. In most cases base- or acid-catalysed reaction routes are applied. The advantages of acid-catalysed reaction routes is that free fatty acids can be converted, which is not the case for base-catalysed routes. The downside of the acidic approach are lower reaction rates (Siddiquee and Rohani, 2011b).

In the production route of In Situ transesterification the lipid extraction and biodiesel conversion are implemented in a single process step. The solvent for lipid extraction and catalyst for the transesterification are added simultaneously to the sludge and after successful formation of biodiesel FAME is removed by suitable measures like e. g. solid-liquid separation followed by phase separation (Dufreche et al., 2007).

During thermos-catalytic reforming of the sludge by pyrolysis three substances are produced: hydrogen rich syngas, bio-oil and biochar. Biofuel can be produced from the liquid fraction. Because the oil is not suitable for direct use as fuel in a combustion engine it has to be upgraded by additional treatment prior to its use as biofuel. Here a catalysed hydrogenation treatment is applied, which removes heterogeneous

atoms like e.g. sulfur, nitrogen and oxygen or substitutes them with hydrogen. The resulting product complies with European fuel standards and therefore can be used as diesel (Schmitt et al., 2019)

4.2.4 Possible risks for ww treatment processes when adopting a biofuel production

Possible risks for the wastewater treatment process with an implemented selector for generation of a lipid rich sludge, are rising for the conventional activated sludge tank. If not operated properly, the elimination of carbon and other nutrients in the bioreactor could lead to a substrate depletion, esp. for the denitrification step where a specific C/N-ratio is necessary. Another possible (although improbable) risk is the formation of floating sludge in the conventional part of the plant, which comprises bad settling properties and therefore cannot be removed in the settling tank.

4.3   Pyrolysis Products

Pyrolysis is the degradation of biomass at high temperatures in the absence of air or oxygen. Under these conditions the organic material decomposes and forms vapors, gases and charcoal. The vapors can be condensed to a pyrolysis liquid (bio-oil). The yield and composition of the pyrolysis products depend on the raw material and the particle size, on the pyrolysis temperature, the heating rate, the additives added, the pressure conditions and the duration of treatment. To maximize liquid production, biomass heating and steam condensation must be rapid, at a temperature of typically 300 to 650 $^{\circ}$C, at a vapour residence time of a few seconds (Basu, 2013). This process is called fast pyrolysis. Alternatively, biomass conversion can also be oriented towards the production of charcoal. In this case, the heating rate is slower. Charcoal orientation can be subdivided in two different processes, torrefaction and carbonization. Torrefaction operates at a temperature of 200 to 300 $^{\circ}$C removing the low energy dense volatiles from the biomass, producing a charcoal with an energy density of 20 to 24 MJ/kg for wood. Carbonisation takes place at a higher temperature of 300 to 600 $^{\circ}$C removing most of the volatiles and maximizing the fixed carbon fraction of the charcoal, while lowering the energy yield as compared to torrefaction. Energy density is 30 to 32 MJ/kg for wood (Basu, 2013). Pyrolysis gases are usually used within the process, as a heating source (Serbanescu and Oprescu, 2016).

Applied CleanTech reports that pyrolysis oil from cellulose screenings differs from wood derived bio-oil, it's smell is not aromatic and it can be diluted in Diesel (Becker, 2014). Existing pyrolysis plants use other biomass such as wood residues, green waste, forestry and agricultural residues, grain husks, paper fibre sludge, straw, coconut fibres, wood chips, etc.. Lignocellulosic biomass, especially wood, has been widely studied and used at laboratory, pilot or commercial scale. Where the pyrolysis of biomass is a relatively new concern for Europe and large-scale plants are scarcely in this part of the world (Serbanescu and Oprescu, 2016). Depending on the process and raw material, the objectives of pyrolysis are the production of various charcoals such as activated carbon, vegetable carbon, feed carbon, carbon products for industrial applications or the extraction of bio-oil for the production of heat and power, biofuels or chemicals.

Nettenergy has a small scale (mobile) pyrolysis technology, which can be used locally, near the biomass source. Their Pyroflash technology produces char, a dry bio-oil with a low acidity and water content (typically 3 wt% water and a higher heating value (HHV) of 24 MJ/kg), wood acid and a fuel gas. Typical yields for a wood based feedstock with 10 wt% water are: 10 wt% char, 20 wt% bio-oil, 30 wt% wood-acid and 40 wt% wood gas. The process can operate autarkic, by combusting a fraction of the charcoal and using the fuel gasses to generate electricity. The wood-acid can be used as pesticide in diluted concentrations (Nettenergy, 2018).

Pyreg operates two pyrolysis plants in Germany (Linz-Unkel and Homburg) and one in the USA, which use sewage sludge as input. The output is a phosphorus-containing fertiliser substrate (coke/carbonate), which, according to own statements (Pyreg, 2019), contains phosphorus in a form available to plants. However, there is currently no certificate for use as fertiliser and the content of residual carbon does not allow for final deposition on landfill sites. This decentralised use of sewage sludge at the wastewater treatment plant saves complex logistics and reduces transport costs. This process benefits the environment by binding a large part of the carbon contained in the sewage sludge for the long term and thus reducing CO_2 emissions.

Pyrolysis can also be a process step in a multi-stage process for thermal sewage sludge disposal, as is the case with the Eisenmann pyrobuster, for example. Pyrolysis is the first step, followed by combustion in a two-chamber rotary kiln. This process has been implemented in St. Lorenzen (IT) and Crailsheim (DE). The focus here is on decentralised utilisation of the sewage sludge with simultaneous supply of thermal energy for sewage sludge drying. The process is approximately energy self-sufficient, offers long-term disposal security and independence. The residual material is an inert ash that can be disposed in landfills.

4.3.1 Bio-oil

Pyrolysis liquid (also called "bio-oil") is a dark brown liquid obtained by pyrolysis of biomass. The pyrolysis oil typically has a HHV of about 17 MJ/kg and contains about 25 % water (Bridgewater, 2012). Although of vegetable origin, pyrolysis oil consists of a large number of chemical compounds in contrast to vegetable oils, which are largely composed of fatty acids. Biomass fast pyrolysis liquids are completely different from petroleum fuels in their physical properties and chemical composition (Oasmaa and Peacocke, 2010).

If the pyrolysis process is designed for the production of bio-oil, fast pyrolysis is used due to the high bio-oil yield. Fast pyrolysis needs a high decomposition temperature (300-650°C), fine particle sizes (1 mm and even smaller), rapid heating rates (600–12,000 K/min) and very short residence times (0.5-1 s or less than 0.5s) (Serbanescu and Oprescu, 2016). Typical product yields for fast pyrolysis are 75 % bio-oil, 13 % gases and 12 % char (Ronsse, 2013).

For example, a large-scale pyrolysis plant is located in Hengelo (NL) (see Table 4-3 and chapter 8.4.10). It processes a quantity of 5 Mg/h clean wood, organic residues and wood pellet waste (Serbanescu and Oprescu, 2016). With an oil yield of about 65 %, the output is approx. 3.2 Mg/h. The remaining part is converted into coal and gas (BTG-BTL, 2019).

There are various possibilities for further processing of the pyrolysis oil. Through the co-combustion in natural gas, coal or fuel oil fired boilers, furnaces and turbines, a substitution of fossil fuel by pyrolysis oil can take place. Greenhouse gas savings can make energy supply more sustainable and at the same time reduce dependence on fossil fuel prices (BTG-BTL, 2019). It can also be used as a source of raw materials for the production of chemicals (resins, fertilizers, aromas, adhesives) (Rasul and Jahirul, 2012).

For direct combustion in a boiler or furnace, pyrolysis oil can be used to generate heat. The boiler burner must be replaced by a multi-fuel burner so that pyrolysis oil can be mixed with natural gas/fuel oil. In addition, the pipes and the storage tank must be made of stainless steel. The boiler itself does not have to be replaced (BTG-BTL, 2019). By replacing natural gas with pyrolysis oil from the Empyro plant at the Friesland Campina milk powder production site in Borculo (NL), 10 mil m³ of natural gas are saved every year and direct CO_2 emissions have been reduced by 15 % (BTG-BTL, 2019). Financially, pyrolysis oil is competitive with heating oil, but not with the currently low natural gas prices in most European and US countries. In countries such as Sweden (carbon tax), Finland and Brazil with low biomass prices and higher fossil fuel prices, prices can compete. In addition, more and more countries are using incentives (or mandates for modern biofuels) for pyrolysis oil to replace fossil fuels (SDE+ in the Netherlands, Renewable Heat Incentive in the UK, etc.) to bridge the price gap (BTG-BTL, 2019).

Gas turbines can be used to generate electricity and heat (CHP). Turbines are available on the market (e.g. from OPRA) which can be operated with pyrolysis oil after extensive testing and some modifications to the combustion chamber (BTG-BTL, 2019).

Diesel engines are used as stationary systems, ship and automobile drives. At over 40 %, the electrical efficiency of diesel engines is very high compared to all other decentralised alternatives (BTG-BTL, 2019). The use of pyrolysis liquid in diesel engines is probably the most technically challenging alternative (Oasma and Peacocke, 2010). Problems identified are related to various pyrolysis oil properties such as: particulates (causing erosion), acidity (corrosion), poor lubrication (injection needle friction), thermal instability (deposits), ignition (delay), viscosity (atomization), combustion (emissions) and water content (reduced caloric value) (Pyrowiki, 2019). The direct use of pyrolysis fluids in conventional engines is not possible. Either the engine or the fuel must be modified to avoid damage (Pyrowiki, 2019). Pyrolysis oil can be produced at a price level that is competitive with diesel. Since 2015 the world's first wood-based bio-Diesel refinery in Laappeenranta (Finland) is producing 100,000 Mg/a liquid named BioVerno Diesel. This type of Diesel can be successfully used in any Diesel type engines (Serbanescu and Oprescu, 2016).

Even though fast pyrolysis has reached a near-commercial status, they are not fully at a mature stage (Serbanescu and Oprescu, 2016). Table 4-3 lists the representative biomass fast pyrolysis commercial operational plants in Europe.

Table 4-3:   Representative biomass fast pyrolysis commercial operational plants in Europe (San Miguel et al., 2011; San Miguel et al., 2012, modified)

Technology provider	Since	Reactor Type	Operational plants in europe
BTG & Empyro	2010	rotating cone	Hengelo, NL
Empyro	2015		Borculo, NL
Neste Oil	2007	rotating cone	Porvoo, FI
PyTec	2006	ablative rotating disc	Bülkau, DE
VTT & Fortum	2009	fluidized-bed	Tampere, FI
Fortum	2013	circulating fluidized-bed	Jelgava, LV
Fortum	2015	fluidized-bed	Joensuu, FI
UPM Biofuels	2015	circulating fluidized-bed	Laappeenranta, FI
Wellmann Process Engineering Ldt.	2002	circulating fluidized-bed	Oldbury, UK
Bio Oil Holding NV	2010	twin screw and fluidized-bed	Tesseenderlo, NL
Bio Oil Holding NV	2010	twin screw and fluidized-bed	Delfzijl, BE

As an alternative to energetic use, pyrolysis oil can also be further processed in a refinery. As a result, a number of products can be produced, just as in a petroleum-based refinery. For this purpose, the pyrolysis oil is fractionated and each fraction is prepared so that it is suitable for further processing into fine chemicals, petrochemicals, fuels and energy. The aim is to maximise the value of pyrolysis oil (see Figure 4-4). So far, however, no refinery for bio-oil is known.
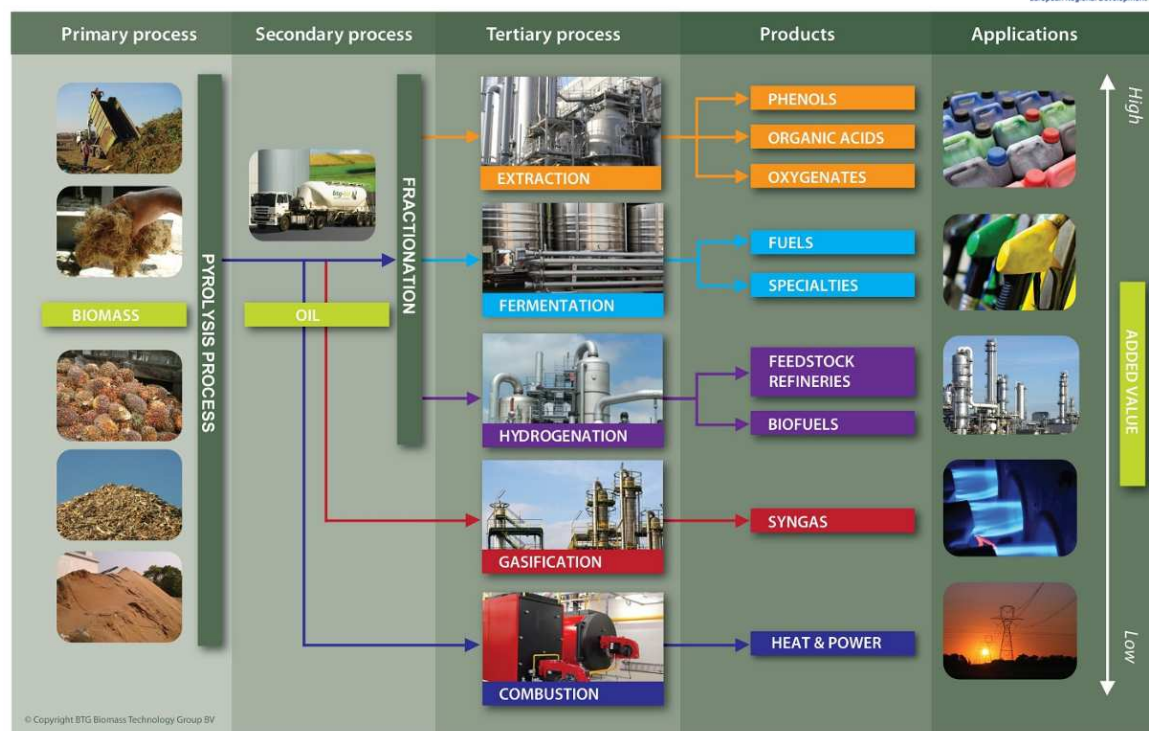
Figure 4-4   Possible application routes for bio-oil (BTG-BTL, 2019)

4.3.2   Biochar

As mentioned above, pyrolysis can be used to produce different types of charcoal for different purposes including activated carbon. Activated carbon consists mainly of fixed carbon and has a highly porous structure. Due to its large inner surface (typical commercial products: 500 to 2,000 m^2/g (Kirk-Othmer, 2004)), it is suitable as an adsorbent in drinking water treatment, wastewater treatment, chemistry, medicine, as well as ventilation and air-conditioning technology. In recent years it has been increasingly used to remove organic micropollutants from the effluent of municipal wastewater treatment plants by adsorption. Various processes have been developed for this purpose, which combine activated carbon in pulverized (PAC) or granulated (GAC) form with the wastewater and, in the case of PAC, separate it again. Activated carbon is mainly produced outside Europe from hard coal or lignite, anthracite, peat, wood, olive stones, nut shells or coconut fibres. In all manufacturing processes, the raw material is dried and carbonised by adding energy and then activated. This activation can take place thermally, by gasification of the carbon at relatively high temperatures or chemically, due to dehydration reactions at significantly lower temperatures (Kirk-Othmer, 2004).

Pyrolytic biochar does not appear to be suitable for activated carbon applications without post-treatment, as it has too little internal surface area in it (Morgano et al., 2018). However, as with other raw materials and manufacturing processes, this can be increased and optimized by activation. The actual suitability of an activated carbon obtained by pyrolysis for the elimination of micropollutants should be ensured by stirring tests (see also chapter 3.5) respectively rapid small-scale column tests (RSSCTs).

If the pyrolysis process is oriented towards the extraction of biochar, slow pyrolysis is carried out. Usual product yields for this type of pyrolysis process are 30 % bio-oil, 35 % gases and 35 % char (Ronsse, 2013). The commercialization of slow pyrolysis is not fully realized at large scales. Although industrial biochar production is currently developed in Europe, there is not yet a fully proven technology at large commercial scale (Serbanescu and Oprescu, 2016). A mostly known slow pyrolysis technology in Europe is from Pyreg, producing up to 130 Mg/a biochar. It allows a high flexibility in the feedstock due to the mechanical auger (Serbanescu and Oprescu, 2016; Pyreg, 2019). Several small-scale mobile pyrolysis units exist in Europe, especially in United Kingdom where this process has been the most developed, mainly for biochar production (Serbanescu and Oprescu, 2016).

4.3.3   Acetic Acid

Acetic acid is an important industrial chemical. The most important industrial synthesis for acetic acid is the catalytic conversion of methanol with carbon monoxide under pressure (Monsanto process). Acetic acid for industrial purposes is also obtained by the oxidation of acetaldehyde with air or oxygen or the carbonylation of methanol.

Acetic acid can also be obtained by pyrolysis. The resulting pyrolysis liquid can be fractionated into different product streams. Acetic acid can be produced from the aqueous phase by an extraction step and subsequent distillation (BTG-BTL, 2019). Vinegar, which is obtained from hardwood by pyrolysis, is also known as wood vinegar.

Applications of this acetic acid can be e.g. in the production of acetate salts as cleaning agents etc. (BTG-BTL, 2019). There are also considerations to use wood vinegar as a biological pesticide and biocide (Tiilikkala et al., 2010). Acetic acid can be used as an external carbon source for denitrification in wastewater treatment plants.

As the composition of the pyrolysis liquids is very complex, toxicological investigations of the acetic acid obtained from the pyrolysis of cellulose-rich screenings remain to be carried out in order to develop ideas for further use.

5 Current supply and market uptake of the five CBEs

Until today, supply of the considered five CBEs from wastewater has not been realized in industrial scale and consequently, a market uptake of them from wastewater does not occur yet. However, from the figures presented in the last chapters, it is possible to estimate for each product how much could be produced at NWE's wastewater treatment plants in the future. This is shown in Figure 5-1. Here is important to highlight that this are preliminary calculations only. In the following market potential study, these figures will be investigated in detail and will be compared with the demand for PHA, biodiesel, bio-oil, biochar and acetic acid from the North-West European Market. For this estimation the higher load generated by 280 mil PE from Table 2-3 for wastewater in North-West Europe was taken into account.
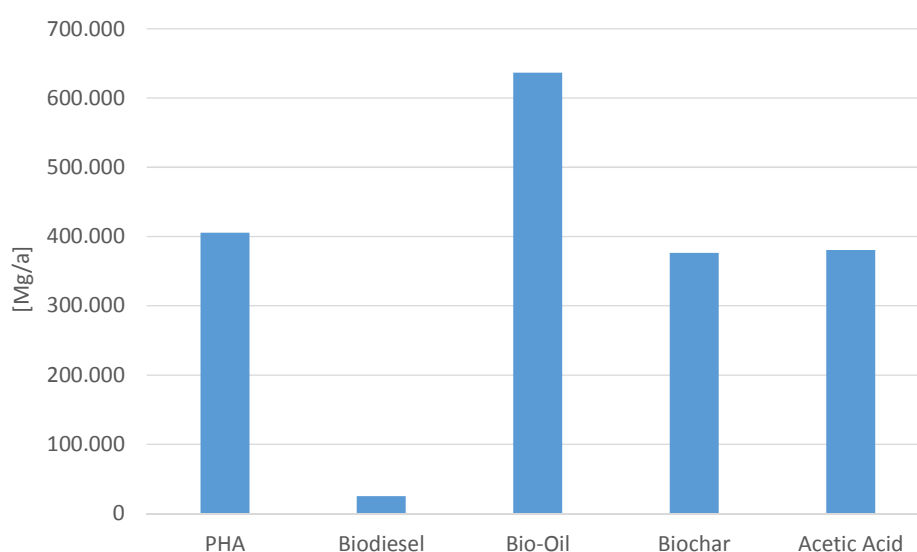

Figure 5-1:   Extrapolation of product quantities in NWE for the five CBEs

With the annual production capacity of 1.4 kg PHA/(PE·a) estimated by Pittmann and Steinmetz (2016) (see chapter 4.1.2), this results in a producible quantity of about 400,000 Mg PHA/a for NWE.

The amount of biodiesel that can be produced from lipids from wastewater in NWE can be calculated with the inhabitant specific amount of 16 g FOG/(PE·a) (see chapter 2.3). It can be assumed that 65 % of the extractable lipids are suitable for biodiesel production (Siddiquee and Rohani, 2011a, 2011b). With a mass conversion coefficient of 0.235 kg biodiesel/(kg saponifiable lipids) (Patiño et al., 2018), the quantity is 251,000 Mg/a. However, not all lipids in the influent of the WWTP are converted into biomass. Assuming a factor of 10 %, the recoverable amount of biodiesel is 25,100 Mg/a. Whether this factor is correct will be determined in the further course of the project in the pilot-scale trials.

For the calculation of the producible pyrolysis products it was assumed that the following quantities are produced during pyrolysis: 6.2 g/(PE·d) bio-oil, 3.7 g/(PE·d) biochar and 3.7 g/(PE·d) acetic acid (Pulsed-Heat, 2019). This results in 636,592 Mg/a bio-oil, 376,543 Mg/a ash/char and 380,671 Mg/a acetic acid.

Here it is not yet clear whether one can add all these amounts or whether one can win one or the other. Basically, the 5 CBEs are won from other shares of the COD, but the technologies may still hinder each other. This is also subject of the following research in the pilot-scale trials.

6 Literature

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7 Abbreviations

ABS	Acrylonitrile Butadiene Styrene
AD	Anaerobic digestion
BMBF	Federal Ministry of Education and Research, DE
BOD	Biochemical oxygen demand
CBE	Carbon based element
CDW	Cell dry weight
CHP	Combined heat and power
COD	Chemical oxygen demand
DM	Dry matter
DS	Dry solids
EFGF	Energie- en Grondstoffenfabriek
EPS	Extrapolymeric Substances
FAME	Fatty acid methyl esters
FOG	Fat, oil, grease
GAC	Granulated activated carbon
HHV	Higher heating value
IEX	Ion exchange
K	Kelvin
LCFA	Long Chain Fatty Acid
LHV	Lower heating value
MAD	Mesophilic anaerobic digester
MAP	Magnesium-Ammonium-Phosphate
N	Nitrogen
NWE	North-West Europe
P	Population
P	Phosphorus
PAK	Pulverized activated carbon
PE	Population equivalent
PE	Polyethylene
PHA	Polyhydroxyalkanoates
PHBV	Polyhydroxy-butyrate-co-valerate
PP	Polypropylene
PS	Polystyrene
RSSCT	Rapid small-scale column test
SAC	spectral absorption coefficient
SBR	Sequencing-Batch-Reactor
SIP	Sludge incineration plant
STP	Sewage Treatment Plant
TPH	Thermal pressure hydrolysis
TRL	Technology Readiness Level
VFA	Volatile Fatty Acids
VSS	Volatile Suspended Solids
WP	Work package
WWTP	Wastewater Treatment Plant

8   Appendix

8.1   Technology example for the recovery of struvite: PEARL™

The PEARL™ process recovers struvite from sludge liquor. In the crystallization reactor that is installed directly after the dewatering unit struvite is precipitated by dosing $MgCl_2$ at the bottom of the reactor and increasing pH by addition of sodium hydroxide. Internal recirculation assures proper mixing and good crystal growth, while the specially designed reactor shape guarantees uniform crystal size and optimum hydraulic conditions (Montag et al., 2015). To increase the phosphorus load in the sludge liquor the waste activated sludge of the wastewater treatment is sent to an anaerobic reactor prior to dewatering, which is designed to rapidly trigger the release mechanism of polyphosphate-accumulating organisms by optimizing conditions for P-release. The waste activated sludge is then sent to the thickening device and the resulting liquid with increased orthophosphate and magnesium concentrations is then fed into the PEARL™ reactor together with the liquor from the dewatering step of the digested sludge. In combination with the specifically designed process for phosphorus release prior to digestion, phosphorus recovery can be significantly increased above 40 % based of the total influent while simultaneously improving sludge dewaterability and digestor capacity (Verfahrenskennblatt Ostara Pearl, 2018).
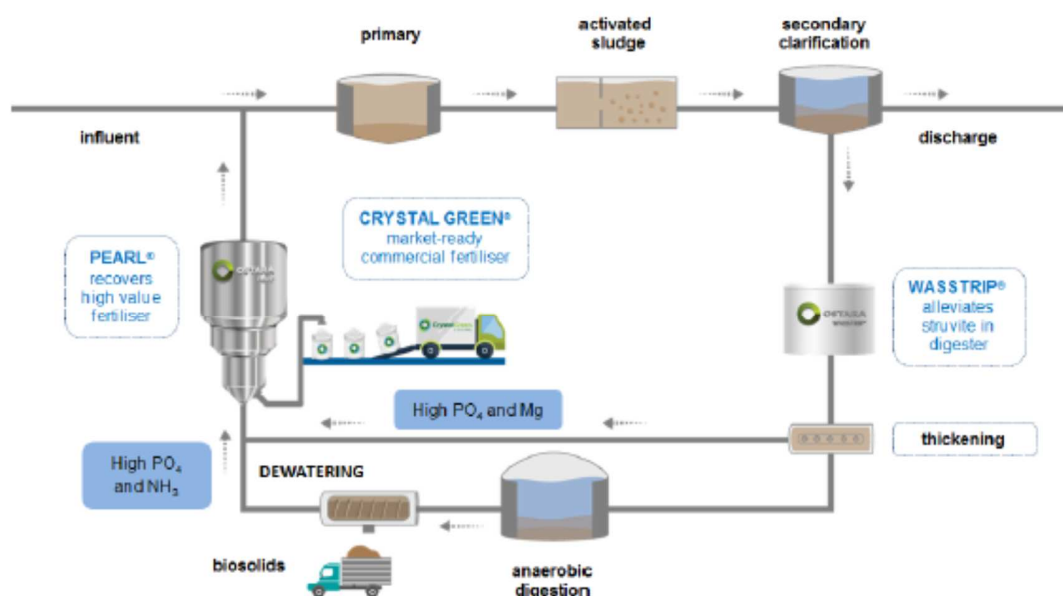

Figure 8-1   P-recovery from sludge water with combination of PEARL™ and WASSTRIP™ Process (Zhou, 2017)

8.2   Technology example for the recovery of phosphoric acid: TetraPhos™

The TetraPhos™-process process treats mono-incineration ash from fluidized bed combustion of municipal sewage sludge with phosphoric acid, then, after separation of acid insoluble residue, purifies the resulting leachate with sulphuric acid, ion-exchange and selective nano-filtration to generate an industrial quality phosphoric acid (brandname REPACID™). The process also enables recovery of iron and aluminium salts as coagulants, for recycling in sewage works phosphorus removal, gypsum intended for production of building material and a residual ash waste that either is used in the cement industry or if the latter is not possible is landfilled. More than 80 % of the ash phosphorus is recovered in the phosphoric acid. The simplified flow sheet of the process is shown in Figure 8-2.
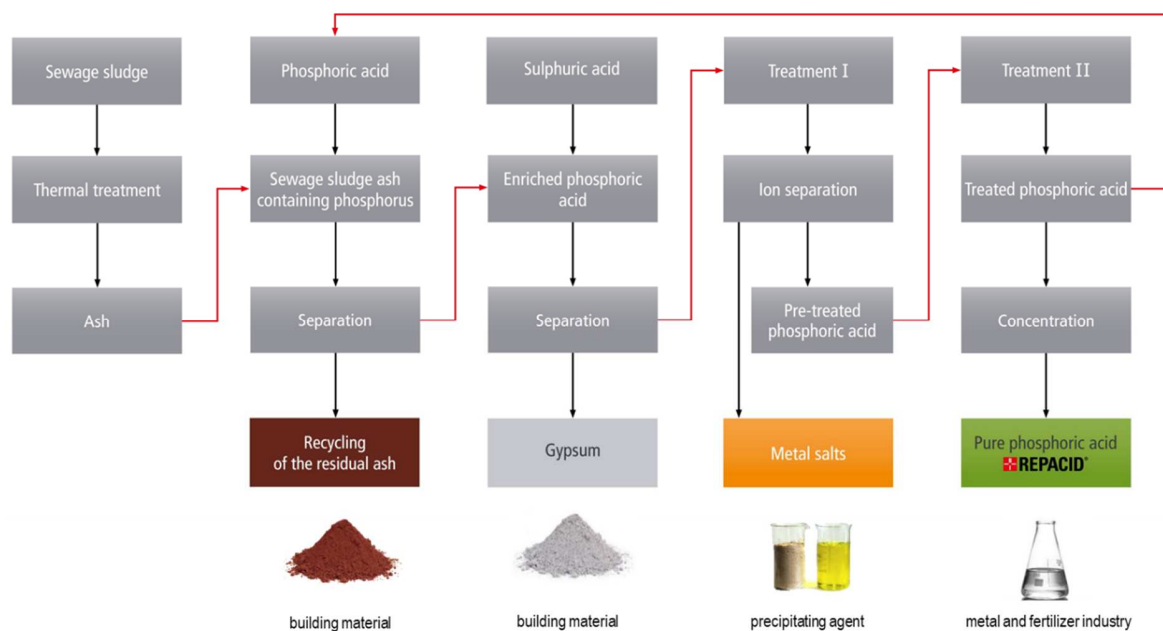
Figure 8-2   Flow sheet of TetraPhos™-process

The rollout of the technology will be implemented as public-private partnership between municipal partners and REMONDIS. First implementation of this model will be a cooperation with HAMBURG WASSER. From 2020 on a large scale TetraPhos™ plant at the WWTP in Hamburg will treat 20.000 Mg of ash annually, thereby recycling more than 1600 Mg of phosphorus. The Hamburg project is supported by the German Federal Ministry for the Environment, Nature Conservation and Nuclear Safety within the scope of the "Umweltinnovationsprogramm".

For ashes resulting from incineration of sludges of municipal origin the process is economical under consideration of all cost (including investments). TetraPhos™ is designed as part of an integrated plant consisting of incineration facility and P-Recycling installation thereby allowing efficient use of waste heat for concentration of REPACID™. The integration of the TetraPhos™ into the wastewater treatment process is shown in Figure 8-3.
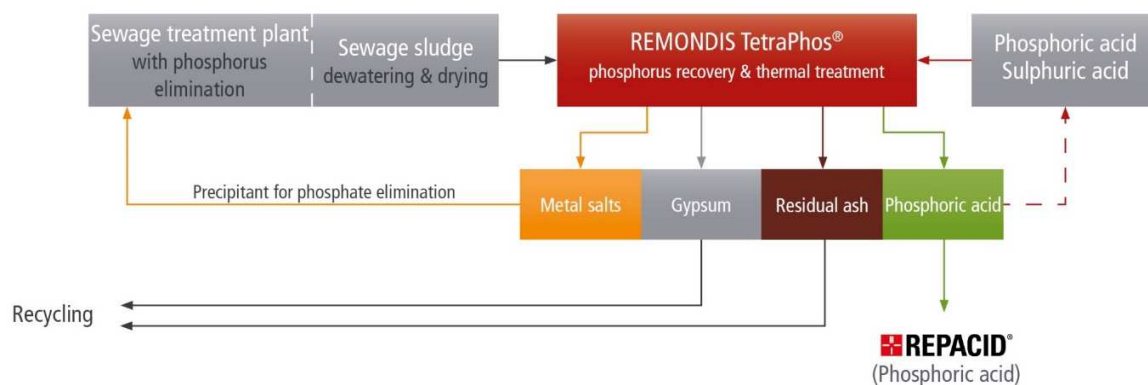

Figure 8-3   Integration of TetraPhos™-process into the wastewater treatment process

8.3 Overview of phosphorus recovery facilities operating or under construction in Europe

Table 8-1 Overview of phosphorus recovery facilities operating or under construction in Europe (Kabbe and Kraus, 2017)

Technology	Location and operator	Operational since	Recovered product
Onsite wastewater treatment plants			
AirPrex®	MG-Neuwerk (DE), Niersverband Wassmannsdorf (DE), Berliner Wasserbetriebe Echten (NL), Drents Overijsselse Delta Amsterdam-West (NL), Waternet Uelzen (DE), SE Uelzen Salzgitter Nord (DE), ASG Wolfsburg (DE), SE Wolfsburg	2009 2010 2013 2014 2015 2015 2016	Struvite
ANPHOS	Land van Cuijk (NL), Aa en Maas	2011	Struvite
EloPhos®	Lingen (DE), SE Lingen	2016	Struvite
EXTRAPHOS (Budenheim)	MZ-Mombach (DE), Wirtschaftsbetrieb Mainz	2017 (pilot)	DCP
Gifhorn	Gifhorn (DE), ASG	2007	Struvite/CaP
NASKEO	Castres (FR)	2015	Struvite
NuReSys®	Harelbeke (BE), Agristo 2x Niewkuerke (BE), Clarebout Potatoes Waasten (BE), Clarebout Potatoes Geel (BE), Genzyme Leuven (BE), Aquafin Schiphol Airport (NL), Evides Land van Cuijk (NL), Logisticon Apeldoorn (NL), Vallei & Veluwe Braunschweig Steinhof (DE), SE|BS / AVB	2008 2009/12 2012 2014 2013 2014 (pilot) 2015 2016 2018/19	Struvite
PEARL® (OSTARA)	Slough (UK), Thames Water Amersfoort (NL), Vallei & Veluwe Madrid (ES), Canal de Isabel II	2013 2015 2016	Struvite (Crystal Green®)
PHORWater	Calahorra (ES), El Cidacos	2015 (demo)	Struvite
PHOSPAQ™	Olburgen (NL), Waterstromen Lomm (NL), Waterstromen Nottingham (UK), Severn Trent Water Tilburg (NL), Waterchap de Dommel	2006 2008 2014 2016	Struvite
PhosphoGREEN (SUEZ)	Aaby (DK), Aarhus Water Marselisborg (DK), Aarhus Water Herning (DK), Herning Water	2013 2018 2016	Struvite
REPHOS®	Altentreptow (DE), Remondis Aqua (dairy)	2006	Struvite
STRUVIA™	Helsingør Southcoast (DK)	2015	Struvite
Stuttgart	Offenburg (DE), AZV Mobile Pilot – MSE Mobile Schlammentwässerungs GmbH	2011 (demo) 2015	Struvite (after acid leaching)
Downstream wastewater treatment plants and ash treatment			
EcoPhos	Varna (BG), DecaPhos Dunkerque (FR), EcoPhos	2016 2017	H_3PO_4/DCP/ MCP
Fertilizer industry	Various companies already apply or consider use of secondary P sources	tested and intended	Commercial fertilizer
MEPHREC	Nürnberg (DE), SUN	2016 (demo)	P-slag
TetraPhos®	Hamburg (DE), Hamburg Wasser/Remondis Aqua	2015 (pilot) 2019 (full scale)	H_3PO_4

8.4   Fact sheets

The following fact sheets describe WWTPs that have already implemented the recovery of a resource particularly well resp. projects for the recovery of recyclable materials from wastewater.

8.4.1   WWTP Apeldoorn (NL) – an example for the recovery of energy

Description of the plant

The Apeldoorn WWTP is a mid-sized WWTP of approximately 350.000 PE. It uses activated sludge and was constructed in its present form in 2003. In 2009 the SEA (Slibgisting Externe Afvalstromen) or Digestion of external waste streams) was added. This meant a significant increase in biogas production, as well as an increase in load for the WWTP.

In 2014 the WWTP was converted in an Energy- and Raw materials factory[1]. This was done by improved energy efficiency and the introduction of thermal pressure hydrolysis (TPH). Therewith sludge degradation was improved, and biogas production was increased.

At present the WWTP has an energy (heat and electricity) surplus, this is provided to the neighbouring residential area for the city heat grid. Also the surplus electricity is delivered to the electricity grid.

Description of the recovery process

Through the TPH process sludge is "cracked" at high temperatures and pressure (Figure 8-4 and Figure 8-5). Cracked sludge will be easier to digest. As is shown in Figure 8-6 starting 2009 sludge (in tonnes DM) is increasing due to the introduction of the SEA (externals waste digestion) and decreases with the introduction of the TPH in 2014. 2012 was a year with unreliable data. Biogas production has, as expected, increased by 20 %.

Figure 8-4:   TPH Sustec

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[1] An EFGF is a WWTP that produces more energy than it requires itself, also it produces raw materials from wastewater, nowadays this is mostly struvite ($Mg(NH_4)PO_4 \cdot 6(H_2O)$) (magnesiumammoniumphosphate).
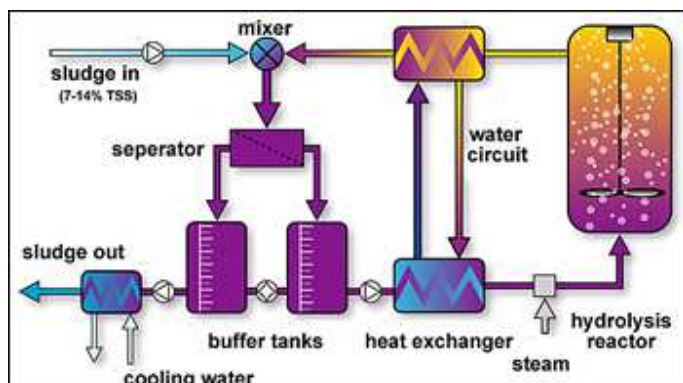
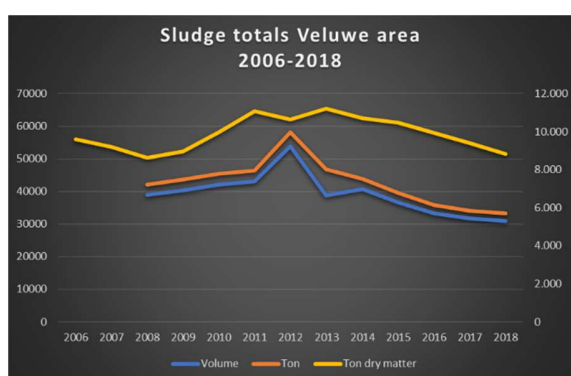*Figure 8-5: Schematic of the TPH of the WWTP Apeldoorn*

Figure 8-6:   Dry matter sludge

Lesson learned

Technologies introduced are complex and require additional skills from the organisation. Also once the WWTP started to provide surplus heat to the heat grid, it suddenly became a supplier. This meant that the operators needed to adapt to this. Delivery reliability could not be guaranteed, this meant that our heat had to be commercialized at a low price. Especially with the provision of heat, reliability is required, or back up facilities are needed.

Surplus electricity is delivered at a fixed price to the electricity grid. Since this is more and more common practice, this causes no major issues.

What is needed to organize well is the process of SDE+ (Subsidie Duurzame Energie + or Subsidie Sustainable Energy +). The financial business case depends on it and knowledge is needed in the organization to manage this.

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8.4.2 Minworth Sewage Treatment works, Birmingham (UK) - an example for the recovery of renewable heat

Description of the plant

Minworth STW, located in Sutton Coldfield near Birmingham, UK is Severn Trent Water's largest sewage treatment works (STW) and treats the sewage of approximately 1.7 mil people and the sludge of approx. 2.5 mil people from the greater Birmingham area.

The site has sixteen conventional mesophilic anaerobic digesters (MAD) that process approx. 5,000 m^3/d of sewage sludge. In 2018 a thermal hydrolysis plant was commissioned that pre-treats the indigenous and imported sludges prior to digestion. Approx. 75,000 m^3/d of biogas is produced and circa 2/3rds of this is directed to combined heat and power (CHP) engines that produce renewable electricity and heat. The electricity is used on site with any surplus being exported to the electricity grid. The heat is used to maintain the temperature in the MAD at about 38°C.

Figure 8-7: Minworth Sludge treatment plant. Showing the new THP plant in the foreground and the gas bags and MAD plant in the background

In 2014 the biomethane injection to grid plant was built and commissioned, this was the first site in the UK to produce biomethane from sewage at commercial scale. The biomethane upgrading plant produces approx. 19,000 m^3/d of biomethane which is enough renewable gas to heat and power 4,000 homes.
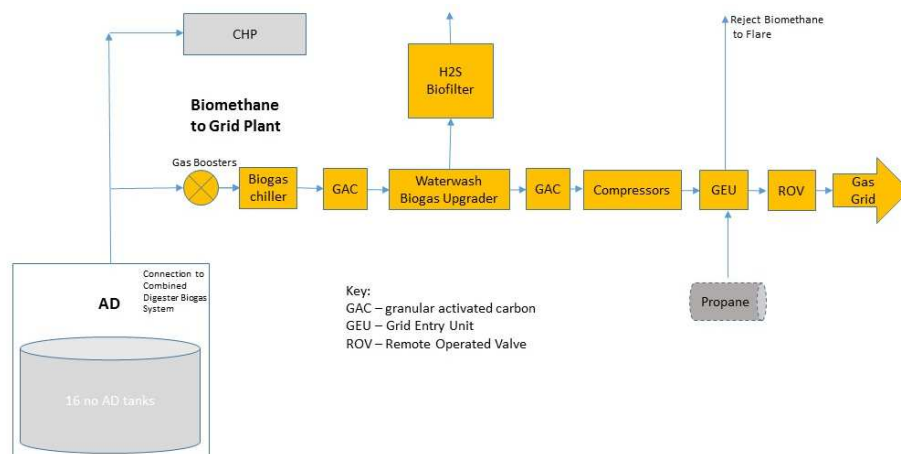
Description of the process

Figure 8-8: Biomethane to grid schematic

The Plant at Minworth is composed of 4 main processing steps:

1. **Pre-conditioning:** Gas coming off the MAD plant at Minworth is saturated with water as well as containing impurities such as siloxanes. The pre-conditioning plant uses chilling and reheating units to dry the gas and activated carbon filters to remove siloxanes.

2. **Carbon dioxide and hydrogen sulphide removal**: This is the key biogas upgrading step - the removal of CO_2 is necessary to ensure that the product gas goes from approx. 60 % CH4 at the inlet to 97 %+ CH_4 at the outlet. At Minworth Malmberg water scrubbing technology is used. The water scrubber utilises the different absorption rates of CO_2 and CH_4 in water at pressures between 4 and 7 bar. The biogas water scrubber utilises the different partial pressures of the gases when the system is operated at approx. 6 bar. At this pressure, the CO_2 readily dissolves into the water whilst the methane passes through. This process is undertaken in a large, packed column to ensure that the gas makes adequate contact with the water and the CO_2 is almost completely removed. Hydrogen sulphide is also extracted at this point because its absorption rate is at a lower pressure than the CO_2. The final step in this piece of plant is to dry the gas again to ensure it conforms to Gas Safety (Management) Regulations (GS(M)R) levels. To ensure the process remains sustainable the water is then depressurised in a second column. As the pressure drops to atmospheric levels, the CO_2 is released in gaseous form and is driven off using air. The water then returns to the system for reuse in the main scrubbing column. Over prolonged periods there is a small amount of build-up of CO_2 in the water. This water is discharged to the sewer and back to the works inlet and is replaced with fresh water from the mains.

3. **Pressurisation:** Minworth was the first site in the UK to connect into the medium pressure tier of the National Gas Grid. This pressure tier typically operates at between 7 and 32 bar. At Minworth the West Midlands Local Transmission System (LTS) operates at around 17 bar. The LTS offers a gas producer advantages in terms of available capacity and reduced propane consumption through 'energy blending'. The product gas leaving the upgrading plant is typically 3-5 bar and so there has to be additional compression before injection into the grid. At Minworth this has been achieved through the use of a single stage compressor provided by Wartsila. These compressors are operated oil free to prevent ingress from oil based lubricants into the gas that may disrupt the National Gas Grid.

4. **Gas testing and injection:** The gas is tested and injected through a Grid Entry Unit. The gas is tested using a gas chromatograph to show it has both the composition and physical properties to match the gas in the grid before it is injected. The gas is predominantly methane and provides a very clean alternative to Natural Gas. This testing reviews calorific value, hydrocarbon dew point, pressure, temperature, trace element (O_2 and H_2) percentages and combustibility factors. At Minworth this is all carried out in a single containerised module provided by Elster. If the gas CV is found to be lower than the input value from the local gas grid, propane is dosed into the gas stream to raise the CV. There is 12 tonnes of propane storage in place on site so that if the gas requires supplementing, STW have cover for 5 days. Prior to injection into the grid, the gas is odorised with methyl mercaptan to ensure there are no issues with any of its physical components. The gas is now at a point where it is almost identical to the gas in the National Grid network.

Figure 8-9: Biomethane Upgrading plant at Minworth (left) and Wartsila Gas compression unit (right)

Project Drivers / Lessons learned

The CHP at Minworth supplies the anaerobic digestion plant and a liquor treatment plant with low-grade heat to ensure its continuous operation all year round. Prior to the biomethane to grid plant being built, for most of the year the site operated at a substantial heat surplus and was forced to waste thermal energy to the atmosphere. This was both inefficient and a waste of a valuable resource. CHP production efficiency drops to 40 % from 80 % when heat from the engines is not being recovered. Injecting biomethane ensures that 97 % of the energy stored in the biogas is exported from site and it can achieve much higher final conversion efficiencies when utilised in bulk elsewhere in the National Grid Gas Network.

The economic viability of biomethane to grid projects are heavily influenced by renewable energy incentives. In 2011 the UK Government recognised the need for providing greater amounts of renewable heat and it introduced the Renewable Heat Incentive (RHI). This scheme financially supported biogas producers to upgrade and inject biogas into the national gas network as biomethane. Severn Trent have gone on to build additional biomethane to grid plants at some of our food waste digestion sites, however the business case remains dependant on accessing the RHI.

Literature

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8.4.3 WWTP Wuppertal Buchenhofen (DE) - an example for the recovery of heat

Description of the plant

The wastewater treatment plant (WWTP) Buchenhofen operated by the Wupperverband has a design capacity of about 600,000 PE. The digestion of the sewage sludge takes place in 3 digestion tanks with a total volume of 18,300 m³ (data sheet of the WWTP). In a combined heat and power plant (CHP), electricity and heat are generated from the biogas. Furthermore there is a hydroelectric power plant and a photovoltaic system, so that around three quarters of the electricity required is generated from renewable energy sources (Flyer Klärwerk Buchenhofen, 2019). At the site in Buchenhofen is also located the central sludge incineration plant (SIP) of the Wupperverband with a capacity of 34,000 t DS/a. Here the sludge of the large WWTPs operated by Wupperverband is combusted together with external sludge from other operators. The plant is equipped with a steam turbine for power production (Kolisch et al., 2014).

Implementation and operation of a local heat grid at a WWTP

Recently, only the heat production of the CHPs at Buchenhofen WWTP was available via a local heat grid for heating the raw sludge to digesting temperature and to cover the heat demand of the digester itself and of the operational buildings. However, during wintertime fossil fuels were needed in addition to cover the overall heat demand. By an optimisation of the heat supply system and connecting the sludge incineration plant to the existing heat grid, an additional heat capacity of about 1.5 MW in heat can be provided for internal as well as for external consumers (Kolisch et al., 2014). By this heat capacity theoretically about 450 households could be supplied with heat. Indeed only a portion of this heat is needed for full self-coverage in heat of the plant and external consumers can be connected to the local heat grid. In addition, excess heat can be used during summertime for cooling purposes. In this concern an adsorption chiller was installed which now converts a portion of the heat for cooling the central operational building.

Figure 8-10: Heat grid at Buchenhofen

As external consumers the central laboratory building of Wupperverband, some residential buildings and an industrial sewage plant operated by Bayer AG without own heat production are located in an economic distance. The heat grid was split into two circles with different temperatures. In the first section a new heat grid from the SIP to the screen building and the operational building was constructed (compare Figure 8-10, green line). The CHP continued to supply all other consumers with heat by the existing grid (yellow line). The filtration building, which was previously not connected to the heating system, was now also been connected to the CHP heat circle. The two heat circles can be reconnected in case of insufficient heat supply due to stops or failure of the generators or higher heat demand in wintertime. If necessary the oil driven boilers can be taken into operation as redundancy. The second step comprised the connection of the residential buildings, the laboratory and the Bayer WWTP by a new piping to the new SIP heat circle. Start-up of operation was in March 2014.

The costs for realisation of the first two construction sections of the heat network at Buchenhofen WWTP were calculated to about 1.4 m €. It is expected that Wupperverband will save on site including the laboratory about 60,000 l of fuel oil by the heat grid. Moreover 60,000 l of fuel oil can be saved by the industrial wastewater treatment plant of Bayer and the connected residential buildings. Furthermore, about 18,000 l of propane gas per year are saved at the filtration building. The current oil price is about 0.80 €/l, that for propane gas is about 0.50 Ct/l. The total financial saving is up to 110,000 € per year by the heat grid (Kolisch et al., 2014). The total emission factor of fuel oil including indirect emissions by production and transport is 3.13 kg CO_2/l (Öko-Institut, 2007). For liquid gas the factor is 1.90 kg CO_2/l. Assuming these factors, the CO_2-emission of the site can be reduced up to 380 Mg/a by the heat grid (Kolisch et al., 2014).

Lesson learned
- Municipal WWTPs with separate anaerobic sludge stabilisation and operation of CHPs usually achieve full self-coverage of the site's heat demand.
- A modern energy management based on a detailed heat and power balance allows to optimise the external energy supply and the use of fossil ressources.
- New approaches allow to convert digesting WWTPs from consuming sites to energy suppliers. Local heat grids may be one successful means in this concern.
- New approaches are especially needed in order to cover the high amount of excess heat that occurs at digesting WWTPs during summer. Drying of sludge or logs may be a suitable and economic means in this direction.

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8.4.4   WWTP Berlin Waßmannsdorf (DE) – an example for the recovery of phosphorus

Description of the plant

The wastewater treatment plant Waßmannsdorf operated by Berliner Wasserbetriebe treats approx. 230,000 m³ of wastewater per day in dry weather conditions. It is equipped with an increased biological phosphorus elimination as well as a pre denitrification process. The daily sludge volume is 2,000 m³/d. The digestion of the sewage sludge takes place in six digestion tanks with a volume of 8,000 m³ each (data sheet of the wastewater treatment plant Waßmanndorf, Gnirss et al., 2012).

Description of the recovery process

In the 1990s operational problems in the field of sludge treatment led to strong incrustations with magnesium-ammonium-phosphate (MAP), which crystallized in pipelines and pumping stations and quickly led to blockages. This problem was solved by targeted MAP precipitation in the digested sludge tank (Gethke et al., 2018). For this purpose, CO_2 is stripped via a planar aeration in the bottom of the tank. This increases the pH value and shifts the solubility equilibrium. At the same time magnesium chloride ($MgCl_2$) is added as precipitant and MAP precipitates. In the meantime, a separate tank has been installed for this purpose, which allows the precipitated MAP to be discharged from the tank during operation. The precipitated product is then fed into a washing process (Gnirss et al., 2012).

The efficiency of MAP precipitation varies between 70 and 85 %. Relative to the total phosphorus load in the inflow of the wastewater treatment plant, the efficiency of phosphorus recovery is 3.5 to 7.3 %, corresponding to 0.5 to 1.5 Mg/d MAP (Gnirss et al., 2012).
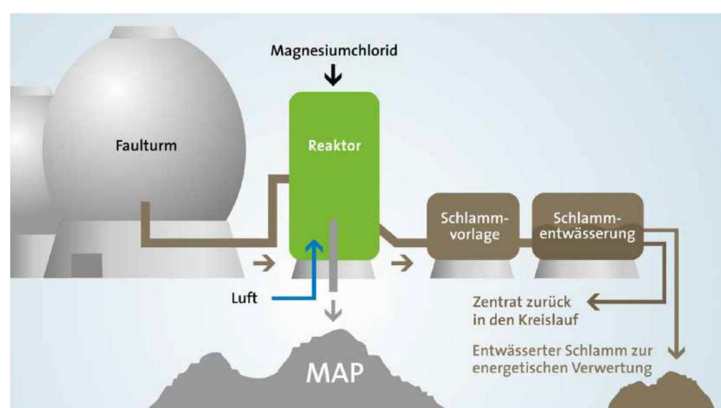

Figure 8-11: Process for obtaining MAP (Product sheet MAP)

Marketing of the product

The precipitation product is marketed as a mineral slow-release fertilizer under the name "Berliner Pflanze". Since 2008, it has been approved according to the EU Fertilizer Regulation (VO 2003/2003). The price is between 1.75 and 5 €/kg depending on the quantity purchased (Info flyer Berliner Pflanze).

Table 8-2:   Composition of the fertilizer (Info flyer Berliner Pflanze)

Magnesium oxide (MgO)	12 %
Total Nitrogen (N)	5 %
Phosphorus oxide (P_2O_5)	23 %
organic (organic carbon compounds)	20 %
crystal water	20 %
carbonates (inorganic carbon compounds)	10 %
other minerals such as calcium (Ca), trace nutrients	10 %

Lesson learned

- REACH registration is expensive and time-consuming for the "first user"
- Investments for optimising the operation of the sewage treatment plant (no MAP precipitation in the pipes and pumps, improvement of biological phosphorus elimination by reducing phosphorus back load) can be refinanced in small shares with the sale of the fertilizer.
- Sales revenues for fertilizer strongly dependent on world market price for phosphorus
- GreenTec Awards 2015

Literature

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8.4.5   WWTP Kloten Opfikon (CH) - an example for the recovery of nitrogen

Description of the plant

The wastewater treatment plant Kloten/Opfikon is located near Zurich Airport and is currently being expanded. It is dimensioned for 54,500 PE, but is currently loaded with an average of 86,000 PE (Projekt-vorstellung - Ausbau ARA, 2016). It had thus reached its hydraulic and biological capacity and was operated above the planned load limit. Since the optimisation potential had been exhausted, the expansion of the plant was started in 2017. The biological treatment process is carried out using the Nereda® process. In this process, the wastewater is pumped into reactors following mechanical treatment. There, microorganisms in the form of granules clean the wastewater biologically. The system promises to require significantly less space, energy and precipitants compared to conventional processes. The Nereda® process is already in operation at KA Sarneraatal, the first plant in Switzerland. (Newsletter - ARA Ticker Nr. 2, 2018). The new Water Protection Ordinance also obliges the operator to eliminate micropollutants. This challenge will be met with the installation of ozone reactors with subsequent filtration (Projektvorstellung - Ausbau ARA, 2016). Since 2011, nitrogen in the form of ammonium sulphate has been removed from the process water from sludge dewatering by stripping and sold as fertilizer.

NH₃-Stripping

At the WWTP Kloten/Opfikon, the stripping process for recovering nitrogen from process water is being carried out on a fully technical scale for the first time in Switzerland (Bryner, 2012). The temperature is raised to 60 to 65°C by means of a heat exchanger and, after pre-treatment, it passes through a stripper and an adsorber column (see Figure 8-12).

During stripping, volatile components in liquids (in this case nitrogen) are expelled with the aid of air. To do this, the pH of the process water must be raised to convert ammonia into ammoniac, which is comparatively volatile. This is usually done by adding larger quantities of lye. Eawag has developed a new process for this purpose, in which the CO_2 contained in the process water is also first stripped out, which already leads to an increase in the pH value from 7.2 to 8.6. The addition of lye can thus be reduced by 50 % (Liebi, 2014). The stripped ammoniac is collected in sulphuric acid solution. This produces ammonium sulphate, which can be used as a liquid fertilizer in agriculture. Around 90 % of the nitrogen in the process water can be removed in this way (Liebi, 2014). The process relieves the conventional elimination of nitrogen in the main stream and helps to save energy (Bryner, 2012).
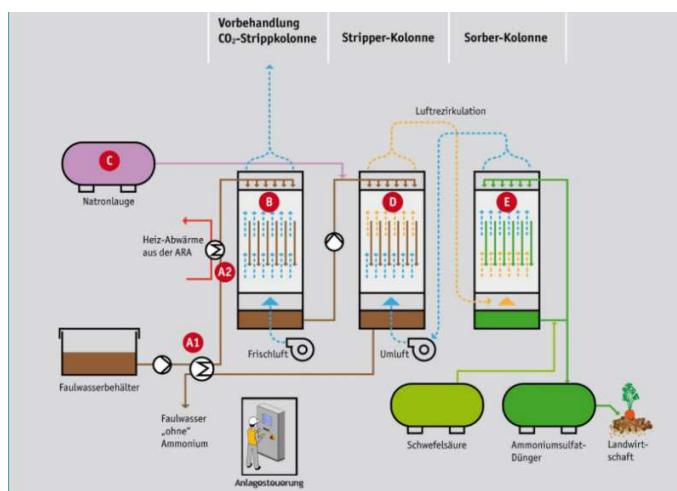
Figure 8-12: Process diagram of ammonia stripping in the WWTP Kloten/ Opfikon (Liebi, 2014)

The product is a 40 % ammonium sulphate solution with 8 % nitrogen and 8.5 % sulphur. The WWTP Kloten/Opfikon has a 10-year purchase agreement with a fertilizer dealer. The product is remunerated at the equivalent of 0.42 €/kg N (Garstenauer, 2018). The price is regulated dynamically in the contract and is adjusted to the nitrogen fertilizer prices. The product was approved by the fertiliser approval office under the name "Brinamon" and is applied using the CULTAN (controlled uptake and longterm ammonia nutrition) method (Garstenauer, 2018).

Lesson learned

- The nitrogen recovery system runs stably and meets the projected output. In 2016 / 2017, 241 respectively 299 Mg of ammonium sulphate solution were produced and sold as fertilizer in agriculture (Geschäftsbericht 2016/ 2017).
- The cleaning of the stripper columns must be given special attention, as the increase in the pH value leads to considerable amounts of precipitation products in the strippers (Liebi, 2014)
- The problem of deposits and incrustation of the filling material in the CO_2 stripping column could not yet be solved definitively. Experiments with pre-precipitation to remove solids and phosphate with subsequent separation of the sludge via a lamella clarifier did not produce the desired results (Geschäftsbericht 2016/2017).

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8.4.6 WWTP Epe (NL) – an example for the recovery of Kaumera (bio-polymer from NEREDA sludge)

Description of the plant

Epe Sewage Treatment Plant in the Netherlands was the first full scale domestic wastewater treatment plant in the Netherlands to install the innovative Nereda sewage treatment technology, which treats wastewater with the unique feature of aerobic granular biomass. Invented by the Delft University of Technology, Nereda was developed in partnership with Royal Haskoning-DHV, the Dutch Foundation for Applied Water Research and the Dutch Water Authorities.

The treatment plant, operated by the water authority Waterschap Vallei en Veluwe, treats wastewater produced by the town of Epe and the surrounding area, including industrial and domestic waste. It needs to meet the clear water requirements of around 54,000 people. Prior to the construction, a four year long pilot trial was carried out and the data findings were used to design the full scale plant.
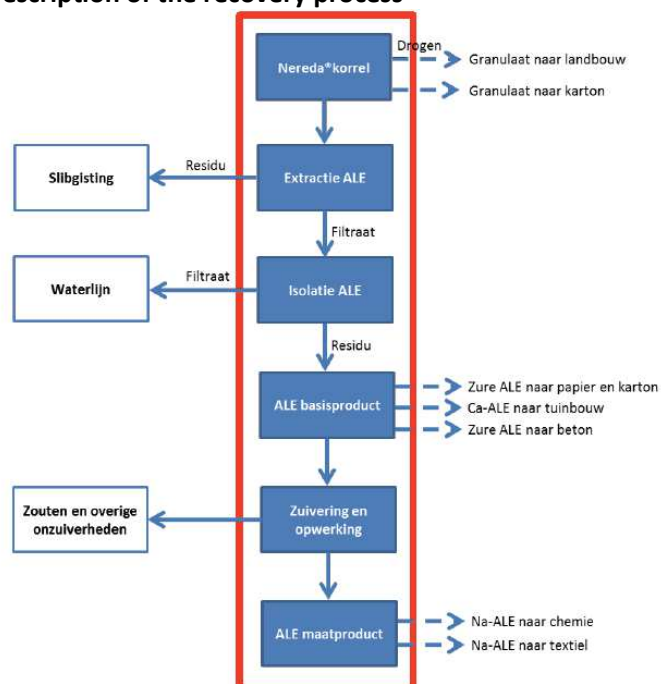

Description of the recovery process

Figure 8-13: Schematic description of the KAUMARE (formerly known as "ALE"); in Dutch

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There is no commercial technology available to extract Kaumera from NEREDA sludge. Hence a totally new, dedicated process needed to be developed to realise this. The innovative extraction process comprises the following steps:

1. Concentration: thickening the granular sludge to the desired dry weight for further processing;
2. Buffering: to buffer the granular sludge between the primary treatment process and the extraction installation;
3. Disintegration: to disintegrate the granules;
4. Sludge heating: to increase the yield of the extraction process;
5. Carbonate dosing: dosing system for the extraction chemicals;
6. Mixing: to have an even distribution of heat and Na_2CO_3 in the processing tank;
7. Separation: for separating the centrate and the residual sludge;
8. Buffering and mixing: temporary storage and mixing of the Kaumera containing centrate;
9. Acid dosing: for flocculation of the Kaumera in order to separate it from the water phase;
10. Separation: for separating the flocculated acid-Kaumera from the water phase.

The acid-Kaumera is refined into end products which are directly applicable. The final purity and quality will depend on the specifications set by the endusers, but basically process steps foreseen are:

1. Conversion of insoluble polysaccharide salts into the soluble Na-Kaumera;
2. Conversion of Na-Kaumera to alginic acid or Ca-kaumera by dosing of acid or Calcium Carbonate;
3. Conversion of Ca-alginate to purified NA-Kaumera by mixing with alcohol/water solution (in which the Na-salt will not dissolve;
4. Drying of Na-Kaumera;
5. Kaumera powder production.

These steps are comparable to regular alginate production, but will be applied to a new type of raw material.

Lesson learned

Kaumera is a totally new product and is as of now only possible to produce from NEREDA sludge. This means that application is not easy to find, there's not a ready market for the product. So apart from optimizing recovery technologies also a market needs to be created.

This is a time consuming process, where the market asks for kilo samples, while lab production capacity is grams per day. Lesson learned is to work closely together with partners willing to organize the market development.

Other lesson is that the technology and product are so new that the development process will encounter several drawbacks, and the partnership will need to be able to cope with this.

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8.4.7 WWTP Beemster (NL) – an example for the recovery of cellulose

Description of the plant

Beemster Sewage Treatment Plant (STP) is designed for a load of 192.000 PE (150 g COD) and a FFT (Full Flow to Treat) of 3,600 m³/h, designed to meet strict nitrogen and phosphate requirements. The waterline consists of a coarse screening (6 mm), finescreens, an anaerobic selector, an oxidation ditch, 4 clarifiers, sludge dewatering with 2 centrifuges and an effluent pumping station.

Description of the recovery process

In the sewage treatment process, toilet paper ends up in sewage sludge. This waste stream is eventually incinerated, which means the loss of good cellulose fibers. By using these fibres as a resource, a STP can contribute to a circular economy.

The finescreen installation built on the Beemster STP filters out undissolved components from the sewage water. A filter mesh is used with a mesh width of 0.35 mm. The finescreen is installed behind the coarse screen, which has removed undissolved components larger than 6 mm from the influent.

Cellu2PLA

The focus for this project (Cellu2PLA) was on harvesting cellulosic screenings to convert this cellulose first in glucose and then in lactic acid for the production of bioplastics (PLA). Cellu2PLA stands for the conversion of cellulose towards poly lactic acid (PLA). Or less complicated: making bioplastic out of used toilet paper!

Plastic waste is a major problem worldwide. Oil based plastic that ends up in nature has a long degradation time. When using bioplastics instead, the impact on the environment will decrease as bioplastics normally degrades faster. For the alternative (bioplastic), crops are currently used as a raw material. By deploying a waste stream as resource, competition with food is avoided.

The screened material was first sterilised with steam at 130 °C. Than enzymes were added that form glucose out of cellulose. After sieving out the solids from this glucose solution, lactic acid can be formed through fermentation/polymerization. After polymerization, lactic acid becomes a bioplastic.

Unfortunately, Cellu2PLA didn't succeed in producing PLA out of cellulose. Due to the use of a faecal/bacteriologically contaminated raw material, uncontrolled bacterial growth (and therefore formation of undesired components) turned out to be a problem. But still, with the knowledge that is gained, the project contributes to a bio-based economy. It is demonstrated that glucose can be formed

out of used toilet paper. Due to bacterial contamination there was no stable process in the glucose plant, but it showed that this technique has potential. Economically however, PLA produced out cellulose from sewage water can't compete with PLA from feedstocks like sugarcane or corn at his moment.

The achieved results

The effect of the fine-screen installation on the purification process was monitored during 2017. The results are as follows:

- the finescreen removes 37 % of the undissolved components from the wastewater;
- approximately 73 % of the resulting cellulosic screening consists of toilet paper (cellulose);
- the organic components that are filtered by the finescreen, no longer enters the aeration tank and don't have to be biodegraded, reducing the energy consumption for aerating wastewater by 9 %;
- 23 % less secondary sewage sludge is produced.

The energy consumption for aerating wastewater and dewatering sludge is decreasing. But because the fine-screen installation itself also consumes energy, the reduction in consumption over the entire STP is no more than 3 %. Due to the lower sludge production, the number of transport movements for sludge processing is decreasing and less sludge needs to be burned. This has a positive effect on greenhouse gas emissions. The over-all effect of using finescreens on STP Beemster, is a reduction in CO_2-emission of about 360 ton/year. Compared to the total CO_2 emission of the STP this is 12 %
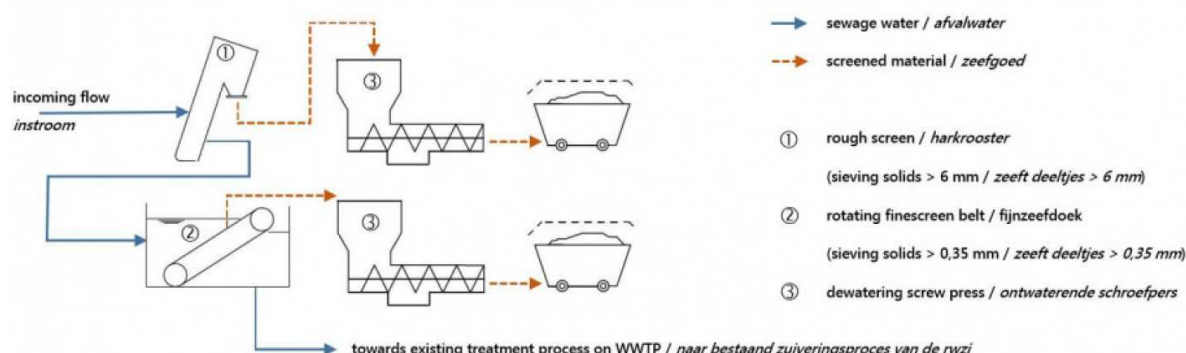

Figure 8-14: The process of harvesting cellulosic screenings

Lesson learned

The project Cellu2PLA ended, but the harvesting of cellulose continues. The water authorities are now looking for other ways to give toilet paper a second life. New initiatives arise quickly, like using it as a binder for asphalt, pressing board material from it and using the cellulose fibers for isolation material. The Dutch water authorities support these initiatives, to eventually make cellulose from wastewater a valuable resource.

Results:

Energy consumption	9 %	▽
Secondary sludge production	20 %	▽
Harvested cellulose	860 kg/d	

Literature

Layman's Report, Cellu2PLA, Toilet paper becomes bioplastic, 01-01-2018

8.4.8   SMART-plant - an example of the production of PHA

About the project

The project SMART-plant (**S**cale-up of low-carbon footprint **MA**terial **R**ecovery **T**echniques in existing wastewater treatment **PLANT**s) started in 2016 and will run for four years. In a consortium of 25 partners from Europe and Israel, the SMART-Plant project is testing existing technology approaches for the recovery of recyclable materials in wastewater treatment plants on a large scale in real plants. This allows to close the value chain for different recyclable materials by energy- and eco-efficient solutions and to achieve circular recycling. At 5 wastewater treatment plants, 7 pilot systems will be optimized over a period of more than 2 years and tested under real conditions. In addition, there are 2 technologies for the post-processing of recyclable materials. The recovered products include biopolymers (polyhydroxyalkanoates - PHA), cellulose, plant nutrients and fertilizers as well as intermediate products that are processed into commercially usable end products.

PHA is to be obtained both in wastewater in the mainstream and in sludge treatment in the side stream. By controlling the biological processes, a bacterium is enriched which forms and stores biopolymers (PHA) from the organic substance. These can then be extracted. The mainstream SCEPPHAR process (Short - Cut Enhanced Phosphorus and PHA Recovery) (SMARTech2b) is used in wastewater to produce PHA and struvite while ensuring adequate quality of the treated wastewater. PHA is also recovered in sludge treatment by the SCEPPHAR sidestream process (SMARTech5). PHA is a raw material for many products in the chemical industry. It can, for example, also be processed together with the cellulose sludge also obtained in the project to form composite materials (Sludge Plastic Composites - SPC), which can be used in the construction sector.

Figure 8-15: SMARTech 2b and fermentation unit of SMARTech 5 (http://www.smart-plant.eu)

WWTP Manresa (Spain)

The pilot plant for the recovery of PHA from wastewater in the mainstream is operated at the Manresa wastewater treatment plant (Spain). In the pilot plant, about 10 m³/d of wastewater will be treated in 3 cycles of 8 hours. It has a reaction volume of approx. 8 m³. It consists of two SBR with a volume of 2,500 l each, one for heterotrophic bacterial growth and one for autotrophic nitrifiers growth, an interchange

vessel and a chemical system for P recovery as struvite. PHA is recovered from the anaerobic purge of the SBR. The main objectives in the operation of the pilot plant are:

- treat 10 m³/d wastewater after primary treatment
- achieve an effluent with P < 1 mg/L and TN <10 mg/L without addition of external carbon source nor FeCl$_3$ for P precipitation
- remove nitrogen via nitrite with a removal up to 90 %
- recover around 50 % of phosphorus in the influent as struvite
- produce a waste sludge with PHA content up to 30 % without addition of external carbon source.

WWTP Carbonera (Italy)

The pilot plant for PHA recovery from sludge will be tested and validated at the Carbonera wastewater treatment plant (Italy). It will treat the total throughput of about 40 to 50 m³/d of sludge water in a sidestream and will enable the recovery of 0.7-0.8 kg PHA/d. The nitrogen from the sludge water is to be removed in an energy-efficient way with simultaneous recovery of PHA and struvite. The following sub-processes are considered:

- Sewage sludge fermentation under alkaline conditions (pH around 10) to improve the production of volatile fatty acids (VFA) and release of nitrogen and phosphorus in soluble form (ammonia and phosphate).
- solid/liquid separation of fermentation products and recovery of struvite from sludge water by addition of Mg(OH)$_2$ to improve precipitation
- Ammonium conversion to nitrite carried out in an SBR
- Selection of PHA-storing biomass in an SBR by the alternation of aerobic and anoxic conditions
- PHA accumulation using a feed-batch reactor to maximize the cellular PHA content of the biomass removed from the selection step. The system is based on two SBRs for nitrogen removal in combination with enrichment and storage of PHA in sewage sludge.

Sludge Plastic Composites (SPC)

The cellulose sludge and PHA recovered in the project will be processed into a novel composite material - sludge plastic composite (SPC). The pilot plant will be built in London (UK). For this purpose, an existing pilot plant which has previously processed a composite material of wood and plastic (WPC) will be retrofitted. SPC is expected to be comparable to conventional WPC and suitable for use in construction and other industrial applications.

Literature

http://www.smart-plant.eu

8.4.9 Sapoval (FR) - an example of the recycling of wastewater containing lipids

Description of the areas of expertise

Sapoval is an engineering and consulting company located in Albi, France. Based on their know-how, they offer tailor-made solutions for optimized management of effluents rich in fat, oil and grease (FOG, Figure 8-16) both for industrial and urban purposes.

Figure 8-16: Sapoval areas of expertise (adopted from www.sapoval.fr)

According to the needs, they install and commission pretreatment tools (grease traps, screens, floatation tanks, etc.). Furthermore, they support management and valorization of the FOG-waste originating from food industry (agriculture, farm machinery, food processing and marketing, foodservices and restaurants) as well as urban sewage system. They are also involved in research and development of energy recovery routes for waste material, as well as new innovative technologies for improving of the wastewater treatment processes (effluent recovery e.g.). Sapoval participate on the process development for anaerobic digestion in France in collaboration with construction companies and biogas plants. Particularly, they are working on the integration of waste material rich in fats aiming at higher fermentation-grade with a greater volume of produced biogas) they are working on the development of recovery routes for FOG-waste recovered from wastewaters. After saponification (conversion of FOG into soap and alcohol at presence of sodium hydroxide and heat), this waste is used as substrate for microorganisms with aim to improve functioning of biological processes. Additionally, they are working on the development of raw material recovery pathway for FOG) Sapoval is involved in development of innovative technologies to improve performance of existing wastewater treatment processes.

Process description and real examples of deployment

Via the saponification process, the SAPO'FIX unit transforms FOG into an odorless liquid effluent that can be directly treated by a conventional WWTP or an anaerobic digester (methanizer) decomposing the waste

at simultaneous fuel production. They offer their solutions also in a mobile version for in situ collection and processing of FOG waste. Thanks to their tailor-made consulting, design and training packages, Sapoval is meeting customer needs (Figure 8-17).

Figure 8-17: Examples of Sapoval's partners and customers in food industry and wastewater treatment processes (adopted from www.sapoval.fr)

They offer a range of solutions for food industry companies (e.g., Menguy's, Arcadie Sud-Ouest (slaughter), Maison Samaran (local food producer), SEPPIC (personal-care products)) to improve waste management at simultaneous waste valorization. They also provide collection and recovery of greasy waste from large kitchen facilities, management of effluents and maintenance of sewerage in case of clogging or overflow (e.g., Kyriad Hotel, McDonalds, Eurest, Sodexo, Compass Group (food service companies), municipality of Albi).

Lesson learned
- Sapoval is a company located in NWE-region focusing on recovery of raw materials from waste and heading towards circular economy
- It could be said that their goals are similar to ours, however, Sapoval is using saponification process for recovery of FOG in contrast to our aim of using transesterification for biodiesel production

Literature
www.sapoval.fr

8.4.10 Hengelo (NL) – example of a commercial fast pyrolysis plant

The Biomass Technology Group (BTG) has a commercial scale fast pyrolysis plant in the town of Hengelo in the Netherlands. The plant operates using a first generation biomass feedstock, using a 25 MW(th) fast pyrolysis process, the biomass is converted into pyrolysis oil, electricity and process steam. The pyrolysis oil is being utilized in a natural gas co-boiler, saving 10 mil m^3 of natural gas annually.

Process description

The wet biomass feedstock, typically clean wood with a particle size of approx. 3 mm is stored in a silo. From the silo the biomass is transported to a low pressure steam dryer. In the dryer, the biomass is dried from 10 to 12 %wt down to 5 %wt before being fed to the reactor, at a capacity of 5 Mg/h. In the pyrolysis reactor (a rotating cone type), sand, as the heat transferring medium is intensively mixed with the biomass. At the reaction temperature of 400 to 600 °C, the biomass stats to decomposes, leading to a solid char and vapor fraction. In several steps the solids are separated from the vapor phase. The vapor is led to a spray condenser where the oil is separated from the non-condensable gas, and collected at the bottom of the condenser. The liquid pyrolysis oil is pumped to a storage tank, from where it can be loaded onto a tank truck. The solid particles, sand and char are transported, as well as the non-condensable gas to a fluidized bed combustor where all char, and combustible gas is combusted to supply the energy necessary to reheat the sand before reusing it in the reactor. Char is therefore not a separate product.

The excess heat from the combustor is used to generate steam in the heat recovery boiler. The mineral fraction, that was present in the biomass, is removed at the bottom of the boiler. From the produced process steam approx. 5.8 MW(th) is utilized at a neighboring AkzoNobel plant, and while approx. 1.6 MW(th) is used internally, from which the bulk is utilized in the steam dryer. Further, process steam is used in a steam turbine to generate 650 kW(el), of which the process uses approx. 40 % and the other 60 % is sold to the grid. A schematic flow sheet is presented below.
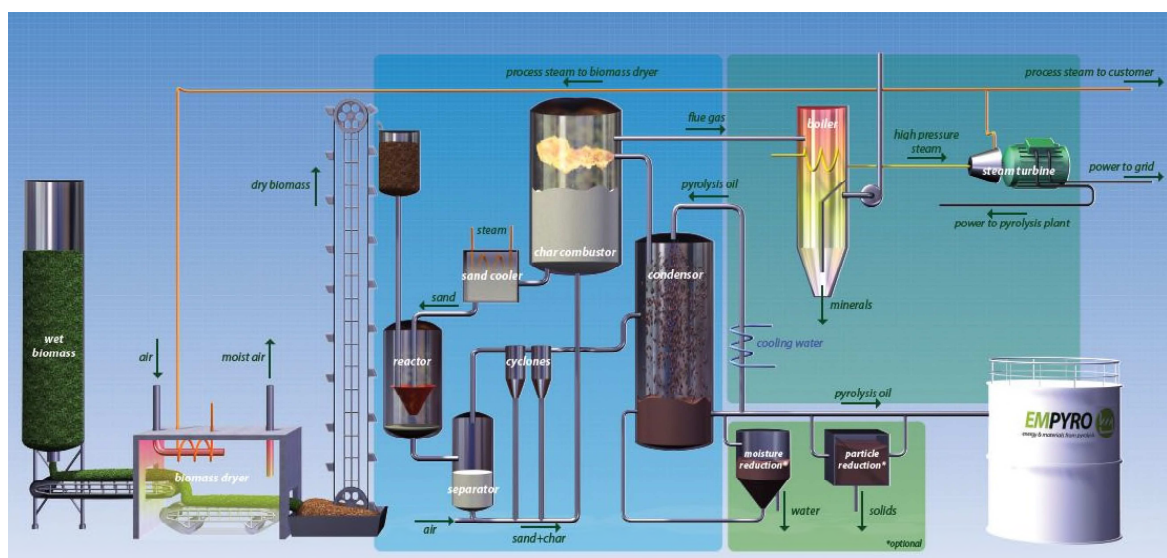

Figure 8-18: Schematic flow sheet of the Empyro fast pyrolysis plant

Pyrolysis oil

From the 5 Mg/h feedstock, the plant produces 3.2 Mg/h of pyrolysis oil, leading to a yield of 65 %wt. The pyrolysis oil has a water content of 22 to 24 %wt, and a lower heating value (LHV) of 16 MJ/kg leading to a thermal capacity of approx. 15 MW. The pyrolysis oil is being exported to a plant of FrieslandCampina, a Dutch global dairy company in Borculo. The transporting is done by tank truck from, Hengelo to Borculo, which is approx. 30 km. At the plant in Borculo the generated process steam is being used in their milk powder process.

The pyrolysis oil it is being co-fired with natural gas in a steam boiler, producing high pressure process steam with a pressure of 21 bar. A schematic flowsheet is presented below. Using the pyrolysis oil, FrieslandCampina annually saves 10 mil m^3 of natural gas. With a LHV of 8.8 kWh/m³ for the natural gas, this is would be a savings of 88 GWh(th) per year.
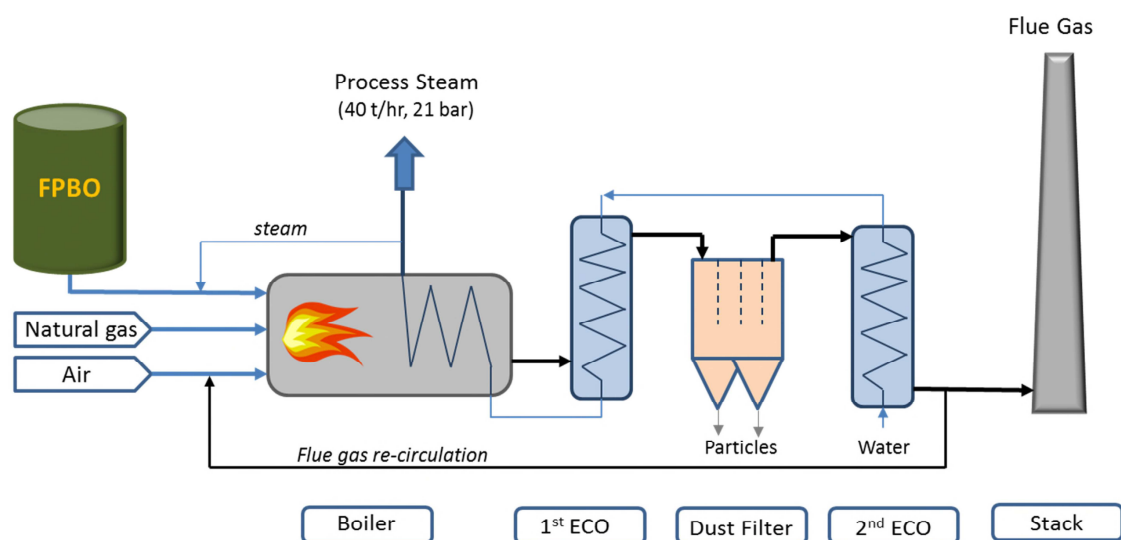

Figure 8-19: Schematic drawing of the steam boiler at FrieslandCampina

Literature

van de Beld, B. (2015): Commercial Scale Fast Pyrolysis Oil Production and Utilisation, IEA conference - Berlin (2015)

van de Beld, L., G. Muggen, G. (2015): EMPYRO: IMPLEMENTATION OF A COMMERCIAL SCALE FAST PYROLYSIS PLANT IN THE NETHERLANDS, 23rd European Biomass Conference and Exhibition, 1-4 June 2015, Vienna, Austria

9   Contribution to this report

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FR: Natureplast, NTP

DE: REMONDIS Aqua Industry

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