

Final report

1. Project details

Project title	Urban Waste Hydrofaction
File no.	64019-0075
Name of the funding scheme	EUDP 2019-II Biomass
Project managing company / institution	Steeper Energy Aps
CVR number (central business register)	33383835
Project partners	Aalborg University RenoNord Aalborg Forsyning, Kloak Aalborg Forsyning, Renovation
Submission date	25 August 2021

2. Summary

Steeper Energy's Hydrofaction® technology platform is being demonstrated in Norway, at an industrial scale, for conversion of forestry residues. The primary objective of this project was to address key technical risks related to new high-ash waste feedstocks (e.g. sewage sludge and household waste) and to advance the technology readiness to enter this new market. Upon the outset, Hydrofaction® was expected to be commercially viable for paid-for-wastes at a comparable scale to the existing demonstration project.

Pre-identified risks in critical components due to high ash in these feedstocks including extensive wear in the high-pressure pumping system have been addressed by building functional prototypes and testing them in dedicated experimental facilities. Homogeneous slurries were produced from these feedstocks and successfully processed through Hydrofaction®. Unforeseen challenges (e.g., product separation) were observed and solved. Preliminary mass-, elemental - and energy balances were established for both sewage sludge and pulped municipal waste (bio-pulp). Destruction of pathogens, endocrine disruptors and microplastics was also observed. Separation of valuable mineral fractions was successfully undertaken. Of the phosphorous contained in the feedstock, 93% ends up in the mineral product with the remainder in the water phase. The mineral product may be used directly as a fertilizer or soil amendment product or may be further processed into conventional phosphorous products such as diammonium phosphate, struvite or hydroxy apatite. Ammonia required to produce these compounds was recovered from the process water. The oil yield from sewage sludge was lower than expected likely due to losses of low-boiling compounds during separation and concentration of the water phase; whereas, oil yields from bio-pulp exceeded expectations. Notwithstanding the project grant was nearly 50% less than originally requested, the project met technical and commercial milestones articulated in the project application. The Hydrofaction® solution for these feedstocks is ready for a techno-economic assessment including a final engineering study.

Danish Summary

Hydrofaction® teknologien er under demonstration i Norge for omdannelse af affaldsstrømme fra skov- og papirindustrien. Nærværende projekt har sigtet mod en afdækning af en række kritiske tekniske risikofaktorer relateret til nye og problematiske affaldsstrømme fra byer såsom spildevandsslam og husholdningsaffald med højt aske indhold. Sådanne affaldsstrømme har en negativ pris og derfor rentable i en mindre skala. Projektet kunne derfor bane vejen for en tidlig markedsintroduktion på et nyt marked i en skala sammenlignelig med demonstrationsanlægget i Norge. Risici i relation til højt slid i specielt højtrykspumpen og trykaflastningspumpe er blevet adresseret ved at bygge en testfacilitet med funktionelle prototyper af disse kritiske anlægskomponenter til undersøgelse af disse forhold. Fremstilling af homogene og pumpbare slurries er blevet demonstreret for begge fødestrømme og omsætning af disse i Hydrofaction® processen er blevet demonstreret. Uforudsete udfordringer i relation til separation af produkterne fra processen er observeret og en ny løsning er blevet udviklet og etableret. Præliminære masse-, element-, og energi balancer er blevet etableret for både spildevandsslam og pulped husholdningsaffald (biopulp) inklusive destruktion af pathogene bakterier og vira, medicinrester og mikroplastik indholdt i disse inputstrømme, og som udgør et potentielt miljømæssigt problem ved tilbagetilføring til fødekæden, for eksempel ved udbringning af udrådnet slam fra bioforgasning på marker. 93 % af fosforindholdet i inputmaterialerne ender op i mineralproduktet fra processen, og det resterende i vandfasen fra processen. Mineral pro-

duktet kan potentielt anvendes direkte gødningsprodukt eller jordforbedringsmiddel eller kan processeres til konventionelle fosfor produkter som diammonium fosfat, struvit eller hydroxyapatit. Ammoniak for produktion af de to førstnævnte produkter kan genvindes fra procesvandet som demonstreret i projektet. De målte olieudbytter fra spildevandslam har været lavere end forventet formentlig pga., tab af flygtige komponenter under separation og koncentrering af vandfasen. I modsætning hertil er olie udbytte fra biopulp højere end forventet. Overordnet set er alle tekniske og kommercielle milestones i projektet opnået, og projektet reduceret risici relateret til sådanne fødestrømme betydeligt. Urban Waste Hydrofaction[®] for disse input strømme er nu klar til næste fase af kommercialiseringen, som vil være et techno-economic assessment studie inkl. et final engineering studie af et konkret anlæg.

3. Project objectives

The Hydrofaction[®] technology for conversion of forestry residues to green transportation fuels for the heavy transport sector is under demonstration at industrial scale in Norway. This project has focused on de-risking of central process steps related to conversion of new high ash containing urban waste streams to green transportation fuels by Hydrofaction[®], and thereby advancing the technology as an attractive circular solution superior to alternatives for this new market.

The specific objectives being addressed in this phase of the project are:

- To produce and verify a homogeneous and stable pumpable slurry from urban waste streams with well-defined rheological properties
- To establish preliminary mass- & energy balances from Hydrofaction[®] on urban waste slurries
- To demonstrate destruction of microplastic and endocrine disruptors from urban waste slurries
- To verify that erosive effects from high ash content urban waste slurries can be kept within acceptable limits for critical components
- To debottleneck the pilot plant for future longterm operation

4. Project implementation

The project period was extended from 18 to 21 months due delays in deliveries of certain key parts due to the Covid-19 lockdown. However, despite the difficult external conditions over past 18 months with Covid-19, the overall project has to a large extent been executed according to the original plans. However, physical project meetings and dissemination activities have been affected by the Covid-19 pandemic. The dissemination activities will carry on beyond the project completion.

5. Project results

The primary objective of the project was to address key technical risks related to the use of Hydrofaction for a new high-ash Urban Waste feedstocks, thereby advancing the technology into a new market. The specific project objectives have been obtained, namely:

The project has demonstrated that homogeneous pumpable slurries can be produced from urban waste stream such as sewage sludge and household waste, and that these can be converted by the Hydrofaction® process. Further destruction of microplastics, endocrine disruptors as well as pathogens have been demonstrated. These represents key sales points compared to alternative technologies for the same feedstocks such as anaerobic digestion. The oil yields for sewage sludge were lower than expected, whereas the oil yield for pulped household waste were higher than expected.

The risks of excessive wear due to erosive effects from the feedstock in the high pressure pump and in the pressure let down system was identified as critical risks that needed validation for advancement of the technology for commercial implementation. Prototypes of the high pressure pump and the pressure let down pump designed for minimization of wear and an experimental set-up for wear testing of the prototypes under representative conditions were successfully constructed and operated. The pumps have been tested for more than 50.000 pressurization-depressurization cycles on sewage sludge and have been proven to be a functional solution for controlling flow and pressure in the system. However, not without challenges and issues to overcome. Developments and improvements include a new piston rod seal lubrication system, development of an axial bearing and lubrication system as well as modification of stems and balls of the actuated control valves. Further tests are required to advance the design for commercial implementation.

An unforeseen challenge related to process product separation, dehydration and demineralisation of the renewable crude oil to an upgradable oil quality for both feedstocks as it has been found that the degassed product forms a stable emulsion that does not separate into distinct oil-, water and mineral phases by the normal gravimetric procedure developed for processing of ligno-cellulosics. A staged disc bowl centrifuge solution including solvent addition and acidification of the product emulsion was successfully developed in vendor shops, and a new disc bowl centrifuge experimental set-up capable of performing all the sequential has been established at Aalborg University.

The technical risks have been addressed in two major work packages WP2: Sorting, pretreatment & handling of urban waste streams, and WP3: Processing of Urban Waste Streams in HTL, whereas a dissemination activities were executed in a dedicated work package WP4: Dissemination. The results of the individual work packages and tasks are described in further details in the following.

5.1 WP2: Sorting, pretreatment & slurry preparation

WP 2.1 Test and validation of commercial sorting and pre-treatment technologies for household waste to meet Hydrofaction® demand

Two commercial technologies for sorting and pretreatment of the green bags from household waste have been identified. The Danish Gemidan Ecogi technology (www.ecogi.dk) and the Italian Tiger Depack technology (www.tigerdepack.com).

Pulped household waste (Green bags) has been procured from Gemidan in Frederikshavn. During the Gemidan Ecogi pulping process, the green bags are shredded and plastic, glass, stone and metals

are separated from the food waste with high efficiency (99,9+%, 95+% of bio-organics recovered in pulp). The dewatering and final contaminant removal is in the current configuration of the Ecogi process in Frederikshavn is performed using a 6 mm screen, and currently results in a bio-pulp with 17-18 wt % dry matter. A finer particle size/screen (e.g. 2-3 mm) and a higher dry matter content (20-25+ wt %) is desirable for commercial operation, but has not been possible from the commercial unit due to the small amounts required for the present project.

Not surprisingly it was found that the Ecogi Bio-pulp contains contaminants and fibres with a size up to 6 mm. Whereas 6 mm may be acceptable for a commercial Hydrofaction[®] plant, it is too large for the 6 mm inner diameter process pipes used in the pilot plant at Aalborg University. Removal of the contaminants by filtering of the bio-pulp through a finer filter was attempted, but this pathway failed as the filter load was high and bio-pulp proved to be too viscous for efficient filtration.

Tests of wet hammermilling of the Ecogi bio-pulp as received followed by subsequent concentration/dewatering in leased pilot facilities at vendors (Euromilling + Flowcom) was successfully tested, and the biopulp was thereby successfully converted into a homogeneous, pumpable feed slurry with a dry matter content in the neighbourhood of 30 % DM. However, further optimization and integration of the Hydrofaction process plant with Ecogi technology is required as the organic lost to the water effluent from the decanter centrifuge was too high (about 1/3 of the total organics). The produced feed slurry has been processed in the pilot plant in Aalborg as described below under task 3.2.



Figure 1: Modified hammermill at Euromilling Test Facility.

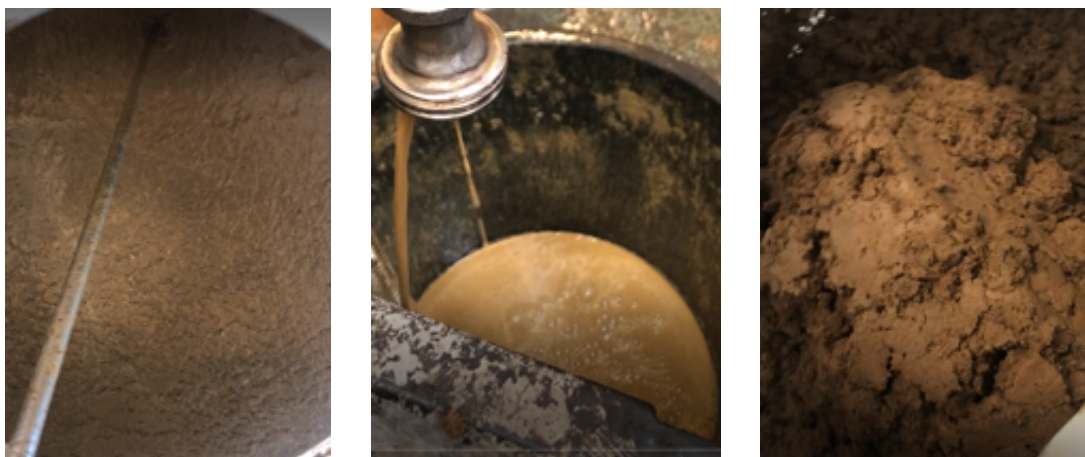


Figure 2: Biopulp after hammermilling (Left), wet fraction from decanter (middle), dry fraction from decanter (right).

WP2.2 Test and validation of slurry preparation from waste water treatment sludges & sorted household waste

Sewage sludge samples withdrawn from various locations of the waste water treatment plant has been procured from the waste water treatment plant at Aalborg Øst and tested both in laboratory and pilot plant tests.

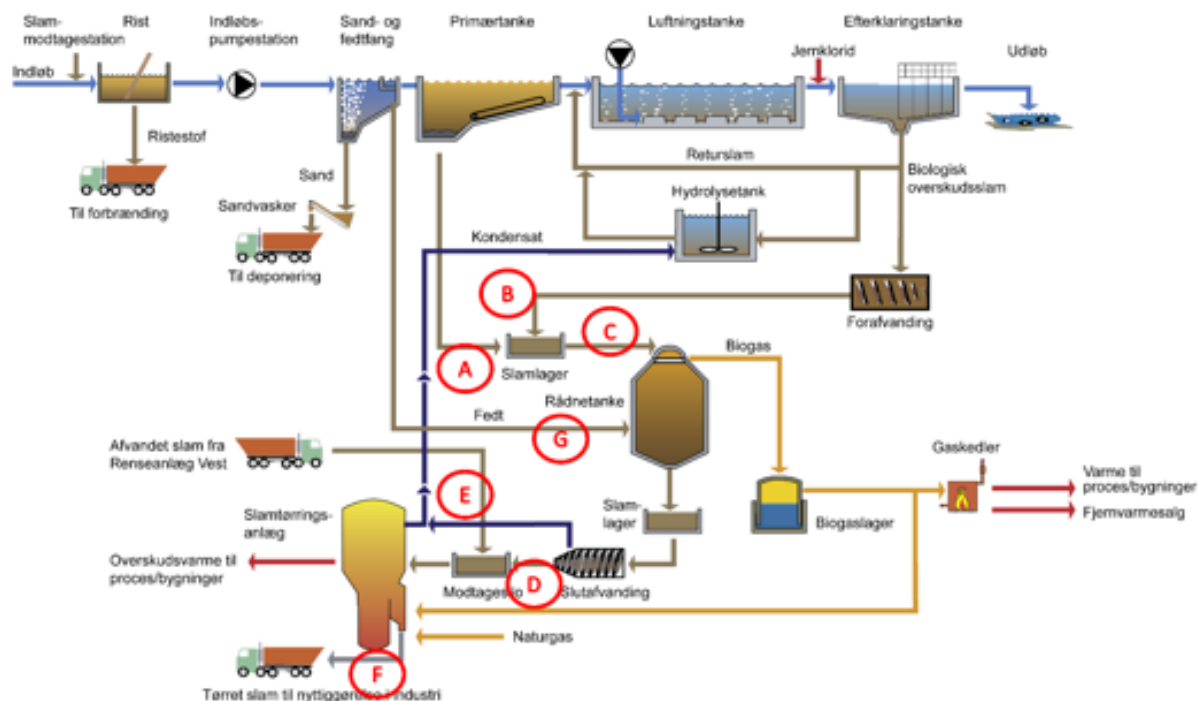


Figure 3: A simplified process flow diagram of Aalborg Rensningsanlæg Øst with indications of sludge sampling points.

Sample name	DM	Ash Dry basis	pH
	%	% DM	-
Sludge A - Primary sludge	4.4	19.9	5.6
Sludge B – Dewatered surplus sludge	5.1	22.7	6.1
Sludge C – Digester feed	4.6	20.0	5.8
Sludge C, Farsø – Dewatered digester feed	23.0	23.3	5.5
	24.9	21.0	5.9
Sludge D – Dewatered & digested sludge East	27.4	31.0	7.2
Sludge E - Dewatered & digested sludge West	26.8	31.5	7.9
Sludge F – Dried digested sludge	91.9	73.1	
Sludge G – Fat	7.6	22.2	

Table 1: Basic analysis of different sludge types sampled.

Homogeneous and pumpable feed slurries have been produced from sludges C, E and F with and without recirculation of water phase concentrate from task 3.3. Further feed slurries of mixtures of wood and sewage sludge with wood concentrations 25 wt % and 75 wt % have been prepared. The sludges have been processed in the pilot plant as described under Processing of Urban Waste Streams in HTL.

WP 2.3 Prototype tests of high pressure pump and pressure let down system to minimize wear

Potential wear, in particularly the high pressure pump and the pressure let down system due to the high mineral content of urban waste feedstock such as sewage sludge, represents a critical technical risk for commercial exploitation. Hence, prototypes of the high pressure pump and the a pressure let down pump designed for minimization of wear and an experimental set-up for wear testing of prototypes of high-pressure pump (HPP) and pressure reduction units was established. The capacity of the experimental set up is circa 500 kg slurry per hour. A schematic process flow diagram is shown in figure 4.

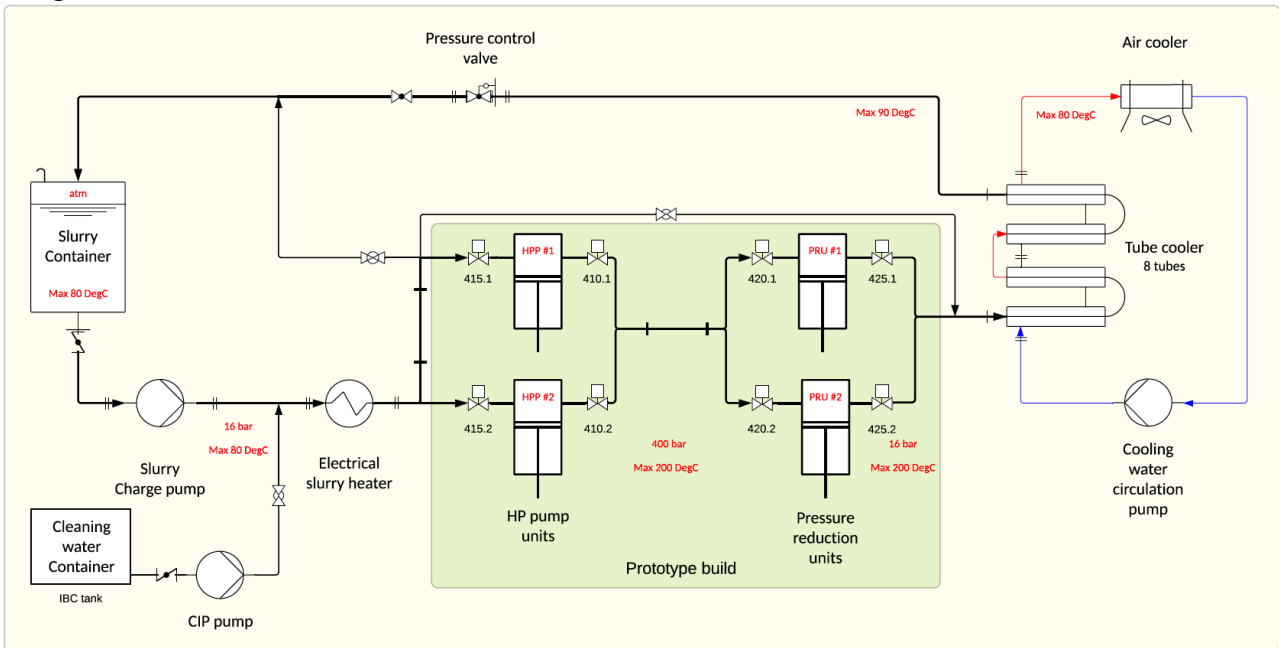


Figure 4: Process Flow Diagram of experimental set-up for wear testing of urban waste feed.

The experimental system comprises slurry container, slurry charge pump, an electrical slurry heater, the high pressure pump and the pressure reduction pump and a sludge cooler that cools the depressurized before returning it to the slurry container.



Figure 5: Photos of experimental set-up for wear testing of high pressure pump & pressure reduction pump.

Pump design

The high pressure pump and the pressure let down pump prototypes are identical and comprises two low stroke speed hydraulically driven pistons with air driven actuated valves (410.1, 410.2, 415.1, 415.2, 420.1, 420.2, 425.1, 425.2) for charging and discharging of the slurry as shown below in figure 6. Each piston further comprises piston positioner for detection of piston position and also used for measuring the volume flow. Further each piston is equipped with pressure sensors before and after the actuated valves

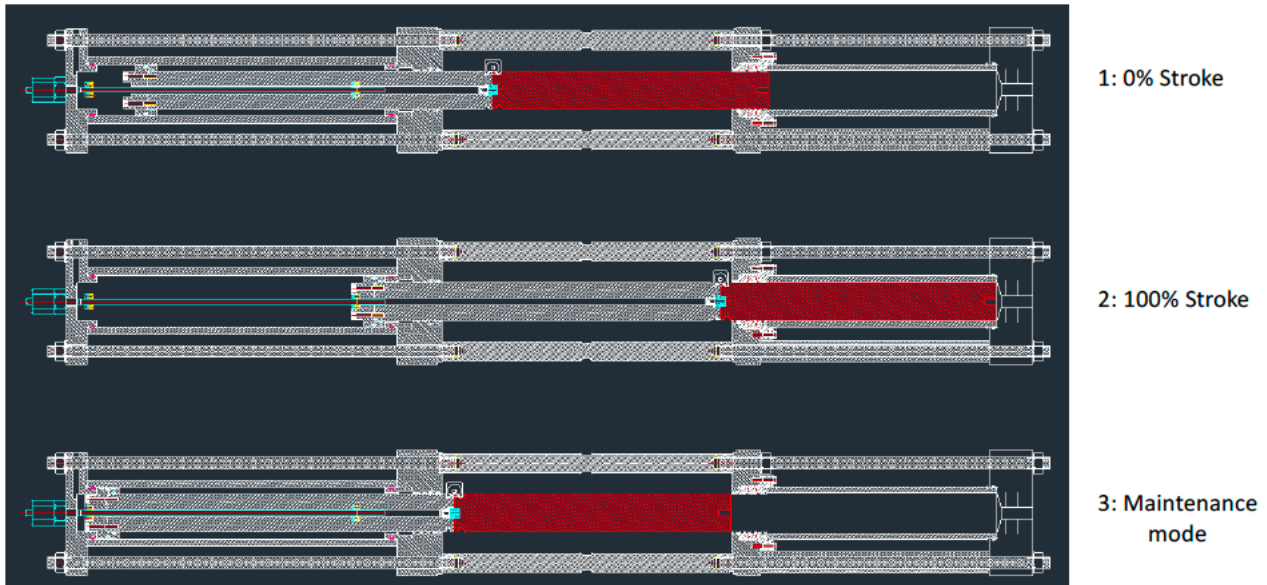


Figure 5: Principle of high pressure pump/pressure let down pump.

At 0% stroke the pump is filled with slurry from the charge pump and ready to pressurize media but before opening the outlet valve, the pressure inside the pump must be equal to pressure in process to avoid DP across outlet valve when opening. This is to decrease wear of valve seals.

At 100% stroke the pump is getting ready to receive slurry for charge pump, but prior to opening inlet valve the piston is retracted just enough to equalize pressure to charging pressure to reduce wear. A commercial pump is envisioned to comprise an additional piston and to allow for easy and safely replacement of seals during operation without having to dismantle the entire pump. An electrical switch is present to indicate maintenance position which also locks inlet and outlet valves in closed position.

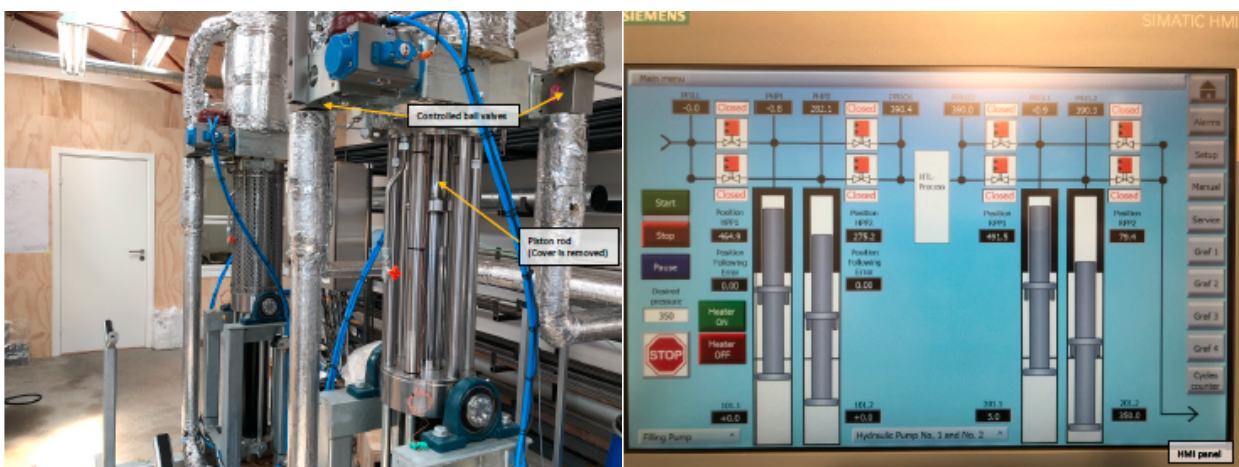


Figure 6: Photo of the high pressure pump (left), and screen dump of the pump control in the control system.

Test results

The pumps have been tested for more than 50.000 pressure depressurization cycles on sewage sludge. As foreseen wear due to the high mineral content of sewage have proven to be an issue and has required various optimizations and developments during the course of project:

1. Lubrication of piston rod seals

Piston rod seals (see figure 7) are normally lubricated by the media. However, water and sewage sludge proved not to provide sufficient lubrication of the piston rod seals on both the high pressure pump and the pressure let down pump, and this lead to pump failure. A solution with a stuffing box with grease to the lubricating oil ports was installed. However, though this solution proved to work initially the viscosity of the grease was too high to ensure sufficient refill of the grease over time. Hence, the grease was replaced but a lubricating oil that flows to the lubrication ports from a small container. Since the installation of this lubrication system no issues have been observed on the piston rod seals.

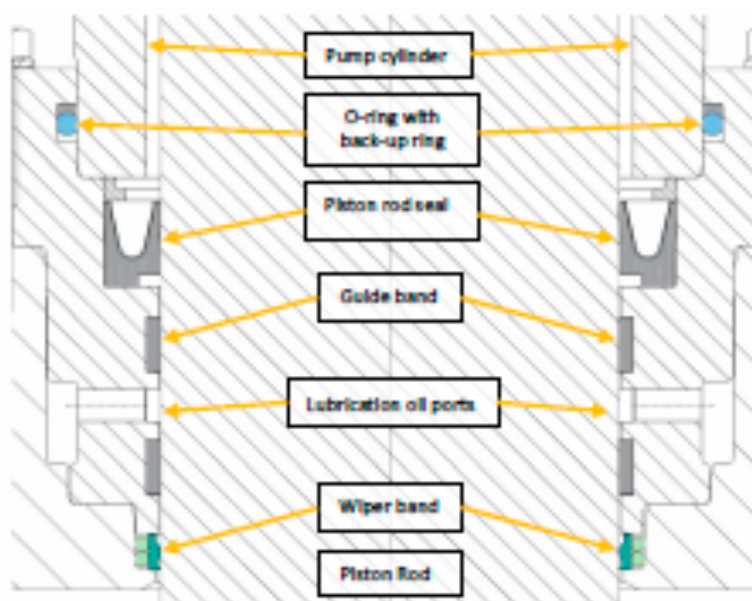


Figure 7: Piston rod seal principle.

2. Rebuild of actuated control valves

The actuated control valves quickly showed issues with an increased slip/stick torque to turn the valves during operation. It was found that issues were that trust washers (see figure 8) are being worn out even after a limited number of cycles. Various trust washer materials including AMPCO45, Ampcoly83 and PEEK CR30 were tested without success. Hence, it was decided to rebuild the valve gland (pos. 7) by inserting an axial bearing to replace the original trust washer. The axial forces are thereby transferred from the ball stem (pos. 1) to the bearing via the sleeve (pos. 9). The packing gland is equipped with grease nipples for easy lubrication of the axial bearing (pos. 6). The new design has reduced the slip/stick torque by more than an order of magnitude, and have proven to work for more than 50.000 pressurization/depressurization cycles.

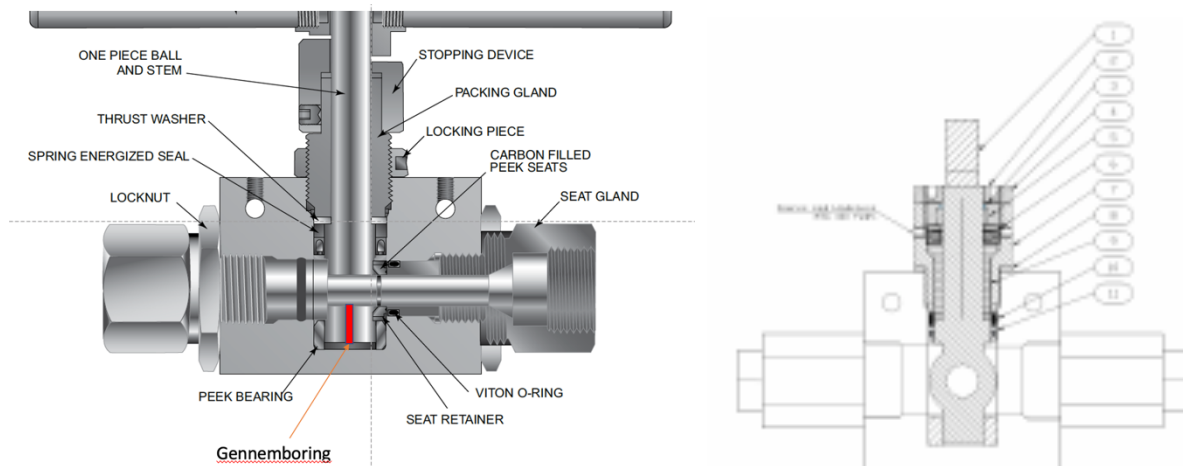


Figure 8: Principle of original actuated ball valve with trust washer (left) and new activated ball valve with axial bearing.

3. Further validation required to advance to commercial scale

The activated control valve design was intended to minimize wear by avoiding high velocities through the valves, when opening for charging. Though significantly reduced velocities, this has not been possible with the current valve design as the stem and ball of the ball valves used are hollow. This hollow volume will be pressurized, released and equalized when opening for charging, and this may result in local high velocities and related wear over time. Hence, full advantage of the low wear pump design is still to be obtained.

Further scale up issues to commercial scale is difficult as ball valves with a bore diameter of 1+” at the design pressure of 400 bar at a temperature of 125+ °C are not readily available and yet to be identified.

Hence, shear seal valves as shown in figure 9 below are considered, which doesn’t have the disadvantages of the current ball valves are considered. However, test of such valves in the prototype set-up for a prolonged period is required before a commercial pump can be released.

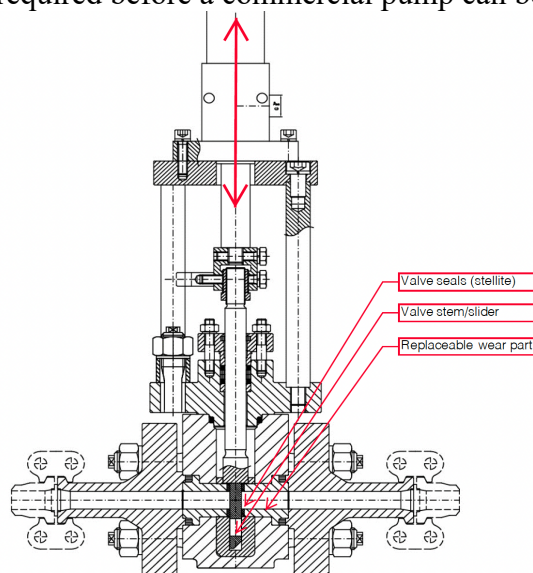


Figure 9: Principle of shear seal valve.

5.2 WP3: Processing of Urban Waste Streams in HTL

The pilot plant at Aalborg University has been intensively used for processing of and optimization of operating protocols urban waste streams produced in WP 2 so as to produce product samples for detailed analysis, establishment of preliminary mass- and energy balances and validating of the destruction of pathogens, medical traces (endocrine disruptors) and microplastics. The pilot plant is shown in figure 10 and a process flow diagram is shown in figure 11.



Figure 10: The pilot plant at Aalborg University.

The processing of the urban waste streams with high inorganic content has taken its starting point Steeper Energy's operating protocols optimized for ligno-cellulosic feedstocks such as forestry residue i.e. conversion pressures and temperatures of approximately 335 bar and 400 °C.

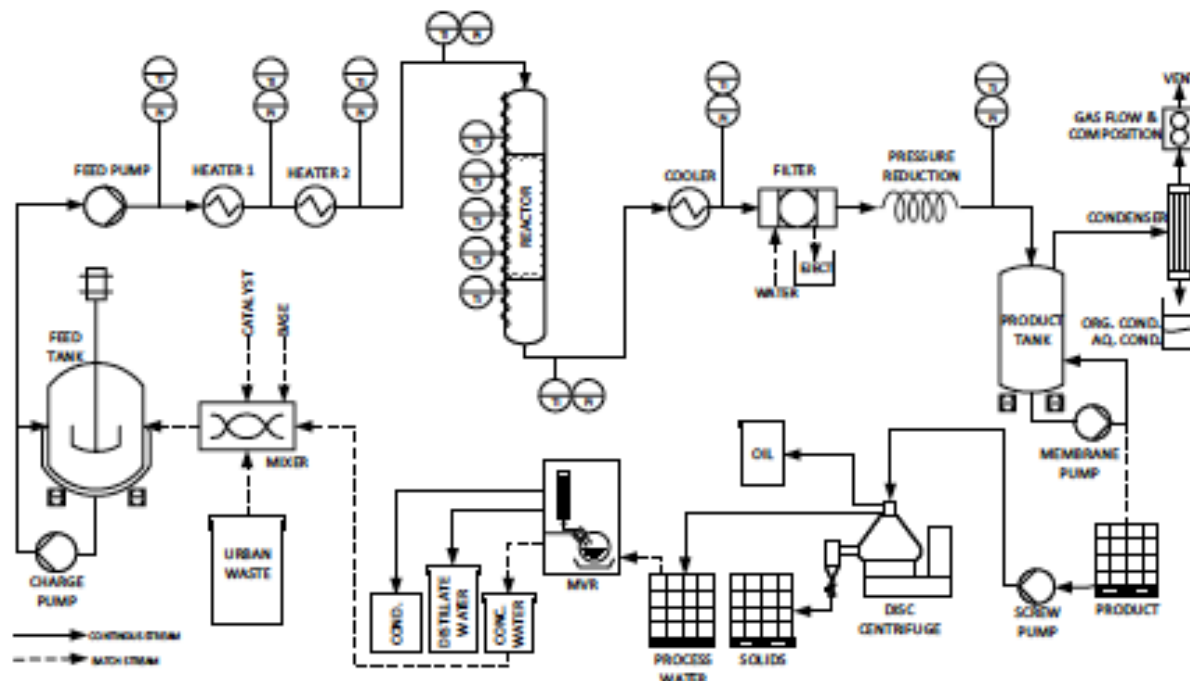


Figure 11: Process flow diagram of the pilot plant at Aalborg University.

The urban waste feedstock enters the plant in the mixer where it is mixed with other slurry ingredients such as homogeneous catalyst and caustic for pH adjustment, and eventually recycled water phase concentrate from the MVR recovery unit (external). Oil recirculation to improve rheological properties of the slurry and the conversion chemistry is normally part of the ligno-cellulosic recipe, but are not applied for Urban Waste Feedstocks as the slurries would become too dilute due to the very high moisture content of these feedstocks. The feed slurry is loaded into feed tank from where it is continuously fed to the plant by the charge pump. The feed slurry is pressurized to 320-335 bar by the feed pump and heated to a reaction temperature in the range 350-410 °C two serially connected heaters, before entering the reactor zone. Subsequently the reactor effluents are cooled before entering a filtration unit and depressurized in a capillary system prior to degassing and separation of the reactor effluent.

Sewage sludge is a living biological material that contains pathogenic bacteria and vira e.g. hepatitis and Covid-19 and others. Hence, handling and working with sewage in general have required a number of initiatives to be implemented for safety reasons including vaccination of all personnel, area classification into contaminated and noncontaminated areas, procedures to avoid cross contamination (e.g. change of clothes and shoes), disinfection of contaminated areas and a high level of personnel hygiene. In order to further minimize the personal contact with the sludge a mink fodder wagon (The “sludge wagon”) has been leased and modified. The sludge wagon allows for collection of up to 1200 kg sludge at the waste water treatment plant, transport to the pilot plant and conveying to the feed slurry mixer without direct personnel contact with sludge.



Figure 12: Left: Collection of sludge at the waste water treatment plant using the sludge wagon, Right: Conveying sludge at the pilot plant at Aalborg University.

The overall strategy for of the high inorganic content urban waste feedstock has been to carry the inorganics all the way through the process to the separation system without sedimentation. In order to do so the flow rate was successfully increased by approximately 50 % compared to the normal operating conditions used for woody biomass. Further a new reactor prototype was successfully designed, constructed and commissioned, and has allowed for an increased availability of the plant.



Figure 13: New reactor installation at the pilot plant at Aalborg University.

The major processing challenge observed for all urban waste streams has been separation of the degassed product mixture. Product separation, oil dehydration and demineralisation to an upgradable oil quality is, for wood processing, performed by a three stage solvent - and acid assisted gravimetric procedure. It has not been possible to apply this separation procedure as the degassed product mixture for all the feedstocks tested forms a stable emulsion that does not separate into distinct oil-, water- and inorganic phases within a reasonable time by gravimetric means. The stable emulsions are

believed to be caused by specific surfactants being formed by these feedstocks and/or the significantly different oil to ash ratio compared to lignocellulosic feedstock (1-2 vs. 30).

Hence, significant efforts have been invested in developing a new separation solution suitable for products from urban waste feedstocks. Separation activities have included solvent addition, acidification, filtration and different types of centrifugation at vendor shops, and in leased equipment. A working solvent- and acidification solution has been developed by replacing the three gravimetric separators in the normal separation system for wood by three phase disc bowl centrifuges. The solvent- and acidification assisted centrifuge solution has been proven to reduce the mineral content by 97-99 %. A new experimental set-up for this purpose comprising a disc bowl centrifuge modified for ATEX operation and capable performing all the sequential steps has been purchased and commissioned.



Figure 14: Separation tests at GEA Facility, Oelde, Germany



Figure 15: New disc bowl centrifuge set-up for product at Aalborg University.

Recovery and recycling of catalyst and water soluble organics from the separated process water while purifying the effluent water to a dischargeable quality constitute an important part of the Hydrofaction® process, but the so-called recovery unit is not installed at the pilot plant at Aalborg University. Hence, the recovery unit has been tested both in laboratory tests and in pilot scale in a falling film evaporator equipped with mechanical vapor recompression which makes the recovery very energy efficient. The pilot tests included separating the process water into a water phase concentrate comprising the alkaline catalysts and high boiling water-soluble organics, and a distillate water effluent stream, and recycling the concentrate to the slurry preparation step in 4 consecutive cycles as further described below under mass- and energy balances. A key difference observed compared to wood processing is the presence of ammonia in the first part of the distillate. About 70 % of the nitrogen in the sewage ends up in the process water and particularly in the form of ammonium salts. Whereas the water phase was concentrated 5 times for recycle the tests also resulted in operational challenges related to a high gas production due to ammonia being evaporated from the concentrate, which resulted in problems with entrainment of droplets of concentrate to the distillate stream. However, more than 80 % of the nitrogen in the form ammonium salts in the water phase may be recovered by stripping it off prior to entering the recovery unit as described under nutrient recovery potential.



Figure 16: Left: Pilot test of recovery unit at Envotherm, Middle: Product sample with a water phase on the top and a bottom oil sand emulsion on the bottom, Right: Water effluent (distillate phase) after recovery unit.

5.2.1 Urban waste feedstocks processed in the pilot plant

The urban waste feedstocks described in section 5.1. WP2: Sorting, Pretreatment & Slurry preparation has been processed in the pilot. An overview of the different feedstock tested is shown table 2. As indicated in the table most efforts have been directed undigested sewage sludge (sludge C), and bio-pulp produced from household waste by the ecogi technology. The compositions of these feedstocks are shown in table 3, and detailed product analysis under mass- and energy balances.

Feedstock	Amount processed kg	Oil Production hours	Dry matter content of feed slurry wt %	Recirc. of water phase	Comments
1. Sludge C	6885	245	19-21	Y	The undigested sludge from Farsøe has been used for both preliminary benchmark test, parameter screening tests and steady state tests with water phase concentrate recirculation performed. The degassed product mix forms a stable emulsion that does not separate gravimetrically within reasonable time. New separation process developed.
2. Sludge E	385	13	20	Y/N	Initial parametric testing. Sludge processed as received without catalyst, with catalyst and at lower temperatures. Water phase from previous run partly recycled. Pressure build up in reactors without catalyst, smooth operation with catalyst with a significant change in the H ₂ /CO observed in the gas (0,5 → 50). Operation at lower temperatures was not successful and led to viscous oil and operational problems due to blocked capillaries in pressure let down system. Degassed product mix a stable emulsion that does not separate gravimetrically within reasonable time.
3. Sludge F	50	2-3	18	N	Operation with issues such as pressure build up over reactors and blocked capillaries in pressure let down system. The dried sludge turned out to have a higher inorganic content than previously measured (73 wt% inorganic measured vs. 30-33 wt % ash in previous samples).
4. 75 wt % Sludge C + 25 wt % wood	250	10	20.5	N	Smooth operation similar to 1. Degassed product mix a stable emulsion that does not separate gravimetrically within reasonable time.
5. 25 wt % Sludge C + 75 wt % wood	250	10	20.5	N	Smooth operation similar to 2. Degassed product mix a stable emulsion that does not separate gravimetrically within reasonable time.
6. Ecogi Biopulp	4160	145	18/27	Y/N	Ecogi biopulp was hammermilled and processed as received (only hammermilled), as well concentrated in a decanter centrifuge with an without recycle of water phase. Degassed product mix forms stable emulsion that does not separate gravimetrically within reasonable time. New centrifuge separation developed.

Table 2: Overview of urban waste feedstocks processed in the pilot plant.

	Unit	Sewage, #1	Sewage, #2	Sewage, #3	Sewage, #4	Biopulp (wet)	Biopulp (de- watered)	Biopulp (wet)	Standard
ID#		NA	203	250	1033	321	318	1080	
Sampling		07.10.19	11.05.20	25.05.20	16.03.21	15.06.20	15.06.20	07.04.21	
Carbon	wt. %	52.2	50.0	50.4		52.9	55.4		ASTM D 5291
Hydrogen ^a	wt. %	7.8	7.4	6.7		6.5	6.9		ASTM D 5291
Oxygen ^a	wt. %	31.5	33.9	34.3		39.6	35.1		Balance
Sulphur	wt. %	1.0	0.9	0.9		0.3	0.3		ASTM D 1552 A
Nitrogen	wt. %	7.5s	7.6	7.8		2.5	2.7		ASTM D 5291
Ash ^b	wt. %	23.0	19.2	19.4	22.5	11.1	11.9	8.4	ASTM D 482
Phosphor ^c	wt. %	2.7	0.5	0.5		-	-		M-0071 DS 259/ICP
Chloride ^c	ppm	1470	100	60		-	-		SS-EN 15408 (mod.)
Potassium ^c	wt. %	0.1	0.1	0.1		-	-		M-0071 DS 259/ICP
HHV ^{a,b}	MJ/kg	22.9 ^d	25.4 ^d	24.7 ^d		24.7 ^d	24.5 ^d		ASTM D 240
DM ^c	wt. %	24.4 ^e	19.5	20.8	18.8	18.6	25.4 ^e	18.7	

^a correction for moisture underestimates hydrogen and overestimates oxygen content and HHV as volatile organics will be lost in moisture analysis, ^b ash given on dry basis, ^c as received basis including ash, ^d large variation observed, ^e Measured by moisture analyzer and not by drying in oven, and may not be accurate due to high moisture content

Table 3: Elemental composition and higher heating value of different urban wastes streams on dry ash free basis unless otherwise specified.

The higher heating values for both sewage sludge in the table are higher than measured on the same feedstocks in parallel laboratory studies at Aalborg University [1,2] and other literature reports [3,4]. Indications from parallel studies at Aalborg University are Higher Heating Values (HHV) in the range 21,0-22,5 MJ/kg for undigested sewage sludge and 22,5-23,5 MJ/kg for the bio-pulp [1,2]. Similar data ranges can be found in the Phyllis2 database e.g. 20-23,5 MJ/kg for sewage sludge with an average around 22,5 MJ/kg and 21-24 MJ/kg with an average around 23,2 MJ/kg for the wet organic fraction of household waste. It has not been possible to establish the cause of this deviation and validate the correct numbers within the project period as further discussed in section 5.3.1.2.

In addition to the parameters listed above pathogens, medical traces, micro- and macro plastics contents have been measured. These numbers are given under the section Pathogens, Micropollutants and Microplastics later in the report.

5.2.1.1 Rheological properties

The feed slurries all follow a non-Newtonian shear thinning behavior, where slurry viscosity is a function of the shear rate (flow rate) and the surface temperature at the heat transfer surface. The data portfolio coming out of continuous campaigns at the pilot plant provides, besides oil yields and product distributions, also critical design data on e.g. slurry viscosity for heat exchanger design. The pressure drops be converted into slurry viscosities. Selected raw data from the urban waste slurries with different dry matter contents are given in Figure 17.

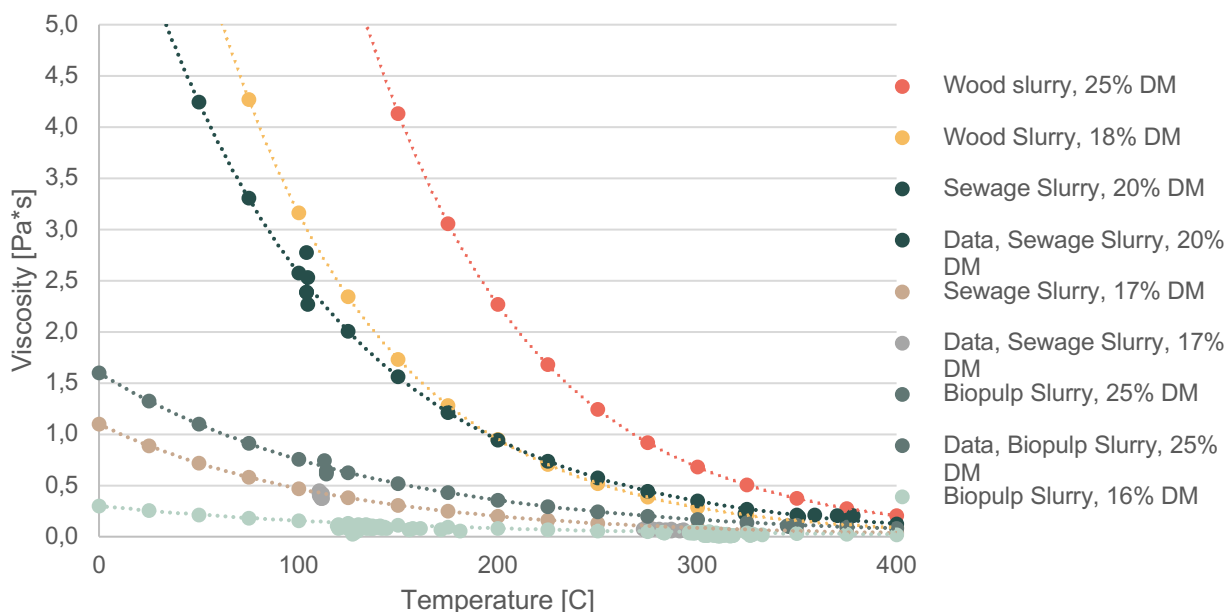


Figure 17: Slurry viscosity raw data and fits from urban waste and wood slurries at different dry matter contents. The data are all corrected to a shear rate of 200 s^{-1} .

The data indicate that independent of feedstock type, there is an exponential relation between viscosity and temperature of the heating surface. Further urban waste slurries in general are characterized by a lower viscosity than forestry slurries.

5.2.1.2 Mass-and Energy balances

Two focus urban waste streams have been selected for establishment of physical-chemical data on feed stocks, products from the process and effluent streams as well as preliminary mass-, elemental and energy balances. In addition to these, the recovery potential of phosphorous and other nutrients and the destruction of pathogens, micropollutant and microplastics during the experimental campaigns have also been investigated. The physical-chemical data of the focus urban waste streams were given above in Table 3, and the results from the various campaigns are presented below.

Sewage Sludge

A campaign was performed on undigested sewage sludge from the waste water treatment plant at Farsø (Sludge C) with recirculation of a concentrate of water phase from the previous cycle produced in the Envotherm recovery unit to the next cycle. The operating pressure for the conversion was in the range 320-335 bar the conversion temperature in the range 390-400 °C. Potassium carbonate was added as catalyst to the first cycle and make up potassium carbonate was added to each cycle so as keep the potassium constant at approximately 2,8 % during the experimental run. Further make-up sodium hydroxide was used to control the pH of the process water after separation to around 8.5.

The conversion data for Hydrofaction® of sewage sludge campaign are listed in Table 4, including mass, energy and a comprehensive list of elemental balances. The data represents average values of four mass balances, two from 3rd cycle using the Sewage #2, and two from 4th cycle processing Sewage #3 in Table 3. Table 5 lists the composition and energy content of the gaseous product from these mass balances.

		Slurry	Sewage	Oilsand	Lights	Gas	Eject	Water	Sum
Mass balances									
Total (ar)	wt.%	100%	90.7%	12.5%	0.2%	3.3%	2.2%	81.4%	99.5%
Total (db)	wt.%		100%	44.4%	0.9%	18.1%	3.2%	31.9%	99%
Organic (daf)	wt.%		100%	29.1%	1.0%	23.1%	1.2%	43.3%	98%
Energy & Elemental balances									
Energy	%		100%	44.5%	1.4%	4.4%	1.7%	33.6%	86%
C	wt.%		100%	45.4%	1.4%	12.9%	1.7%	26.0%	87%
H	wt.%		100%	9.9%	0.2%	0.8%	2.1%	90.7%	104%
N	wt.%		100%	23.3%	0.4%	0.0%	1.1%	66.9%	92%
S	wt.%		100%	35.0%	2.7%	7.1%	2.2%	13.9%	61%
O	wt.%		100%	5.3%	0.0%	3.2%	2.0%	90.8%	101%
Inorganics ^a	wt.%		100%	74.2%	0.0%		7.9%	10.4%	93%
Fe	wt.%		100%	78.0%	0.0%		13.3%	-0.1%	91%
P	wt.%		100%	93.0%	0.0%		9.0%	2.0%	104%
Ca	wt.%		100%	92.7%	0.0%		9.2%	-0.1%	102%
K ^a	wt.%		100%	27.8%	0.0%		3.1%	56.7%	88%
Al	wt.%		100%	80.6%	0.0%		8.5%	0.0%	89%
Mg	wt.%		100%	108.9%	0.0%		10.6%	-0.6%	119%
Na ^a	wt.%		100%	49.9%	0.0%		5.9%	22.2%	78%

^a including catalysts added to slurry

Table 4: Mass, energy and elemental balances of Hydrofaction[®] oil production, based on average values of the 3rd and 4th cycle runs MB20-014, MB20-015, MB20-018 and MB20-019.

Component	vol.%	vol.% [*]	wt.% [*]	HHV MJ/kg	Standards
CO ₂	77.5%	82.0%	89.0%	0.00	SS-ISO 6974
CO	0.3%	0.3%	0.2%	0.02	SS-ISO 6974
CH ₄	3.4%	3.6%	1.4%	0.79	SS-ISO 6974
C ₂ H ₄	0.8%	0.9%	0.6%	0.30	SS-ISO 6974
C ₂ H ₆	1.3%	1.4%	1.0%	0.54	SS-ISO 6974
C ₃	2.3%	2.4%	2.6%	1.32	SS-ISO 6974
C ₄	2.6%	2.7%	3.9%	1.91	SS-ISO 6974
MeOH	0.1%	0.1%	0.0%	0.01	SS-ISO 6974
ETOH	0.0%	0.0%	0.1%	0.02	SS-ISO 6974
Acetone	0.3%	0.3%	0.4%	0.14	SS-ISO 6974
H ₂	5.5%	5.8%	0.3%	0.41	SS-ISO 6974
H ₂ S	0.4%	0.4%	0.3%	0.06	SS-ISO 6974
O ₂	0.4%				
N ₂	1.7%				
Total	96.6%	100.0%	100.0%	5.52	

^{*}Air free and normalized

Table 4: Steady state gas composition for the dry product gas. The gas analyses were performed by the certified analysis laboratory at SP Technical Research Institute of Sweden, Sweden. Based on the given composition, the elemental content of the gaseous product is 32.5 wt.% C, 2.2 wt.% H, 0.0 wt.% N, 0.3 wt.% S and 65.0 wt.% O.

As seen from Table 4, the total mass balances (ar: as received including water, db: total mass balance on a dry basis i.e. both organics and inorganics, daf: dry ash free basis = organic balance without inorganics) all have a closure of approximately 100%. However, whereas most of the balances have a reasonable closure the carbon and energy balances only close to 87 and 86 %, respectively. Hence, follow-up screening tests have been performed in 2. quarter 2021 for validation of oil yields and evaluation of different operating parameters including non-catalytical processing and subcritical

conditions. The key operating conditions and performance indicators are compared for the different processing conditions in Table 5.

		Sludge II	Sludge III	Sludge IV
Processing Conditions				
Catalysts	-	Yes	No	Yes
Rec. Concentrate ^a	-	Yes	No	Yes
Reaction Temperature	C	400	400	350
Reaction Pressure	Bar	320-330	320-330	250
Performance Indicators				
Oil yield (daf)	wt. %	30.2 (±2.7)	34.7 (±2.0)	32.9 (±0.9)
Gas yield (daf)	wt. %	23.2	13.0	10.2
Gas HHV	MJ/kg	5.5	4.5	2.1
Oil HHV (daf)	MJ/kg	37.4	36.5	36.1
Energy recovery				
Oil	%	45,9-58,5 ^b	51,0-60,3 ^b	48,6-58,1 ^b
Gas	%	5,2-6,1 ^b	2,4-2,7 ^b	0,9-1,0 ^b
MCR (daf)	wt. %	11.4	15.6	14.9

^a from Sludge II campaign, ^b the high value of the ranges is based on an assumed HHV of 21 MJ/kg for the sewage sludge

Table 5: Operating conditions and key performance indicators from processing of sewage sludge at different conditions.

As seen from Table 5, the oil yields are of the same magnitude for the different operating conditions, although the oil yields without recycled concentrate and catalyst and subcritical conditions comes at the expense of a lower oil quality. Notable are also the significantly lower gas yields without catalyst and at subcritical conditions, and even at the Sludge II the gas yield and higher heating value are also significantly lower than normally observed at same conditions for forestry residues of 41 % and 7,8 MJ/kg.

However, the oil yields and energy yields obtained for sewage sludge are in all cases lower than expected and lower than obtained in a micro-batch laboratory study by Aalborg University in a parallel project at similar conditions with water phase recirculation [1]. The reason for this may be the missing carbon in the balance. This is believed to be due to insufficient recovery of low boiling organic compounds from the water phase during the separation and concentration of the water phase. If these this hypothesis is correct the oil yield, energy recovery in the oil and carbon efficiency by circa 1/3, whereby the results would be close to those measured by Aalborg University in another study. However, it has not been possible to validate this within the project period.

Ecogi Biopulp

Biopulp was tested as feedstock in preliminary once-through pilot runs i.e without recirculation of water concentrate. Both wet (as received) and dewatered (by decanter centrifuge) biopulp were processed of 18.3 % and 25.4 % dry matter, respectively. The differences in composition of the biopulp before and after dewatering were shown in Table 3. Both types of biopulp were processed at conditions similar to the Sludge II campaign i.e. 320-330 bars pressure and a temperature of 390-400 °C using 2,5 wt % potassium carbonate and 4.0 wt % sodium hydroxide added as catalysts. The degassed products didn't separate spontaneously. However, it was possible to separate and demineralize the products using the solvent and acid assisted

Steady mass balances were established for each Biopulp type. Table 6 lists the conversion data for wet bio-pulp as received, while Table 7 represents the conversion of dewatered bio-pulp.

		Slurry	Biopulp	Solids	Oil	Lights	Gas	Eject	Water	Sum
Mass balances										
Total (ar)	wt.%	100%	95.0%	3.9%	13.7%	0.1%	3.4%	1.6%	77.4%	100%
Total (db)	wt.%		100%	8.8%	47.1%	0.3%	19.2%	2.9%	18.3%	97%
Organic (daf)	wt.%		100%	3.8%	48.7%	0.3%	19.3%	1.9%	29.4%	103%
Energy & Elemental balances										
Energy	%		100%	5.7%	62.0	0.5%	5.0%	4.6%	12.7%	91%
C	wt.%		100%	4.5%	58.9%	0.4%	12.3%	3.0%	24.4%	104%
H	wt.%		100%	2.9%	14.4%	0.1%	1.1%	1.5%	82.5%	103%
N	wt.%		100%	4.1%	43.2%	0.2%		3.0%	84.2%	135%
S	wt.%		100%	4.1%	57.6%	1.2%		2.7%	26.2%	92%
O	wt.%		100%	3.0%	8.9%	0.0%	3.0%	0.9%	84.5%	100%
Inorganics ^a	wt.%		100%	15.0%	10.2%	0.0%		3.5%	55.9%	85%

^a including catalysts added to slurry

Table 6: Mass, energy and elemental balances of MB20-026 reflecting once-through Hydrofaction[®] of wet biopulp. For this table balances refer to the biopulp that constitutes about 95% of the slurry. The Gas composition for this mass balance is assumed identical to MB20-027.

		Slurry	Biopulp	Solids	Oil	Lights	Gas	Eject	Water	Sum
Mass balances										
Total (ar)	wt.%	100%	95.0%	4.8%	20.2%	0.1%	4.8%	1.6%	68.5%	100%
Total (db)	wt.%		100%	8.7%	47.3%	0.3%	20.0%	2.2%	13.6%	92%
Organic (daf)	wt.%		100%	4.1%	49.3%	0.3%	22.7%	1.4%	24.1%	102%
Energy & Elemental balances										
Energy	%		100%	10.7%	59.8-%	0.5%	3.8%	3.4%	9.6%	88%
C	wt.%		100%	5.1%	55.5%	0.4%	12.0%	2.1%	17.4%	93%
H	wt.%		100%	3.7%	20.8%	0.1%	1.1%	1.6%	74.9%	102%
N	wt.%		100%	5.1%	49.6%	0.2%	0.0%	2.0%	52.9%	110%
S	wt.%		100%	5.1%	60.2%	1.3%	0.0%	2.3%	18.7%	88%
O	wt.%		100%	3.7%	15.0%	0.0%	4.7%	1.0%	79.4%	104%
Inorganics ^a	wt.%		100%	16.7%	12.7%	0.0%		3.1%	42.9%	76%

^a including catalysts added to slurry

Table 7: Mass, energy and elemental balances of MB20-027 reflecting once-through Hydrofaction[®] of dewatered biopulp. For this table balances refer to the biopulp that constitutes about 95% of the slurry.

Table 8 lists the gas composition from processing of the dewatered biopulp containing 22 vol.% hydrogen.

Component	vol. %	vol. %*	wt. %*	HHV MJ/kg	Standards
CO ₂	71.5%	72.8%	93.9%	0.00	SS-ISO 6974
CO	0.3%	0.3%	0.3%	0.03	SS-ISO 6974
CH ₄	1.2%	1.2%	0.6%	0.32	SS-ISO 6974
C ₂ H ₄	0.5%	0.5%	0.4%	0.19	SS-ISO 6974
C ₂ H ₆	0.9%	0.9%	0.8%	0.43	SS-ISO 6974
C ₃	0.8%	0.8%	1.1%	0.55	SS-ISO 6974
C ₄	0.8%	0.8%	1.4%	0.66	SS-ISO 6974
MeOH	0.0%	0.0%	0.0%	0.00	SS-ISO 6974
EtOH	0.2%	0.2%	0.3%	0.08	SS-ISO 6974
Acetone	0.0%	0.0%	0.0%	0.01	SS-ISO 6974
H ₂	22.0%	22.4%	1.3%	1.88	SS-ISO 6974
H ₂ S	0.0%	0.0%	0.0%	0.00	SS-ISO 6974
O ₂	0.2%				
N ₂	0.8%				
Total	99.2%	100.0%	100.0%	4.15	

* Air free and normalized

Table 8: Steady state gas composition from MB20-027 (dewatered biopulp). The gas analyses were performed by the certified analysis laboratory at SP Technical Research Institute of Sweden, Sweden. Based on the given composition, the elemental content of the gaseous product is 29.3 wt.% C, 2.2 wt.% H and 68.5 wt.% O.

The data for the both types of biopulp are very similar in terms of conversion data and elemental distribution as shown in Table 6 and Table 7. The gas yields are similar to the gas yield obtained in the Sludge II campaign. Oil yields approaching 50 wt.% was obtained from both wet and de-watered biopulp, and is exiting and exceed expectations. Further, preliminary tests with aqueous product recirculation have proven to increase the yield beyond 50 wt.% by reducing the loss of organics to the process water. However, whereas most balances have closures close to 100 %, the energy balances and the indicated energy recoveries in the oil are relatively poor. As indications are that the higher heating value of the crude oils produced from the bio-pulps have heating values around 38,7 MJ/kg as presented under product characterization higher energy recoveries in the crude oils are expected. The most likely cause of the relatively low energy recoveries in the oil is that the measured values of the bio-pulp feedstocks are too high as discussed above in relation to Table 3. Using the 23,2 MJ/kg measured by Aalborg University in a recent paper [2] on the same bio-pulp results in an energy recovery of 83 %, which is close to the reported energy recovery of 84,4 % from microbatch experiments presented in the paper, but without water phase recirculation.

5.2.2 Batch experiments

Batch experiments of various catalysts candidates for liquefaction of biopulp were conducted in an attempt to reduce the heteroatom content to a level to enable spontaneous gravimetric separation. All experiments were performed with biopulp as the feedstock. However, since the biopulp was collected from the plant in three different moments, the name “biopulp 1” refers to the first batch of biopulp, “biopulp 2” to the second batch and “biopulp 3” to the third. The biopulp was processed “as received” without adding any water nor pre-drying the biomass. The effect catalysts tested were potassium hydroxide, sodium hydroxide, potassium carbonate, sodium carbonate, zero valent iron, raw bauxite residue (raw BR, also named raw red mud), reduced bauxite residue (BR), nickel nitrate hexahydrate, and calcium oxide. The catalyst loading was set to 10% (i.e. the weight of the catalyst corresponds to 10% of the dry biopulp feed). In case of iron, an additional experiment was performed with 20% catalyst loading (exp. 8). All experiments were performed in the subcritical range. For the experiments with alkali catalysts, nickel-based catalyst, and calcium oxide, the reaction temperature and residence time were respectively set to 350 °C and 15 minutes. A lower temperature and a longer residence time (300 °C and 30 min) were instead adopted for the experimental runs with iron-based catalysts (exp. 7, 8, 9, 10). Moreover, two experiments (exp. 1 and exp. 6) were done at the described process conditions but without any catalyst in order to establish reference values. Table 9 provides an overview of the HTL batch experimental activities.

	Feedstock		Catalyst		Temperature (°C)	Time (min)
	Type	Dry matter (wt%) _{db}	Type	Loading (wt%) _{db}		
1	biopulp 1	17	No catalyst	-	350	15
2	biopulp 1	17	KOH	10%	350	15
3	biopulp 1	17	NaOH	10%	350	15
4	biopulp 1	17	K ₂ CO ₃	10%	350	15
5	biopulp 1	17	Na ₂ CO ₃	10%	350	15
6	biopulp 2	16	No catalyst	-	300	30
7	biopulp 2	16	BR-Fe(0)	10%	300	30
8	biopulp 2	16	BR-Fe(0)	20%	300	30
9	biopulp 2	16	Raw BR (Fe ₂ O ₃)	10%	300	30
10	biopulp 2	16	Reduced BR (Fe ₃ O ₄)	10%	300	30
11	biopulp 3	17	Ni(NO ₃) ₂ ·6H ₂ O	10%	350	15
12	biopulp 3	17	CaO	10%	350	15

Table 9. List of the HTL batch experiments performed using biopulp as feedstock with details on the process conditions and catalyst used.

5.2.2.1 Iron catalyst synthesis from residue

Red Mud or bauxite residue (BR) is an industrial waste stream generated from processing of bauxite to aluminum by the Bayer process. It contains Fe_2O_3 (around 50%), SiO_2 , TiO_2 , and CaO metal oxides, and was investigated as a potential replacement of the alkali based catalysts used in Hydrofaction[®]. The BR was tested “as-received”, but was also reduced to enhance its activity, mainly by conversion of iron oxides to an Fe(0) state. Biopulp (BP) was used for the carbothermic reduction by its microcarbon residue for the synthesis of the BR-Fe(0) catalyst. The synthesis was performed by mixing BR with the biopulp and heated to 700 °C in a quenching furnace under N_2 atmosphere. For comparison purposes, reduced bauxite residue (RED-BR) was also synthesized by reducing dry BR in a stainless steel batch reactor at 400 °C under H_2 pressure for 30 min.

Table 10 shows the results of the batch experiments. Compared to the blank tests and alkali catalysts (hydroxides and carbonates), the iron based catalysts and CaO resulted in a higher biocrude yield and a slightly lower oxygen content relative to the references, but in all cases significantly lower yields and higher oxygen content than obtained in the pilot plant, and spontaneous gravimetric separation was not obtained.

Experiment	Yield	C	H	N	O	H/C	O/C	ash	HHV	ER
No catalyst	30.7	73.9	9.8	3.4	12.9	1.58	0.13	0.5	36.2	52.0
KOH	31.3	75.1	10.0	4.4	10.5	1.59	0.10	4.3	37.1	54.3
NaOH	26.3	74.4	10.1	4.3	11.2	1.62	0.11	1.1	36.9	45.4
K_2CO_3	35.0	75.7	10.3	4.9	9.1	1.62	0.09	1.5	37.9	62.0
Na_2CO_3	32.9	74.3	10.2	4.8	10.7	1.64	0.11	1.6	37.1	57.0
No catalyst	38.6	75.8	10.5	4.0	9.7	1.65	0.10	0.6	38.2	62.2
BC-Fe(0) 10%	43.7	76.4	11.1	4.1	8.4	1.72	0.08	1.4	39.4	72.7
BC-Fe(0) 20%	42.1	76.0	10.6	3.9	9.5	1.66	0.09	0.7	38.5	68.4
Raw BR	39.0	75.4	10.9	3.7	10.0	1.72	0.10	1.9	38.7	63.7
Reduced BR	32.3	74.8	10.5	3.4	11.3	1.67	0.11	0.1	37.8	51.3
$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	33.6	75.6	10.2	4.1	10.1	1.60	0.11	0.8	37.7	53.4
CaO	46.2	77.0	10.0	4.8	8.2	1.54	0.08	4.9	38.1	74.2

Table 10. Oil yields, elemental compositions, atomic H/C and O/C ratios, ash contents, calorific values (HHV), and energy recoveries (ER) of the biocrudes obtained from liquefaction of biopulp. Values are reported on dry ash-free basis (daf).

5.2.3 Product characterization

The products from the Hydrofaction[®] process have been subjected to detailed analysis, and the results are presented in the following. The main products are visualized in Figure 18.



Figure 18: Photos of products from Hydrofaction[®] of sewage sludge. Left: Solid product. Middle: sewage oil. Right: Water effluent.

5.2.3.1 Oil

Table 11 provides oil assays and distillation profiles for representable oils derived from sewage sludge and biopulp. The properties for forestry derived oil are given for comparison.

Property	Unit	Sewage Oil ID 641	Biopulp Oil ID 725	Forestry Oil	Standard
Elemental composition (daf):					
Carbon	wt. %	77.4	80.1 ^b	81.4	ASTM D5291
Hydrogen	wt. %	9.8	10.3 ^b	8.7	ASTM D5291
Nitrogen	wt. %	3.7	4.0 ^b	0.10	ASTM D5291
Sulphur	wt. %	0.8	0.9 ^b	0.01	ASTM D1552 A
Oxygen	wt. %	8.2	5.6 ^b	9.8	By difference
H/C mole-ratio (daf)	-	1.51	1.53	1.28	Calculated
HHV (daf)	MJ/kg	37.4 ^a	38.7 ^a	38.6	ASTM D240
Mineral content	wt. %	0.17 ^a	< 0.1 ^a	< 0.01	ASTM D 482
Metals	ppm	901			M-0071 DS 259
Micro carbon residue (daf)	wt. %	11.4 ^a	7.9 ^a		ASTM D4530
Water content	wt. %	0.99	2.4 ^a	0.8	ISO 6296
Density @ 15 °C	kg m ⁻³	999.0			ASTM D4052
Density @ 40 °C	kg m ⁻³			1057.2	ASTM D4052
Kinematic viscosity:					
@ 40 °C	mm ² /s	141.9		17360	ASTM D445
@ 50 °C	mm ² /s	81.5			ASTM D445
@ 60 °C	mm ² /s			1545	
@ 100 °C	mm ² /s	12.4			Calculated
Total acid number	mg KOH /g	67			ASTM D664 A
Pour point	°C	-3		24	ASTM D97
Flash Point	°C	36 ^a		59	ASTM D7236

^a measured by Steeper Energy ^b measured by Aalborg University

Table 11: Characteristics of Hydrofaction® oils produced from urban waste streams. All analyses of the oils were performed by the certified laboratory Uniper, Sweden, unless otherwise specified.

As seen from Table 11, the nature of the waste feedstock affects the corresponding oil quality. The urban waste oils contain relatively high amounts of nitrogen, sulphur and inorganics, which is also inherent to the respective feedstocks. The higher amount of proteins, lipids and fats in sewage and biopulp compared to forestry residues results in a higher molecular H/C ratio and a lower micro carbon residue compared to forestry derived oil.

5.2.3.2 Gas

Volatiles carried with the gas product are condensed to an aqueous and an organic condensate fraction before the dried gaseous product is measured in a gas flow meter along with the online analysis of CO₂, H₂, CH₄, CO and O₂ concentrations. Detailed gas compositions are analyzed by SP Technical Research Institute, SE by sampling in dedicated gas bags, and are shown in the tables 3 and 8.

As mentioned above indications are the Urban Waste Streams produces approximately 50 % less gas on a dry ash free basis than lignocellulosic feedstocks like forestry residues at similar process conditions. Further the higher heating value is 30 to 45 % lower than forestry residues.

The major constituent of the gas is CO₂, which may be recovered as liquid CO₂ at 60-70 bars pressure by simple two stage condensation. The liquid CO₂ is a convenient form of CO₂ that may be further converted into e-fuels. Hence, the technology is P2X ready.

5.2.3.3 Process Water

The development of key parameters with recycle of water concentrate is shown in Figure 19 and 20, and a detailed analysis of the process water as well recycled water concentrate and distillate from Recovery unit tests are shown in Table 12.

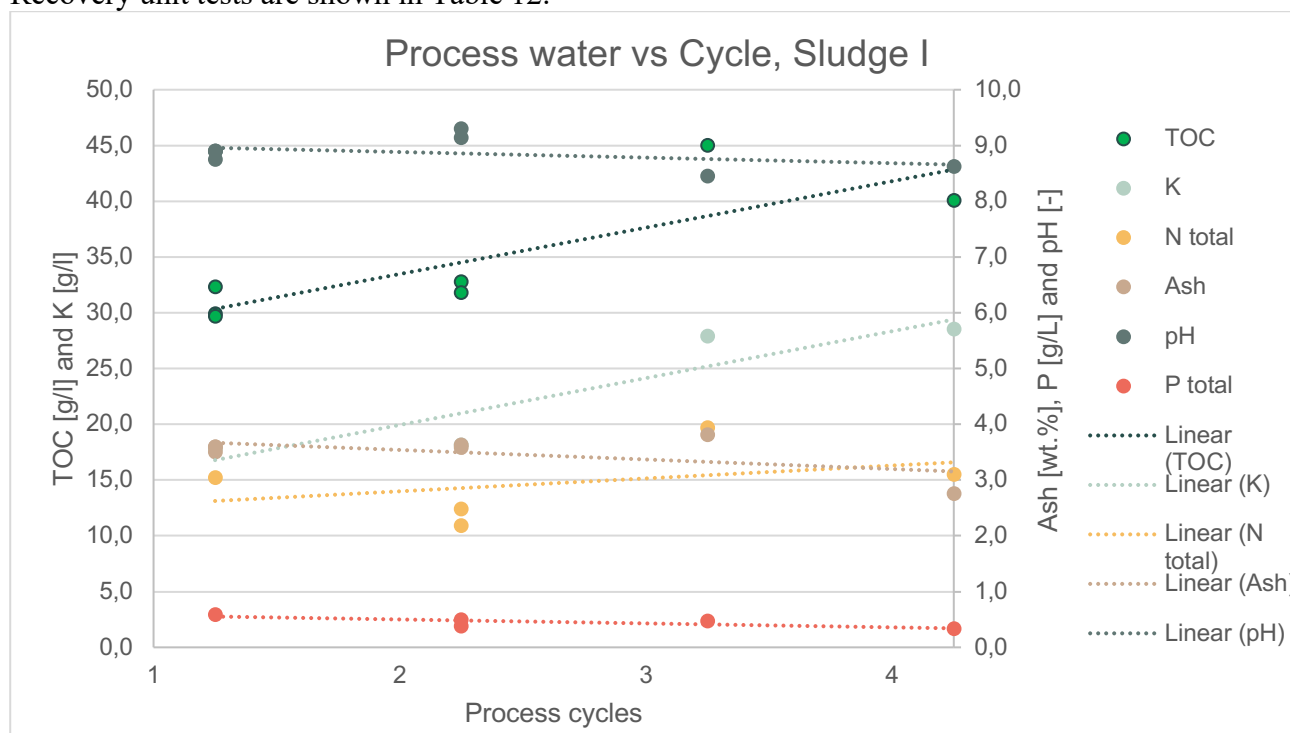


Figure 19: Key parameters in the process water vs. cycle number

As seen from Figure 19, the TOC concentration in the process water increases over the first 2-3 cycles and stabilizes, whereas other parameters are relatively constant. A similar trend is observed for the major organic compounds in figure 20.

The detailed analysis of the process water, water concentrate and the water effluent in table 10 shows that the TOC concentration is reduced by 92,8 % while recovering the homogeneous catalyst and the majority of the water-soluble organics. The organic acids stay in the water concentrate that the residual TOC in the distillate whereas low boiling organics like C1-C4 alcohols and ketones constitute the majority of the residuals. The TOC content in the water effluent is too high for direct discharge. However, the recovery is performed without flashing off ammonia & low boiling prior to the recovery unit. Hence, it is believed that water effluent can meet environmental requirements for discharge to sewer or recipient. Alternatively, a polishing step such as a membrane bioreactor is required prior to discharge.

Compounds	Process Water	Water concentrate	Water distillate (Water Effluent)
Cycle	4	4	4
pH ^a	8,64	6,91	9,79
TOC ^a - g/L	46	138	3,31
Density - g/L	1038	1135	1040
Total Inorganics, wt %	2,255	12,38	0,0
K ^a , wt%	11,9	59,9	0,4
Total N ^a , g/l	10,75	14,98	0,325
NH ₄ ⁺ , g/l	8,895	5,06	0
Total P ^a , g/l	0,325	0,36	0
Eddikesyre/Acedic acid mg/l	24650	129000	103
Myresyre/Formic acid, mg/l	180	748	<10
Propionsyre/Propionic acid, mg/l	8700	36000	65
Smørsyre/Butyric acid, mg/l	8150	0	30
Isosmørsyre/Isobutyric acid mg/l	2750	30800	<10
Isovaleriansyre/Isovaleric acid, mg/l	1175	8100	10
Valeriansyre/Valeric acid, mg/l	2900	0	23
Benzene, mg/l	0,1485	4200	0,00065
Toluene, mg/l	3,375	8800	1,49E-03
Ethylbenzene, mg/l	1,8475	0,0139	1,59E-03
o-xylene, mg/l	0,0936	0,158	2,97E-03
m+p-xylene/P-Xylene, mg/l	0,1815	0,054	2,08E-03
Napthalene, mg/l	0,0349	0,00361	3,44E-02
Methanol, mg/l	1245	0,00468	930
Ethanol, mg/l	575	0,0091	910
1-Propanol, mg/l	80	0	230
Propan-2-ol, mg/l	32	0	42
n-Butanol, mg/l	200	0	200
Isobutanol, mg/l	52,5	0	180
Acetone, mg/l	730	7	510
MEK, mg/l	300	0	80
Isobutyl methyl, mg/l	4	14	
Ethyl acetate, mg/l	0	4	<0.005
Phenol, mg/l	94	0	1,1
2.3-Dimethylphenol, mg/l	0,145	0	
2.4-Dimethylphenol, mg/l	2,95	73	
4-Chloro-2-methylphenol, mg/l	0	0,033	
4-Chloro-3-methylphenol, mg/l	0	0,65	
4-Chloro-2-isopropyl-5-methylphenol, mg/l	0	0	
2-phenylphenol, mg/l	0	0	
2.5-Dimethylphenol, mg/l	0,32	0	
2.6-Dimethylphenol, mg/l	4,95	0	
3.4-Dimethylphenol, mg/l	0,26	0,44	
3.5-Dimethylphenol, mg/l	17	0,93	
2-Methylphenol, mg/l	8,5	0,206	
Diethyl ether, mg/l	0	7,6	<0.05
3-Methylphenol, mg/l	31,5	3,3	
4-Methylphenol, mg/l	2,05	0	
3-ethylphenol, mg/l	0,205	21	
2.3.5-trimethylphenol, mg/l	1	1,4	
2.3.6-trimethylphenol, mg/l	0	0,41	
2.4.6-trimethylphenol, mg/l	1,95	0	
3.4.5-trimethylphenol, mg/l	0	3,7	
2-Naphthol, mg/l	0,031	0,16	
NH ₃ , mg/l	11150	0	0
Identified of TOC	52,4%	71,2%	51,7%
Hydrogen content / identified TOC, %	1,09%	2,58%	12,15%
Oxygen content / identified TOC, %	4,19%	13,15%	36,22%
Total Acids, mg/l	48505	217648	231
Total GC-MS	3389	138	3083
Acid Carbon / TOC, %	48,6%	71,1%	6,98%
Identified mass/TOC, %	9,5%	28,0%	100,1%
HHV (estimated), MJ/kg	2,11	5,56	0,09

Table 12: Characteristics of process water as well as the water concentrate and water condensate from recovery unit tests.

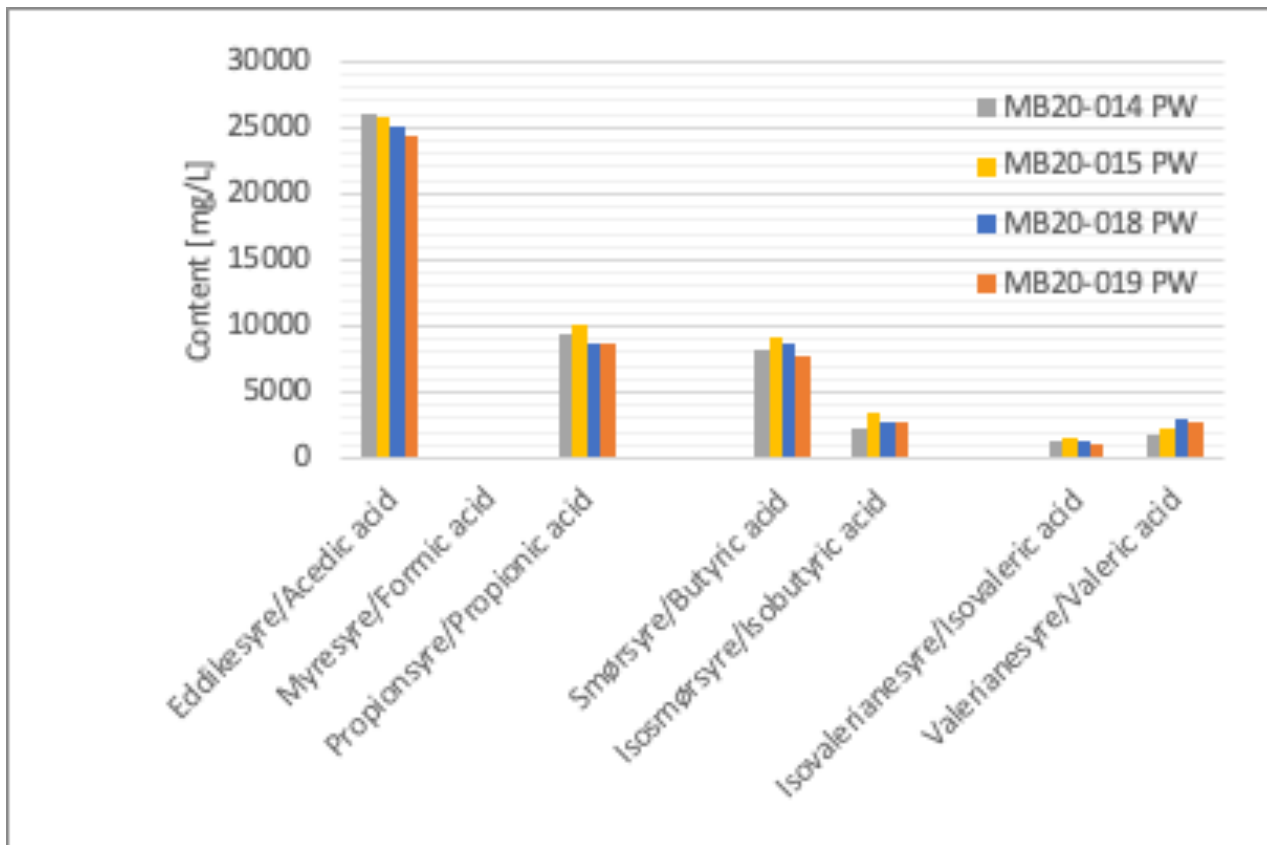


Figure 20: Organic acids content in process water vs. cycle number.

5.2.3.4 Mineral product

The Oilsand derived from conversion of sewage sludge was subjected to the two stage solvent and acid assisted centrifugation process developed thereby resulting in a crude oil with the composition presented in Table 11 and a mineral product with the composition and inorganics distribution given in Table 13. A visual impression of the mineral product is given in the left picture of Figure 18. Based on the difference in dry matter and inorganics, there is 23.4 % combustible matter in the solids. The HHV of 35.5 MJ/kg on dry ash free basis and a H/C molecular ratio of 1.45 indicates that the sewage oil that was not fully recovered during separation. It is expected that the organic content of the solid product can be significantly reduced or eliminated by optimizing the separation efficiency or added further washing/purification steps.

The major inorganic elements can be derived from the ICP elemental analysis given in Table 13. Phosphor comprise 32% of the detected inorganics, iron represents 26%, calcium 14%, potassium 9%, sodium 8%, aluminum 6% and magnesium represents 4%, which sums up to 99 %. Except sodium and aluminum, these are all critical plant nutrients, indicating that the mineral product from Hydrofaction® of sewage could be a sustainable and valuable fertilizer. Sodium and aluminum can reduce the soil quality if present in too high concentrations, but the concentrations of both are relatively low, and the presence of calcium outweighs the negative impact of sodium.

DM	Ash	HHV	HHV (daf)	C	H	N	S	O	H/C		
98.6 wt.%	75.2 wt.%	8.3 MJ/kg	35.5 MJ/kg	17.3 wt.%	2.1 wt.%	1.2 wt.%	0.48 wt.%	3.7 wt.%	1.45		
Metals by ICP [mg/kg]											
Al	P	Sb	As	Ba	Pb	B	Cd	Ca	Cr	Co	Fe
13800	78100	4.05	17	369	52	15	5.35	34000	60	5.52	64200
K	Cu	Hg	Mg	Mn	Mo	Na	Ni	Se	Tl	V	Zn
22500	320	0.8	10600	370	16	19000	47	2	2.04	23	1660

Table 13: Composition and inorganics distribution in the solid product derived from sewage sludge. Analysis carried out by certified laboratory according to DS204 (DM), ASTM D482 (inorganics), ASTM D240 (HHV), ASTM D5291 (Elemental composition) and DS259 (ICP).

Trace heavy metals are also present in the solid byproduct, and of these cadmium is of most concern as it is harmful to human health. In Denmark, there are specified limits for the concentrations of certain heavy metals in both commercial fertilizers and in biosolids from e.g. sewage sludge used for agricultural purposes. The limit values for the latter is given in Table 14 and compared to the actual concentrations in the solids derived from Hydrofaction® of sewage. For the four heavy metals Cd, Hg, Pb and Ni, a limit value is both given per kg dry matter and per kg phosphorous. However, according to the legislation it is sufficient to meet one set of limit values if all heavy metals fulfill the same set [5]. Thus, it can be concluded that the concentrations of heavy metals in the mineral product are all below the limit values for agricultural use and even for gardening use in private households.

	Limit values in Denmark		Avg. of Sewage #1, 2, 3		Solids from Hydrofaction® of Sewage	
	mg/kg DM	mg/kg P	mg/kg DM	mg/kg P	mg/kg DM	mg/kg P
Heavy Metals						
Cadmium, (Cd)	0.4	100	• 1.0	✓ 78	• 5.4	✓ 69
Mercury, (Hg)	0.8	200	✓ 0.1	✓ 11	✓ 0.8	✓ 10
Lead, (Pb) ^a	120	10000	✓ 11	✓ 923	✓ 53	✓ 666
Nickel, (Ni)	30	2500	✓ 9	✓ 769	✓ 48	✓ 602
Chromium, (Cr)	100	-	✓ 14	•	✓ 61	•
Zink, (Zn)	4000	-	✓ 544	•	✓ 1684	•
Copper, (Cu)	1000	-	✓ 79	•	✓ 325	•
Arsenic, (As) ^b	25	-	✓ 4	•	✓ 17	•
Pollutants						
Linear alkylbenzene sulfonates, (LAS)	1300		•		•	•
Polyaromatic hydrocarbons, (PAH)	3		•		•	•
Nonylphenol ethoxylates, (NPE)	10		•		•	•
Diethylhexyl phthalate, (DEHP)	50					•

^a The limit for lead is 60 mg pr. kg DM for use in private gardening, ^b The limit for arsenic only applies for use in private gardening.

Table 14: Limit values for heavy metals in waste products used for agricultural purposes [5]. The concentrations given in Table 1 are given for comparison and related to the limit values.

As a conclusion, the mineral product contains critical plant macro- and micronutrients that, if available in the right form for plant uptake, may provide an attractive direct pathway for recovery of

phosphorous and other nutrients from sewage sludge. Whereas the mineral product may require further purification and characterization of residual organic carbon containing compounds, the concentration of heavy metals is acceptable for both agricultural purposes and use in private gardening.

5.2.4 Phosphorous & other nutrients recovery potential

Phosphorous from urban feedstock processing concentrated in the mineral fraction of the oilsand product after the Hydrofaction® process with a recovery of a 93-95 % by weight as shown in the Table 4. The remaining phosphorous ends up in the aqueous phase from the Hydrofaction® process. Hence, the Hydrofaction® process has a high potential for recovery of phosphorous and other nutrients from these phases.

Phosphorous is an essential resource that are becoming limited [7-11]. Hence, both phosphorus and phosphate rock have been identified by the European Commission as critical raw materials, based on its supply risk and the economic importance for EU operators, and recovery of phosphorous constitute a key element of EU plan for a climate-neutral, resource-efficient and circular economy [11]. Urban Waste feedstock and Sewage Sludge in particular have a high concentration of phosphorous, typically 2-6 % by weight, and recycling and recovery of phosphorous is expected to be mandatory for waste water treatment plants in the future with Germany being the first country to put Sewage Sludge Regulation in place to gradually close the phosphorous cycle and reduce the dependence of phosphorous imports [9]. The regulation requires operators of larger sewage plants (population equivalent of > 50,000) to recover phosphorus from sewage sludge. To develop and optimise respectively recovery processes, the regulation provides for transition periods. Large sewage plants (population equivalent of > 100,000) have 12 years to comply with the new regulation. The transition period for smaller sewage plants (population equivalent of 50,000 to 100,000) is 15 years. By the end of 2023 the operators of sewage plant of the above sizes must have submitted their phosphate recycling concept to the regulatory authorities [9].

Hence, preliminary tests of potential pathways for recovery of phosphorous from the mineral product and aqueous phases have been conducted in addition to the detailed characterization though this activity was cut off with the modification and reduction of the original project.

The mineral product may be used directly as a fertilizer or soil improvement product (Biochar) for agriculture or gardening as described above, or may be further processed into conventional marketable phosphorous products such as struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$), Diammonium Phosphate ($(\text{NH}_4)_2\text{HPO}_4$, DAP) or hydroxyapatite ($\text{Ca}_5(\text{PO}_4)_3(\text{OH})$).



Figure 21: Photos of struvite (left), Diammonium Phosphate (middle), hydroxyapatite (right).

In addition to the phosphorous and depending of the feedstock 55-86 % of the nitrogen contained in the feedstock ends up in the process water phase prior to the recovery unit mainly as dissolved ammonia salts. Preliminary tests indicate that more than 80 % of the nitrogen content in the process water may be recovered as an aqueous ammonia solution by flashing and/or stripping prior to entering the recovery unit.

Depending of the separation conditions part of the phosphorous may leached from the mineral phase to the acid wash water phase during separation of the Oilsand intermediate product by the two stage acid and solvent assisted disc bowl centrifugation as shown in figure 22. Hence, investigations were performed to maximize the leaching of phosphorous to the acid wash water phase in the separation step using different acids, concentrations and resulting pH. As seen from left figure the leaching of phosphorous increases with decreasing pH up to a maximum concentration of about 3-3.5 g/l with Citric Acid (CA) & Sulphuric Acid (SA) performing best. However, for optimal oil and minerals separation the pH of the wash water needs to be maintained in the range 3-5 as shown in the figure to the left. Hence, a two stage process is required for efficient oil and mineral separation as well as a high recovery of phosphorous from the mineral phase is required.

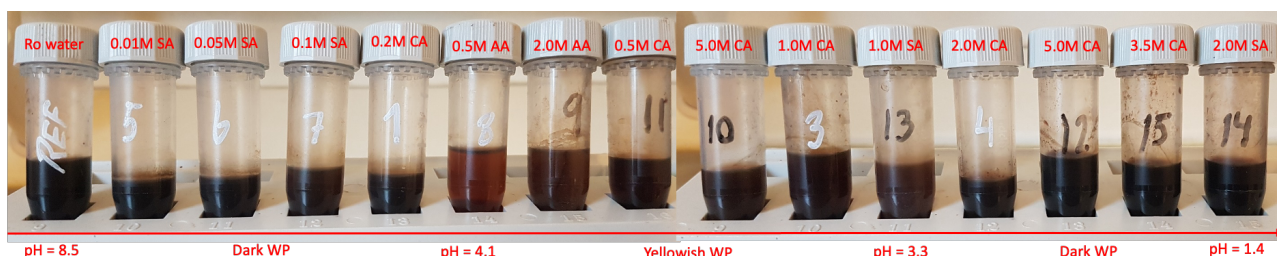
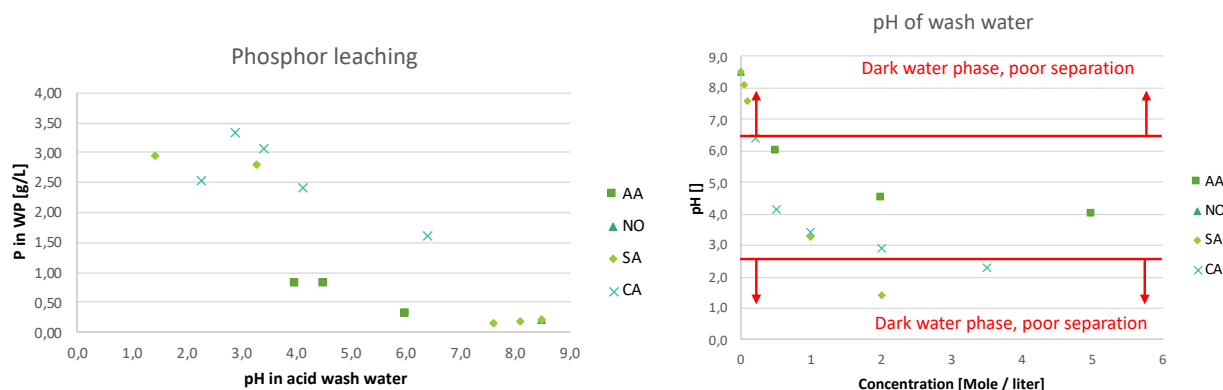


Figure 22: Concentration of phosphorous in the acidic wash water vs. pH (left), Optimal pH range with respect to separation of phases.

Potential pathways for recovery of phosphorous in the form of conventional marketable phosphorous products from the mineral product and aqueous phases by demonstrating the synthesis of Diammonium phosphate $(NH_4)_2HPO_4$, DAP & struvite $(NH_4MgPO_4 \cdot 6H_2O)$ from leaching of phosphorous from the mineral phase, as well synthesis of hydroxy apatite $Ca_5(PO_4)_3(OH)$ from process water prior to the recovery step.

Diammonium Phosphate was synthesized by mixing 50ml of acidic wash water from the separation having a P-content of 1.9 g/l was mixed with 1.7 g of MB19-028 aqueous gas condensate containing ammonia and adjusting pH to 8,8 by addition of NaOH resulting in a light brownish precipitate. 0.18 g precipitate contained 18.2 wt % (dry) P after filtration and drying.

Struvite was synthesized by mixing 50ml of acidic wash water from the separation having a P-content of 1.9 g/l was mixed with 1 g of MB19-028 aqueous gas condensate containing ammonia and 0.75 g of $MgCl_2$ adjusting pH to 8,2 by addition of NaOH resulting in a light brownish precipitate. 0.12 g precipitate contained 15.5 wt %(dry) P after filtration and drying.

The synthesis of Diammonium Phosphate from phosphorous leached from the mineral product is shown in figure 23.



Figure 23: Mixing acidic wash water with precipitation chemicals (left). Diammonium phosphate (top right) and struvite (bottom right) product after filtering and drying.

Synthesis of hydroxyapatite ($Ca_5(PO_4)_3(OH)$) from process water

Synthesis of hydroxyapatite from the process water prior to the recovery unit was demonstrated using CaO and $CaCl_2$ as precipitation chemicals. The effects of stoichiometric ratio, pH for precipitation, residence time and temperature on phosphate precipitation were investigated. 1 M NaOH and H_2SO_4 solutions were used to adjust the pH of the samples as shown in Table 15 and 16.

Phosphate recovery was determined by measuring the phosphate contents of aqueous phases before and after the treatment using HACH kit LCK 349. After the treatment, the samples were kept for 1.5 hours to settle the precipitates. The samples were then filtered with 5-13 μm filter paper, and then dried at 105 °C overnight. The precipitates were analyzed with x-ray diffraction (XRD), FTIR and scanning electron microscopy (SEM) techniques.

Sample No.	Initial pH	Final pH	Reaction time (h)	Temperature (° C)	Amount (g/L)	Phosphor Recovery (%)
1	8.7	11	2	25	0.66	25.5
2	8.7	11	2	25	6.6	88.1
3	8.7	11	Overnight	25	6.6	87.4
4	8.7	11	2	25	3.3	45.7
5	8.7	10.5	2	25	6.6	53.1
6	8.7	10.5	2	35	6.6	48.1
7	8.7	10.5	2	52	6.6	20.9
8	8.7	12	2	25	3.3	58.4
9	8.7	8.7	2	25	3.3	15.2
10	8.7	8.7	2	25	13.2	99.5

Table 15. Experimental plan followed for the phosphate recovery from HTL aqueous phase using $CaCl_2$ under different conditions.

Sample No.	Initial pH	Final pH	Reaction time (h)	Temperature (° C)	Amount (g/L)	Phosphate recovery (%)
11	8.7	11	2	25	0.37	0.6
12	8.7	11	2	25	3.67	57.7
13	8.7	10.5	2	25	3.67	18.2
14	8.7	10.5	Overnight	25	3.67	86.7
15	8.7	7.5	2	25	3.67	38.2
16	8.7	8.7	2	25	8.33	57.6
17	8.7	8.7	2	25	15	84.7

Table 16. Experimental plan followed for the phosphate recovery from HTL aqueous phase using CaO under different conditions. Figure 24 shows the process water samples under treatment for the recovery of phosphate using CaCl₂ and CaO.



Figure 24 Aqueous phase samples during the treatment process for the phosphate recovery with (1) CaCl₂ and (2) CaO.

As seen from Table 15 and 16, the phosphorous recoveries range from 15 to 99,5 % by weight with pH and the amount of precipitation chemicals the strongest factors, whereas no influence of the reaction temperature and reaction time on the recovery of the phosphate were observed. The effect of the precipitation chemicals is further illustrated in figure 25 below.

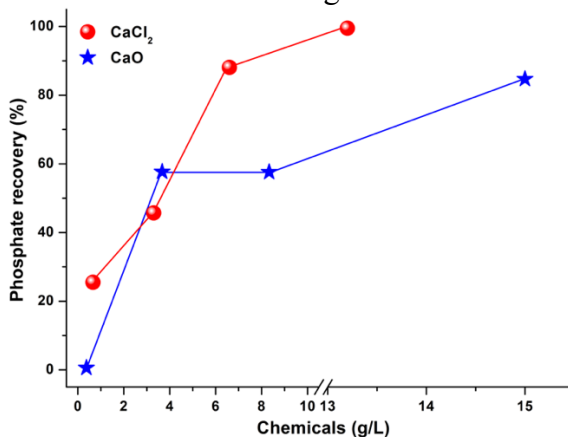


Figure 24 Phosphorous recovery with respect to chemical quantities.

The preliminary tests have demonstrated that conventional phosphorous products can be produced from the leaching of phosphorous from the mineral phase and from the process waters. pH is important for both the leaching and the precipitation step. The leaching of the phosphorous from the mineral phase after separation second stage is optimally performed using sulphuric acid at a pH around 2 as further reduction of the pH leads to dissolution of more contaminants. Similarly, a higher pH for precipitation of diammonium phosphate or struvite leads to more efficient precipitation, but more contaminants also ending up in the phosphorous product at a given stoichiometric ratio. Optimum seems to be precipitation at a pH in the range 8 to 9. Nitrogen recovered from process in the form of ammonia is sufficient to provide the ammonia for production of diammonium phosphate and struvite. Further work is required to determine the optimum.

5.2.5 Pathogens, micropollutants & microplastics

Destruction of endocrine disruptors/micropollutants (pharmaceuticals, personal care products, hormones, biocides etc), microplastics and pathogens represents unique sale points compared to competing technologies such as anaerobic digestion, which is known to have moderate to no reductions of such micropollutants and microplastics. As anaerobic digestate is typically used as fertilizer, these hazard micropollutants and microplastics may become hazards to the entire food chain, and thus an area of concern. Hence, an objective of the present project was to validate the destruction of these micropollutants and microplastics or at least validate that the water effluent and mineral/phosphorus products are free or significantly reduced in such compounds and their metabolites (breakdown products).

5.2.5.1 Destruction of pathogens

The destruction efficiency of pathogens in sewage sludge in Hydrofaction® process was evaluated using Culture-based and DNA sequencing approaches. Two sewage samples (S1, S2), and Hydrofaction® products (reactor outlet: H3, ash: H7, oil: H9 and water phase Hx) were tested using soil and tap water as control samples. For the culture-based approach, undiluted and 1:100 diluted samples were prepared using multiple solvents (MEK, THF, miliQ water) and tested in selective media plates to detect the presence of *E. coli* and coliform. While MEK and THF solvents resulted in high solubility of the Hydrofaction® samples, these chemicals significantly damage the detection plates, jeopardizing the significance of the results. When using miliQ water as solvent, expected results were observed for control samples (~200 coliform colony forming units -CFU for soil and no *E. coli* or coliform was detected in the tap water sample). However, no apparent *E. coli* or coliform forming units were visually observed in the Hydrofaction® products tested. Further dilution of the Oil and H7 sample is suggested to reduce the presence of residues in the detection plate. The sewage samples (S1 & S2) exhibit significant amounts of *E. coli* and coliform colonies that were categorized as too high to be counted and that further dilution is necessary to get accurate CFU counts.

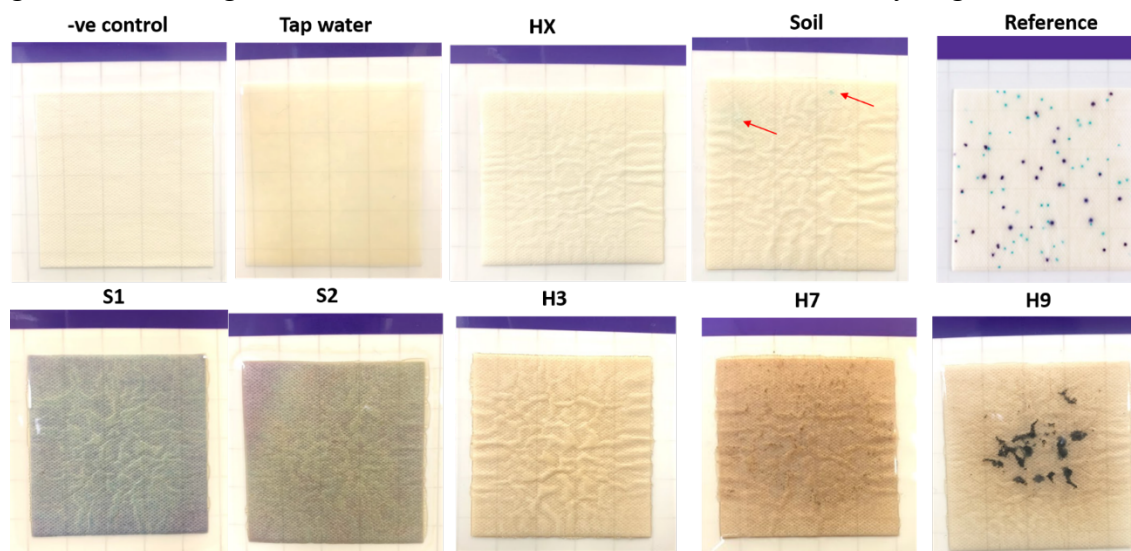


Figure 25: *E. coli*/Coliform selection MC-Media Pad®. Coliforms develop blue/blue-green colored colonies and *E. coli* develop purple-colored colonies as indicated in the reference picture. Negative control: no sample added; Soil: soil sample collected outside the Life Sciences Innovation Hub building. Tap water and Hx samples were not diluted while the rest of the samples (Soil, S1, S2, H3, H7 and H9) are in 1:100 dilutions in sterile MQ water. HX, H3, H7 & H9: no colony forming unit (CFU) detected; Soil (coliform): 200 CFU/g considering dilution; S1&2: too numerous to count (TNC). All samples were filtered with sterile 0.5 µm syringe filter and incubated at 37 °C for 48 h.

The genomic DNA extraction and sequencing approach, genomic DNA was extracted from all samples provided and, absorbance-based methods estimated its concentration and purity. Samples S1, S2 and soil exhibited the purest and highest DNA concentration, as indicated in figure 26 and table 17. No trace of genomic DNA material was observed in all Hydrofaction® products tested (detection limit 5 ng), suggesting a significant reduction of genomic DNA concentration compared to the sewage feedstock (figure 26).

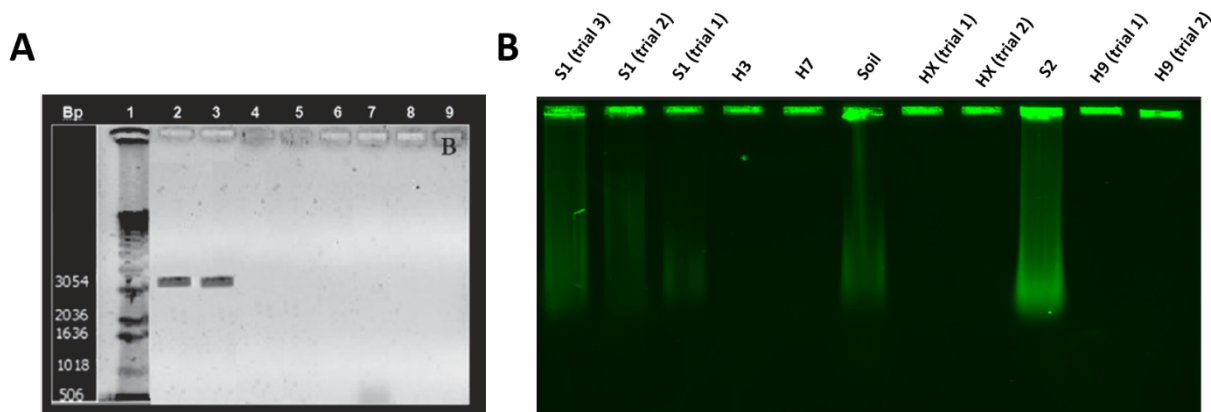


Figure 26. DNA extraction before and after HTL. **A**, agarose gel showing 95-97% removal of plasmid DNA after HTL (wells 4-9) versus the swine manure feedstock spiked with plasmid-transformed *E. coli* (wells 1-2). **B**, 0.5% agarose gel containing extracted genomic material from Hydrofaction® feedstock and downstream products. No trace of genomic material was detected in samples H3, H7, HX and H9. Highest trace of genomic material detected in S2 sample. Samples S1 and Soil exhibited the presence of genomic material but with lessen degree as judge by the well’s intensity. Agarose gel run at 70v for 40 min, stained with Ethidium Bromide (EtBr). Minimum visual detection limit of agarose gel stained with EtBr is ~5 ng of DNA/RNA.

UV Spectrometry			NanoDrop Measurements		
Sample	Dilution	Abs260 nm	Dilution	Conc. (ng/μL)	Abs 260/280
S1 (Trial1)	1:100	-	-	98 ± 3	1.2
S1 (Trial2)	1:100	0.04	-	125 ± 5	1.4
S1 (Trial3)	1:100	0.11	-	156 ± 2	1.9
S2	1:100	0.21	-	405 ± 6	1.7
H3	1:100	-	-	27 ± 4	0.9
H7	1:100	1.49	-	1503 ± 13	0.8
H9 (Trial1)	1:100	-	-	12 ± 3	1.4
H9 (Trial2)	1:100	0.06	-	15 ± 5	1.3
HX	-	0.02	-	-	-
Soil	1:100	0.15	-	96 ± 2	1.8

Table 17. UV Spectrometry: Absorbance at 260nm corresponding to the absorbance profile of samples represented in figure 3. Accurate measurement considered between 0.1-1 absorbance unit (AU). Absorbance at 260 nm (Abs₂₆₀) normally used to estimate nucleic acid (DNA/RNA) presence. NanoDrop® Measurements: commonly used absorbance-based method to estimate DNA concentration and purity in purified samples. Pure DNA considered between ~1.7-1.9 values of Abs₂₆₀/280 ratio.

5.2.5.2 Destruction of micropollutants (pharmaceutical traces)

Complex quantitative analysis of different pharmaceutical traces in sewage #2 and #3 before Hydrofaction® and of 4 different process water samples after Hydrofaction® was carried out by SGS Analytech, DK.

Table 18 lists the compounds and concentrations detected in the different samples. Due to the complex sample matrix of particularly the sewage sludge, the number of different pharmaceutical traces detected for was limited compared to the process water. The percentage of destruction is also given in Table 18, indicating how efficient the pharmaceutical traces were destroyed during pilot processing.

Compounds	3 rd Cycle			4 th Cycle			3 rd Cycle			4 th Cycle		
	Sewage #2 ng/kg	WP, MB14 ng/l	De-struction %	WP, MB15 ng/l	De-struction %	Sewage #3 ug/kg	WP, MB18 ng/l	De-struction %	WP, MB19 ng/l	De-struction %		
Atenolol	<1000					<1000						
Diclofenac	31600	<50	100%	<500	100%	28400	<500	100%	<50	100%		
Fenoprofen		<100		n.d.			n.d.		n.d.			
Ibuprofen	31700	n.d.	100%	n.d.	100%	15100	6120	59%	7530	50%		
Indomethacin		<100		<500			n.d.		n.d.			
Ketoprofen	<1000	n.d.		n.d.		<1000	n.d.		n.d.			
Naproxen	5000	<100	100%	n.d.	100%	3200	<500	100%	<1000	100%		
Paracetamol	<1000	>3000	-200%	6500	-550%	<1000	n.d.		n.d.			
Bezafibrate		<100		n.d.			<100		<500			
Etofibrate		<50		<100			<50		<50			
Sulfamethoxazol		<100		n.d.			<500		<100			
Fenofibric		<50		<500			<50		<50			
Fenofibrate		500		n.d.			<500		259			
Gemfibrozil		<50		<50			<100		<500			
Carbamazepine	5000	<100	100%	n.d.	100%	5800	1000	83%	720	88%		
Pentoxifylline		<100		<100			<500		<100			
Diazepam		<50		<100			<50		<50			
Iopamidol							n.d.		n.d.			
Amidotrizoic							n.d.		n.d.			
Iodipamide							<50		<50			
Dihydroxy... ^a		<100		n.d.			<1000		<50			
Atenolosäure		n.d.		n.d.			<100		<100			
Candesartan		<50		<50			<50		<50			
Oxazepam		<100		n.d.			<100		<100			
Metoprolol	14000	120	99%	<100	100%	12100	<20	100%	<50	100%		
Clofibrilic		<500		<500			<50		<100			
Primidon		n.d.		n.d.			n.d.		n.d.			

n.d. = not detectable, ^a 10, 11-Dihydro-10,11-dihydroxycarbamazepin

Table 18: Pharmaceutical traces detected in sewage sludge before and process water (WP) after Hydrofaction®. Analysis carried out by certified laboratory SGS AnalyTech Miljølaboratorium, Denmark.

The analysis of pharmaceutical traces presented above refer to sewage sludge, and similar analysis have not been done on the biopulp. However, compounds originating from cosmetics, inks, paper, and food preservation are also present in the biopulp and are reported to be highly bioactive, extremely persistent towards degradation and possible endocrine disruptors [6]. These compounds are also expected to be destroyed by the severe operating conditions of Hydrofaction[®]. Hence, this could be a more sustainable approach to the current use where the digestate from anaerobic digestion of biopulp, which is likely to contain persistent and harmful pollutants, when spread for agricultural purposes.

5.2.5.3 Destruction of micro-plastics

Microplastics are present in urban waste feedstocks such as sewage sludge and bio-pulp. Typical such feedstocks are digested by anaerobic digestion and the digestate is applied to agricultural fields thereby leading to undesired contamination of soils and potential introduction of microplastics to the food chain.

The hypothesis was that the severe process conditions applied in Hydrofaction[®] would significantly degrade and reduce microplastics in effluent streams thereby making Hydrofaction[®] an attractive circular solution for conversion of such feedstocks not only from energy point of view, but also from an environmental point of view.

Therefore, the fate of microplastics in urban waste streams were investigated in the Hydrofaction[®] process exemplified by sewage sludge. The main steps involved in the recovery of the Hydrofaction[®] products are illustrated for sewage in figure 27 below, and the green boxes indicated the samples analyzed for microplastics.

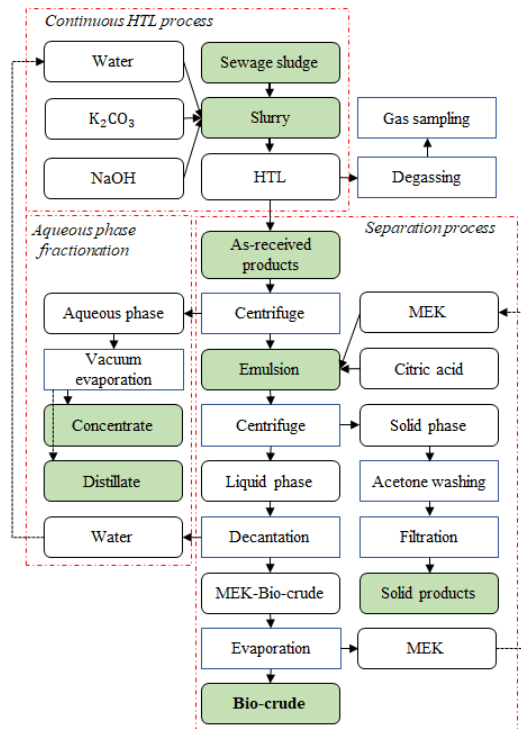


Figure 27 An overview of the Hydrofaction[®] process, product separation and aqueous phase fractionation. Microplastic extraction and quantification were performed on the phases highlighted in green.

The presence of microplastics was investigated in both feedstocks (sewage sludge and slurry) and Hydrofaction® products (as received products, concentrate, distillate, emulsion, mineral product, and biocrude). After being extracted, microplastics were quantified by Fourier Transformation Infrared Spectroscopy (FTIR). Particles of 10-500 µm were quantified using Focal Plane Array (FPA) based micro-FTIR (FPA-µ-FTIR) imaging combined with an automated analysis of the generated spectral image, while Attenuated Total Reflection (ATR)-FTIR was used for MPs >500 µm. 18 polymer types were identified: PP, PE, PEsT, PU, cellulose acetate (CA), polyvinyl chloride (PVC), polystyrene (PS), PA, pan acrylic (PAN), POM, PC, polyvinyl acetate (PVaC), acrylonitrile butadiene styrene (ABS), alkyd, acrylic, epoxy, phenoxy resin, and poly(ethyle acrylate). The most diverse polymeric composition was found in the sewage sludge where 17 types of polymer were identified, while only 11 types were identified in the other investigated products (15 types in the slurry, 3 in as-received products, 4 in the emulsion, 3 in concentrate, 8 in distillate, and 8 in solid residue). Microplastics were detected in all matrices with the only exception of the bio-crude where no microplastics was detected (see Figure 28, below). Results from particle concentration showed that MPs are mainly concentrated in the minerals (Figure 28, graph A), while results from mass concentration showed that there is a decrease in the mass concentration when comparing the Hydrofaction® products respect to sewage sludge indicating that Hydrofaction® significantly decrease the presence of microplastics.

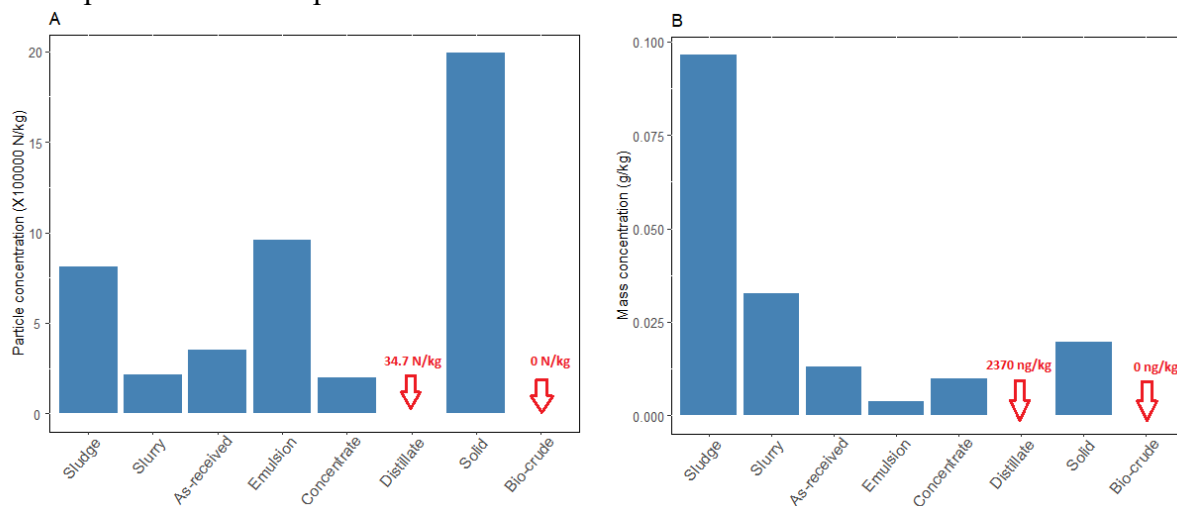


Figure 28 The concentration of microplastics in analyzed matrices: A) particle concentration and B) estimated mass concentration. The concentration is per wet weight of the sample matrices except the matrix ‘Solid’

The figure shows the polymer composition based on microplastics number (A) and mass concentration (B). The polymer composition between samples was quite heterogeneous, with the exception of sewage sludge and slurry, which were quite comparable in composition. The main polymers in sewage sludge (by particle number) were PP, PU, PE, PEsT, and Alkyd (a polyester resin modified by the addition of fatty acids and other components, and mainly used in paints) which alone accounted for the 80% of the detected polymers in sewage sludge. In terms of microplastics numbers (see graph A), PP dominated in the samples after the Hydrofaction® process. The exception was in the distillate. However, it should be observed that the total concentration in this phase was quite low, and thus uncertainty on the polymer composition was correspondingly high. In terms of mass (graph B), PP was the dominant polymer in emulsion and solid phases. This shows that PP is a resilient plastic to be destroyed in Hydrofaction®.

Some polymers (CA, POM, PVaC, PC, alkyd, phenoxy resin, poly(ethyle acrylate) were instead not detected in the Hydrofaction® products, indicating a considerable or even total degradation in the Hydrofaction® process.

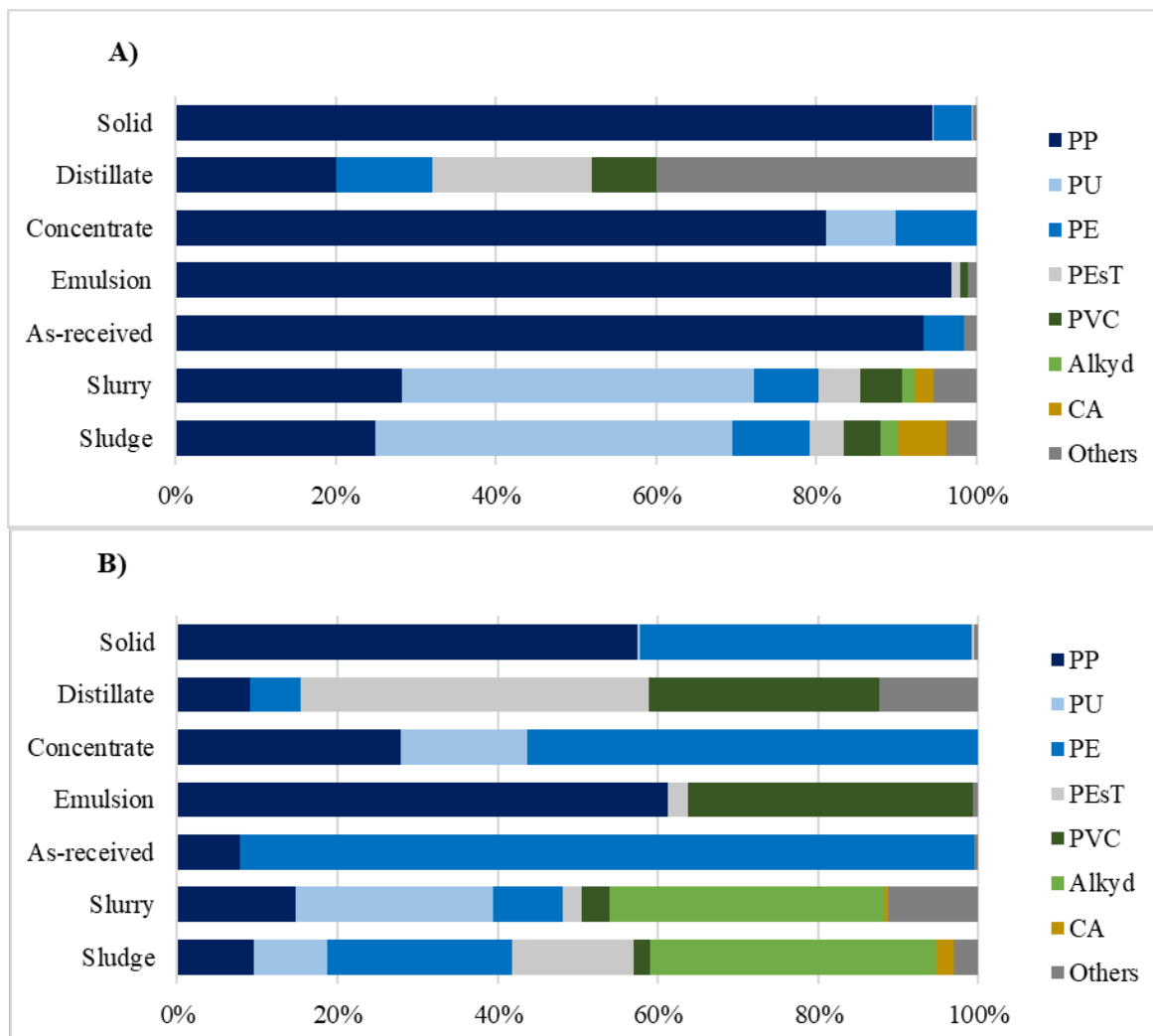


Figure 29 Distribution of the identified polymers based on: A) particle concentration and B) estimated mass concentration. ‘Others’ includes the following polymers: acrylic, PS, PA, PAN, POM, epoxy, PC, PVaC, ABS, phenoxy resin and poly (ethyl acrylate).

A reduction of microplastics of about 76 % in terms of microplastics number and 97% in terms of microplastics mass was achieved by processing of sewage sludge in the Hydrofaction® process. The difference in reduction of the number of microplastics versus their accumulated mass was caused by microplastics being smaller after the Hydrofaction® process. The Hydrofaction® process was found to decrease the particle size of the more resistant polymers, such as the PP, PE, and PU which resulted more resilient to the Hydrofaction® process.

Hence, the Hydrofaction® process is able to not only convert sludge to biocrude but also to mitigate the issue of the sludge being polluted by microplastics, creating residual products substantially free of microplastics compared to the original raw material. Similar results have been found for macro- and microplastics in biopulp in a parallel study outside this project.

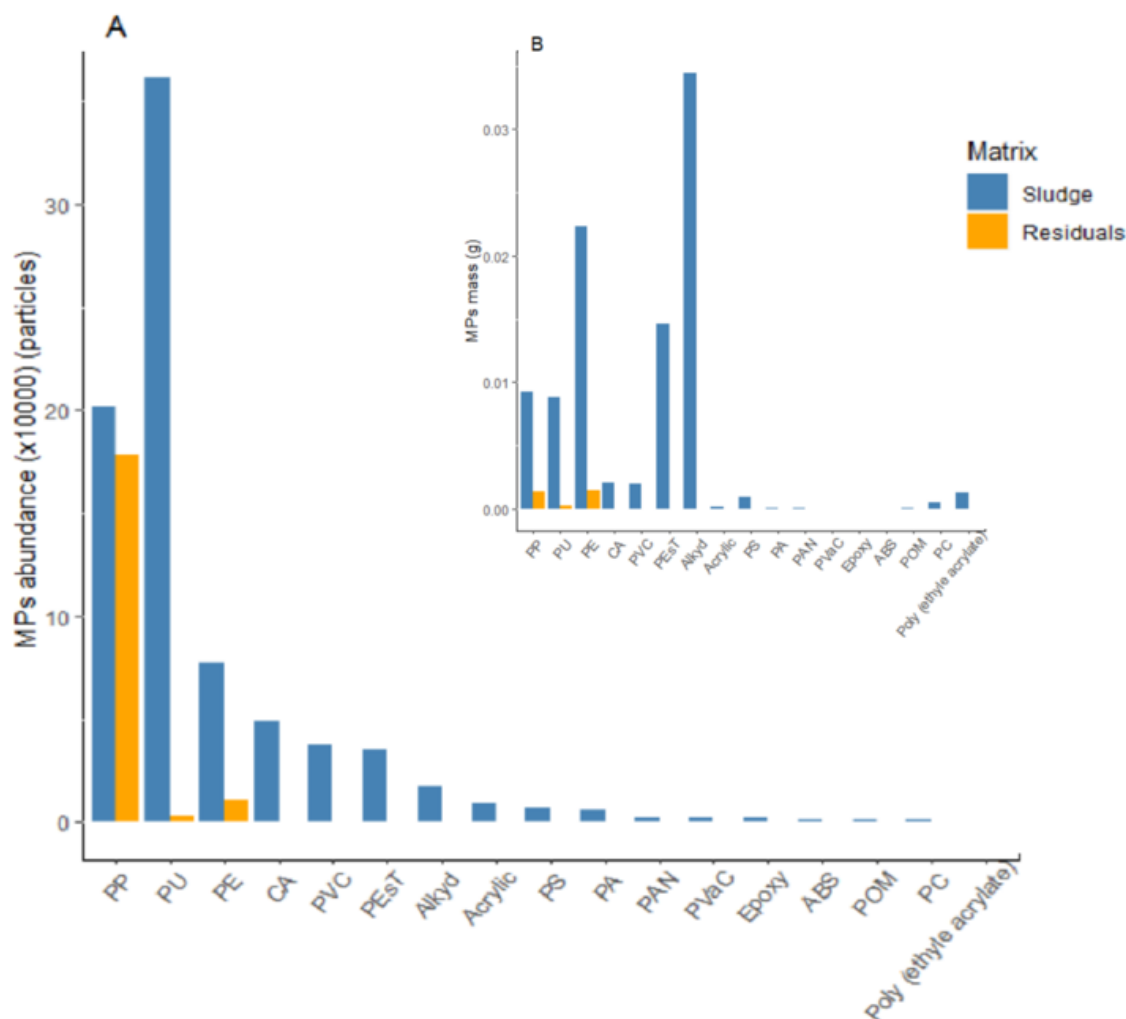


Figure 30: Microplastics load reduction over Hydrofaction® process A) by number and B) by mass.

5.3 WP4: Dissemination

Dissemination activities have been affected by the Covid-19 pandemic, but will be continued beyond the project according to the health regulations. The following list covers all dissemination activities, including presentations at workshops and conferences, and scientific output.

Presentations:

- presentation to DANVA (invited speaker for the yearly “Nytårstaffel”), January 2020
- presentation at SEGES workshop, “Produktion af bio-olie og transportbrændstoffer gennem HTL”, January 2020
- presentation to High School Talent Academy, “Fremtidens bæredygtige brændstoffer” February 2020
- presentation on HTL at IDA meeting, November 2020
- presentation “Catalytic hydrothermal liquefaction of wet municipal solid waste”
- presentation “Economic evaluation and cost reduction potentials in hydrothermal liquefaction”, PNNL workshop, January, 2020
- presentation “Introduktion til biokonverteringsprocesser”, EDC workshop, April 2021

- HTL plant visit by Consulting Engineers (COWI), June 2020
- Scientific paper submitted to the Journal of Cleaner Production, entitled “Removal of microplastics from sewage sludge by a continuous hydrothermal liquefaction process”

In addition to the above project activities Steeper Energy has participated and self-financed activities outside the project, where Urban Waste Hydrofaction has been presented as a new application under development as part of general presentations. Selected presentations include:

- “Hydrofaction – Recovering Energy from Sewage Sludge to Advanced Biofuels”, Presenter & Exhibitor at WEFTEC 2019, (Single largest annual water quality exhibition in the world), 21-25 Sept. 2019, Chicago, USA
- ” Hydrofaction - Advanced Biofuels for Long Haul Transport from Organic Waste”, Biofuels International Con. & Expo, Oct. 22.-23. 2019, Brussels, Belgium.
- “ The Path to Commercialization of Hydrofaction®”, Scaling up 2019, Nov 4.-6., 2019, Ottawa, Canada.
- Hydrofaction® - Transforming Bio-Organic Waste into advanced bio-fuels”, Virtual presentation, Bio4Fuels Days, November 18., 2020
- “Hydrofaction®: a leading technology for the efficient conversion of sustainable biomass into renewable transportation fuels”, IEA Bioenergy Workshop, Nov. 18-19th, 2019, Brussels, Belgium.
- “Panel discussion with the speakers: Industrial status and market situation; Challenges on the pathway towards commercialization of HTL; and, Next steps – cooperation possibilities – building a platform”, H2020 HTL Expert workshop, Nov. 19th, 2019.
- “Hydrofaction® A Leading Technology for the Efficient Conversion of Sustainable Biomass into Renewable Transportation Fuels“, RSB Annual Meeting (Roundtable on Sustainable Biomaterials), Berlin, Germany.
- Exhibitor at Globe Forum 2020, Feb 10.-13, 2020, Omaha, Nebraska, USA.
- ” Transforming Organic Residuals in co-processable feedstocks with Steeper Energy’s Hydrofaction®. Joint European Workshop: Co-processing as a route to deployment of advanced bio-fuel production, virtual presentation April 14.-15., 2021
- ” Hydrofaction®, transforming organic waste into advanced biofuels and other valuable resources”, US DOE-BETO Workshop on Advancing Synergistic Waste Utilization on Biofuel Feedstocks: Preprocessing, Co-products and sustainability, virtual presentation April 14.-15., 2021.
- ”Forestry and Ag Residues to Competitive SAF via Steeper Energy’s Hydrofaction®”, The Coordinating Research Council (CRC) Aviation meeting, May 3.
- “”, eFFECT Seminar Organized by Worley, May 17.-18., 2021.

Further, as an indirect result of the project Steeper Energy’s 100% owned subsidiary Steeper Energy Canada and the City of Calgary signed a “Memorandum of Understanding (MoU)” with the intent of The City and Steeper Energy to see the development of a demonstration plant at the Pine Creek Wastewater Treatment Plant in South-East Calgary, and a press release was submitted announcing the MoU, <https://steeperenergy.com/2020/07/02/memorandum-of-understanding-mou-signed-by-the-city-of-calgary-with-steeper-energy-canada-ltd>.

6. Utilisation of project results

- *Describe how the obtained technological results will be utilised in the future and by whom.*

Steeper Energy

The main objective has been to derisk central process steps related to the use of the Hydrofaction[®] process for urban waste streams.

The use of the results will form part of Steeper Energy's future product development and hence expected to come into final use with the waste handling utilities. This may be through direct sales to waste handling utilities or through an intermediate technology and service provider already established in the waste handling area as a provider to the waste handling utilities.

The separation process that form part of the project and the results of this will first of all allow for implementation of the Hydrofaction[®] process in the waste handling area, where significant content of minerals/inorganics typically is part of the feedstock. Further the ability to separate the mineral part of the output stream will allow for expanding the potential feedstock options to other areas with a similar high mineral content in the feedstock.

Aalborg University

Aalborg University is a center of hydrothermal research at global level. The project results will be used in the continued research & development of hydrothermal liquefaction technologies for advanced biofuels, and boost applied research and education carried out in this field e.g. by attracting future projects and the best students also in the future. Furthermore, the participation in the project will continued development in applied research and engineering education within energy and related fields, maintain and strengthen links to cutting edge industrial development.

Aalborg Forsyning, Renovation & Kloak, RenoNord

The project has resulted in a broader and more detailed understanding of the Hydrofaction[®] technology as part of their assessment of potential circular technologies for urban waste management with creation of high value products.

- *Describe how the obtained commercial results will be utilised in the future and by whom the results will be commercialised.*

The project has shown that the value proposition of the waste handling for the waste handling utilities is primarily a question of reaching the goals measured in terms of a sufficiently clean output to meet the legal and political requirements. The implementation of the possible new technologies and the risks and benefits achievable through these is often of secondary interest. Technology and service operators established in the field and having a value proposition that apart from delivering the technology and to some extent also operating service, that provided the results legally and politically required, has a commercial focus in competition with other technology and service operators. This is likely to be a future entry road to the waste handling market. Another possible entry road could be accessing the potential market in areas with no existing waste treatment and hence a recognised need for this

and at the same time no/lower barriers towards against existing technologies and the risk of not delivering the legal and politically required level of waste treatment.

- Did the project so far lead to increased turnover, exports, employment and additional private investments? Do the project partners expect that the project results in increased turnover, exports, employment and additional private investments?

Revenues and increased turnover from the project have yet to come, and are awaiting the Hydrofaction[®] Demonstration plant for forestry residues in Norway to get online in the fall as well as further development of the engineering design and business cases based on the project results.

However, as an indirect measure Steeper Energy has more than doubled its staff from 14 to 33 employees during the course of the project partly due to the project, and this organizational growth is expected to continue in the coming years. The staff is circa 50/50 splitted between Steeper Energy Denmark and its 100 % owned Canadian subsidiary.

- *Describe the competitive situation in the market you expect to enter.*
 - *Are there competing solutions on the market? Specify who the main competitors are and describe their solutions.*

Sewage Sludge

About 50 % of all waste water treatment plants in Denmark, i.e. the larger plants are equipped with an anaerobic digestion solution, whereas the anaerobic digestion solution is typically not viable for smaller WWT plants that uses technologies like aerobically stabilization before land field application. The common solution for larger WWT plants is anaerobic digestion to produce a biogas that is typically used to supply part of the energy for the WWT plant. The digested sludge comprising about 50 % of the volume may be dried and incinerated e.g. in Cement production.

The overall energy balance for these methodologies is negative as evaporation of 60-70 wt % of water from the dewatered digestate is required. There is a growing regulatory pressure for recycling and recovery of phosphorous and more circular solutions in general which is expected to be mandatory for waste water treatment plants in the future. Germany has the first country to put Sewage Sludge Regulation in place to gradually close the phosphorous cycle and reduce the dependence of natural phosphorous imports, and it is expected that other countries will follow. Implementation of such regulations may drive the technology solutions towards more expensive mono-incineration like Biofoss Lynetten combined with leaching of phosphorous from the combustion ash.

Household waste & Biopulp

The Gemidan sorting plant in Frederikshavn has a capacity of 50.000 ton per annum, whereof about 25.000 ton is current used. A new Gemidan plant with a total capacity of 120.000 ton per annum has been established in Ølstykke, and will treat a part of the organic waste from Copenhagen and Zealand. The sorted and pulped waste is currently supplied to anaerobic digestors, where it is typical co-processed with manures. The operation and economics of anaerobic digestors relies of the use of the digestate being recycled to agricultural

land as a fertilizer or soil amendment product, which may be problematic due to the increased level of micropollutants such as microplastics from the household waste being introduced to the food chain through the digestate.

Urban Waste Hydrofaction® may be used directly on the bio-pulp or eventually in combination with digestate from anaerobic digestion thereby providing a more energy- and resource efficient solution with less negative environmental impact.

Technology solutions such as pyrolysis and gasification could potentially provide complete thermal destruction of the bio-organics and opportunities for phosphorous recovery in the form of biochar residual and residual ash. However, both technologies have lower quality output products, lower carbon and energy efficiency, due to a requirement for a very dry incoming feedstocks, which is opposite to the very wet characteristics of sewage sludge and biogenic fraction of household waste that may be utilized directly or almost directly in Hydrofaction®.

Hydrothermal liquefaction has attracted a lot of attention over the last decade as a highly prospective solution for direct thermochemical conversion of a wide range of bio-organic feedstocks to high value liquid fuels. It is widely accepted (e.g. US DOE) to be an exceptionally cost and resource efficient technology for production of biofuels with the greatest potential GHG mitigation for heavy transport [16].

The main competitors within hydrothermal liquefactions include Licella (AU), Genifuel Corporation (US), Reliance (IN), ENI (IT), Southern Oil (AU), Solray (NZ), Exxon/Mobile (US), who are all in a development or pre-commercial scale for different types of feedstocks. Steeper Energy is widely recognized as being in the forefront of development and commercialization with the Norwegian demonstration plant coming online in Q4-2021. The Hydrofaction® technology is a robust and efficient implementation made up from of the whole value chain from feedstock preparation, high pressure pumping, heat recovery, pressure let-down, product separation, by-product recovery as well upgrading Hydrofaction® oil to advanced biofuels. Presently 149 patents have been granted (99 pending) across 25 patent families covering the whole value chain.

- *Describe entry or sales barriers and how these are expected to be overcome.*

Wastewater treatment and waste treatment is in most contexts a publicly operated activity that aims at providing legally and political defined objectives. Predictability in outcome is in most cases paramount to the stakeholders and to a lesser degree the interest in potentially achieving further improvements. Often a requirement to a prior reference site over a longer period, e.g. 5 years successful operation, is expected before investment decisions are made for new technologies. Additionally, in many cases, the task is outsourced to trusted third-party engineering firms or technology and service providers (TSP). These entities are capable of delivering the required results according to Service Level Agreements (“SLA”), where the TSP has a significant influence on the actual technology implemented in order to achieve the results defined by the SLA. Furthermore, TSP’s often identify, implement and service the chosen technological solution where long term profits are generated from service contracts supporting traditional (and often inferior) solutions.

Most large TSP's have linkages to a broader (international) market of solutions, beyond the individual local municipality or waste management company. Thus the TSP is a likely in-road to the market, locally, nationally and internationally, assuming Hydrofaction® represents a competitive advantage and not a threat to the TSP existing business model.

The project results provide evidence that there will be a value proposition beyond the results expected by the end-user (municipality), that will likely be attractive to TSP's in the waste management space and hence a collaboration with such stakeholder will be attractive for Steeper Energy to investigate for future commercialization and proliferation.

- *How does the project results contribute to realise energy policy objectives?*
Hydrofaction® produces a renewable crude oil that can be refined into fuel components for use in heavy transportation e.g. long-haul road, sea and air transportation. The project has broadened the market by enabling utilization of new high ash urban waste streams as input to Hydrofaction®.

The adoption and roll out of Hydrofaction® for the Urban Waste market utilizing those urban wastes that are currently incinerated (reference year 2019), can directly contribute to the Danish Energy Policy goals. Namely, the technology directly positively contributes to Denmark's energy security, green transition and independency of fossil fuels by 2030 as well as towards a fossil free energy supply in 2050, climate and environment, cost efficiency, economic growth, and employment.

More specifically:

a) Security of supply

The project contributes to the security of supply by utilizing urban waste resources with high inorganic content for production of renewable crude oil that can be refined into advanced biofuels for the heavy transportation, e.g. long-haul road, sea and air transportation. In 2019 a total of 460.000 ton (dry ash free basis) of biogenic waste from households was incinerated, and 140.000 ton of sewage sludge (dry ash free basis) was processed. These arising could potentially be converted in 276.000 ton of oil or 5,9 % of the Danish crude oil import in 2020.

b) Fossil Fuel independence

Denmark is no longer self-sufficient in the crude oil production, and imported 4,66 million ton of crude oil in 2020. As mentioned under section a) Security of supply, 5,9 % of the Danish crude oil import in 2020 could potentially be from Urban Waste resources alone.

c) Climate and Environmental considerations

The perspective of the Hydrofaction® technology in a waste management context is a circular solution that converts low-value waste material into high-value resources with a potentially significant CO₂ reductions in Denmark, EU and globally. Urban waste further provides a solution where nutrients may be recycled with high efficiency, and having a significant destruction of micropollutants such as pathogens, microplastics and pharmaceutical traces as an additional benefit. Hence, Urban Waste Hydrofaction® provides a solution that have climate, environmental and health effects.

Table 19 below shows a roll out of plants in Denmark and globally along with the GHG emission reduction, phosphorous recovery generation of indirect and direct jobs. Basis for the calculation is 50.000 dry ton mixed Urban Waste per annum (TPA) in same ratio as the arisings, transport distance to plant up to 100 km, weighted yield of oil 46 % (49,6 % and 34,5 % respectively), share of renewable electricity 80 %, low grade heat used for district heating, crude oil upgraded to 70 % diesel and 30 % marine fuel. Carbon intensity baseline based on substitution of diesel (CI: 94 gCO₂e/MJ) only i.e. feedstock considered carbon intensity savings from eventual replacement of anaerobic digestion not included. The ratio of direct to indirect jobs is based on temporary construction jobs only, and is based on WASTED 2014 [17].

Year	Installed Capacity t(daf)/year	Renewable crude oil produced t(daf)/year	Renewable Diesel Produced t/year	GHG Reductions tCO ₂ e/yr	Phosphorus Recovered (t/year)	Employment (Full-time Equivalents)	
						Direct	Indirect
						2022	0
2023	0	0	0	0	0	10	0
2024	50.000	23000	20.240	66.279	128	14	162
2025	50.000	23000	20.240	66.279	128	24	162
2026	100.000	46000	40.480	132.558	256	38	324
2027	150.000	69.000	60.760	198.837	384	52	487
2028	200.000	115.000	101.200	331.394	640	66	649
2029	300.000	138.000	121.440	463.952	896	94	973
2030	400.000	184.000	161.920	596.510	1152	122	1298

Table 19: Urban Waste Hydrofaction[®] plant roll out and effect GHG emissions, Phosphorous recovery and employment.

As seen from the table 8 plants each having a capacity of 50.000 (daf) tonne per annum could potentially be built in Denmark from Urban Waste resources alone thereby contributing to a GHG reduction of nearly 600.000 tonnes of CO₂ by 2030. In addition to this 1152 ton of phosphorous could potentially be recovered from these resources.

d) Energy efficiency

Hydrothermal liquefaction generally, specifically Hydrofaction[®], has an unprecedented energy efficiency rooted in the fact the no latent heat needs to be added for evaporation, a very high carbon conversion efficiency (all carbon converted including lignin) and very high selectivity towards the desired oil product combined with extensive heat recovery between the incoming feed stream and outgoing product stream. Subject to the higher heating values of the urban waste feedstocks being validated, the preliminary mass- and energy balances of energy contained being recovered in the desired crude oil product

from the urban waste feedstocks is 60 % and 83 %, respectively with room for further improvement. An additional 13 % of the input energy is recoverable as district heat, whereby the overall energy efficiency may exceed 95 % for the biopulp, when the district heating option is utilized.

e) Growth and employment effects

As shown in the roll out plan in table 19, 122 direct and 1298 indirect jobs may be created in DK from the roll out of the Urban Waste Hydrofaction plants alone.

The project has underlined the significant opportunity for Steeper Energy at both an European and global scale. Hydrofaction[®] projects based on urban sourced wastes offer a financial attractive solution and could provide a significant future Danish export. Denmark is regarded as a lead adopter and showcase for this type of technology, as such, it is important to use this position for the future international commercialization of Hydrofaction[®].

Steeper Energy is identified as a leading company within the HTL space and through a strong IP position created over time the international commercialization of Hydrofaction[®] is likely to be a solid and lasting commercial position, especially in the areas of waste destruction/conversion and transformation of woody residues to advanced biofuels. Steeper Energy is already advancing the commercial availability of Hydrofaction[®], e.g. through its engagement with Norwegian Silva Green Fuel, and the existing industrial scale demonstration facility.

7. Project conclusion and perspective

7.1 Conclusions

The project has addressed the central process steps related to conversion of high ash bio-organic waste streams from urban sources by:

- 1) Demonstrating that pumpable homogeneous slurries can be produced from biogenic portion of urban waste streams including sewage sludge and household wastes.
- 2) Identifying and validating that commercial aggregation and sorting technologies for household wastes can produce a homogenous pumpable slurry satisfactory for the Hydrofaction[®] process (i.e., commercially pretreated biopulp from sorted organic household waste can be directly used in Hydrofaction[®]).
- 3) Establishing critical design data, such as rheological properties of slurries, for future commercial engineered design of urban waste Hydrofaction[®] plants;
- 4) Addressing risks of potential extensive wear in the high-pressure pump and the pressure let down pump including:
 - a) The design and construction of an experimental set-up comprising functional prototypes of the high-pressure pump and pressure let-down system addressing potential critical risk of high-wear from the high-ash containing urban waste streams; and
 - b) Solutions to minimize wear by controlling the pressure drop over the valves when opening and closing.

- 5) Demonstrating the processability and conversion of two urban sourced waste streams, at the continuous pilot plant at Aalborg University, including:
 - a) Identifying and solving challenges related to product separation:
 - i) as the degassed product results in an emulsified product that does not separate into phases via gravimetric methodologies that then required;
 - ii) a two-stage solvent- and acid assisted disc bowl solution has been developed and installed at Aalborg University.
 - b) Establishing a comprehensive assay of physical/chemical data on input, product, and effluent streams such as feed stocks, feed slurries, renewable crude oil, process water streams, gas and mineral fraction; and,
 - c) Establishing preliminary data such as mass, elemental, and energy balances for relevant urban waste feedstocks.
- 6) Demonstrating essential destruction of micropollutants such as pathogens, endocrine disrupters, other trace pharmaceuticals and microplastic from sewage sludge (a key sales advantage when compared to alternative technologies such as anaerobic digestion).
- 7) Demonstrating and evaluating the recovery potential of phosphorous and other mineral nutrients in the Hydrofaction[®] process such that:
 - a) Of the phosphorous contained in the feedstock, 93% by weight ends up in the mineral phase with the remainder ending up in the process water;
 - i) The mineral product may be used directly as a fertilizer or soil amendment product or may be upgraded to a conventional phosphorous product like diammonium phosphate, struvite or hydroxyapatite by leaching using sulfuric acid and precipitating the product at alkaline conditions;
 - ii) Recovery of phosphorous from the mineral and process water in the form of conventional phosphorous products have been demonstrated by preliminary synthesis of the conventional phosphorous products; and,
 - b) Of the nitrogen contained in the feedstock, 56-86 % by weight ends up as nitrogen compounds such as ammonium salts in the process water such that:
 - i) More than 80 % of the nitrogen is recoverable as an aqueous ammonia solution by flashing and/or stripping it off from the water phase; and;
 - ii) The quantity of recoverable ammonium corresponds to more than the double of the stoichiometric amount for synthesis of phosphorous compounds.
- 8) The oil-yield from sewage sludge is lower than expected, whereas the oil yield from bio-pulp is approaching 50 % by weight of the dry ash free part of the bio-pulp even for a once through process without water phase recirculation.
- 9) The lower oil yield from sewage sludge is likely due to insufficient recovery of low boiling organic compounds from the process water during separation and concentration of the process water, which is consistent with the relatively poor carbon balance closure (87 %) for the sewage sludge run, and micro-batch studies performed by Aalborg University in a parallel project [1].
- 10) The energy yields for both sewage sludge and bio-pulp are expected to have been affected by errors or insufficient measured samples for higher heating values, such that:

- a) The measured values are significantly higher than measured by Aalborg University on the same feedstocks in parallel studies [1,2], and data reported in databases [3,4];
 - b) Assuming the same heating values as measured by Aalborg University the energy recoveries are close to expectations; and,
 - c) It was not possible to validate the higher heating values within the project period.
- 11) The gas yields and heating values from both sewage sludge and biopulp conversion are similar; however, less than half of values measured for lignocellulosics at same operating conditions.

In summary, one may conclude that the project has met all technical and commercial milestones initially articulated in the project application and have significantly de-risked the technology for the processing of such feedstocks. Please note that these conclusions must be subject to further validation of the loss of low-boiling point organic compounds as well as validation of the heating values for the feedstocks.

7.2 Perspectives

The urban sourced waste feedstocks at the center of this project are both typically regulated by the state and generally controlled, collected, and managed by municipalities or by private operators under municipal contracts. However, these two feedstocks must be processed with Hydrofaction[®] subject to their unique attributes and outputs. Hence, the application of Hydrofaction[®] in relation to the two feedstocks have different commercial and regulatory drivers that could affect overall commercial attractiveness.

7.2.1 Sewage sludge

The primary value of Hydrofaction[®] as it may be applied to sewage sludge will be as a circular-solution for waste destruction. The project as proved the destruction of essentially all micropollutants as well as the recovery and recycle of minerals and nutrients. While the project has also proved the possibility for the production of green transportation fuels, due to the typical scale of waste-water treatment plants (and characteristics of the sludge) fuel production cannot be seen as a driver nor a substantial contribution to future commercial project profitability.

Further, future Hydrofaction[®] plants are likely be co-located with the waste-water treatment plants to limit the aggregation and transport of sewage sludge to a centralized facilities to be co-processing sewage sludge with other urban waste streams are likely to be both cost prohibitive and unpopular with citizens and at this point thought not to be a valid assumption to improve economies of scale for such projects.

During the course of the project, a MoU with City of Calgary was signed related to the development of significant-scale project at its Pine Creek waste-water treatment centre. Overall that demonstration project has been delayed due in large part to market conditions and other constraints associated with Covid-19 restrictions. A future decision to move forward with the Pine Creek Project is pending subject to engineering/feasibility study based on this project's results and market testing to validate commercial attractiveness to either the City of Calgary and/or commercial waste management companies.

Additional issues related to the commercial application of Hydrofaction[®] into the destruction and transformation of sewage sludge is also related to the deep connection and management of these

feedstocks by municipal governments. These customers or regulators (and influencers) are notorious around the western world for both very long decision cycles and conservatism related to new technology adoption. Furthermore, it is often the case that (and has been already Steeper Energy's experience) that unique solutions to wastewater/sludge treatment must still be tendered to the general marketplace possibly leading to significant investment in sales and project development activities to have Hydrofaction[®] adopted only to find other HTL vendors invited to participate in a tender or indeed finding that without significant competitive solutions more traditional vendors will be chosen.

As a result of the learnings gained through this project, Steeper Energy is now actively seeking waste management and/or waste-water companies as active joint venture or corporate partners to enter this particular market.

7.2.2 Biopulp

Biopulp, in contrast to sewage sludge and to some surprise, has proven to be a very attractive opportunity from both a waste management and a green transportation fuel perspective. Biopulp conversion via Hydrofaction[®] has demonstrated very high biocrude oil yields with high energy density (or inherent energy recovery from the incoming feedstock). Combined these observations with similar micropollutant destruction as seen with sewage sludge, as well as the possibility for nutrient and mineral recovery, the application of Hydrofaction[®] in this waste management sector represents an attractive circular solution. Furthermore project economics are enhanced by way of the cost and socially acceptable opportunities to aggregate feedstocks allowing implementation of meaningful scale for production of green transportation fuels. To improve economic scale beyond that possible with biopulp alone, future commercial projects may be supplemented or made larger by accepting other feedstocks such as the fiber fraction from anaerobic digestion and/or other agricultural, food & beverage production and/or other commercial and industrial waste streams.

The learnings and experiences from the processing of feedstocks with high inorganic and high nitrogen content will be very value for evaluating such co-processing options. Additional work and optimization of both operations and future engineered designs will be undertaken by Steeper Energy. Experience and input to be gathered by the Tofte, Norway *Industrial Scale Demonstration Project* associated with smaller scale applications of Hydrofaction[®] will also be critical for advancing the design of a commercial solution for the biogenic portion of municipal solid wastes.

Opportunities to partner with waste management companies and co-locate at Materials Recycling Facilities in large municipalities across Europe and America offers the promise of a very large sector specific business opportunity that is compelling to customers and shareholders alike.

Steeper Energy is currently in early, confidential, discussions with companies currently working on the periphery of the urban waste management business who aspire to offer a more comprehensive solution to utilizing biogenic wastes aggregated in urban areas and are potential early entrants to this market with a Hydrofaction[®] based solution/offering.

7.2.3 Next Steps

This project has produced a plethora of excellent results that will lead to further developments and innovations for applications, processes (operations) and apparatus (physical plant modifications and upgrades). The road to commercialization of Hydrofaction[®] in either of both of the urban waste streams studied within this project requires some additional technology development and more im-

portantly the development of commercially relevant relationships with partners and potential customers/host facilities. Furthermore, deeper understanding of regulatory imperatives such as the emerging new waste directives in Denmark, European Union as well as America will drive geographic focus and early technology implementation.

Steeper Energy is planning future engineering studies to improve its understanding of the generic techno-economic analysis of projects in both sewage sludge and biopulp. The company is already investigating specific business case development while searching for sectoral and/or project partnerships to validate economical attractiveness and launch an early commercial project.

Finally, the industrial-scale demonstration project at Tofte, Norway, is to be operational from Q4 2021, will yield further operational experience, data, and know-how. This demonstration project happens to be very close in scale (representative-scale) to future commercial projects in the urban waste market and will be of significant assistance and importance to potential strategic partners, joint project owners and customers for Hydrofaction® in these market applications.

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